Solid|Liquid SEPARATION

Scale-up of Industrial Equipment

Richard Wakeman and Steve Tarleton

Solid/Liquid Separation: Scale-up of Industrial Equipment

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Preface

Recent years have witnessed a dramatic and accelerating increase in the number of books written about many aspects of solid/liquid separation, so that now there is a substantial and impressive choice available to those seeking information on what, nonetheless remains the Cinderella technology, especially in terms of the attention it receives at both undergraduate and postgraduate level in the universities around the world.

This book is not intended as merely another addition to this growing library, but is conceived with the more ambitious objective of serving as an experimental handbook to which the person in industry can turn for detailed guidance on how to tackle practical solid/liquid separation problems. As such, it is unique in devoting its main attention to exactly those aspects of the technology which have hitherto received little or none excepting in the occasional short article in a journal; the description of equipment is therefore subordinated to the consideration of how to plan, conduct and interpret experiments, while theoretical aspects are introduced only when required for this primary purpose. The last book on this topic ("Solid/Liquid Separation Equipment Scale-Up", edited by D.B. Purchas and R.J. Wakeman) was published in 1986 – this book has been out-of-print for many years but it brought together methods that were in use at that time by a selection of companies. The preface here echoes what was written by Derek Purchas and Richard Wakeman in 1986 since the situation in respect to the standardisation of test procedures remains largely unchanged almost 20 years later.

This book collects together the test procedures used in different sectors of the solid/liquid separation equipment industry, identifying the types of tests carried out and what might typically be done with the resulting data when faced with a new application to assess. The procedures

described are based on established methods that are determined by industrial practice. The style of the book has a strong practical emphasis and is intended to act as a reference text for engineers concerned with applications evaluation of equipment or its scale-up.

Solid/liquid separation has many features in common with solid/gas (or air) separation, but also certain clear points of difference. One of these remains the virtual total absence of any standardized test procedures for the liquid sector, whereas fully defined test methods have long been established for a significant part of the air sector. This is not to say that no efforts have been made in this direction for liquids, for the contrary is true with certain very specific applications such as hydraulic fluid systems; however great the effort, in practice the solid/liquid field is still relatively untouched by standardization.

The test procedures described in the pages which follow are not claimed to fill this gap, although perhaps their assembly and publication may serve as a small and useful step along what will inevitably be a long and stoney road if standardization is ever to be achieved in this most complex industrial field. Even if they lack the éclat of a 'standard' status, these procedures have two significant merits. Firstly, there is the very fact that they have been written down in such detail; secondly, each procedure is firmly based on the established methods used by at least one major manufacturer of the corresponding type of industrial equipment.

Industrial practice is the dominant theme. This deliberate industrial bias also explains the choice of units which may vary from one methodology to the next, in the belief that this facilitates communication between an international team of authors from 4 countries and (it is hoped) a worldwide readership. It is correspondingly appropriate that the authors are drawn predominantly from those who are at the peak of their own industrial field, while the few others are university based contributors who enjoy very strong and close links with industry.

The intention of the authors is that this book shall not merely be read, but that it shall be used as a practical manual. For this reason, efforts have been made to evolve a uniformity of style and structure. The use of a standard system of symbols throughout all the chapters would obviously have contributed to this objective – in practice, the number of symbols required made this practical step impossible, so each chapter incorporates its own nomenclature.

This book is therefore offered as a working manual rather than as an educational text. Even so, it is hoped that it will be of use, and perhaps even serve as a stimulus, to those teaching and working in the

universities, since they will surely find a remarkable chasm between the narrow interpretation of solid/liquid separation as it is generally taught in universities around the world, and the industrial realities which are revealed in the following pages; those with discerning eyes will see here as immense but incomplete canvas, where the gaps which await their attention are as revealing as those small areas so far completed in comparative detail.

Richard Wakeman and Steve Tarleton

September 2005

Disclaimer

In all of the procedures described in this book the authors and their Companies and the editors have made considerable efforts to ensure the suitability, accuracy and safety of the tests for use. The information and guidance provided in this book is of a general nature only and whilst efforts have been made to ensure its accuracy no guarantee is given nor responsibility accepted that it is without error or is applicable in all circumstances. Neither the editor, the authors nor their Companies will accept liability for any loss resulting from the use or misuse of the tests, procedures or methodologies described. The person carrying out the test must be satisfied that further procedures and precautions as may be necessary or prudent are carried out.

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1 Solid/liquid separation equipment selection

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There are many solid/liquid separation techniques that have established general application within the process industries, and there are a few which are currently in their early stages of commercial exploitation. The dividing line between the two categories is open to dispute, and difficult to identify in a field which is noted for innovation and rapid development. The enormous choice of solid/liquid separation equipment is bewildering to the non-expert and selection of appropriate equipment is thus problematic to the design engineer. It is often difficult to identify the most appropriate separator without extensive previous knowledge of a similar separation problem.

The purpose of this chapter is to provide guidance on what form of small scale tests and results analysis are appropriate for the initial selection of equipment. The basis for the selection and ranking of potentially suitable equipment for a particular separation is described through knowledge of experimental data, selection charts and an expert system approach. Additional tables indicate how ranked equipment can be further short-listed for further investigation.

1.1 Methods of equipment selection

Although a number of different approaches to equipment selection have been proposed, the overall procedure can be summarised by the flowchart shown in Figure 1.1. The basic principle is to use a limited amount of data about the process and some preliminary knowledge of the separability of the feed together with a form of inference mechanism such as a selection chart or table. This combination allows the identification of a range of equipment that could be expected to carry out the required duty. If necessary, the equipment list can be shortened

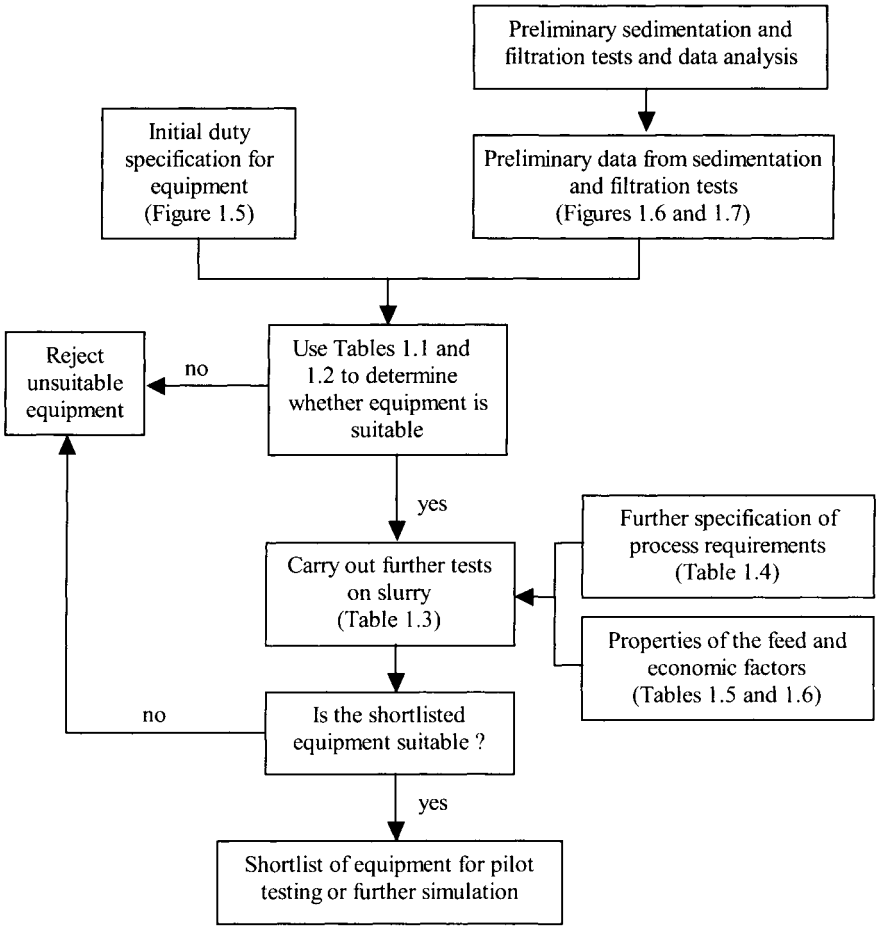

Figure 1.1 Flowchart for the selection of solid/liquid separation equipment

by performing further small scale test work more germane to the identified equipment. The final shortlist of equipment contains those items of equipment that are worth further evaluation through pilot testing and/or computer simulation.

To carry out the steps shown in Figure 1.1, a number of different equipment selection schemes have been proposed. The information available may be categorised into five groups (Wakeman and Tarleton, 1999):

(a) General information (Hicks and Hillgard, 1970; Maloney, 1972; Alt, 1975).

(b) Non-ranked table: Equipment is placed in a list relative to some characterising parameter or performance indicator such as feed

concentration, particle size in the feed or standard cake formation time (Flood *et al*, 1966; Davies, 1970; Purchas, 1970, 1972a, 1978, 1981; Hawkes, 1970; Emmett and Silverblatt, 1974, 1975; Day, 1974; Dahlstrom, 1978; Gaudfrin and Sabatier, 1978; Trawinski, 1980; Purchas and Wakeman, 1986; Pierson, 1990).

(c) Ranked table: Equipment performance is rated by one or more indices to produce ranked lists (Davies, 1965; Purchas, 1972b; Fitch, 1974, 1977; Moos and Dugger, 1979; Komline, 1980; Ernst *et al*, 1987, 1991). Indices are typically related to operational parameters such as solid product dryness, crystal breakage, cost etc.

(d) Logic diagram: A decision tree guides a user through a series of yes/no choices towards a potentially suitable generic class of separation equipment (Davies, 1965; Tiller, 1974; Pierson, 1990).

(e) Expert system: Rule-based computer programs select and rank potentially suitable separation equipment (Korhonen *et al*, 1989; Ernst *et al*, 1987, 1991; Garg *et al*, 1991; Tarleton and Wakeman, 1991, 2005) – only the latter has become commercially available (in the Filter Design Software (2005) package (FDS)).

The charts, tables and general information contained in categories (a)–(d) can be used as guides toward an initial selection of solid/liquid separation equipment. The better contributions consider a wider variety of possible eventualities, and indicate clearly where decisions must be made. The charts and tables have generally been devised by experts to be fairly comprehensive, and are of greatest value to the solid/liquid separation expert. They also illustrate the near impossibility of combining comprehensive descriptions with usability. Without 'expert' guidance it is extremely difficult for an end-user to correlate information and decide which equipment is more suitable for any particular application. Subsequent chapters in this book describe approaches to testing solid/liquid separation equipment and serve as a guide to the tests that need to be undertaken.

With the widespread availability of personal computers, it is obvious that the development of computer software is an ideal way to handle the complexities involved in equipment selection. Whilst rule-based expert systems appeared to provide the optimum solution at first, it later became apparent that inherent restrictions would prevent their widespread application. Thus, interactive personal computer software has been developed to commercial standards and this led to the release of FDS. The underlying philosophies used within the selection module of FDS are described in this chapter.

1.2 Test procedures

The general procedure developed by Purchas provides a valuable, non-specialist, guide through the complex and confusing area of equipment selection (Purchas and Wakeman, 1986). The basis of that procedure has been adopted here, and extended to include a wider range of separation methods as well as a method for the ranking of potentially suitable equipment.

The method requires some limited information about the separability of the suspension, which is provided by filter leaf tests and/or jar sedimentation tests. Similar alternative tests are described in later chapters of this book.

1.2.1 Jar sedimentation tests

The ability of the suspension to settle and the settling rate can be easily determined from a jar test. The settling rate depends on a number of factors – effective particle size, the specific gravity difference between the solid and liquid phases, the flocculated or dispersed state of the suspension (which is itself a complex function involving surface electrical properties, adsorption of charged ions, and so on), and the viscosity of the liquid.

A bulk sample of the slurry to be evaluated should be sub-sampled and poured into a 1 litre measuring cylinder, and its behaviour noted. The general features of its behaviour that should be observed are:

1. Any indications of the tendency to flocculate;
2. The way that the solids settle: (a) quickly with a clear mud line with the solids apparently descending 'en masse' to leave a clear supernatant liquid lying above the suspension, or (b) slowly with solids building up from the bottom and leaving a cloudy supernatant liquid;
3. After a 24 hour settling period, the amount of settled solids.

The objective of the sedimentation test is to determine the initial (constant) rate of settling, clarity of the supernatant liquid and the final proportion of sludge. In more detail, the sedimentation test and analysis procedures follow a sequence dependent on the nature of the test slurry.

1. Determine the solids concentration in the slurry sample before commencing the jar sedimentation test(s).

2. For a preliminary test, to determine if flocculants or pretreating chemicals need to be used, fill a one litre graduated measuring cylinder to the one litre mark with a sample of slurry. Shake to ensure uniform dispersion of the particles and then place the cylinder on a rigid, level surface and allow the slurry to settle. At suitable intervals record the suspension-supernatant interface height from the millilitre graduations on the cylinder and note the corresponding elapsed time. Allow settling to continue until at least ten sets of time *vs.* height readings have been taken and/or settling is finished. If the supernatant liquid has poor clarity or the settling rate is excessively low (e.g. <0.1 cm s^{-1}), consideration should be given to modifying the feed slurry by pretreatment as indicated in 3. and repeating the sedimentation test. If the settling test appears to be satisfactory with a reasonable settling rate and good supernatant clarity then proceed to 4.
3. Add flocculants or pretreatment chemicals as necessary (more information on the choice of pre-treatment chemicals can be found in Chapter 2). A preferred technique is to place the required amount of diluted flocculant into an empty beaker, pour the measured amount of feed slurry rapidly into the flocculant, and then promptly pour back and forth repeatedly. If the concentration does not need further adjustment, immediately introduce the sample into the graduated measuring cylinder. During settling record the time vs. suspension-supernatant interface height as in 2.
4. Note the volume of sludge at the bottom of the measuring cylinder after the slurry has finished settling, and check the acceptability of the supernatant liquid.
5. The settling rate is determined by plotting a graph of suspension-supernatant interface height *vs.* time, identifying the set of points which comprise the initial linear portion and calculating the gradient of a line of best fit. The proportion of sludge is the ratio of final sludge height to initial suspension height.
6. Repeat steps 1–5 as necessary to ensure data accuracy and reproducibility.

The suspension-supernatant liquid interface location is recorded at time intervals to build up a plot of the interface height *vs.* time, as shown in Figure 1.2. A detailed discussion of the significance of such a plot can be found in other texts (for example, Wakeman and Tarleton (2005)). The volume of the settled solids after 24 hours (or some shorter time if the settling has completed), expressed as a percentage of the original volume, should also be recorded.

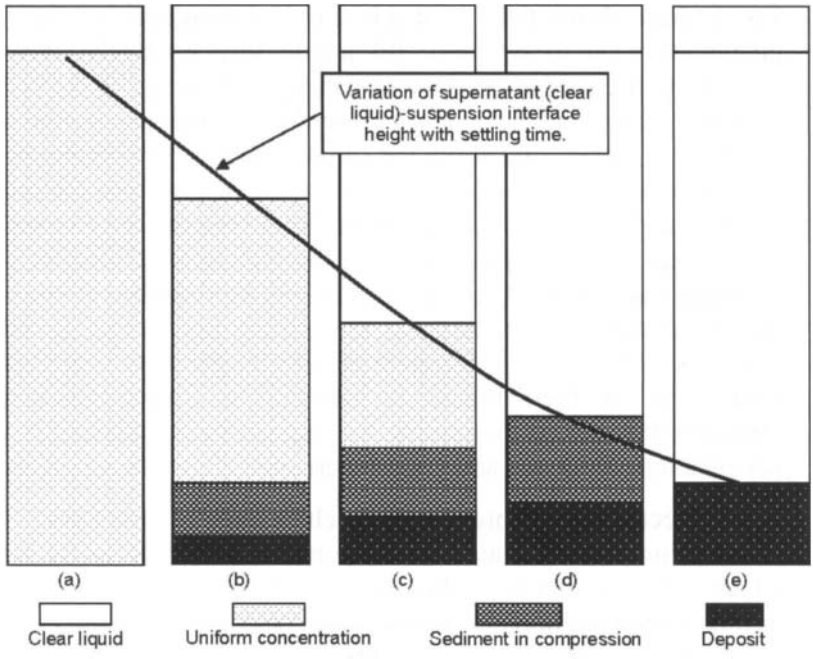

Figure 1.2 Sedimentation of a suspension in a jar test

1.2.1.1 Example calculation of settling rate from a jar test

The following data were obtained from a settling test using a jar with an inside diameter of 6.5 cm.

Settling time (s)	*Suspension volume* (cm^3)	*Interface height*[†] (cm)
0	1000	30.1
10	910	27.4
20	830	25.0
30	750	22.6
40	660	20.0
50	580	17.5
65	500	15.1
90	410	12.4
130	370	11.2
180	340	10.3
220	332	10.0
250	332	10.0

[†] = 4 × (suspension volume)/(π 6.5^2)

From these data, estimate the settling rate and the proportion of sludge obtained.

Plot the interface height against the settling time, Figure 1.3.

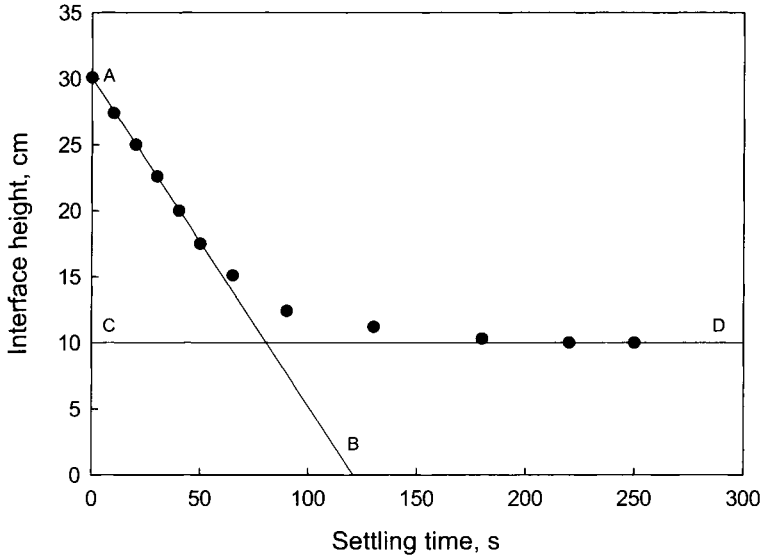

Figure 1.3 Variation of suspension-supernatant interface height with settling time

From the graph:

The settling rate is given by the line AB as $-\frac{30-0}{0-121.1} = 0.248$ cm s^{-1}.

Using line CD: The proportion of sludge in the jar is $\frac{10}{30.1} \times 100 = 33.2\%$.

1.2.2 Leaf filter tests

The filtration rate is controlled by several factors, including the effective particle size in the feed, the flocculated or dispersed state of the suspension, the viscosity of the liquid, and the pressure applied during the filtration.

Filtration tests can be carried out with quite simple apparatus to determine cake formation data. The objectives of the tests are often twofold. Firstly, cake formation rate is required for preliminary equipment selection purposes, and secondly specific resistance or filterability coefficient data are needed for filter sizing and filtration rate calculations.

Figure 1.4 shows a vacuum test filter apparatus system that can be used to obtain the data. The drainage characteristics of the normal ceramic perforate bottom Buchner funnel are poor and make it unsuitable for these tests; it is preferable to use a filter funnel with a porous sintered bottom, or ideally a top-fed leaf assembly equipped with a filter cloth. To simplify subsequent analysis of the data it is usual to operate the apparatus at a constant pressure difference (or vacuum). An equivalent pressure driven apparatus is easily devised for obtaining data using higher pressure differences across the filter. More sophisticated vacuum or pressure leaf apparati can be used if a well equipped separations laboratory is already available. However, the use of complicated equipment at the early stage of slurry assessment is probably not justified, but it is wise to use the same type of filter medium in the tests as is used at full scale so that any medium effects are incorporated into the experimental results.

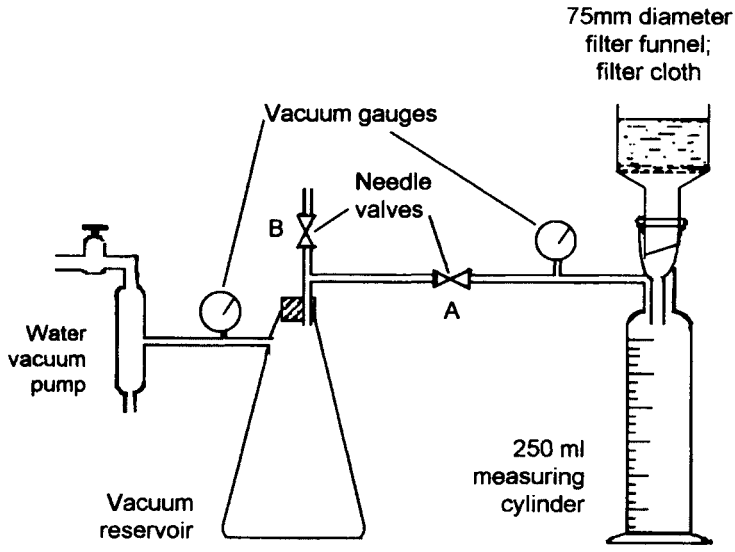

Figure 1.4 Test apparatus showing a top-fed leaf filter for obtaining vacuum filtration data

The general procedure to obtain the filtration data is as follows:

1. Determine the solids concentration in the slurry sample before carrying out the filter test(s).
2. With the needle valve A in the vacuum line closed, adjust the bleed needle valve B to give the required level of (constant) vacuum.

This is likely to be in the region of 50 to 70 kPa (380 to 530 mm Hg). In the wastewater treatment industry the standard used is 49 kPa (386 mm Hg).

3. Pour the well stirred slurry sample into the filter funnel containing the filter cloth and open the needle valve A in the vacuum line so that a pre-set vacuum level is achieved as rapidly as possible in the graduated cylinder. It is better not to pour the sample directly onto the cloth, but to feed it onto a perforated plate located 2 to 3 cm above the cloth. The plate acts as a distributor to (i) prevent solids approaching the pores in the cloth at an unrealistically high velocity and thereby causing unexpected plugging of the pores, and (ii) spread the feed over the full area of the funnel, enabling formation of a cake of more uniform thickness.
4. Monitor the filtration test by recording the filtration rate by measuring the volume of filtrate collected at various time intervals. The intervals between recording the measured volumes need not be constant but may be increased progressively to compensate for the gradual drop in filtrate flow rate.
5. If the cake form rate is too slow (e.g. of the order of cm h^{-1}) it may be desirable to add flocculants or pretreatment chemicals to the slurry and repeat the filtration tests as appropriate. For slow filtering slurries it may be necessary to use smaller measuring cylinders.
6. When ten or more sets of volume-time readings have been obtained fully open both valves to break the vacuum. There should be some surplus unfiltered slurry visible on top of the cake at this stage. If there is not, it is likely that the cake will have started to deliquor and subsequent cake moisture measurements will be erroneously low, whilst leaving the surplus on the cake will lead to incorrectly high moisture measurements.
7. Pipette the excess slurry from the surface of the filter cake.
8. If possible, measure the thickness of the filter cake.
9. Remove as much of the cake as possible from the filter, weigh it, dry it and reweigh it. From these measurements calculate the ratio of the mass of wet cake to the mass of wet cake.

1.2.2.1 Example calculations from constant pressure test data

The following data were obtained from a leaf filter with an area of 45 cm^2 using a pressure difference of 70 kPa (data taken from Wakeman and Tarleton, 2005). The ratio of the mass of wet cake to mass of dry

cake was measured and found to be 1.34, the densities of the solid and liquid phases are 2640 and 1000 kg m^{-3} respectively, and the solids concentration in the feed slurry is 132 kg m^{-3}.

Filtration time, t (s)	*Filtrate volume, V* (ml)	*Cake thickness*† (cm)
0	0	0
170	141	0.324
275	200	0.460
340	230	0.529
390	252	0.580
457	285	0.656
527	320	0.736
589	341	0.784
660	370	0.851

†Calculated data.

Estimate the cake formation rate from these data.

The mass fraction of solids in the slurry (s) is obtained using:

$$s = \frac{c'}{c' + \rho_l\left(1 - \frac{c'}{\rho_s}\right)} \tag{1}$$

where c' is the solids concentration in the feed (kg m^{-3}), and ρ_l and ρ_s are the densities of the liquid and solids respectively (kg m^{-3}).

$$s = \frac{132}{132 + 1000\left(1 - \frac{132}{2640}\right)} = 0.122$$

The filtrate volume collected (V) is related to the thickness of cake formed (L) by:

$$V = \frac{\rho_s\left(1 - m_{av}s\right)}{s\left[\rho_s\left(m_{av} - 1\right) + \rho_l\right]} AL \tag{2}$$

where m_{av} is the ratio of the mass of wet cake to the mass of dry cake and A is the filtration area of the test filter.

$$V = \frac{2640(1-1.34\times0.122)}{0.122[2640(1.34-1)+1000]}45L = 429.3L$$

i.e. $L = 0.0023V$ cm

The cake thicknesses shown in the last column in the above table are calculated from this expression. The rate of cake formation decreases with time in a constant pressure filtration; in this case it decreases from 0.0019 cm s^{-1} over the period 0 to 170 s down to 0.0013 cm s^{-1} over the period 0 to 660 s; i.e. the cake formation rate is on the order of 0.001 cm s^{-1}.

1.3 Initial selection procedures

An initial list of equipment that could satisfy a particular duty requirement is drawn up in the following way (this follows the first few boxes in the flowchart on Figure 1.1). The first step is a preliminary specification of the separator duty to define three characterising letter codes. The second and third steps involve the fairly rudimentary bench scale tests described in the preceding section. The analysis of the test data yields overall settling and cake formation rates as well as four more characterising letter codes. Although it is not necessary to collect additional information, it is recommended that as much data as possible are obtained from the tests as these can aid later refinement of an equipment list. The characterising letter codes defined for required duty, settling and filtration are then used in conjunction with selection codings to give a list of equipment that is potentially suited to the required separation objective(s).

1.3.1 Specification of duty

The first step in defining a selection problem is to specify the general requirements of the process. An initial specification can be quite limited and is essentially confined to the process scale, mode of operation, and overall objective of the separation. These objectives and unavoidable restrictions can be specified before any experiments are undertaken. Other specifications, such as the need for filter sterility or the possibility of toxic or flammable hazards, can be considered at a later stage in the selection process.

The principles involved in duty specification are shown in Figure 1.5. Each specification is identified by a characteristic letter, so that a

group of letters specify the nature of a problem. For example, a large scale batch operated system for the recovery of untreated solids would be coded as *adg*.

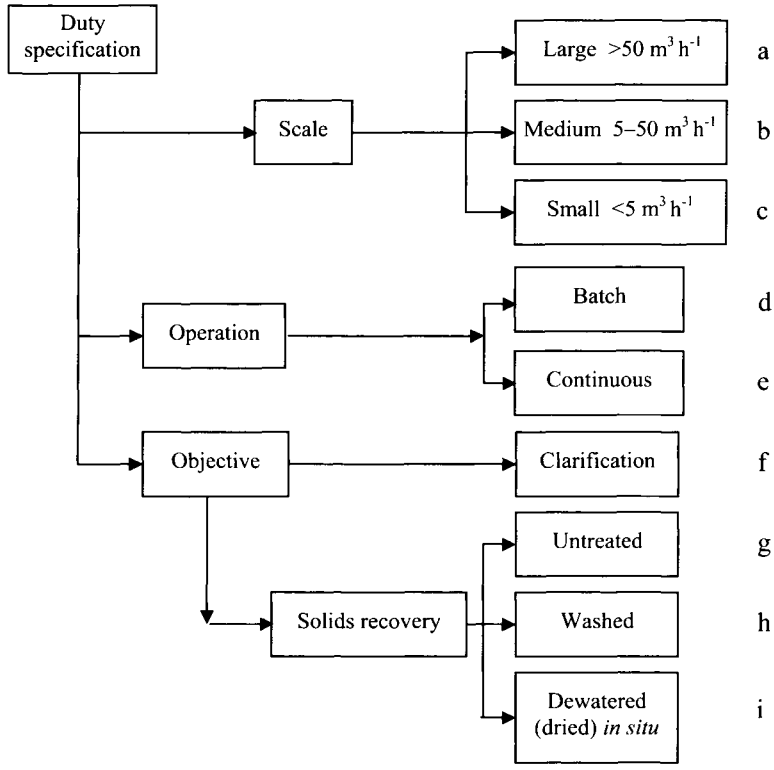

Figure 1.5 Coding the duty specification (Purchas and Wakeman, 1986)

1.3.2 Specification of settling characteristics

The results from the procedure in Section 1.2.1 are coded using a second set of characteristic letters as shown in Figure 1.6. For example, a slurry which settles at 3 cm s^{-1} to yield a clear liquid and a sediment whose volume is 15% of the initial slurry volume would be coded *BEG*.

1.3.3 Specification of filtration characteristics

The results from the test procedure in Section 1.2.2 are coded using a third set of characteristic letters, as shown in Figure 1.7. For example, a slurry which forms a cake at the rate of the order of cm min^{-1} is

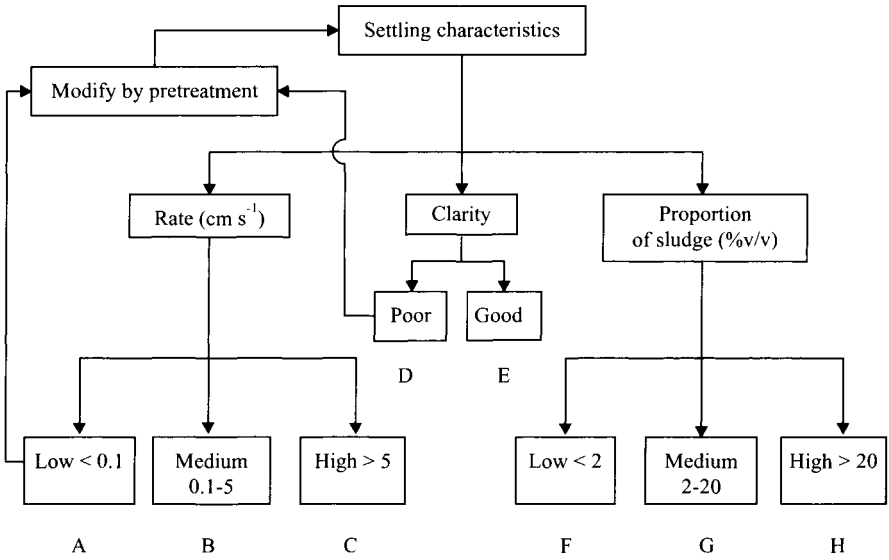

Figure 1.6 Coding the slurry settling characteristics (Purchas and Wakeman, 1986)

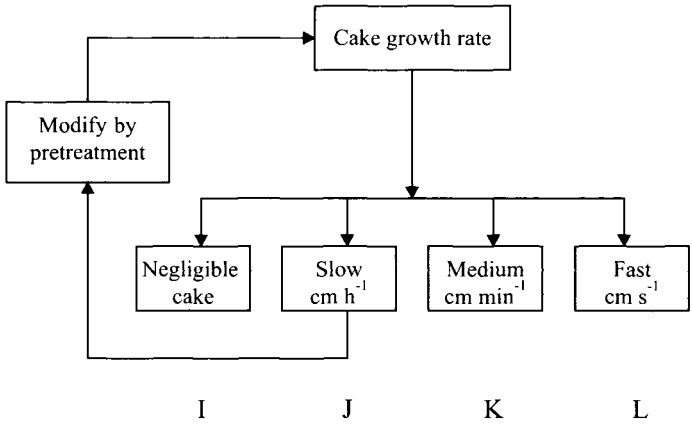

Figure 1.7 Coding the slurry filtration characteristics (Purchas and Wakeman, 1986)

coded *K*. Combining this with the settling characteristics (e.g. *BEG*) gives a total preliminary description of the separation characteristics of the slurry (e.g. *BEG*, *K*). If the proposed duty is simply to thicken a slurry then it is not necessary to carry out a filtration test. However, for a total separation of the solid from the liquid (as obtained in a filter, for example) both settling and filtration tests need to be performed.

1.4 Tables of equipment

The duty specification, jar sedimentation and filtration tests enable the slurry settling and filtering characteristics to be broadly classified, and a selection problem to be specified through a series of letter codings. In order to select and rank equipment from this information it is necessary to provide charts and/or tables that relate equipment performance to the letter codings.

In Table 1.1, generic classes are listed and associated with the letter codings for duty specification, jar sedimentation and filtration performance. Within each generic class a wide range of different types of separator exists, for example, more than ten sub-classes of pressure filters are identified in Table 1.1. Whilst each sub-class may contain a variety of types, they tend to differ in detail of design rather than possess major differences related to their fields of application. Naturally, designs from different manufacturers will differ but almost all will fit into a sub-class. It therefore becomes important to identify the sub-class of equipment that will be suitable for any specific application.

Inspection of Table 1.1 permits identification of those types of equipment which are potentially suitable for the specific duty, thereby indicating in which areas more detailed testing should be concentrated to generate further data for design purposes. It is possible to specify two letter code combinations for which equipment types are not identifiable, these are (i) '--- C-- I' and (ii) '--- --H I'. The series represented by (i) and (ii) indicate a high settling rate (C) or large sludge volume (H), both of which would be incompatible with negligible cake formation (I). If experimental results appear to lead to these combinations, the integrity of the experimental tests and subsequent calculations should be checked.

Whilst many classes of solid/liquid separation equipment will permit most functions to be performed (e.g. cake formation, cake washing, etc.), not all will execute a function with the same degree of effectiveness. The basis of relative performance indices for equipment was set out by Davies (1965) and Moos and Dugger (1979); their methodologies have been adopted in Table 1.2. The suitability of the selected equipment is also related to typical particle size ranges and feed concentrations. Although the latter information has been used in the preliminary selection, values have been implied through the use of other data such as initial settling rate. It is at this point that the engineer can check the compatibility of the equipment with the feed particle size distribution and slurry concentration. The relative performance criteria for the selected equipment are based on a scale

Table 1.1 Classification of equipment according to suitability for duty and slurry separation characteristics.

Type of equipment	*Duty specification*	*Separation characteristics*	
		Settling	*Filtering*
Gravity thickeners and clarifiers			
• circular basin thickener	a, b or c d or e g or h	B or C E F or G	
• settling tank or lagoon thickener	a, b or c d or (e) f or g	A, B or C (D) or E F, G or H	
• circular high capacity thickener	a or b e f	B or C E F or G	
• deep cone thickener	a or b e f	B or C E F or G	
• lamella separator	a, b or c e f	(A), B or C (D) or E F or G	
• clarifiers	a, b or c d or e f or g	A, B or C E F	
Hydrocyclones			
• conical reverse flow or circulating bed	a or b e f, g or h	B or C D or E F or G	
Sedimenting centrifuges			
• tubular bowl	b or c d f or (g)	A or B D or E F	
• basket bowl	b or c d f or g	(A) or B D or E F, G or H	
• disc stack (self clean, manual or nozzle discharge)	a, b or c d or e f or g	A or B D or E F or G	
• scroll decanter	a, b or c e f, g, (h) or (i)	(A), B or C (D) or E F, G or H	

Table 1.1 *continued*

Type of equipment	*Duty specification*	*Separation characteristics*	
		Settling	*Filtering*
Filtering centrifuges			
• basket (pendulum)	b or c d g, h or i	A, B or C D or E G or H	J, K or L
• basket (peeler)	a, b or c d g, h or i	A, B or C D or E G or H	K or L
• cone screen (slip discharge)	a e g or i	C E G	L
• cone screen (vibratory/ oscillatory or tumbling)	a e g or i	C E H	L
• cone screen (worm screen)	a e g or i	C E H	K or L
• inverting bag	b or c e g, h or i	A, B or C D or E G or H	J, K or L
• pusher	a or b e g, h or i	B or C E G or H	K or L
• baffle	b or c e g, h or i	B or C E G or H	K or L
Vacuum filters			
• single leaf (vacuum Nutsche)	c d g, h or i	A, B or C D or E F or G	J, K or L
• single leaf (tipping pan)	c d g, h or i	A, B or C D or E F or G	J, K or L
• multi-element leaf	a, b or c d or e g, h or i	A, B or C D or E F or G	J or K

Table 1.1 *continued*

Type of equipment	*Duty specification*	*Separation characteristics*	
		Settling	*Filtering*
Vacuum filters			
• horizontal belt, rotary table or rotary tilting pan	a, b or c e g, h or i	A, B or C D or E F, G or H	J, K or L
• rotary (vacuum) drum (bottom fed)	a, b or c e f, g h or i	A or B D or E F, G or H	I, J or K
• rotary (vacuum) drum (top fed)	a, b or c e g, (h) or i	C E G or H	L
• rotary (vacuum) drum (internal fed)	b or c e g or i	B E G or H	J or K
• rotary (vacuum) disc	a, b or c e g or i	A or B D or E G or H	J or K
Pressure filters and presses			
• single leaf (pressure Nutsche)	a, b or c d g, h or i	A or B D or E F, G or H	J, K or L
• multi-element leaf (horizontal element)	b or c d g or h	A or B D or E F or G	J or K
• multi-element leaf (vertical element)	a, b or c d f, g, h or i	A or B D or E F or G	I or J
• multi-element (tubular candle)	a, b or c d f, g, h or i	A or B D F or G	I or J
• filter press	a, b or c d f, g, h or i	A or (B) D or E F, G or H	I or J
• sheet filter	a, b or c d f	A D F	I

Table 1.1 *continued*

Type of equipment	*Duty specification*	*Separation characteristics*	
		Settling	*Filtering*
Variable volume (pressure) filters and presses			
• diaphragm filter press	a, b or c	A or B	J or K
	d	D or E	
	g, h or i	G or H	
• tube press	a, b or c	A or (B)	J or K
	d	D or E	
	g, (h) or (i)	G or H	
• expression press	a or b	A	I or J
	d or e	D or E	
	g	H	
Continuous (pressure) filters			
• belt press	a, b or c	B or C	J
	e	D or E	
	g or i	G or H	
• vertical diaphragm filter press	a or b	A or B	J or K
	e	D or E	
	g, h or i	G or H	
• rotary (pressure) drum	b	A or B	(J) or K
	e	D or E	
	g, h or i	G or (H)	
• rotary (pressure) disc	a or b	A or B	(J) or K
	e	D or E	
	g or i	G or H	
Miscellaneous pressure filters			
• cartridge filter	b or c	A or B	I
	d	D or E	
	f	F	
• bag filter	a, b or c	C	I or J
	d or e	E	
	f or g	F or G	
Precoat filters			
• precoat rotary (vacuum) drum	a, b or c	A	I or (J)
	e	D or E	
	f or (g)	F or (G)	
• precoat (pressure) filter and filter press	b or c	A	I
	e	D	
	f	F	

Table 1.1 *continued*

Type of equipment	*Duty specification*	*Separation characteristics*	
		Settling	*Filtering*
Depth filters			
• deep bed (pressure fed) sand	a, b or c e f	A D F	I
• deep bed (gravity fed) sand	a or b e f	A D F	I
• deep bed (fibre)	b or c d or e f	A D or E F	I
Classifiers			
• hydraulic	a, b or c e g or h	B or C E G or H	L
• mechanical	a, b or c e (f), g or h	B or C E G or H	L
• screen (sieve bend)	a, b or (c) d or e f, g or h	(B) or C E F or (G)	I, J, K, or L
Membrane filters			
• dead-end	b or c d or e f	A or B D or E F	I
• low shear crossflow ultrafilter	b or c d or e f, g or (h)	A or B D or E F	I
• low shear crossflow microfilter	(a), b or c d or e f, g or (h)	A or B D or E F or G	I or J
• high shear crossflow	b or c e g or (h)	A or (B) D or E F or G	J or K

Table 1.1 *continued*

Type of equipment	*Duty specification*	*Separation characteristics*	
		Settling	*Filtering*
Miscellaneous separators			
• flotation	a or b	A or B	
	e	D or E	
	f or g	F	
• strainer	a, b or (c)	A	I
	d or e	D or E	
	f	F	
• single leaf (gravity Nutsche)	(b) or c	B or C	K or L
	d	D or E	
	f, g, h, or i	F, G, or H	
Force field assisted separators			
• low gradient or low intensity magnetic	a, b or c	A, B or C	I or J
	d or e	D or E	
	f, g or (h)	F or G	
• high gradient magnetic	a, b or c	A, B or C	I or J
	d or e	D or E	
	f, g or (h)	F or G	
• high voltage electric	b or c	A	I
	e	D	
	f	F	

'()' around a letter index indicates a marginal choice

from 0 to 9, with larger numbers indicating better performance. The letter designations 'C' and 'S' indicate whether the solid is generally discharged in the form of a cake or a slurry.

Whilst this information provides useful guidance, there may be some specific designs of separators that fall outside of the guidelines. Nonetheless, once an initial selection of equipment has been drawn up using Table 1.1, the list can be sensibly ranked using Table 1.2. Ranking the list or considering some of the other basic equipment characteristics may point to some types being unsuitable for an application.

Table 1.2 Relative performance characteristics of solid/liquid separation equipment.

	Performance indices				*Feed solid properties*	
Type of equipment	*Solids product dryness & state*[†]	*Washing*	*Liquid product quality*	*Crystal breakage*	*Particle size (μm)*	*% by mass solids in feed*
Gravity thickeners and clarifiers						
• circular basin thickener	1 S	2	5	9	0.1–500	< 20
• settling tank or lagoon thickener	1 S	–	5	9	0.1–500	< 20
• circular high capacity thickener	1 S	–	5	9	0.1–300	< 15
• deep cone thickener	1 S	–	5	9	0.1–300	< 15
• lamella separator	1 S	–	5	8	1–150	< 15
• clarifiers	1 S	–	6	9	1–50	< 15
Hydrocyclones						
• conical reverse flow	1 S	2	4	7	5–200	2–40
• circulating bed	1 S	–	4	7	2–500	2–25
Sedimenting centrifuges						
• tubular bowl	3 S	–	6	5	0.1–100	< 5
• basket bowl	2 S	–	5	5	0.1–100	< 5
• disc stack (self clean)	2 S	–	–	6	0.1–100	0.05–2
• disc stack (manual discharge)	2 S	–	–	6	0.1–100	0.05–2
• disc stack (nozzle discharge)	2 S	–	–	6	0.1–100	0.5–10
• scroll decanter	4 C	3	4	3	1–5000	4–40
Filtering centrifuges						
• basket (pendulum)	9 C	6	5	6	10–1,000	4–30
• basket (peeler)	9 C	6	5	5	2–1,000	4–30
• cone screen (slip discharge)	7 C	5	4	4	80–10,000	10–40
• cone screen (vibratory, oscillatory or tumbling)	8 C	5	4	3	100–10,000	10–40

Table 1.2 *continued*

	Performance indices				*Feed solid properties*	
Type of equipment	*Solids product dryness & state*[†]	*Washing*	*Liquid product quality*	*Crystal breakage*	*Particle size (μm)*	*% by mass solids in feed*
Filtering centrifuges						
• cone screen (worm screen)	9 C	5	4	4	60–5,000	10–40
• inverting bag	9 C	6	5	6	2–1,000	5–30
• pusher (single stage)	9 C	7	4	4	40–7,000	10–40
• pusher (multi- stage)	9 C	8	4	4	40–7,000	10–40
• baffle	9 C	5	5	4	100–7,000	10–40
Vacuum filters						
• single leaf (vacuum Nutsche)	6 C	8	7	8	1–500	1–10
• single leaf (tipping pan)	7 C	9	7	8	20–80,000	5–30+
• multi-element leaf	5 C	5	7	8	1–100	5–30+
• horizontal belt or rotary tilting pan	7 C	9	7	8	20–80,000	5–30+
• rotary table	7 C	8	7	8	20–80,000	10–30+
• rotary (vacuum) drum (bottom fed)						
belt discharge	6 C	7	7	8	1–200	1–20
knife discharge	6 C	7	7	8	1–200	1–20
roller discharge	6 C	7	7	8	1–50	1–10
string discharge	6 C	7	7	8	1–70	1–10
• rotary (vacuum) drum (top fed)	5 C	2	7	8	1–600	10+
• rotary (vacuum) drum (internal fed)	5 C	–	7	8	10–600	10+
• rotary (vacuum) disc (cloth covered)	4 C	–	6	8	1–700	5–20
• rotary (vacuum) disc (ceramic)	4 C	–	9	8	1–700	5–20

Table 1.2 *continued*

		Performance indices			*Feed solid properties*	
Type of equipment	*Solids product dryness & state*[†]	*Washing*	*Liquid product quality*	*Crystal breakage*	*Particle size* (μm)	*% by mass solids in feed*
Pressure filters and presses						
• single leaf (pressure Nutsche)	6 C	8	8	8	1–200	< 1–20+
• multi-element leaf (horizontal element)	5 C	8	8	8	1–100	< 1–20+
• multi-element leaf (vertical element)	5 C	6	8	8	0.5–100	< 1–20
• multi-element (tubular candle)	5 C	7	8	8	0.5–100	< 1–20
• filter press	6 C	8	8	8	1–100	< 1–30+
• sheet filter	N	–	9	–	0.1–80	<< 1–5
Variable volume (pressure) filters and presses						
• diaphragm filter press	8 C	8	8	7	1–200	0.3–30+
• tube press	8 C	4	7	7	1–200	0.3–30+
• expression press	6 C	–	6	5	1–200	10–80
Continuous (pressure) filters						
• belt press	8 C	7	7	7	1–200	0.2–30+
• vertical diaphragm filter press	8 C	8	8	7	1–200	0.2–30+
• rotary (pressure) drum	6 C	6	7	7	1–100	5–30+
• rotary (pressure) disc (cloth covered)	5 C	–	6	8	1–100	5–30+
• rotary (pressure) disc (ceramic)	7 C	–	9	8	1–100	5–30+
Miscellaneous pressure filters						
• cartridge filter	N	–	9	–	0.4–50	< 0.1
• bag filter	6 C	–	4	7	10–300	0.2–10

Table 1.2 *continued*

		Performance indices			*Feed solid properties*	
Type of equipment	*Solids product dryness & state*[†]	*Washing*	*Liquid product quality*	*Crystal breakage*	*Particle size* (μm)	*% by mass solids in feed*
Precoat filters						
• precoat rotary (vacuum) drum	4 CN	5	8	8	0.5–100	< 1
• precoat (pressure) filter and filter press	5 CN	–	9	–	1–40	< 1
Depth filters						
• deep bed (pressure fed) sand	N	–	8	–	0.2–60	< 0.1
• deep bed (gravity fed) sand	N	–	8	–	0.2–50	< 0.1
• deep bed (fibre)	N	–	8	–	0.1–40	< 1
Classifiers						
• hydraulic	3 C	3	3	5	50–2,000	4–40
• mechanical	4 C	2	3	4	100–3,000	4–40
• screen (sieve bend)	5 C	4	5	4	45–100,000	20–40
Membrane filters						
• dead-end (leaf or tubular element)	N	–	9	8	0.1–10	< 1
• low shear crossflow ultrafilter	1 S	2	9	–	0.001–0.05	< 20
• low shear crossflow microfilter	1 S	2	9	6	0.05–20	< 20
• high shear crossflow	2 S	4	6	4	0.1–20	< 25
Miscellaneous separators						
• flotation	S	–	–	8	< 300–2,000	1–20
• strainer	N	–	7	–	5–200	< 0.1
• single leaf (gravity Nutsche)	4 C	7	7	9	100–10,000	1–10

Table 1.2 *continued*

		Performance indices			*Feed solid properties*	
Type of equipment	*Solids product dryness &state*[†]	*Washing*	*Liquid product quality*	*Crystal breakage*	*Particle size (μm)*	*% by mass solids in feed*
Force field assisted separators						
• low gradient or low intensity magnetic	3 C	2	4	8	< 40–4,000	5–20
• high gradient magnetic	1 S	2	4	8	< 400	< 10
• high voltage electric	1 S	-	7	8	< 20	< 10

A '-' performance index may be taken to mean either zero (that the equipment is not effective) or that the equipment is not suitable for that particular duty.

[†] State of solids product: S ≡ slurry or free flowing, C ≡ cake, N ≡ solids not generally recoverable.

1.5 Computer software for equipment selection

Filter Design Software (2005) (FDS) has been developed from the successful p^C-SELECT software that has been used by numerous filtration equipment and user companies since it was launched in 1991. It is an interactive computer software package that allows even the novice involved in separation technology to make a preliminary choice of larger scale separation equipment from simple laboratory tests and knowledge of the required duty. FDS is a result of a collaboration with companies in the equipment supply, pharmaceutical, fine chemical and mining industry sectors where the software has been tested during its development.

FDS is industry tested, intelligent and interactive software that is designed to be user friendly for those users who may not be familiar with the bewildering choice that exists amongst solid-liquid separation equipment, and is also a valuable tool for the solid-liquid separation equipment expert. It is an aid to equipment specification and is also a training and educational tool for use by both industry and academia – so it contains many explanations, pictures and diagrams of equipment. The simulation procedures have in-built constraints that arise due to equipment design features that affect the operation of the equipment, along with many guides to aid correct input of data.

FDS is a sequence of interlinked modules that can be used independently from one another. The full set of FDS modules offers many capabilities, including:

- A catalogue and explanation of the main operational and design features of 70+ equipment types;
- Full analysis capabilities of leaf filter, jar sedimentation and expression test results to give the relevant parameters for scale-up and simulation of solid-liquid separation equipment;
- Comparison of data sets from a range of tests or trials;
- Simulation of 20+ types of vacuum and pressure filters;
- The ability to import data files from other software (e.g. Excel® spreadsheets);
- Web access to equipment suppliers.

The equipment catalogue is designed for use as a reference manual that gives technical information and diagrams and photographs about all of the generic types of equipment stored in FDS.

The equipment selection procedures enable selection from more than 70 solid/liquid separation equipment types, including all those listed in Table 1.2. Selection follows the procedures described in Sections 1.2 to 1.4 and is possible from either limited or from more extensive information – obviously, the more information that is available about the feed and the process the better the selection.

The *Selection Module* in FDS has many key features that enhance its capability, making use of computer and web technology to enable the user to make informed selections of equipment, and include:

- User specification of the process and the duty that the equipment will be expected to perform;
- Automatic comparison of the specifications with the FDS database;
- Ranked listing of solid/liquid separation equipment that match the specifications;
- General and detailed information about solid/liquid separation equipment – technical design and operational information about each equipment item in the list is provided in a descriptive format on screen, together with diagrams and photographs;
- Facility to prioritise the equipment list according to process criteria (such as cake washability, cake dryness or the damage likely to be caused to the particles);

- Identification of equipment in the list that is marginal for technical reasons;
- Customisable list of web addresses of equipment suppliers and facility to paste suppliers addresses to a web browser.

The *Data Analysis Module* in FDS automates the analysis of jar sedimentation and filter test data, as well as other types of filter test results. The tests carried out early in the assessment of a solid-liquid separation problem – for either equipment selection or to gather design data – are either a constant pressure leaf filter test (sometimes pilot scale tests may be available) and/or a jar sedimentation test. A piston press test is sometimes carried out to assess compression or consolidation effects on the separation. Whichever test is done, FDS enables rapid analysis of the measured data.

Even with well conducted tests, sometimes some of the necessary input data are missing yet the best possible analysis must be done with what information is available. FDS deals with this situation in two ways. Firstly, when the input data are entered they are checked as far as is possible and if FDS suspects that the data may be incorrect it warns the user or does not accept the data. In many cases FDS displays a range of acceptable values for the data as a guide to what is realistic. Secondly, the calculation sequences within FDS are hierarchical. Depending on which data are missing, a sequence of assumptions are made in order to carry out the calculation. The key features of the *Data Analysis Module* include:

- Analysis of constant pressure filtration test data;
- Analysis of jar test sedimentation data;
- Analysis of piston press or consolidation data;
- Checking of input data to protect against possible incorrect data entry;
- Interactive graphical presentation of data to enable the user to input interpretations of the measured data;
- Output data in graphical or tabulated format;
- All data required for process design, simulation and scale-up (and much more) are output;
- Data from several analyses can be compared and correlated.

Two simulation models are available – the *Vacuum Filter Simulation Module* and the *Pressure Filter Simulation Module*. These provide the

calculation sequences for 10 types of vacuum filters and 11 types of pressure filters.

1.6 Shortlisting equipment for pilot scale testing

Although the procedures described in the preceding sections are able to produce a short list of equipment, it is possible to further refine equipment choice. This requires consideration of additional selection parameters (indicated through Tables 1.3 to 1.6) and provides a basis for deciding which types of solid/liquid separation device merit the time and cost of executing detailed testing.

The selected shortlist of equipment may be further shortened if the applications problem is more precisely defined or if additional laboratory experiments are carried out. The additional tests, indicated on Table 1.3, are dependent on the type of separator in the selected list. For example, if the initial selection has not uncovered magnetic separations as potentially suitable then there is no point in attempting to carry out the magnetic tests shown on Table 1.3. It is likely that some form of filter will be on the initial shortlist, and further filter leaf tests will be necessary. Whether these are pressure leaf or vacuum leaf tests depends on the filter type(s) on the selected list, but in either case it may be prudent to consider the need for flocculants at this stage (refer to Chapters 2, 5 and 6 for more information about flocculants and vacuum and pressure filter tests). The effects of pressure will need to be evaluated for pressure filter applications. Carrying out the appropriate tests in Table 1.3 will provide more information about the slurry and/or eliminate some equipment from the initial shortlist.

Many other facets of the process and the materials being separated may need to be taken into account during refinement of the selected equipment list. Table 1.4 is an *aide-mémoire* that details aspects of the process to be considered at this stage. Not all will be relevant during any one selection procedure, but against each aspect is a comment which may have relevance to the final choice of equipment.

Also, it is essential to take more account of the properties of the solids and liquids to be handled during the separation; these are listed in Tables 1.5 and 1.6. The properties of the slurry are also important, but any particular property of the slurry will normally be dominated by the corresponding property of one of the phases to be separated. Many properties of the slurry (e.g. solids concentration in the slurry, filtrate fluxes, settling and filtration rates, slurry flow characteristics, particle

Table 1.3 Further tests to be performed on a suspension. (Preliminary selection has identified a range of equipment types that are likely to be suitable. Further tests need to be performed only for those equipment types identified in the preliminary list.)

Process	*Nature of test*
Settling	Centrifugal. Using a laboratory centrifuge with an acceleration of about 1000 g, and various spinning times, determine the consistency of the settled solids (firm paste, flowable, etc.) and the time taken for the solids to settle and the supernatant liquid to clear. These tests are helpful, but not necessarily essential.
Filtration post-treatment	Cake washing. If washing is envisaged a preliminary indication of the likely ease of washing is required at this stage. Feed wash liquid to the cake, measure solute concentration in the washings as a function of the washing time. A total volume of wash equal to the volume of formed cake should usually provides adequate data for this stage in the selection. Cake dewatering. (Dried in situ). Suck or blow air through the cake for various times, and measure the moisture content of the cake at these times.
Magnetic	Immerse a hand magnet (~ 0.2 Tesla) protected by a plastic bag in a beaker of slurry and determine the mass of (ferromagnetic) solids that collects at the poles of the magnet. The amount of paramagnetic material in a slurry can be measured only with more specialised equipment generating ~1 Tesla.
Flotation	Used to recover small amounts of suspended solids, and requires particles to be hydrophobic so that they attach to air bubbles. Hydrophobicity can be conferred by use of chemical additives in the suspension. 'Natural' hydrophobicity can be determined by bubbling fine air bubbles through about 1 litre of suspension; any hydrophobic particles should float.
Electrofiltration	Requires the particles to be small and charged; a measure of the zeta potential is helpful. Assessing the potential of electrical assistance is relatively difficult and requires specialist apparatus.

size distribution and their state of aggregation in the slurry, and pH) will have been ascertained in a well designed experimental programme. It is also important to perform some kind of sensitivity analysis to assess the likelihood and extent of variations in flow rate and solids content of the feed slurry, the particle size distribution in the feed, and temperature. These can have a marked effect on equipment performance, are greatly influenced by process operations upstream of the separator, and may have a profound influence on the economics of both the separator choice and the overall process.

Table 1.4 Process requirements to be considered during equipment/process selection.

Property	*Comment*
Scale of operation	Total slurry volume to be separated affects equipment choice. For large volumes, either large scale equipment or greater numbers of smaller units are needed. This is particularly true when the feed solids concentration is low, giving large liquid volumes to be handled. It may be necessary to consider a staged separation to concentrate the solids to an intermediate level before a complete separation is attempted (e.g. use a thickener to pre-concentrate the feed to a vacuum filter).
Required phase	Reason for separation is to remove the solid from the liquid phase. Either or both phases may be the desired product (in the case of many waste treatment processes neither are necessarily 'wanted' products). Identification of uses for either 'product' of the separation can affect economics and the need for subsequent treatment.
Solid product	This may come from the separator as either a cake or as a slurry. Downstream processes or uses may decide the best solids product moisture content (e.g. only a narrow range may be permissible if the solids are to be briquetted). It is possible for solids product size distributions to differ from feed distributions.
Liquid product	The solids content and/or the degree of saturation by soluble impurities are important properties of the liquid product. It is preferable for liquids not to be close to saturation.
Washed solids	Cake washing may be needed to improve the purity of the solids product, or to increase the recovery of the liquid phase. Solids product purity may be improved by reslurry washing when the wash time is constrained unduly by the equipment design (e.g. this may occur with rotary drum filters). Reslurrying involves additional equipment for re-suspending the cake solids and re-filtering. Washing may have a great effect on process economics, such as when the wash liquor is a solvent that must be regenerated.
Dewatered solids	See 'solid product' above.
Process integration	*With downstream processes*: Downstream processes often put a requirement on some of the solids properties (e.g. moisture content, continuity of solids production). Very dry cakes can lead to solids handling difficulties. *With upstream processes*: Upstream processes determine if the separator needs to be operated in a batch or continuous mode, or if it will have to handle a range of products. The type of process used to form the particles (e.g. reactor, crystalliser, etc.) will often control the size distribution of the solids, which in turn has a marked effect on separation rates.

Table 1.4 *continued*

Property	*Comment*
Process integration	*With ancillary equipment*: Pumps, instruments, feed and effluent tanks need to be specified. Pump type has an effect on filtration rates and filter sizing; use of the incorrect type may cause solids product degradation.
Use of additives	*pH reagents and coagulants.* pH reagents used for acidity or alkalinity control. Both types of chemical additive may affect the state of dispersion (i.e. aggregation) of the particles. *Flocculants.* Generally polymers, used to aggregate fine particles to improve their settling and/or filtration rates. *Filter aids.* Used as a precoat to prevent blinding of filter media, or as body aid to add bulk to a feed and to produce a more permeable filter cake. Only useful when the solid is not a desired product. In cases where the solid is the product, separation of the filter aid is usually required after filtration. *Surfactants.* Have some use to assist dewatering of the filter cake by lowering the surface tension of the liquid and/or changing the contact angle (wettability) on the solid.
Equipment reliability	Experiences of operating equipment for the separation of similar slurries are helpful here. Users of a large range of equipment types should build up a reliability data bank.
Space availability	New equipment may need to fit into an existing plant. Need to consider headroom and floor area, together with feed and discharge port elevation/orientation.
Product value	See entries in Tables 1.5 and 1.6.
Special requirements	See 'toxicity', 'volatility' and 'flammability' in Tables 1.5 and 1.6.

Table 1.5 Properties of the solid phase to be considered during equipment/process selection.

Property	*Comment*
Chemical composition	The chemistry of the system should be known so that possible reactions due to changes of pH, solvent, temperature or pressure may be anticipated. This requires identification of the solids present, which may not always be possible.
Solids concentration	Best operation of most separator types is obtained for a limited range of solids concentration in the feed slurry. If solids recovery is required from a low concentration feed, staged separation should be considered. This often involves pre-concentration before filtration, and does not necessarily imply the use of similar types of separator at each stage.

Table 1.5 *continued*

Property	*Comment*
Size distribution	Size and size distribution of the particles should be determined – these have a marked effect on sedimentation and filtration properties. Many separators operate most effectively over a limited size range.
Density	The density difference between the particles and the liquid controls sedimentation (or flotation) rates. Small differences may make some centrifugal techniques unsuccessful (due to short residence times). Porous particles tend to have a lower density.
Particle shape	Shape affects particle packing density and specific surface, which has a marked effect on pressure rise in constant rate filtrations (or filtrate flux decline in constant pressure processes). Needle shaped particles lead to lower pressure losses than equi-axial particles. Platelet shaped particles can be difficult to wash and dewater.
Particle strength	Zones of high shear in equipment (e.g. centrifuges) will fracture brittle particles, leading to a broadening of the particle size distribution and (generally) a reduction in cake porosity.
Solubility	Filtration from saturated (or near saturated) solutions can cause nucleation and crystal formation in filter media, and hence blocking in the media or feed pipe work. During washing, the solubility of the particles in the wash liquor must be taken into account to avoid excess losses.
Toxicity	Closed equipment is desirable for handling toxic systems to avoid creating a health hazard. Manual handling of separation products should be avoided wherever possible.
Chemical reactivity	Reactive substances may be explosive or flammable, and require the selection of completely enclosed equipment. Special seals, flameproof motors and appropriate switchgear are needed. Cake dewatering may need to be done using gases other than air, and nitrogen purging of equipment may be needed prior to discharging products. (After nitrogen purging, care must be taken to avoid air (oxygen) condensation on equipment and products, together with sources of ignition).
Sterility	Ingress of contaminants should be avoided and may require fully enclosed separator designs. Contamination prevention of product(s) during and after discharge also requires special consideration.

Table 1.5 *continued*

Property	*Comment*
Abrasivity	Abrasive solids cause rapid wear of equipment when its design causes high velocities and/or rapid changes of direction of the solids flow (e.g. as in the entry zones of scroll and decanter centrifuges). Materials selection then becomes of greater importance.
Magnetic properties	The response of the solids to magnetic fields may enable selection of magnetic separators. Ferromagnetic substances respond to fairly weak fields (~0.2 Tesla) such as those generated from permanent magnets. Paramagnetic and diamagnetic substances respond only to high intensity fields (>1 Tesla).
Surface properties	Surface charge affects the state of dispersion of the particles, and is pH dependent. Close to the iso-electric point aggregation tends to occur. Surface charge determines adsorption of flocculants and dispersants. Surface hydrophobicity/philicity can be modified to make particles respond to flotation.
Value	The product value, combined with the scale of operation, has a bearing on equipment selection. If the solid is the desired product, addition of filter aids may not be allowable.

Table 1.6 Properties of the liquid phase to be considered during equipment/process selection.

Property	*Comment*
Chemical composition	Chemical composition of the liquid phase affects separability of the particles. Electrical conductivity may determine suitability of electrically assisted separation processes. Paramagnetism may prevent use of high intensity separation methods.
Density	See Table 1.5.
Temperature	Temperature affects liquid viscosity, effectiveness of flocculants, dewaterability, and may influence materials of construction.
pH/ionic strength	Affects extent of aggregation and choice of flocculants and dispersants by altering electrical charge on the particle surfaces. Plays an important role in determining corrosivity of liquid, which in turn affects choice of materials of construction.
Viscosity	Controls rate of sedimentation/filtration. Higher rates are obtained at lower viscosities, which may be achieved by elevating the temperature.

Table 1.6 *continued*

Property	*Comment*
Toxicity	See Table 1.5.
Volatility	Containment of volatile liquids by fully enclosed equipment is essential, particularly if toxic or flammable dangers are associated with the vapours. Variation of the vapour pressure of the liquid with temperature and pressure is important, especially with vacuum filtration equipment. Vapour formation in or just downstream of the filter medium or in vacuum lines and pumps should be avoided.
Flammability	Containment of flammable vapours by enclosed equipment is essential. Hoods may be suitable under some circumstances.
Sterility	See Table 1.5.
Surface tension	Combined with the particle/liquid contact angle, controls the moisture content of dewatered filter cakes.
Value	See Table 1.5. The greater the value of the liquid, the more important it may be to prevent losses. If the liquid is the desired product, the addition of solute additives (e.g. flocculants) may not be permitted.

Careful consideration of the above factors should lead to the elimination of further equipment from the initial shortlist and identify those separators worthy of pilot scale testing and/or detailed computer simulation. However, there is a further category of information that may be useful at this point, that is information based on the accumulated experience of equipment users and suppliers. Much of this type of information exists as proprietary knowledge owned by equipment suppliers and users, and clearly only that data in the public domain can be accessed. Purchas (1981) and Pierson (1990) provide coverage of a wide range of separations equipment, with particularly useful information provided by Purchas. Applications details of centrifuges (Day, 1974; Ambler, 1971; Moyers, 1966), hydrocyclones (Svarovsky, 1984), classifiers (Hawkes, 1970), gravity filters (Pierson, 1990), pressure filters (Emmett and Silverblatt, 1974), filters with compression (Blagden, 1975), vacuum filters (Blagden, 1975; Dahlstrom, 1978; Moos and Dugger, 1979), crossflow filters (Porter, 1990) and magnetic separators (Watson, 1990) are also available in the public literature. More information is given in Chapters 3 to 8 of this book.

1.7 Conclusions

The procedures outlined in this chapter enable the non-expert to make rational decisions based on expert knowledge, without the need to consult an expert in the earlier stages of solving the problem. This is important in solid/liquid separation – not least because the expert is often a representative of an equipment manufacturing company whose job it is to sell a particular type of separator! Taking filters as an example, many types are usually capable of carrying out a particular filtration, but probably only a few types will be most suited to the task. It is wise to have an insight into which these are before consulting an expert.

The procedures outlined, or used in Filter Design Software (2005), enable rapid analysis of data and exploration of alternatives in the earlier stages of the selection process. It puts the engineer in a position to ask more penetrating questions of whichever expert may be consulted, and reduces expenditure on unwarranted pilot scale test work too early in the selection process.

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2 Chemical pre-treatment

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A major consideration for any solid/liquid separation operation is the requirement for pre-treatment chemicals. The type of pre-treatment required is dependent upon a large number of factors with the key ones being; the suspension to be treated, the solid/liquid separation techniques being used, any throughput constraints, and the outputs required.

The majority of the pre-treatment chemicals used increase the effective particle size of the solids to be separated, by various aggregation mechanisms. It is important to understand the basic theory of the behaviour of particles in suspension in order to identify the most appropriate chemicals to evaluate. This will be discussed in Section 2.1.

The main chemical groups used will be discussed in some detail. Guidance will be provided in terms of the product types to consider under different circumstances. The importance of mixing and application techniques will be highlighted, as will some of the pitfalls to avoid.

Towards the end of the chapter a number of laboratory protocols will be described to illustrate the most appropriate methods to use for specific solid/liquid separation equipment. This will be aimed at optimum product choice rather than scale-up considerations, as this is being dealt with elsewhere in the book.

2.1 Basic theory of suspensions

When in suspension, most particles exhibit a surface charge. This is illustrated by the fact that when a voltage is applied, particles migrate to the pole of opposite charge. In aqueous systems, most particles migrate to the anode, and therefore exhibit an overall negative charge.

This surface charge can arise for a number of reasons: ionisation of surface groups, uneven distribution of constituent ions, ionic substitution, or specific adsorption of ions. As particles approach each other, the presence of the electrical charge causes repulsion between them, and any tendency to agglomerate, which is usually observed as coagulation or flocculation, is opposed.

The effective range of the electrostatic repulsive forces is relatively large, of the order of 300 nm. Within any suspension system, other forces operate, such as Van der Waals forces of attraction. These forces are relatively strong but they are also short ranged, in the order of 5 to 10 nm.

A result of these opposing forces is that, as particles approach each other, a build up of potential energy is observed (Shaw, 1989). However, if the particles can be brought closer together this potential energy and the so called barrier to coagulation can be overcome. Figure 2.1 provides a diagrammatic representation of this effect.

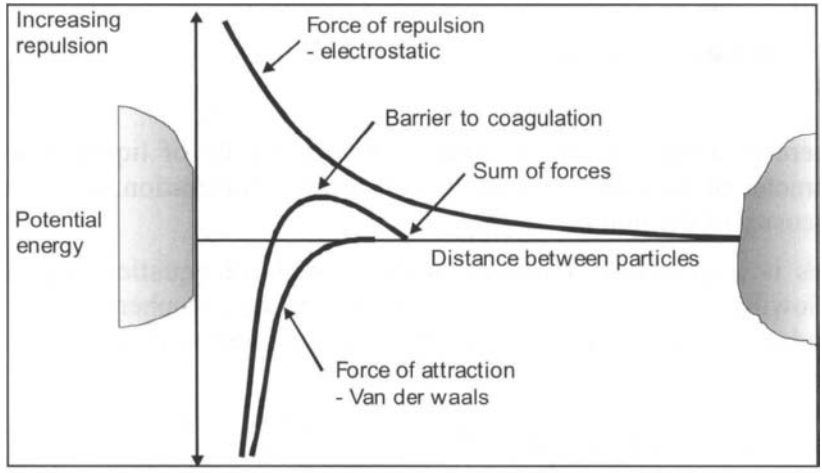


Figure 2.1 Potential energy diagram – as two particles approach one another

Once the particles are close enough agglomeration will result and the apparent particle size will be increased. The barrier to coagulation can be overcome in a number of ways:

- *Mechanical Agitation* – using mechanical agitation puts energy into the system and can cause coagulation by forcing particles closer together. It is observed most frequently when dealing with low solids systems.

- *Reduction of electrostatic charge* – This can be brought about in a number of ways. (i) Addition of inorganic salts – these provide a shielding effect and reduce the thickness of the double layer (Moss, 1978). (ii) Adjustment of pH – particularly observed in inorganic systems e.g. where the solid is a mineral. The pH can be adjusted to the isoelectric point, or point of zero charge when "self-coagulation" can be observed (Greenwood, 2003). (iii) Addition of inorganic or organic coagulants. Both of these entities have the effect of neutralising the charge on the surface of the particles or causing partial, localised charge reversal on the particle surface (Gregory, 1973).
- *Physical bridging between the particles* – High molecular weight polymer flocculants are large enough to adsorb onto a number of different particles and bring them together physically.

The benefit of increasing particle size, with respect to a sedimenting centrifuge, can be illustrated by the use of Stokes Law that states:

$$\text{Settling Velocity} = \frac{(\rho - \rho_0) d^2 G}{18\mu} \tag{1}$$

where ρ is the density of solid, ρ_0 is the density of liquid, d is the diameter of the particle, G is the centrifugal acceleration, and μ is the viscosity of the liquid.

This is a simplistic approach in that the above equation makes the following assumptions: the shape of the particle is spherical, the suspended solids content is dilute, and the suspension is in a quiescent state. However, although this deviates somewhat from the practical situation, it does identify an exponential relationship between particle size and settling velocity. Therefore any means of increasing the apparent particle size should have a significant impact on solid/liquid separation efficiency.

2.2 Pre-treatment chemicals

2.2.1 pH modification

Adjustment of pH can be undertaken for a variety of reasons:

- To bring the pH into a range that is acceptable for the plant process e.g. to prevent corrosion.

- To bring the pH into a range that is acceptable for effluent disposal or for re-use of the liquor in the plant.
- To precipitate soluble components e.g. to remove heavy or transitions metals from a system.
- To promote coagulation by achieving as far as possible the point of zero charge.

The reagents used to adjust pH include simple mineral acids, alkalis such as sodium hydroxide or calcium hydroxide (lime) and some of the inorganic coagulants such as ferric chloride or aluminium sulphate. Where multivalent metal salts are used to adjust pH then they can have the dual effect of inducing coagulation through charge neutralisation, as described in Section 2.2.2. The alkaline reagents such as sodium hydroxide and, more notably, calcium hydroxide are also useful as metal precipitants. They can cause bulk precipitation of a number of metals by formation of the hydroxide salts that can then be removed by standard solid/liquid separation techniques. In some cases, to achieve very low discharge consent limits on residual metal ions, it may be necessary to use this as a first step in a process, where the liquor is then further treated in a polishing step that could include specific propriety chemicals, slow sand filtration (Muhammad *et al*, 1997) or biological means (Weijma *et al*, 2002).

pH modifiers are relatively easy to handle as they are supplied as either low viscosity solutions or as easily dissolved/dispersed solids. However, some caution in handling is required since, by their very nature, they are often corrosive to skin tissue and eyes as well as equipment, in their concentrated or solid forms. This is due to the fact that they are inherently acidic or alkaline. Consideration must also be given to the possibility of chemical reactions that could occur as well as the simple adjustment of pH. This has to be assessed on a specific case by case basis.

2.2.2 Inorganic coagulants

The vast majority of the reagents in this category are the salts of multivalent metal ions, which includes Ca^{2+}, Fe^{2+}, Fe^{3+} and Al^{3+}. These coagulants work by neutralising the surface charge, which as stated earlier is invariably negative, thus reducing the barrier to coagulation and effecting aggregation. These are very cost effective chemicals but do have some disadvantages e.g. most have a fairly narrow effective pH range and also affect the pH of the system in their own right. Aluminium sulphate and ferric chloride are acidic in nature and there-

fore lower the pH of the system, calcium hydroxide, on the other hand raises the pH. It is therefore sometimes necessary to use other pH modifiers to correct the balance. The products are cost effective, but have to be used at relatively high dose levels, some also, as part of their mode of action, precipitate and add solids to the system, thus adding to the volume of solids to be disposed of.

They are particularly effective in dilute solids systems, e.g. polishing filtration, low solids effluent treatment, etc. where their high dose level and tendency to work via a precipitation mechanism greatly assists in solids capture and this effect can, at times, be difficult to obtain by any other reagent.

A further specialised group of products are the polyaluminium chlorides (PAC). These are particularly effective inorganic coagulants and are generally used in similar situations to the more traditional products described above. In solution they form a net structure that assists in solids capture and they have a tendency to form larger flocs than aluminium sulphate, the closest comparison from the standard inorganic coagulants (Malhotra, 1994). In addition PAC tends to be more dose efficient than aluminium sulphate.

2.2.3 Organic coagulants

Organic coagulants are generally synthetic polymers that have molecular weights from around 250,000 up to 1,000,000. Due to the normal overall negative charge of most systems these organic coagulants are usually highly cationic in nature. There are a number of different chemical types but most common are based on either quaternised polyamines or polyDADMAC (poly diallyl dimethyl ammonium chloride). Rather than reducing the overall negative charge as occurs with the inorganic flocculants, it is believed that these products work by a charge patch mechanism. This is shown diagrammatically in Figure 2.2.

Organic coagulants are useful in applications similar to those described in Section 2.2.2. They tend not to be effective in heavy metals removal but can be useful in removal of colour (humates) from drinking water (Greville, 1997) and some textile effluents.

Organic coagulants are also, at times, used in combination with high molecular weight flocculants. In particular, the addition of a coagulant improves filtrate and centrate clarities, when the flocculant treatment alone is unable to attain acceptable levels. A further common combination treatment is the pressure belt filtration of mineral slurries, where the coagulant is added after an anionic flocculant. In this application the

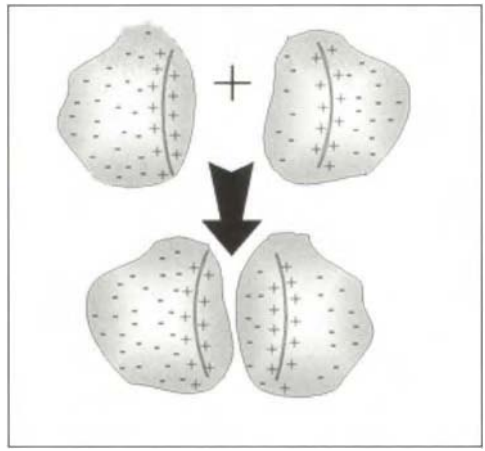


Figure 2.2 Envisaged charge patch mechanism

coagulant appears to strengthen the floc formed resulting in improved cake formation and discharge.

2.2.4 Bridging flocculants

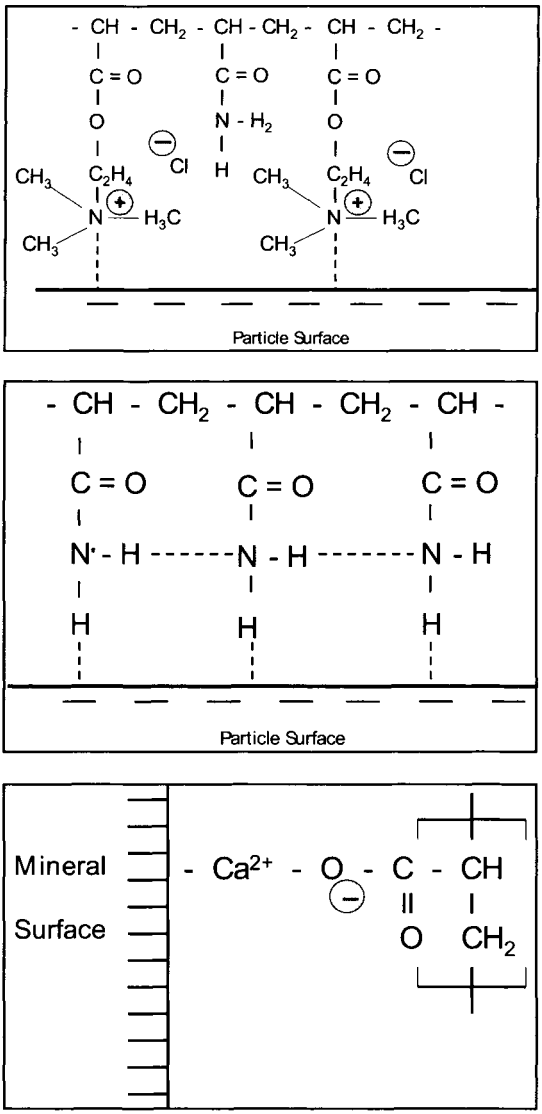
2.2.4.1 The mechanism of bridging flocculation

Bridging flocculants as described below are of sufficiently high molecular weight to attach to one or more particles essentially simultaneously (Deason, 1987). This is in contrast to coagulants previously discussed that affect each particle independently of the others. In order to bring about flocculation the flocculant must adsorb onto the solid particles and this is brought about in one of three main ways:

- *Electrostatic Attraction* – for this to be effective the flocculant must carry a charge that is opposite to that of the particle surface. This is the most likely mechanism occurring when treating organic materials such as sewage sludge or paper effluent with cationic flocculants.
- *Hydrogen Bonding* – δ positive charge on the non-ionic flocculant causes adsorption on to the negative sites on the particle. It is likely that non-ionic products such as polyacrylamide and polyethylene oxide adsorb via this mechanism. Whilst the individual hydrogen bond may be quite weak the overall adsorption can be strong due to the relatively high number of bonds that can be formed.
- *Salt Linkage* – on mineral surfaces, the surface charge is uneven mainly as a result of the availability of metal ions. The negatively charged pendant groups on anionic copolymers can form salt

linkages with the cationic metal ion. This is the main reason why it is possible to flocculate mineral particles that carry an overall negative charge with anionic flocculants.

Each of these modes of adsorption is illustrated in Figure 2.3.



(a) Electrostatic attraction of a cationic polymer onto a negatively charged surface.

(b) Hydrogen Bond formation of a non-ionic polymer onto a negatively charged surface.

(c) Salt linkage formed between anionic polymer and positively charged metal ions at an overall negatively charged mineral surface.

Figure 2.3 Modes of adsorption

Whatever the mode of adsorption, it is anticipated that the flocculant becomes attached to one or more particles but leaves loops and tails in

solution that can become attached to other particles or to more sites on the same particle. As time progresses and greater adsorption occurs then the particles are drawn closer and closer together forming a floc. The size of the floc formed is dependent upon the particle size of the material being treated and the characteristics of the flocculant applied. The effect of the flocculant characteristics is described in more detail below.

In the vast majority of cases flocculation is an almost instantaneous effect, the limiting factor tends to be the rate at which the substrate (which at times can be viscous) and the polymer solution (also often viscous) mix together homogeneously. It should also be noted that continued mixing after floc formation will cause a gradual and permanent breakdown of the floc. It is unlikely that the original substrate particle size will be reached but such breakdown can result in the requirement for higher flocculant doses than would otherwise be required.

2.2.4.2 Natural products

The first flocculants that appeared on the market were natural polymers usually derived from crops and include starches and dextrin (Halverson and Panzer, 1980). By and large they have been superseded by synthetic products, as described below but there are still niche markets where the fact that they are natural products can have benefit, for example in treating materials that may be included in animal feedstuffs. It may be that, due to environmental pressures, natural products or natural/synthetic hybrids could gain higher profile in the future as a result of factors such as sustainability, biodegradability, etc.

2.2.4.3 Synthetic polyelectrolytes

Synthetic flocculants based on polyacrylamide and copolymers of acrylamide with anionic and cationic co-monomers have been available for over 40 years. Significant changes to the products have taken place over this period of time, probably the most noticeable being that of increasing molecular weight (Mohammed *et al*, 2000). Such polymers lead to commercially desirable dose efficient dewatering compared to lower molecular weight analogues (Weir *et al*, 2002). The initial products probably had molecular weights in the region of one million, whilst today many of the products used probably have molecular weights of 25 million or more. In fact it is very difficult to actually state molecular weight with any accuracy as it defies even the most sophisticated of analytical instruments to obtain a true measure. Some techniques such as Flow-Field Flow Fractionation are getting closer to

attaining this measure but further development is still required (Hecker *et al*, 2000). The polyacrylamide group of reagents have considerable widespread use in industries ranging from drinking water treatment to sewage treatment and from paper making to copper extraction. Today many of the latest innovations in solid/liquid separation equipment rely on the use of these products.

The secret to their success is their versatility, and this is mainly due to the fact that it is possible to copolymerise acrylamide, which is non-ionic, with ionic monomers (whether anionic or cationic) in any ratio to give products tailored to specific substrates.

There are other polymer types, such as polyethylene oxide, that are used and that have niche applications but it is the polyacrylamides that currently predominate.

The variables that can be controlled by the polymer chemist are ionicity, molecular weight and structure and these are described in further detail below.

2.2.4.4 Ionicity

It is possible to vary the ionic content of the polyacrylamides from 100% cationic through all ratios to non-ionic and through all ratios with anionic monomers to 100% anionic content. Figure 2.4 identifies some of the most commonly available monomers.

When considering possible flocculant choice then it is the chemical characteristics of the substrate that dictates what ionic character is likely to be the optimum. Perhaps surprisingly, the chemical constituents that affect optimum ionicity are not always the solid particles i.e. those being flocculated, but rather the soluble constituents. This can be illustrated in its simplest form by evaluating products on a kaolinite slurry. Moody (1995) has shown that the optimum ionic content changes from 5:95 w/w sodium acrylate:acrylamide to 50:50 w/w sodium acrylate:acrylamide simply by changing the slurry pH from 2 to 7.

Due to the complex nature of suspensions it is difficult to provide hard and fast rules in terms of optimum ionic content for specific substrates, however there is some general guidance that can be given. This is presented in Figure 2.5.

2.2.4.5 Molecular weight and structure

Acrylamide based polymers are manufactured through a radical addition polymerisation (Kulicke *et al*, 1982). The molecular weight of products can be modified by the polymer technologist in a variety of ways. Conditions that can be controlled include:

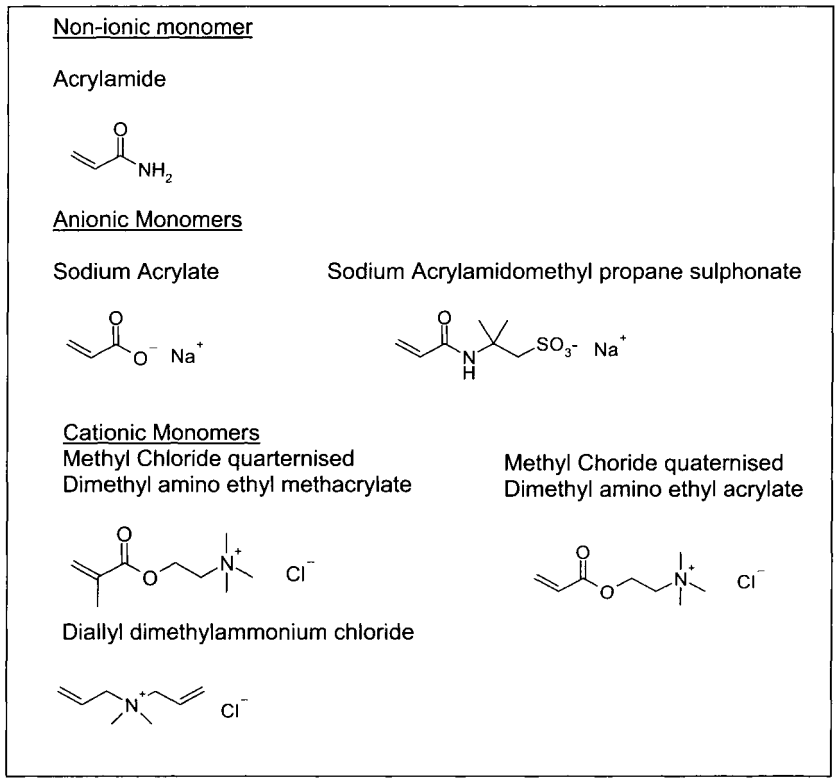


Figure 2.4 Chemical structures of common monomers

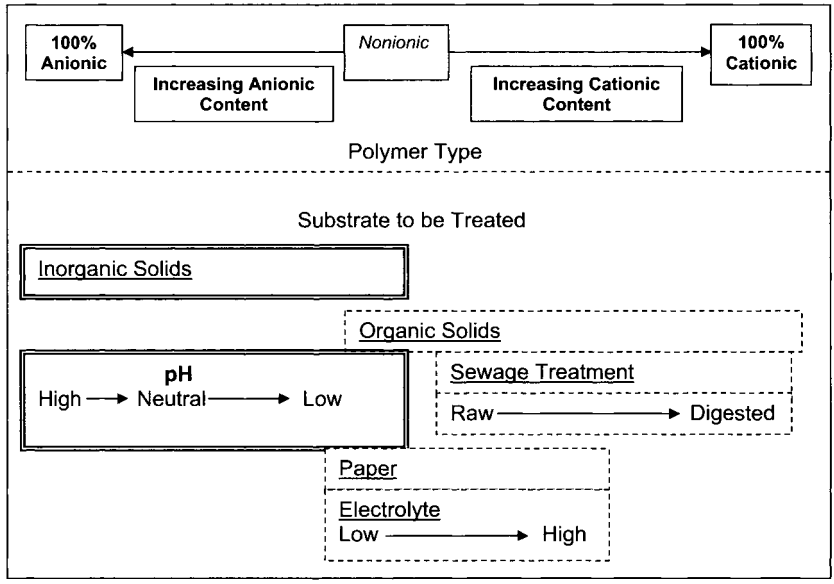


Figure 2.5 General guide to optimum ionic content by substrate type

- Monomer concentration
- Concentration and type of initiators used
- The use of chain transfer regents
- The use of cross linkers
- Temperature.

All of these can affect the average molecular weight and the molecular weight distribution of the polymer produced. The average molecular weight can range from a few thousand up to 25 million or more, but it is only products that have an average molecular weight of at least one million that will act as flocculants.

In contrast to ionicity where the substrate is the determining factor, the choice of molecular weight and structure is usually determined by the solid/liquid separation technique being employed. To illustrate this, if one considers that a typical sewage sludge will probably require a product of 60%w/w cationicity to flocculate it, it does not matter whether it is being dewatered using a belt press or a centrifuge. However, what will change is the optimum molecular weight. A general approach is that the more rigorous and shear intensive the separation process used then the higher the molecular weight and the higher degree of structure is required. Figure 2.6 provides a diagrammatic representation of this.

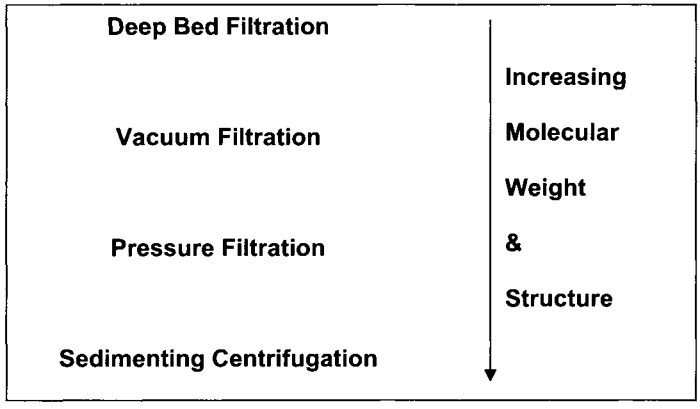


Figure 2.6 General guide to optimum molecular weight by solid/liquid separation technique

Figures 2.5 and 2.6 have provided some very general guidelines to help in choosing the optimum product for an application, however the final choice is often arrived at through empirical testing. The difficulty in being able to accurately predict the most appropriate reagent is due to

the complex nature of the substrates being treated, where the combination of components (often minor components) can drastically affect the outcome.

2.2.5 Surfactants

The generic term surfactants covers an extremely wide range of chemical types from foamers and defoamers, flotation reagents, detergents, etc. The role of surfactants in solid/liquid separation is significantly less than that of flocculants and coagulants described above. There are two main areas where surfactants do find application. Firstly as dewatering aids, mainly in vacuum filtration and in batch pressure filtration where their effectiveness is derived from an ability to lower the surface tension of the liquor and thus enhance the ease of water removal (Besra *et al*, 1998). Details of the mechanism involved has been published by Pearse and Allen (1981). There has been little development in this area in recent times and the sulphosuccinates still prevail in this application.

The other area of use is that of foam control. The creation of excess foam is deleterious to solid/liquid separation processes. Foam can be an environmental nuisance as it can become airborne, it can also trap solid fines. In some cases it is the fines themselves that stabilise the foam. A variety of antifoams and defoamers are available to help prevent foams forming or destabilise foams once they have formed.

2.3 Test protocols

In the protocols described below, the main pre-treatment chemicals under consideration are coagulants and flocculants, as these are by far the majority of the chemicals in use to specifically aid solid/liquid separation.

It should be noted that most of the test methods described are batch tests and these are being used to simulate what, in most cases, are continuous processes.

2.3.1 Substrate preparation and characterisation

2.3.1.1 Substrate sampling and preparation

The correct preparation of the sample of substrate to be treated is absolutely key in ensuring correct product choice. Items that need to be considered include:

- *Bulk sample size* – sufficient sample needs to be taken such that, if possible, all products to be evaluated can be compared on the same substrate over a range of dose levels. If this is not possible then careful thought needs to be given to experimental design. If several different batches of substrate have to be used then it is recommended that products are directly compared at similar doses on the same batch of substrate, rather than using a different batch for each product. The number of "control" tests required also needs some careful consideration.
- *Sample ageing* – the rate of sample ageing needs to be identified. If it is rapid then it may be necessary to use different batches of sample since, in this case, the ageing effects could be more significant than any batch-to-batch variation.
- *Sampling point* – wherever possible the sample obtained should be taken from a point immediately prior to the addition of the pre-treatment chemical under investigation. One particular problem area is that of liquor re-circulation. In closed circuits the liquor being returned can contain some of the pre-treatment chemicals that are under evaluation, so it can prove to be very difficult to obtain samples that are truly untreated. It is also desirable to take the sample from a fast flowing vertically downward or upward flow pipe as opposed to a horizontal pipe or open channel. This is due to the increased possibility of sedimentation or stratifying in a horizontal pipe, particularly in a slow flowing region.
- *Sub sampling* – all actions possible should be taken to ensure that sub samples are representative of the whole and that there is no trend developed as the bulk sample is depleted. A typical example would be that if there is insufficient mixing during sub sampling then there is likely to be a gradual increase in suspended solids content due to settlement, particularly of coarse material.
- *Temperature* – it is important that the tests are conducted at a temperature that is as close as possible to that encountered in the practical situation. Temperature can affect the speed of the separation process itself, the product of choice and the final results obtained in terms of cake solids, etc. Where the substrate is treated at elevated temperatures it is recommended that, as well as conducting the test work at the appropriate temperature, the substrate is not allowed to cool between sampling and testing. The reason for this being that if the substrate is allowed to cool, soluble constituents may precipitate out onto the surface of the suspended solids. On reheating they may not always re-dissolve, thus changing the surface of the solids to be treated. Changing the solid's surface in this way can have a

significant effect on the efficacy of pre-treatment chemicals, since surface adsorption is a major mechanism, and could also result in incorrect choice of product.

- *Other reagents or conditions* – it is necessary to ensure that reagents, other than the one under investigation, that are being used are also present in the substrate at the correct level and at the correct time e.g. it needs to be ensured that pH is correct. Dependent upon where it has been possible to take samples, other reagents may or may not have to be added, for example reagents will have to be added if the only sampling point is before the reagent is present in the system, or if a further reagent is added after the pre-treatment chemical being evaluated.

2.3.1.2 Substrate characterisation

It is important to characterise the test substrate for a number of reasons that includes identifying batch to batch sampling variations that could affect the efficacy of different products, confirming or otherwise that the sample obtained is typical of the substrate to be treated, providing information that may help in choosing the most appropriate pre-treatment chemical. Typical measurements could include:

- Total solids content
- Suspended solids content
- Dissolved solids content
- pH
- Conductivity
- Appearance (colour, odour, etc)
- Ash content or volatiles content.

2.3.2 Reagent preparation

The reagents described above are available in a wide variety of physical forms. In most cases it is necessary to prepare an aqueous solution, if the products are solids or emulsions as supplied, or dilute from a concentrate. In determining the most appropriate solution strength to use there are two main considerations. Firstly wherever possible the reagent solution used should be of a similar strength to that likely to be used in practice on full scale. The second consideration is that of accuracy. The strength and volume of solution used should take into account the ease of weighing/measuring the solid or

concentrate to the accuracy required, and subsequently the volume of the reagent to be added to the test substrate. Both of these factors can result in solutions of lower or higher strength being used in laboratory evaluations compared to the full scale situation, the effect that this may have on ease of mixing and subsequent results is discussed in Section 2.3.3.

For the majority of types of chemicals discussed, the reagents are either readily soluble solids or easy to handle concentrates. The exception to this is the high molecular weight flocculants group where care in solution preparation for both laboratory test work and full scale plant operations is required. These products are hydrophilic, and as soon as they come into contact with water they begin to swell. They continue to absorb water until fully hydrated and then form an homogeneous solution, this can take up to two hours for solid grade products. In the initial swelling stage, if the individual particles are not kept separate they can clump together and form large lumps that can then be very slow to dissolve. To overcome this effect there are specific laboratory techniques, one of which is described in detail below. For full-scale use, there are a number of specialised preparation/dissolving units available on the market.

The normal solution strength prepared in the laboratory usually ranges from 0.25% to 1.0% dependent upon the specific product being prepared. This concentrated solution is usually diluted around ten fold to form the working strength solution.

2.3.2.1 Example

500 cm^3 of make-up water is placed in a 600 cm^3 beaker and stirred using a mechanical paddle type stirrer at a speed sufficient to create a vortex. 2.5 g of dry polymer is sprinkled into the vortex taking care that the polymer doesn't form clumps. (If clumps do form the solution should be discarded). Once all the polymer has been added the stirring speed is reduced to a level that ensures there is full movement of all the solution, but low enough to prevent shearing of the polymer. Stirring is continued for two hours or until a fully homogeneous solution is formed. This solution can then be diluted to the appropriate working strength solution.

If a 40% active emulsion polymer is used in place of the solid grade polymer, then 6.25 cm^3 of product should be expelled into the vortex via a syringe. To ensure that an accurate solution strength can be calculated, the syringe should be weighed before and after the emulsion is expelled so that the exact quantity of emulsion added can be determined.

2.3.3 Mixing and application techniques

It is well known that choosing the correct mixing regime and application technique is as important as ensuring optimum product choice (Gregory, 1981). It is also known that the flocculation efficiency of a fixed dose of flocculant can be minimal if poor dispersal occurs due to insufficient mixing or be similarly minimal due to floc destruction due to excessive mixing shear (Weir and Moody, 2003). The maximum flocculation efficiency occurs with intermediate mixing where efficient flocculant dispersal is achieved and subsequent mixing leads to floc forming collisions. Therefore it is important that when conducting any application test work that both mixing regime and application technique are given careful consideration. As with solution strength, conditions that are likely to be encountered in the full-scale situation should be replicated as closely as possible.

Factors to consider include:

- Solution strength
- Solution viscosity
- Solids concentration of substrate
- Mixing regime – length of time, shear applied, type of mixing
- Single point or multi point addition.

The types of product where these considerations take on most importance are the flocculants whether they are natural products, such as starch, or high molecular weight synthetic flocculants. Flocculation is not a permanent effect. Floc formation, subsequent breakage and reformation occurs over a short, but definitive period of time (Farrow and Swift, 1996). Sufficient mixing needs to occur to provide intimate contact of flocculant and substrate to ensure flocculation occurs, but too much mixing will cause the flocs to break and so reduce the effectiveness of solid/liquid separation. In full-scale plant it is not always possible to ensure that conditions are conducive to optimum flocculation, for example the best flocculation may be achieved at very low solution strengths but this may result in too much water being introduced into the process or insufficient fresh water may be available; when flocculated substrates are introduced into centrifuges they encounter very high levels of shear which cannot be avoided etc. Therefore when looking at any of the application techniques which follow, the first task is to decide the most appropriate mixing regime to use.

2.3.4 Dose level calculations and definitions

Before commencing any test work there are a few basic calculations and definitions that need to be known and understood.

2.3.4.1 Substrate solids concentration

In quoting substrate solids concentration, it is necessary to distinguish between suspended solids content and total solids content (suspended plus dissolved solids). In high suspended solids systems this is less important as the dissolved solids content is only a small proportion of the total solids, however in low solids systems the dissolved solids content can be higher than the suspended solids content. Lack of clarity could result in misleading conclusions regarding throughput, dose level calculations etc.

It is also important to quote the solids content as weight solids per substrate volume (w/v) or weight solids per total substrate weight (w/w). In contrast to the comments above, regarding dissolved solids, in the low solids system the difference between these two measures is often quite small, whereas in high solids systems, particularly those containing high density suspended solids the difference can be significant.

2.3.4.2 Dose level

Often dose levels are seen quoted as parts per million (ppm), but this can be very misleading as it is dependent upon whether this refers to parts per million of total substrate or parts per million of dry solids. In order to clearly distinguish between these two alternatives it is more appropriate to quote dose level as either grams polymer per tonne dry solids (g/tds) or in milligrams polymer per litre of substrate (mg/l). At the start of a series of test work it is possible that the solids content of the substrate is not yet known and it is therefore easier to calculate dose levels based upon mg/l that can subsequently be converted to g/tds as follows:

$$\text{Dose level (g/tds)} = \frac{100 \times M}{S} \tag{2}$$

where M is the dose level (mg/l), and S is the suspended solids content (% w/v).

Calculation of the volume of reagent to add is carried out as follows

$$\text{Volume of reagent required (cm}^3\text{)} = \frac{D \times 100}{C \times 1000} \times \frac{V}{1000} \tag{3}$$

where D is the dose level required (mg/l), C is the solution strength (%), and V is the volume of test substrate (cm^3).

2.3.5 Qualitative screening tests

A very good starting point when encountering a substrate for the first time is to carry out qualitative tests to determine whether or not certain reagents have any effect. Once some effect is observed then it is worthwhile progressing to specific quantitative tests. Since coagulation and flocculation is a very visual effect, simple screening tests can be devised using visual observations. Typically, the required amount of sample (at least 200 cm^3 is recommend, although if sample is in limited supply, useful information can be obtained from as little as 50 cm^3) is taken in a beaker, using glass or clear plastic to facilitate observation. The beaker containing the feed sample is taken in one hand and a beaker containing the measured out polymer solution is taken in the other. The feed is poured from the one beaker into the other and then back and forth from one beaker to the other whilst visual observation is made of the floc build-up. The number of beaker pours is noted (ten times should be sufficient to see an effect, and the effect may be seen with much less, however to simulate the mixing encountered in a centrifuge then 50 pours or more may be required). An alternative is to induce mixing by mechanical stirring using a paddle blade.

Observations that can be made include assessments of floc size, settlement rate, liquor clarity and the speed of effect.

Often, the dose level required is unknown, and since it is possible to overdose with flocculants and coagulants there can be some danger in testing at a single dose. Where dose requirement is unknown a simple approach is to take a single substrate sample and begin by adding a relatively low dose level e.g. 0.5 mg/l, mixing the substrate and flocculant, and then making further additions, doubling the total dose until an effect is seen. If a total dose of >500 mg/l is reached without any observable effect then it is unlikely that further additions will improve the situation.

These tests alone should not be used to recommend a product for full scale testing, but are appropriate in helping to identify the range of products that are worth evaluating in greater detail.

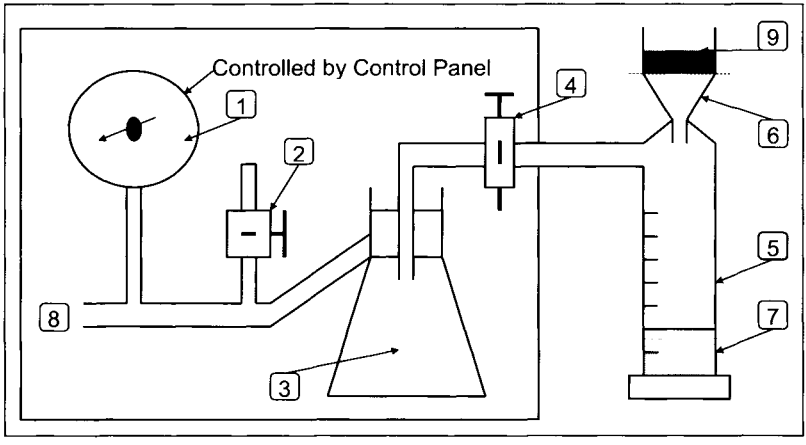
There are a number of sophisticated tools on the market (Barrett and Glennon, 1999) that can more accurately measure particle size (although these are often limited to 1 mm or possible 2 mm maximum size) and in some cases provide images of floc shape etc., but these are for the moment generally limited to use in more fundamental research studies. However, it is anticipated that as the technology develops, they will become increasingly used in the industrial situation.

2.3.6 Vacuum filtration

It should be noted that the first stage of any filtration is cake formation that is then followed by cake dewatering and there are a number of mathematical approaches to modelling this process (Corapcioglu, 1981; Wakeman and Tarleton, 2005). There are two main types of vacuum filtration, rotary vacuum filtration and horizontal vacuum belt filtration (more details are given in Chapter 6). In the former, a main consideration is the efficacy of cake pick up as the filter is submerged into the slurry and solids are drawn onto the filter surface, in the latter the filter is top fed and all the slurry is deposited on the filter bed. However, one problem to be wary of is sedimentation on the belt that can lead to a number of problems including blinding of the cloth. Vacuum filtration has a particular propensity for facilitating cake washing and therefore the test protocols need to be able to accommodate this. Due to the differences in the feed delivery there are different test protocols recommended for horizontal vacuum belt filtration as opposed to rotary vacuum filtration. Having said this, there are a number of aspects that are common to both and will become apparent below.

2.3.6.1 Horizontal vacuum filtration

The most common method used is the Buchner Filtration Test. A typical arrangement can be seen in Figure 2.7. In order that the test mimics the specific process as closely as possible the following need to be established.



1. Vacuum gauge
2. Bleed to Atmosphere
3. Buchner Flask
4. Three-Way Tap
5. Cylinder
6. Buchner/Hartley Funnel
7. Filtrate
8. To Vacuum
9. Cake

Figure 2.7 Buchner filtration apparatus

1. Likely cake thickness – this is dependent upon the substrate solids content and the cycle time, and it is important that the sample size used in the test results in a cake of similar thickness.
2. Cycle time and wash cycles
3. Wash water characteristics if used
4. Vacuum applied
5. Filter cloth type – if possible obtain a used sample of the actual cloth in use. If a brand new sample is obtained this should be conditioned by carrying out a number of tests, washing the cloth in between and discarding the results of the initial tests.

Once these have been established and following the substrate and reagent preparation guidelines given in Section 2.3.1 to 2.3.3 the protocol is as follows.

The appropriate filter cloth is clamped in the filter leaf. The vacuum pump is switched on and the three-way tap is switched to air to allow a build up of vacuum whilst at the same time, leaving the filter leaf open to air. The vacuum is adjusted to the required level.

The required volume of sample (dependent upon the cake thickness required) is sampled into an appropriate size dish or beaker. The contents of the beaker are mixed using a mechanical mixer incorporating a gate stirrer at a speed sufficient to create a shallow vortex. The required dose level of dilute reagent solution is added and stirring continued until a suitable floc structure is formed. The treated slurry is poured carefully into the filter funnel. The three-way tap is turned to apply the vacuum to the filter leaf, and a stopwatch is started. The time taken for the water to disappear from approximately 80% of the surface of the cake is measured (**). This may appear to be a rather subjective measure, but the surface characteristics undergo a very clear visual change at this point. An alternative means of carrying out the test is to measure the filtrate volume against time and record the time taken to release a specified volume of filtrate. A further standardised drying time is allowed dependent on the filter cycle, but it is usually around 1 minute. The tap is turned to release the vacuum.

The cake is removed and the cake moisture content, or solids content, is determined by placing the cake in a pre-weighed beaker, drying at an appropriate temperature (usually around 100 °C) and reweighing (see calculation below).

Cake thickness, filtrate volume with time and filtrate clarity can also be measured.

The filter leaf and cloth should be cleaned thoroughly before commencing the next test.

2.3.6.1.1 Modification to include cake washing

The procedure in Section 2.3.6.1 is followed to (**). At this point the required volume of wash water, containing any reagents necessary, is added and again the time taken for the liquor to disappear from 80% of the surface of the cake is measured. This can be repeated as many times as necessary. Once as many cycles of wash water as required have been applied then the procedure is continued from point (**) to the end.

An additional measurement that may be useful is the volume and chemical analysis (dependent upon the reason for washing) of the wash water following each wash cycle which will give an indication of the efficiency of cake washing.

It is possible to use the Buchner Filtration test to measure more fundamental characteristics of the sludge being dewatered, such as specific resistance to filtration (Osborne, 1976) and also to predict the washing performance attainable (Wakeman, 1972).

2.3.6.1.2 Calculations

$$\text{Cake moisture content (\%)} = \frac{(W_2 - W_3)}{W_2 - W_1} \times 100 \tag{4}$$

where W_1 is the weight of the dish, W_2 is the weight of the dish and wet cake, and W_3 is the weight of the dish and dry cake.

An estimation of throughput can be made as follows:

$$\text{Cake throughput (kg m}^{-2}\text{h}^{-1}) = \frac{W \times 3600}{T \times A \times 1000} \tag{5}$$

where W is the weight of dry cake (g), T is the time taken to 80% removal (seconds), and A is the area of the filter leaf (m^2).

Typical results are shown in Table 2.1. Three flocculants of similar molecular weight (~ 10,000,000) but of varying copolymer ratios were tested on a sample of coal tailings with the following characteristics:

Total solids content = 7.1% w/v
Specific gravity = 1.00
pH = 6.6

Table 2.1 Horizontal vacuum filtration test work.

Product	*Dose* (g/tds)	*80% Dry Time* (s)	*Cake*		*Filtrate Volume* (cm^3)
			Moisture (%)	*Throughput* ($kg\ m^{-3}\ h^{-1}$)	
Blank	–	56.0	32.6	158	315
Product X					
(5:95 NaAc:ACM)	10.5	46.9	23.2	189	355
	17.5	26.5	22.8	377	345
	24.5	18.1	22.9	593	345
Product Y					
(15:85 NaAc:ACM)	10.5	43.3	32.9	246	330
	17.5	18.7	29.4	516	340
	24.5	17.8	23.1	584	362
Product Z					
(30:70 NaAc: ACM)	10.5	31.4	22.8	318	350
	17.5	16.8	20.8	731	340
	24.5	12.9	21.8	911	350

NaAc = Sodium Acrylate; ACM = Acrylamide.

These results show the benefit of all of the flocculants tested, they all provide an increase in throughput of at least four fold, and at the same time give a significantly drier cake. There is a definite trend of improving results with increasing anionic content with Product Z being the product of choice for this substrate in terms of both throughput and cake moisture content.

2.3.6.2 Rotary vacuum filtration

Buchner filtration as described above may be used to test reagents for rotary vacuum filtration but it does not have the ability to fully assess the efficiency of cake pick up, which is a significant factor when considering this application. A better simulation is the inverted filter leaf test (see also Chapter 6), which only requires some minor modifications to the apparatus used and the set up is as shown in Figure 2.8.

The appropriate filter cloth is clamped in the filter leaf. Vacuum is applied and allowed to build up as described in the Buchner filtration procedure.

The appropriate volume of sample, typically 500 cm^3, is taken in an appropriate size measuring cylinder. The cylinder is inverted a number of times to ensure that the sample is homogeneous. The required dose

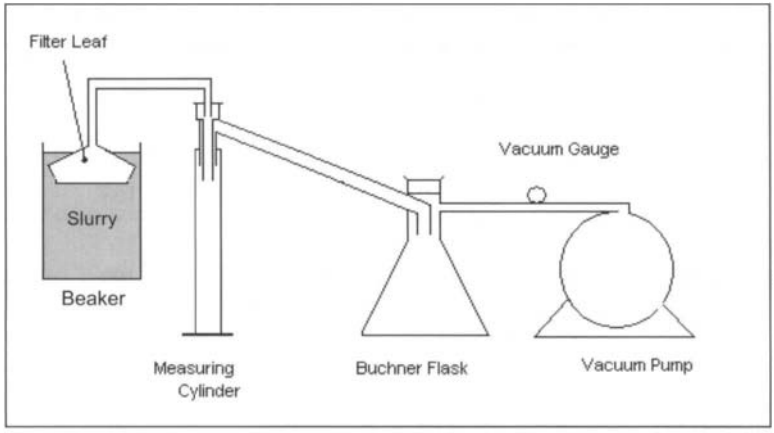


Figure 2.8 Laboratory rotary vacuum filtration apparatus

level of reagent is added and the cylinder inverted four times to obtain efficient mixing. Alternatively, reagent addition and mixing can be carried out directly in the beaker, as described under the Horizontal Vacuum Filtration procedure.

The treated slurry is transferred swiftly, but gently into an appropriate, typically 800 cm^3, beaker and the inverted filter leaf is immersed in the slurry. A gentle up and down motion is maintained in order to minimise solids settlement. Vacuum is applied to the filter leaf, and a stopwatch started.

After allowing the required pick-up time (usually in the range 20 to 60 seconds), the filter leaf is removed from the sample and kept inverted for half the drying time (usually 20 to 60 seconds) before turning the filter leaf upwards for the remainder of the drying time (usually 20 to 60 seconds). The timings used should be matched to the particular rotary drum or disc filter cycle being simulated.

The vacuum is released and the cake removed. The cake moisture content, or solids content, is determined by placing the cake in a pre-weighed beaker, drying at an appropriate temperature (usually around 100 °C) and reweighing (see calculation below). For rotary vacuum filtration, the dry cake yield provides an indication of cake pick up, this is calculated from the weight of the dry cake measured when carrying out the moisture content determination.

Filtrate volume and clarity can also be measured. The filter leaf and cloth should be cleaned thoroughly before commencing the next test.

A useful means of comparing different treatments is to plot graphs of

dose level against moisture content and dose level against dry cake yield for each of the products evaluated. In some instances a plot of moisture content against dry cake yield can also provide further insight. As dry cake yield increases it is anticipated that moisture content would also increase as the filter cake formed is thicker. Therefore a product that increases dry cake yield with the minimum increase in moisture content is of particular interest.

If the effectiveness of wash water is to be evaluated, or a top loading rotary vacuum filter is in use, then it is probably more appropriate to use the Buchner Filtration Test as described in Section 2.3.6.1.

2.3.6.2.1 Calculations

$$\text{Cake moisture content (\%)} = \frac{(W_2 - W_3)}{W_2 - W_1} \times 100 \tag{6}$$

where W_1 is the weight of the dish (g), W_2 is the weight of the dish and wet cake (g), and W_3 is the weight of the dish and dry cake (g).

$$\text{Dry cake yield (kg m}^{-2}) = \frac{(W_3 - W_1)}{1000} \Big/ A \tag{7}$$

where A is the area of filter leaf (m^2).

Typical results can be found in Figures 2.9 and 2.10, where Products Y and Z (as described in Section 2.3.6.1.2) were compared with each other.

Both substrates are coal frothed fines from two different sources. The characteristics are as described in Table 2.2.

In both series of tests the cycle time used was 20 seconds pickup with a total of 40 seconds drying time.

There are a number of interesting points from the comparison of the series of tests. The only apparent difference between the two substrate samples is that Sample 1 is of significantly lower solids content but has a slightly higher specific gravity than Sample 2, which possibly indicates a higher clay content. The results obtained though are quite different, Sample 2 requires a much lower flocculant dose to achieve a similar throughput, but moisture contents are considerably higher than Sample 1. In terms of product of choice then Product Y (lower anionic content) is the more effective product on Sample 1 in terms of both moisture content and throughput, whilst Product Z is the more effective product on Sample 2.

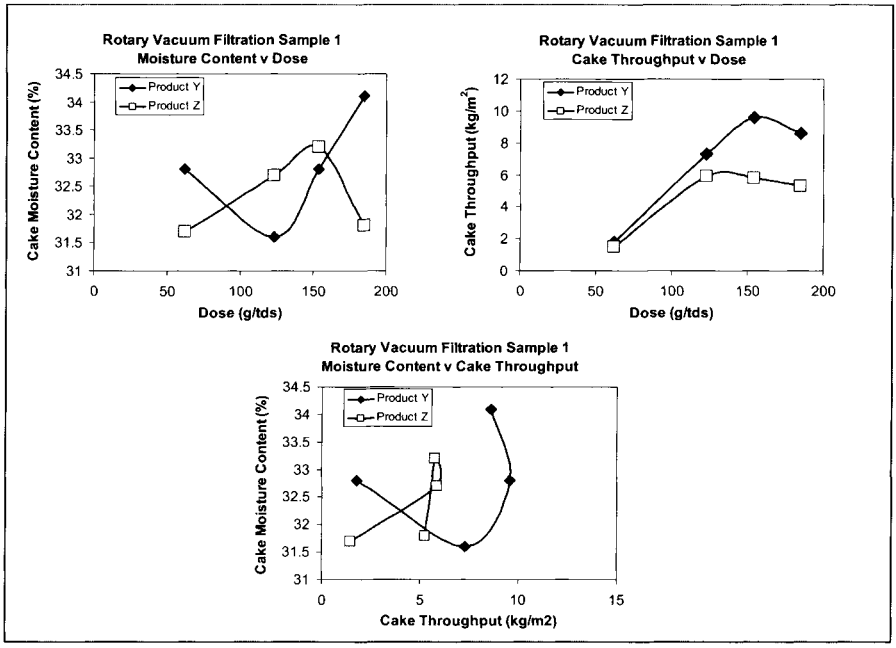


Figure 2.9 Rotary vacuum filtration results - Sample 1

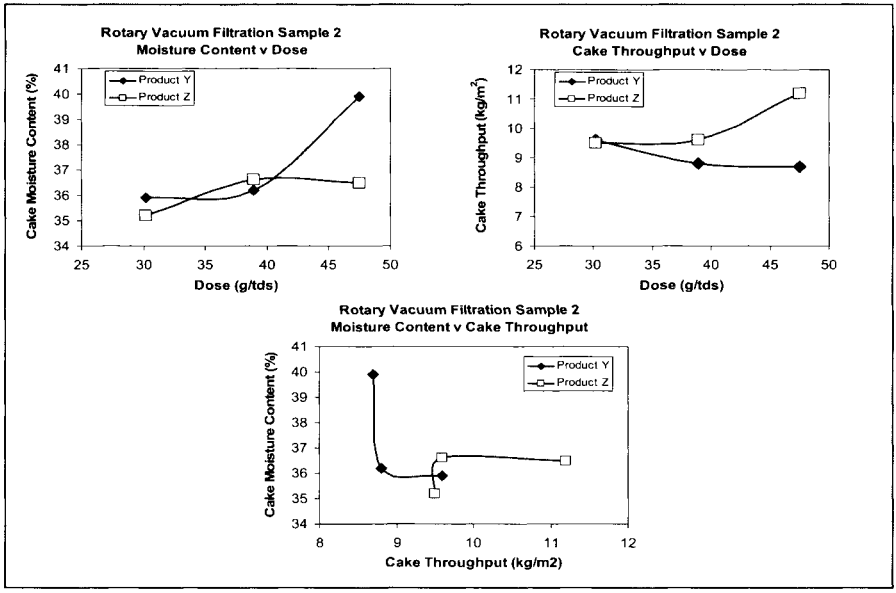


Figure 2.10 Rotary vacuum filtration results - Sample 2

This illustrates that even on two samples of substrate that one would expect to be quite similar in characteristics, different products are required to gain the best solid/liquid separation effect.

Table 2.2 Sample characterisation for rotary vacuum filtration test work.

	Sample 1	*Sample 2*
Total solids Content	16.2% w/v	23.3% w/v
SG	1.05	1.00
pH	6.9	7.0

2.3.7 Pressure filtration

There is a vast array of different types of pressure filters on the market. However, in terms of pre-treatment evaluations it is possible to use a generic procedure for them all. In all cases the substrate will go through a cycle of cake formation, initial dewatering and finally compression dewatering (see also Chapter 5).

A standard test is the piston press test, and this can be modified to suit the particular press being simulated. The two main factors that need to be considered are the level of pressure applied and the cycle time used. The type of cloth in use should be identified and, if possible, a used sample of the actual cloth in use should be obtained (if a brand new sample is obtained this should be conditioned by carrying out a number of tests, washing the cloth in between and discarding the results of the initial tests).

The main disadvantages of the piston press test are:

- The filtration is one sided – generally speaking in a pressure filter whether it is a high pressure batch filter, or a continuous pressure belt filter, a cake is formed and pressure is applied to both sides of the cake. In the laboratory apparatus pressure is applied from one side via a solid piston.
- No shear is applied to the surface of the semi-formed flocculated cake. This is particularly relevant for continuous pressure belt filters, as when the two belts come together, and begin to squeeze the cake, a reasonably high level of shear can be applied. This can have a combination of effects. On full-scale plant the shear applied could result in a higher solids cake being produced than appears possible in the laboratory; alternatively, it could have a detrimental effect and cause floc breakdown that is not observed during laboratory evaluations.

The piston press apparatus is as illustrated in Figure 2.11 and requires a control system that can apply hydraulic pressure in a controlled manner, and also have a means of applying vacuum to move the piston

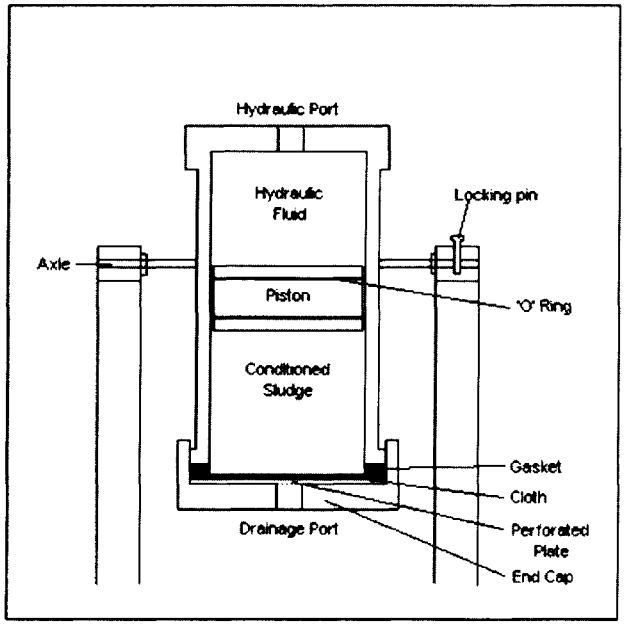


Figure 2.11 Piston press apparatus

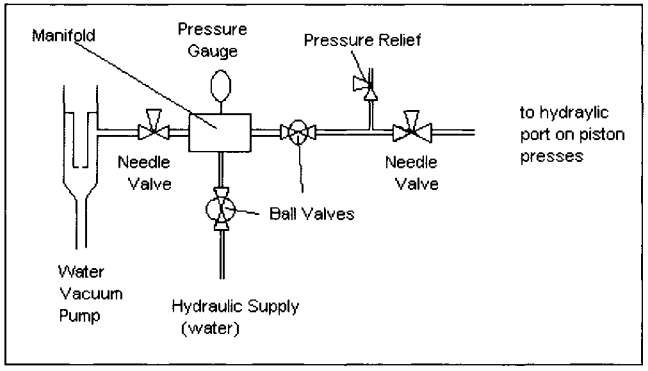


Figure 2.12 Typical control system for piston press apparatus

when empty or when the pressure cycle is complete. An example of a typical control system is illustrated in Figure 2.12.

The apparatus consists of a stainless steel cylinder, with a precision-ground bore. At one end is a hydraulic port and at the other is an end-cap held in place by a threaded collar. Inside the cylinder is a freely moving piston bearing 'O' rings that provide a water-tight seal. The end-cap retains a circle of filter cloth on a perforated plate through which filtrate is conducted to a central drain port. The cylinder is

mounted on an axle, allowing rotation in the vertical plane, and can be locked in either vertical position.

The required volume of slurry is taken (this will depend upon the cake thickness required and the solids content of the slurry) in a suitable beaker and stirred using a mechanical paddle stirrer at a speed sufficient to create a shallow vortex.

The required dose level of dilute reagent is added (in some cases it is useful to add a constant volume of water/solution, see explanation below) and stirred for the required period of time (until a suitable floc structure is formed when using flocculants, or for at least 2 minutes when using surfactants).

If conducting test work for batch pressure filters the treated slurry is transferred to the piston press as described below. However, if the test work is aimed at treatment in a continuous pressure belt filter then, at this point, the treated slurry is placed in a free drainage apparatus (see Section 2.3.8.3, Free Drainage) and allowed to free drain for a specific period of time. The filtrate clarity and volume are measured. The thickened slurry is then transferred to the chamber of the piston press.

A test is conducted by locking the press in position with the end-cap uppermost. The end-cap is removed and the piston is drawn down the cylinder by means of a vacuum pump. Flocculated feed suspension is then charged into the cylinder and the end-cap, with an appropriate filter-cloth in place, is replaced and the collar screwed down tight. By means of an air-hydro pump, the piston is gently raised until filtrate just appears at the drain port. The press is rotated through 180° and locked in position. A measuring cylinder is placed under the drainage port and hydraulic pressure is incrementally raised in a prescribed manner according to the pressure/time cycle required. Throughout the pressure cycle, the volume of filtrate is collected and measured versus time. This can be carried out manually using a suitable sized cylinder and a stopwatch, or by placing an appropriate collecting vessel on a digital balance that is connected to a suitable computer.

On completion of the pressing cycle the pressure is released, the press is rotated, the end-cap removed and the cake retrieved for determination of dry solids content. The whole cake is removed and the solids content determined by placing the cake in a pre-weighed beaker, drying at an appropriate temperature (usually around 100°C) and reweighing (see Section 2.3.7.1, Calculation).

A graph of the filtrate volume collected against time can be plotted. When plotting this graph, one of the following conditions must be followed:

1. If a constant volume of reagent solution was added (i.e. each reagent dose was made up to an equal volume with water and for the untreated sample, an equivalent volume of water was added), then the plot can be simply filtrate collected versus time.
2. If varying volumes of flocculant solutions were added, and for the untreated sample no water was added, then the filtrate volume plot should take this into account (i.e. the volume of solution added should be subtracted from the total volume of filtrate collected).

2.3.7.1 Calculations

$$\text{Cake solids content (\%)} = \frac{W_3 - W_1}{W_2 - W_1} \times 100 \tag{8}$$

where W_1 is the weight of dish (g), W_2 is the weight of the dish and wet cake (g), and W_3 is the weight of the dish and dry cake (g).

From the plot of filtrate volume collected versus time, it is possible to calculate the time taken to achieve a specified solids content (S) as follows:

(1) Weight of dry solids in feed, $D = \left(\frac{W^v \times V}{100} \right)$ (9)

where W^v is the solids content of the feed (% w/v), and V is the volume taken for each test (cm^3).

(2) Weight of water in feed, $H = \left(\frac{W^v \times V}{W^w} \right) - D$ (10)

where W^w is the solids content of the feed (% w/w).

(3) Weight of water removed to achieve solids content

$$= H + F - \left(\frac{D \times 100}{S} - D \right) \tag{11}$$

where H is the weight of water in feed (g), and F is the weight of flocculant solution/water added (g), D is the weight of dry solids in the feed (g), and S is the desired solids content (% w/w).

Since 1 cm^3 of water weighs 1 g, the weight of water removed can be directly read off the plot of filtrate volume collected versus time to give the time taken to remove that volume of water and hence achieve the required solids content.

2.3.7.1.1 Example of batch pressure filtration evaluation

Results from test work conducted on a copper concentrate to simulate a plate and frame type filter can be seen in Table 2.3 and Figure 2.13. Here a flocculant reagent, Product Y was compared with an untreated sample and a surfactant reagent (di-octyl sulpho succinate).

Table 2.3 Results of piston press test work on a copper concentrate.

Product	*Dose* (g/tds)	*Final Moisture Content of Cake*	*Yield* (%)	*Time to achieve Solids Content* (minutes)		
				50% w/w	60% w/w	70% w/w
Blank	-	18.5	93.4	2.1	3.8	4.9
Product Y	49	19.4	96.2	2.3	3.5	3.9
Surfactant	49	18.5	96.8	1.8	3.5	4.6

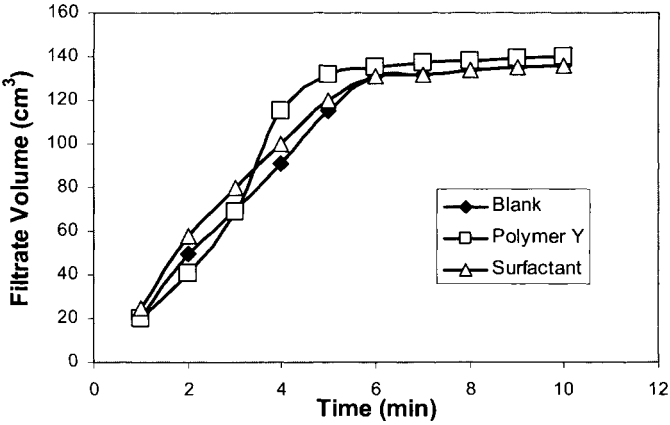


Figure 2.13 Piston press results

The copper concentrate has a total solids content of 43.6% w/w and a specific gravity of 1.41. A total cycle time of 10 minutes was used with a maximum pressure of 9×10^5 Pa. These results show that dependent upon the outcomes required by the plant both the surfactant and the flocculant show benefits.

The surfactant provides an improvement of around 15% in initial dewatering over the untreated sample, in the first few minutes. The flocculant, Polymer Y, on the other hand shows poor performance initially but then is superior to both the untreated sample and the

sample treated with surfactant after around 4 minutes dewatering. After the full cycle is complete neither product shows any improvement over the untreated sample in terms of residual moisture content, but it is likely that either treatment could achieve this level of performance in a shorter time. Both treatments also give a higher yield than the untreated sample, suggesting that both are capable of retaining more of the fine solids than the untreated sample without causing a detrimental effect on residual moisture content.

2.3.7.1.2 Example of continuous pressure belt filter test work

300 cm^3 samples of primary activated sludge were treated with a number of different cationic flocculants. The treated samples were allowed to free drain for 1 minute and then transferred to a piston press for a total of 5 minutes to a maximum pressure of 4×10^5 Pa. The characteristics of the sludge are shown in Table 2.4.

Table 2.4 Characteristics of sludge used in pressure belt filtration test work.

Colour:	BLACK	Viscosity:	12.0 cP
Odour:	RAW	Fibre: Ash:	18.58% 18.62%
pH:	6.08	Particle Size:	
10 mm CST:	1253	-(v, 0.1):	10.53
18 mm CST:	284	-(v, 0.5):	49.55
Dry Solids:	3.39%	-(v, 0.9):	159.3
Organics:	65.18%	Conductivity:	4.49 mS cm^{-1}
Anionic Value:	0.2832 meq/gDS	Surface Area:	3746 cm^2 cm^3

Figure 2.14 shows a plot of the solids content of the cake against flocculant dose level. The flocculants evaluated were all of similar molecular weight of approximately 10 million. They ranged in cationic content as follows:

Polymer K – 20% w/w

Polymer L – 40% w/w

Polymer M – 58% w/w

Polymer N – 76% w/w.

The results indicate that on this substrate there is an increasing performance obtained as the cationic content of the polymer increases

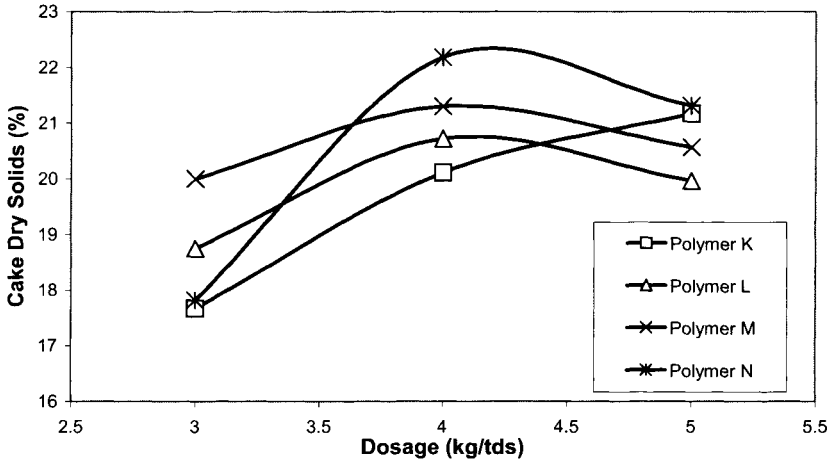


Figure 2.14 Piston press results simulating a continuous pressure belt filter

and for this particular substrate Polymer N is the product of choice, with an optimum dose level of approximately 4 kg/tds.

2.3.8 Centrifugation

Solid/liquid separation and dewatering occurring within a sedimentation centrifuge (see also Chapter 8) are complex dynamic processes and therefore extremely difficult to simulate in the laboratory; consequently, there is no one test which can be used to fully assess the effectiveness of chemical pre-treatments. There are, however, a number of laboratory tests that focus on particular aspects of the process, such as the application of high shear, high *g* force or high compressive force, and these, either alone or in combination, aid in the selection of an appropriate chemical pre-treatment.

The tests, described below, are as follows.

- Beaker test
- Sheared CST
- Free-drainage test
- Centrifugal sedimentometry
- Piston press test.

2.3.8.1 Beaker test

Initial screening of polymers can be carried out by a simple procedure called the beaker (or jar) test (Lecey, 1980; AWWARF, 1989; CIWEM,

1999). Solutions of the polymers to be evaluated are first made up at concentrations of 0.1 to 0.5% in deionised or distilled water. Each solution thus prepared is then applied over a range of doses, via a syringe, to samples of the feed suspension or sludge measured out in beakers, and assessment made of the flocculation produced.

Two methods of mixing the polymer dose into the feed may be used. The first, which is very simple and requires the minimum of equipment, is the beaker-pour technique that was described in Section 2.3.5, but it is also included here for completeness. A beaker containing the measured out polymer solution is taken in one hand and a beaker containing a feed sample is taken in the other. The feed is poured from the one beaker into the other and then back and forth from one beaker to the other whilst visual observation is made of the floc build-up. The number of beaker pours is noted. For centrifuges, up to 50 beaker pours may be necessary to simulate the high shear mixing encountered. Alternatively, mixing may be achieved by mechanical means using a high-speed laboratory stirrer. The stirrer should have a controllable speed and be fitted with a marine or paddle-blade type impeller. Mixing speeds of 500 up to 2000 rev/min, or higher, are used. Typically, mixing is carried out for 10 seconds up to, say, 1 minute. The greater intensity of mixing provided by the laboratory stirrer probably provides better simulation of the high shear conditions inside a centrifuge than does the beaker-pour technique.

Whichever method of mixing is used the object of the test is to observe the extent of flocculation achieved. This entails visual assessment of floc size and structure and clarity of free water (i.e. clarity of supernatant after settling). For centrifugation a large, well-formed floc is required, with diameter as large as 10 to 20 mm, and clear free water. If the floc is small and grainy, a higher polymer dose will be required. If the floc appears slimy and poorly formed, and the free water is cloudy, this is an indication that insufficient mixing has been used and it will be necessary to increase the mixing speed or time, or the number of beaker pours. Typical results are shown in Table 2.5.

In some cases visual assessment of floc size may not be appropriate, for example solid/liquid separation of fermentation broth using a disk-stack centrifuge. Since the flocs cannot be measured accurately with the naked eye it is necessary to use either (a) magnification, namely, microscopic analysis or enlargement of a photographic image, or (b) instrumental particle size measurement. With (b) flocs can be sized directly using a number of techniques such as the Coulter counter and laser light scattering. With both (a) and (b) sizing of the flocs is possible using image analysis computer software.

Table 2.5 Beaker test results.

Mixing speed: 1000 rev/min; Mixing time: 20 s.

Polymer	*Polymer dose* ($mg\ l^{-1}$)	*Description of floc*	*Clarity of free water*
Polymer A	50	small	cloudy
	100	small	cloudy
	150	medium	cloudy
	200	medium	slightly cloudy
	250	large	clear
	300	very large	clear
Polymer B	50	small	cloudy
	100	medium	slightly cloudy
	150	large	clear
	200	very large	clear
	250	very large	clear
	300	very, very large (slimy)	slightly cloudy

2.3.8.2 Sheared CST

During the centrifugation process, particularly in the inlet zone where the feed is accelerated up to drum speed, flocs present in conditioned feed suspensions are subject to high shear forces which, by breaking flocs down irreversibly, can have a detrimental effect on the efficiency of solid/liquid separation and result in dirty centrate (Zurcher, 1974; Gosele, 1980; Bell and Brunner, 1983). It is desirable, therefore, to assess floc physical strength when evaluating pre-treatments for centrifugation. The method consists of adding the pre-treatment chemical to be tested to the feed suspension and subjecting the suspension to various levels of shear, achieved by high-speed stirring, after which the dewaterability of the suspension is assessed by measurement of capillary suction time (CST). The relative floc strength is assessed by plotting CST values versus time of stirring. A feed suspension with strong flocs will show only gradual increase in CST with stirring time, whereas one with weak flocs will show a significant increase in CST with stirring time.

The sheared CST test utilizes the standard CST apparatus, which is used in conjunction with the standard stirrer/timer apparatus, as originally developed by the Water Research Centre for assessing the dewaterability of sewage sludge (Baskerville and Gale, 1968; HMSO, 1984).

The CST apparatus consists of two separate components: the filtration apparatus and the automatic time-recording unit (Figure 2.15). The former is a rectangular piece of absorbent filter paper (No.17 Whatman chromatography grade) sandwiched between two rectangular blocks of perspex. In the centre of the upper block is a circular hole in which a stainless steel cylinder loosely fits, resting on the paper and forming a sludge reservoir; two such cylinders are provided, one of 1.0 cm internal diameter and the other of 1.8 cm diameter. On the underside of the upper perspex block are two engraved circles, diameters 3.2 and 4.5 cm, concentric with the reservoir. This upper block stands clear of the paper by resting on five stainless steel supports 1A, 1B, 2, 3 and 4 in Figure 2.15. Supports 1A and 1B are specially machined probes in line with the first concentric circle and 2 is a similar probe in line with the second concentric circle. Electrical connections are made from these probes to a terminal box and then to the digital stop-clock. The other two supports (3 and 4) are provided to hold the block parallel with the paper. Liquid from the sludge is absorbed by the filter paper in a roughly circular pattern of increasing diameter. When the liquid front reaches the first pair of probes 1A and 1B the timer starts. When the liquid front reaches Probe 2, timing ceases.

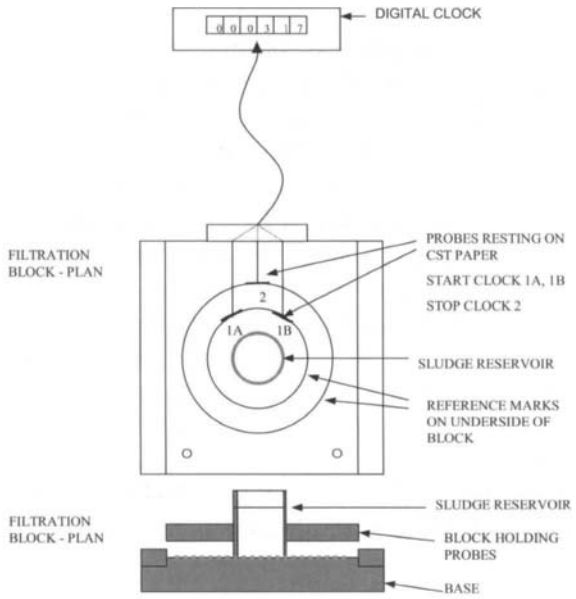


Figure 2.15 Diagram of CST apparatus (Triton Electronics)

The dispersion of polymer solution into sludge, and the subsequent shearing down of the flocs formed is achieved using a Triton Standard Shear Test Stirrer/Timer Type133/131 (Figure 2.16). This unit consists

of a constant-speed stirrer (1,000 rev/min) and a process timer (Gale and Baskerville, 1970; Gale, 1974). The timer, mounted on top of the stirrer, can programme the stirrer to give either fixed (10, 30 or 60 seconds) or variable stir times (1–99 seconds) as determined by the selector switch. Additionally, there is a motor override, allowing continuous stirring, for cleaning the stirring paddle in a beaker of clean water.

Figure 2.16 Standard shear test stirrer/timer (Triton Electronics)

The test is carried out by dosing, via a syringe, various volumes of the conditioning chemical to 100 cm^3 volumes of the feed suspension in a 250 cm^3 beaker and stirring with the stirrer/timer for 10 seconds. The suspension is poured immediately into the reservoir of the CST apparatus, and the CST is measured and recorded. The remainder of the suspension is stirred for a further 10 seconds, and then a second CST is measured. This procedure is repeated using 10 second intervals, or longer if desired.

Control tests may be carried out using the sludge, i.e. feed suspension, as is or with appropriate amounts of water added.

Typical sheared CST results are shown in Figure 2.17, where comparison is made of two polymers, A and B. Polymer A can be seen to provide strong flocs capable of withstanding the applied shear forces,

whereas polymer B can be seen to have given weak flocs that quickly sheared down with subsequent loss of dewaterability. Further, it can be seen that polymer A had not fully dispersed after the initial 10 seconds stirring, requiring a further 10 seconds to achieve minimum CST. This is an indication that polymer A was not fully adsorbed onto the solids after the initial 10 second stirring, the free polymer in solution increasing viscosity and tending to retard the passage of water through the capillaries of the CST paper.

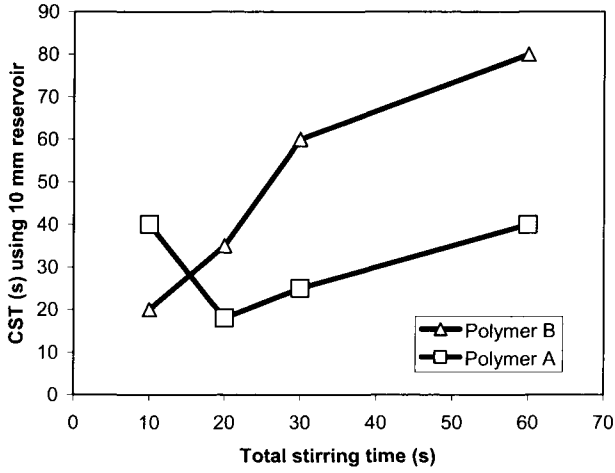


Figure 2.17 Sheared CST test results

2.3.8.3 Free drainage test

Although originally designed to simulate gravity-drainage of suspensions on a filter belt, the free-drainage test is, nevertheless, a useful method for assessing polymer pre-treatments for decanter centrifuges since both mechanical solid/liquid separation processes mentioned have similar requirements from pre-treatment, i.e. large, strong flocs, which readily release free water, and clean filtrate/centrate (Poduska and Collins, 1980; STORA, 1982; WERFRF, 1993; Severin and Grethlein, 1996; Murthy *et al*, 1997).

The free-drainage test apparatus is shown in Figure 2.18, and consists of a purposely-designed funnel assembly positioned over a measuring cylinder. The essential element of the funnel is the section of loose-weave fabric, as used on a full-scale belt press, which serves as filtration medium.

Performing the test involves initially taking a sample of the feed suspension and flocculating it by addition of polymer under controlled

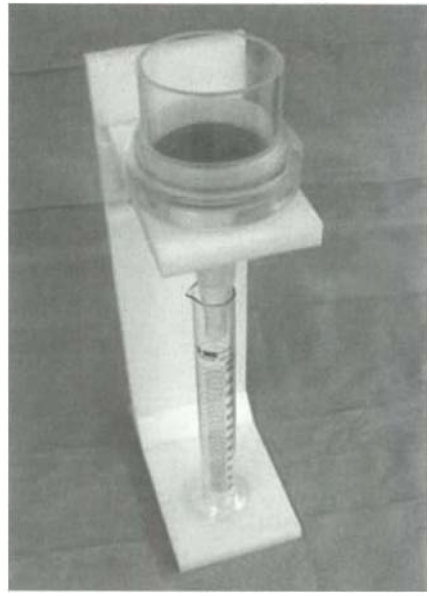


Figure 2.18 Free-drainage test apparatus (Ciba Specialty Chemicals)

mixing conditions, these being achieved either by beaker-pouring or mechanical stirring (as described in Section 2.3.8.1). The flocculated sample is then poured quickly and gently into the funnel and a stopwatch immediately started. Readings of filtrate volume are taken at 5 seconds and, if desired, at successive time intervals as considered appropriate in order to produce a plot of filtrate volume versus time. A note may also be made of filtrate clarity. Each polymer of interest is tested over a dosage range and a plot is made of 5 second filtrate volume versus polymer dose (Figure 2.19). This so-called free-

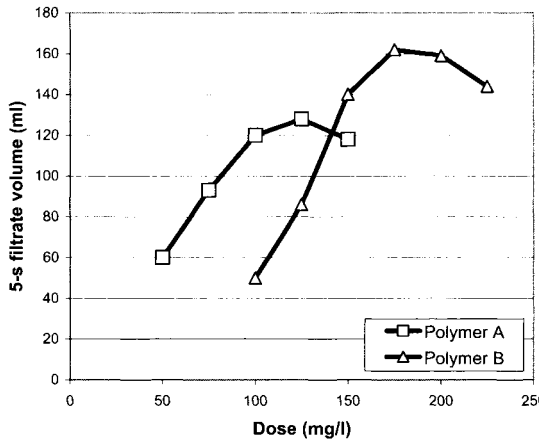


Figure 2.19 Free-drainage curves

drainage curve enables the optimum dose, at which maximum dewatering is achieved, to be determined for each polymer.

2.3.8.4 Centrifugal sedimentometry

Solid/liquid separation tests that assess the ability of solids in a sample to settle and compact under centrifugal acceleration can be conducted using a laboratory centrifuge. Pre-treated samples are spun at an appropriate *G*-force and the behaviour of the suspension is monitored as a function of time and *G*-force. Properties of the suspension that can be measured to give comparative data relating to the effectiveness of flocculants and other pre-treatments are:

- Supernatant quality (cell/particle count, suspended solids content, turbidity)
- Sediment compaction (solids concentration, sediment height).

It should be borne in mind that supernatant quality indicted by this general procedure may be somewhat overestimated compared to the full scale because it is based on a batch-type process, unlike the full scale which is continuous, or semi-continuous, and therefore less efficient in terms of solids recovery.

Vesilind and Zhang (1984) describe a method that measures the compaction of sludge solids, $S_{f(t,z)}$. When solids are centrifuged they settle to the bottom of the tube and an interface between the liquid and the compacted solids is visible. The compaction of solids is calculated as follows:

$$S_{f(t,z)} = \frac{H_0}{H_f} \times S_0 \tag{12}$$

where $S_{f(t,z)}$ is the final compacted solids concentration after t seconds at z centrifugal acceleration (number of gravities) (kg m^{-3}), H_0 is the initial height of sludge (m), H_f is the final height of sludge (m), and S_0 is the suspended solids in the original sludge (kg m^{-3}).

A laboratory centrifuge is used which has a swing-out rotor and holds flat-bottomed graduated tubes of at least 50 cm^3 capacity. Various rotational speeds are used, giving centrifugal acceleration, z, of up to 2000 gravities. After a sample has been spun for a given time and a given speed, the volume of supernatant is measured thus enabling calculation of the compacted solids concentration. Compaction was related to centrifugal acceleration by:

$$S_{f(t)} = S_1 + K_1 \log z \tag{13}$$

where $S_{f(t)}$ is the final compacted solids concentration after t seconds, S_1 is the solids concentration at $z = 1$ (i.e. gravitational acceleration), and K_1 is a proportionality constant.

The term sludge *compactivity*, expressed as $z^{1.5}t$, has been proposed which described the time and energy necessary to achieve a desired compaction under centrifugal force in a given time.

A more sophisticated means of conducting essentially the same test is to use the Lumifuge 114 Separation Analyser (Figure 2.20). This proprietary equipment is described as an analytical centrifuge with an integrated opto-electronic sensor system which allows the local and temporal changes of light transmission to be traced during rotation (Sobische and Lerche, 2002). Up to eight samples can be investigated simultaneously at centrifugal acceleration up to 1200*G*. In preset time intervals the local transmissions are determined over the entire sample length simultaneously whereby transmission profiles are generated. The transmission profiles change during centrifugation according to the progress of separation. The boundary between supernatant and sediment can be traced, thus allowing the dynamic behaviour of the sludge or suspension under centrifugal acceleration to be investigated. A claimed benefit of using the Lumifuge 114 is that it enables sediment volume to be measured under conditions of centrifugal acceleration whereas in common centrifuge tests the sediment volume is measured

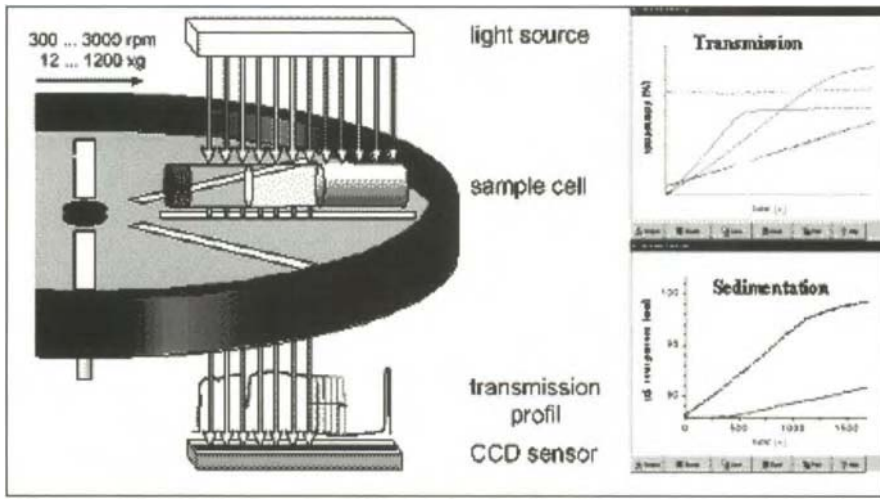


Figure 2.20 Schematic of Lumifuge 114 9 (L.U.M.)

after switching off the centrifuge. Because flocculated solids tend to dilate once acceleration is reduced the level of dewatering predicted by common centrifuge tests is erroneously low.

Flocculant performance stability against high shearing forces, such as may be encountered on the full scale, can also be measured by means of the Lumifuge 114.

An important limitation of compaction tests is that they tend to markedly underestimate the extent of dewatering achieved in practice due to the fact that in a full-scale centrifuge, solids moving along the bowl will experience a final period of drainage resulting in drier cakes. The tests are useful, however, in that they provide estimates of the relative effectiveness of flocculants.

2.3.8.5 Piston press test

Dewatering in the decanter centrifuge takes place in the following stages: sedimentation, compaction, drainage and compression (see Chapter 8). Modern high-solids centrifuges have been designed to accentuate dewatering at the drainage and compression stage in order to produce much drier cakes. These machines are particularly useful in the dewatering of compressible feed slurries. Whilst the dewatering in such devices is too complex to model, some useful information about cake dryness can be gleaned in the laboratory by carrying out piston press tests. These tests provide some insight into how readily the solids will release water when a mechanical squeezing action is applied, as may occur during the all-important drainage/expression stage.

The piston-press test, as described in Section 2.3.7, utilizes a small-scale pressure filtration cell for assessing the dewatering properties of a suspension or sludge when subjected to compression dewatering (Novak *et al*, 1999).

The test method described in Section 2.3.7 is followed. A measuring cylinder is placed under the drainage port and hydraulic pressure is incrementally raised in a prescribed manner reaching a maximum of, say, 4×10^5 Pa after 8 minutes, which is maintained for a further 2 minutes. During the pressing period the volume of filtrate is recorded every 2 minutes.

On completion of the pressing cycle the pressure is released, the press is rotated, the end-cap removed and the cake retrieved for determination of dry solids content.

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3 Deep bed filters

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Deep bed filters are used for the clarification of liquids, often to a very high degree of purity. Normally, the suspensions to be clarified are of relatively low concentration: usually less than 50 mg/litre, and never more than 500 mg/litre. The filtrate quality requirement may allow concentrations as high as 10 mg/litre as in some tertiary sewage treatment, or as low as 0.1 mg/litre (or <1 in Formazin Turbidity Units) as in drinking water supply.

By far the greatest number of deep bed filters is used in municipal engineering for domestic water supply and sewage treatment. Consequently, the installations can be large, for example, some waterworks filter more than half-a-million tonnes of water per day. Therefore, the individual units will be large, of the order of 10 m by 10 m plan area, built in reinforced concrete (Figures 3.1 and 3.2). Smaller units may be in steel vessels 2 m or 3 m in diameter.

It follows that the use of full-scale filters for experimental purposes is not desirable when flexibility is required, for the effort required in making a change in design or operation may be too large to be practicable.

3.1 Operating characteristics

A deep bed filter consists of a container, either open to atmosphere (gravity filter) or a closed pressure vessel (pressure filter), with a floor

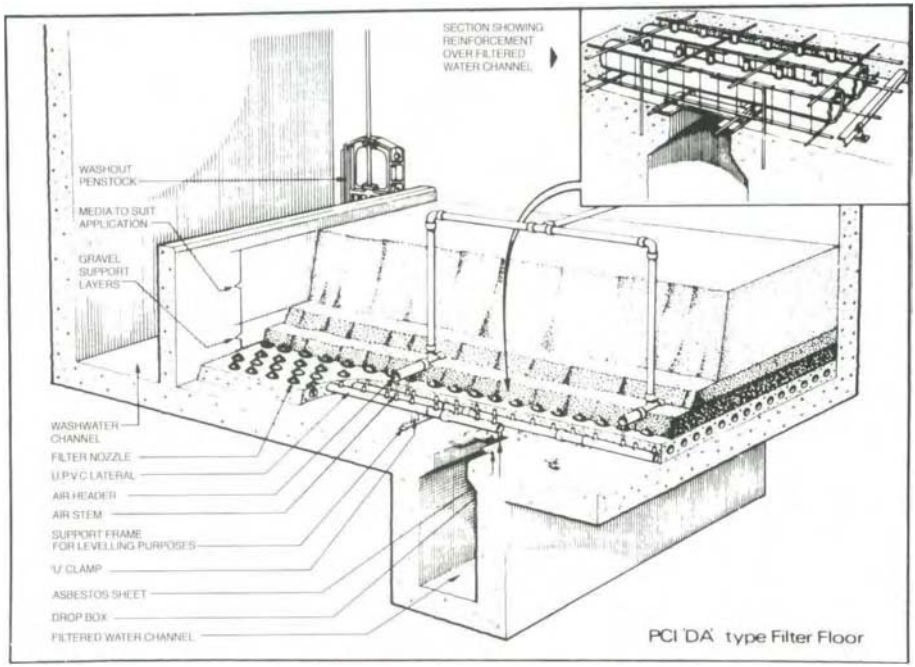


Figure 3.1 A typical deep bed filter constructed in concrete (Paterson Candy International Ltd)

Figure 3.2 Photograph of deep bed filter units

on which granular filter media, usually sand, can rest. The floor may be porous, or perforated with nozzles or other proprietary devices, so that liquid passing through the sand can be collected as the filtrate. Filtration is normally downwards, although some upward flow filters

are used in tertiary sewage treatment. A few designs have flow from top and bottom to a mid-depth filtrate collector, and a few have radial (outwards) flow to a peripheral filtrate collector. Also in recent years there has been some use of horizontal flow filters as a pre-treatment in small community water supply systems.

The process variables to be considered by a designer or an operator are:

(i) Media: type, size (and size distribution), depth;

(ii) Flow direction;

(iii) Rate of flow (usually volumetric flow rate per unit face area), constant or declining;

(iv) Suspension pre-treatment;

(v) Pressure available;

(vi) Washing: rate, duration; need for air scouring or auxiliary wash.

During a filter cycle, which may be from 12 h to 100 h depending on conditions, the media becomes clogged with accumulated deposits. This causes the filtrate quality to vary during the cycle, and leads to an increase in pressure drop (often referred to as the head loss, water gauge). Both of these have limits, one imposed by filtrate quality requirements, the other by the pressure head available across the filter unit.

It is the variation of filtrate quality and head loss, with cycle run time, and their dependence on the suspension characteristics, media and rate of flow which usually require elucidation by tests. Filtration theory can aid the interpretation of such tests, giving useful extrapolations and relationships. However, the theory cannot be predictive as it is based on many idealizing assumptions (e.g. homogeneous, unisize, spherical particle suspensions) which do not hold in practice.

3.1.1 Summary of test programme

The tests which are required to solve filtration problems, either in design or operation, will be considered under the following headings:

(i) Filterability, a laboratory test which is particularly useful for rapid assessment of suspension characteristics, including pre-treatment.

(ii) Models which simulate filtration, in which changes of pre-treatment, media and rate of flow can be readily made.

(iii) Observations of full scale filters which give insight into their internal working and efficiency.

(iv) Media testing: the suitability of granular media for deep bed filtration.

(v) Specialized research techniques.

3.2 Measurement of filterability

The specification of the filtering capability of granular media, other than by building and operating pilot filters or carrying out full-scale tests, could be by defining a filterability property. However, all attempts to measure filterability so far have had to take into account that this is an interactive property depending on both the filter media and the suspension to be filtered. Most filterabilities have been more concerned with the property of the suspension in relation to a given or standard filter material, e.g. lint, micromesh, membranes.

It follows that if filter media are to be tested, they should be assessed in relation to a given, or standard suspension. Undoubtedly, the most desirable suspension would be that to be filtered at the works under consideration. However, that is not always available, and if so, pilot filter tests might be more appropriate and practical. Bearing in mind the diversity of deep bed filtration problems: filtration of aluminium hydroxide flocs, iron flocs, the addition of polyelectrolytes, softening precipitates, algae, organic sewage treatment residuals, it is unlikely that a standard suspension can meet all the needs. Nevertheless, it may be a worthwhile objective for certain limited types of filtration problems. Two possible practical applications of measurement of filterability therefore exist:

(a) Comparison of different media, based on tests with the same suspension.

(b) Characterisation of a given suspension, with various types and degrees of pre-treatment, based on tests with the same media.

3.2.1 Definition of filterability number

Since it is desirable to have filter media which will produce the cleanest filtrate, with the least head loss, operating for the longest

times between washes, at the highest filtration rate, a basis for a filterability test is the filterability number F.

$$F = \frac{H_L C}{C_0 u_1 \theta} \tag{1}$$

where H_L is the head loss, θ is the run time of the filterability test, u_1 is the approach velocity (filter rate), C is the average quality of the filtrate during the test, C_0 is the quality of the inflowing suspension, and F is the filterability number which is dimensionless in consistent units. The minimum value of F represents the best filterability, because it is desirable to have a low H_L, and low C, but be able to accept a high C_0, at high u_1 for a long time θ.

This definition of F is not wholly rational, and so cannot meet boundary conditions such as $C = 0$, but it is intended to operate within a practical range. It has the advantage that the quality of C is not an absolute unit, but can be in any units required by the specification (turbidity, coliforms, Al) as it is the ratio C/C_0 which enters the value of F.

It is not possible, without specifying a standard suspension to state what the value of F should be. However, using a typical set of values for a rapid sand filter for a waterworks: H_L = 2 m, C = 0.1 FTU, C_0 = 20 FTU, u_1 = 10 m h^{-1}, θ = 20 h, the value of F becomes 0.5×10^{-4}. (Note: FTU = Formazin Turbidity Units).

3.2.2 Apparatus

A compact apparatus, requiring only small samples of media (33 g) and of suspension (1 litre) is shown in Figure 3.3 and diagrammatically in Figure 3.4, with typical dimensional details of the major items.

3.2.3 Experimental procedure

A constant weight of media should first be added and consolidated, by gentle tapping, to a constant volume (i.e. a marked level); this ensures constant porosity, as discussed more fully below under consideration of small scale filters (Section 3.3.1).

The apparatus must be flushed and filled with clean water to remove all air bubbles, and the clean water drained to the base of the conical funnel. After addition of the litre sample to the funnel the flow is set at the required filtration rate (e.g. 8.2 cm min^{-1}), and after allowing for displacement of the clean water (40 seconds in the apparatus shown in

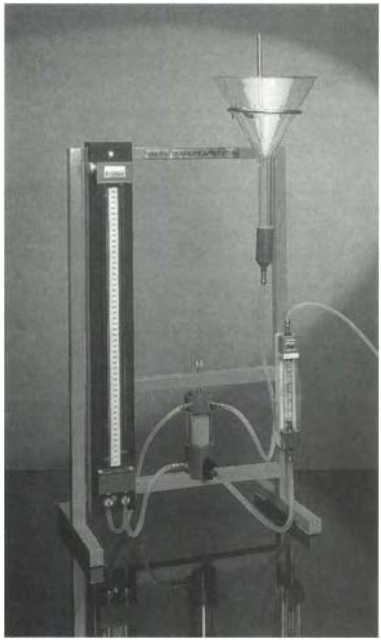


Figure 3.3 Photograph of filterability apparatus (Armfield Ltd)

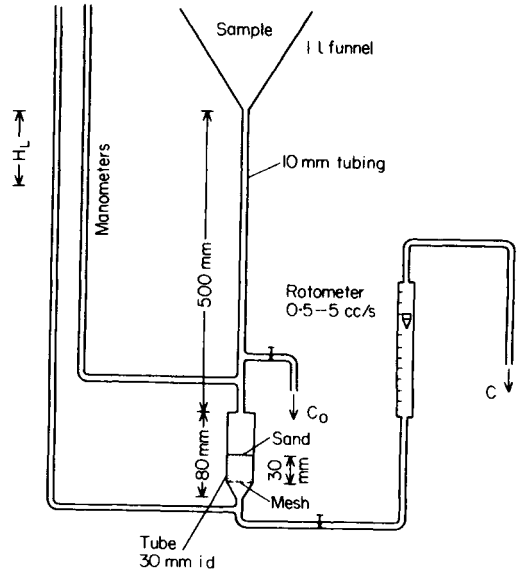


Figure 3.4 Diagram of filterability apparatus

Figure 3.4), the clock is started. The manometer difference should be read when the water level has fallen to the base of the funnel. The diagram shows the other readings which enable the filterability number F to be calculated.

3.2.4 Interpretation of data

The results of a series of laboratory filterability tests are shown in Figure 3.5, where the F value is seen to fall with increasing orthokinetic flocculation, until a minimum F is reached. Orthokinetic flocculation depends solely upon mechanically-induced contact between the original solid particles; the factors which contribute to this are the velocity gradient resulting from the action of an agitator, U_g, the flocculation time, θ_f, and the solids concentration, C_0. These are combined in Figure 3.5 as $U_g\theta_f C_0$, showing a minimum at 0.7; beyond this value further increases in flocculation brought no advantage in the filterability of the resultant suspension.

Similar tests could be made to evaluate the effects of varying times of pre-settlement, or varying polyelectrolyte dosage, etc.

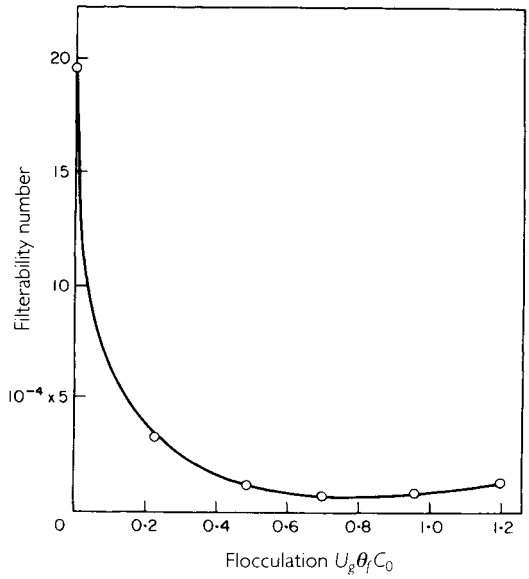


Figure 3.5 Filterability Number, $H_L C/C_0 u_1 \theta$, as a function of prior flocculation, $U_g \theta_f C_0$

3.3 Small scale filters – filter models

The use of models to simulate systems where water movement is concerned has to ensure dynamic similarity between the model and full-scale. This similarity is normally established on the basis of maintaining the same ratio of significant forces in both systems, even though the absolute magnitude of the forces may be smaller in the model system. From this come the well-known dimensionless numbers of hydraulic performance, such as the Reynolds Number (ratio of fluid inertia force to fluid viscous force) and Froude Number (ratio of fluid inertia force to fluid gravity force).

However, in the case of filter models, the criteria of dynamic similarity need not be invoked. In most hydraulic problems the scale of any fluid movement or action (e.g. size of eddies) is limited by the boundaries of the model and of the full-scale unit, and so the geometric relationship of the two has to be taken into account. In a full-scale filter the scale of the fluid action is limited by the boundaries imposed by the filter grains, i.e. by the pore size, and not by the boundary of the filter container. So, if the same size of grains is used in the model, the size of the container is of little consequence.

One precaution, however, is necessary. At the wall of the filter container there will be atypical pores where the grains meet and touch the wall. The number or volume of these boundary pores must be small compared with the number or volume of normal pores in the mass of the filter bed. It has been empirically established that if the wall-to-wall distance in the model is at least 50 times the largest grain size, such boundary effects are negligible.

If then, the pore structure is not scaled, it follows that the suspension particles need not be scaled because the particle actions (the so-called 'transport mechanisms' (Ives, 1975)) in the fluid in the pore should be the same as in full-scale operation. This removes the particular difficulty experienced in sedimentation tanks where it is almost impossible to scale the characteristics of the particles in suspension.

Modern filter theories attribute part of the filtration action (the 'attachment mechanisms' (Gregory, 1975)) to physico-chemical and molecular forces between the particle and grain surfaces, at very close approach. It would be almost impossible to scale these forces, and freedom from this requirement is a great benefit to the use of models in deep bed filtration. Finally, if the pore structure and suspension particles are not to be scaled it follows that there is no necessity to scale the fluid. That is, there is no need to use fluids of different densities or viscosities from those envisaged for the full-scale filter.

Therefore, to use a model to obtain information on deep bed filtration, one should have the same suspension, in the same fluid, flowing at the same rate through the same pore structure as occurs in full-scale. The only aspect of model-making that is required is that the filter unit should be smaller than the full-scale so that it can be constructed at less cost, and be operated with much smaller volumes of suspension to be filtered.

3.3.1 Apparatus

3.3.1.1 Main unit

From a constructional point of view the best form of model filter unit is a tube; if it is transparent so much the better as visual observation is a valuable addition to instrumentation. Suitable acrylic (perspex, plexiglass, lucite) tubing can be purchased; as it is not easily shattered and can be drilled and machined, it is preferable to glass. Such perspex columns are widely used for research studies at universities and initial fears that the sand would scratch the internal surface and render the tube opaque have not been confirmed; such tubes remain quite transparent. Such a tube is suitable for both laboratory and on-site use.

A more robust unit can be made from plastic or metal pipe, complete with waterworks fittings, is shown in Figure 3.6 and diagrammatically in Figure 3.7.

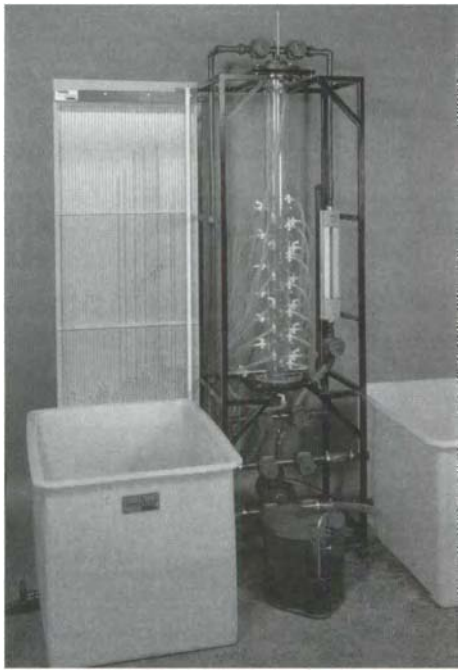


Figure 3.6 Photograph of a model filter (Armfield Ltd)

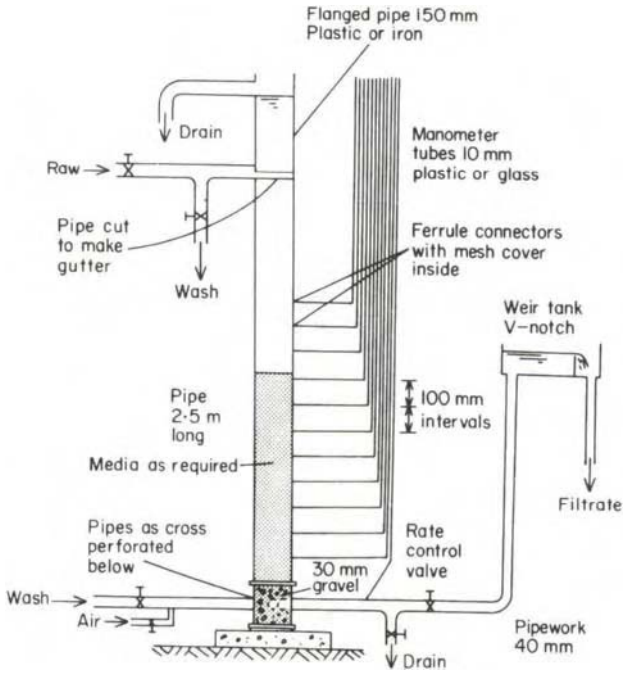


Figure 3.7 Diagram of a model filter, using a pipe and standard fittings

Bearing in mind the 50:1 criterion to avoid boundary effects, a tube should be 5 cm diameter for 1 mm grains or 10 cm for 2 mm grains, the latter being preferable particularly for tertiary sewage treatment. The length of the tube should be at least 1 m, preferably 1.5 to 2.0 m, to accommodate the required depth of media.

Inlet and outlet units for the tube can be attached by flanged or socket-spigot connections with O-rings seals. If the unit is under pressure, restraint for the unbalanced water pressure forces on each end unit can be provided by tie rods between the end units.

3.3.1.2 Feed arrangements

If the model is on-site, the filter influent may flow by gravity to the model. However, in the laboratory the suspension may be in a tank from which it has to be pumped. In either case the influent may be piped directly to the inlet unit of the filter assembly, but it must be borne in mind that the pump may break up flocs or other particle aggregates so that they reach the media in the model in an atypical state. For this reason, peristaltic pumps are preferred for suspensions of fragile particles. If it is desirable to use a constant head tank to supply the model, it should be designed to prevent settlement in the tank.

The filter should incorporate the following:

(i) A baffle near the inlet connection, to destroy the momentum of the influent water so that it does not scour the surface of the filter media. (Note that in Figure 3.7, this effect is achieved by cutting away the top of the feed pipe inside the filter).

(ii) An outlet connection for dirty wash water.

(iii) Valves generally as shown in Figure 3.7.

(iv) If the top of the filter is closed a vent cock at the highest point.

(v) A useful extra item is a socket for a thermometer.

3.3.1.3 Outlet arrangements

The filter media can be supported on a metal mesh which should be soldered into a ring at its circumference. The mesh must be fine enough to prevent the media falling through, but not so fine as to affect the filtration action. B.S. 44 mesh (0.35 mm) in brass or stainless steel is usually suitable. If washing is to be studied with the model, some glass beads (about 1.5 cm diameter) under the mesh will help to ensure uniform wash water distribution.

The outlet unit requires a filtrate pipe connection, wash water inlet, and drain connections, these last two being equipped with valves. The filtered water outlet passes either through a needle valve for manual control, or through a flow control device; in the latter, any changes of flow cause a change in level of water in a small tank which, through a float, either pinches or opens a soft rubber tube, or moves a tapered needle in and out of an orifice.

Flow rate can most easily be read on a rotameter, although orifices, venturis or weirs can be used. For the filtrate the range required would be 5 to 50 $cm^3\ s^{-1}$ for a 10 cm diameter column, corresponding to filtration rates of about 2.5 to 25 $m^3\ m^{-2}\ h^{-1}$. For the wash water, rates up to 150 $cm^3\ s^{-1}$ would be required for a 10 cm column.

If air scour for media cleaning is required, an air pressure line can be connected to the wash water inlet pipe. Again, a rotameter can measure the air flow rate controlled by a needle valve. Suitable needle valves have been obtained from engineer model-maker stores, where they are sold as steam valves for model steam engines.

3.3.1.4 Pressure measurement and sampling

The minimum requirement is an inlet and an outlet manometer, and similar sampling points; the inlet points are in the upper part of the filter tube (above the media), the outlet points being below the media support mesh.

However, it is desirable to have manometer connections through the depth of the filter media at about 5 cm vertical intervals. The connecting tubes should penetrate the column wall and about 0.5 cm into the media to avoid boundary effects, and should have mesh (e.g. B.S. 44) over the ends to prevent media entering. Each manometer should have a valve (laboratory pinch clips are suitable) so that it can be isolated. The manometers can be formed with transparent plastic tubing, fastened to a backing board with a ruled grid for reading the water levels.

If the real manometer levels are too high to be read, a manifold connecting the tops of all the manometers can have air pumped in to depress the water levels; as one is usually concerned with the differences in level (i.e. head loss) this depression of the levels does not affect the validity of the readings. Pressure gauges and mercury manometers are usually too insensitive and are not recommended. A variety of pressure transducers are now available giving an analogue voltage output, which can then be calibrated to give a pressure reading in appropriate units. This can be converted to a digital reading and

input directly into a computer. Pressure transducers have been successfully applied to monitoring backwash and bed fluidisation at laboratory and pilot scale (Hemmings and Fitzpatrick, 1997; Hall and Fitzpatrick, 1999). A differential pressure transducer is a single device that can give head loss (pressure drop) across the whole filter bed. These can be used on full scale or pilot scale filters.

For further details of model filter design see Ives (1966).

3.3.1.5 Filling the filter with media

A filter unit and its associated pipe work should be tested with water under pressure to check for leaks. This also gives an opportunity to remove all air from the system, taking particular care that none is trapped under the retaining mesh at the base of the unit, and that none remains in the manometer connections. Filling the unit through the backwash line and displacing air upwards is usually most effective.

The media should be pre-weighed in the dry condition, so that porosity can be subsequently calculated. However, it should be added in a saturated state to the column, which should have enough water in it so that the media collects under water. This avoids problems with air bubbles clinging to the grains and causing later difficulties because of their reluctance to be removed. Some media, particularly anthracite, may require saturating for as much as 48 hr beforehand, to remove air bubbles. Backwashing of the media at least twice is also desirable before commencing filtration, and then clean water filtration tests should be carried out to check the performance of manometers, rotameters, valves, etc.

After backwashing the media will settle down in an unconsolidated state, but random vibrations may compact it slightly, altering its porosity (and, therefore, its permeability). This is a very important characteristic which affects the operation of the filter very significantly. If the model is simulating some existing full-scale filter, not only the media but also the washing regimes must be identical to establish proper comparability. As the full-scale filter media is unlikely to be consolidated, then consolidation must be avoided in the model which should be protected from vibration or knocks.

If the filter is to be used for some general testing of experimental work, it is desirable to obtain reproducibility by partially consolidating the media by tapping the column gently, to cause the media surface to consolidate down to some predetermined mark.

The following example illustrates how the required quantity of media can be calculated, to allow for consolidation to a controlled porosity.

Media: sand, density = 2650 kg m^{-3}

required porosity = 0.40

sand column length = 1 m

column diameter = 0.1 m

$$\text{Porosity} = \frac{\text{apparent volume} - \text{solid volume}}{\text{apparent volume}} \tag{2}$$

$$\text{Apparent volume} = \frac{\pi}{4}(0.1)^2 \times 1 = 0.007854 \text{ m}^3$$

$$\text{Solid volume} = \frac{\text{mass}}{\text{density}} = \frac{M}{2650} \text{ m}^3$$

$$\therefore 0.40 = \frac{0.007854 - M/2650}{0.007854}$$

hence

$$M = 2650 \times 0.007854\ (1–0.40)$$

$$= 12.488 \text{ kg}$$

So 12.488 kg of sand should be placed in the 0.1 m diameter column and consolidated by vibration until the top surface is 1 m above the retaining mesh. Note that this calculation is independent of the sand diameter.

Details of densities and typical porosities of various granular filter media are given in Section 3.6.2 (see Table 3.2).

3.3.2 Operating procedure

The filtration experiments can commence after clean water tests have established the proper working of the unit. The suspension to be filtered must be introduced into the filter column, via the constant head tank (if used); the time is noted, as zero run time, when the suspension reaches the media surface. In theory, zero run time is different for each level in the media due to the finite displacement time of the suspension through the media depth. But this displacement time is only about 5 minutes for a 100 cm deep bed operating at 8.2 cm min^{-1} approach velocity (about 20 cm min^{-1} interstitial velocity), which is negligible compared with the run time which will be several hours.

Half-hourly readings, for a filter run lasting less than 24 h (longer intervals for longer runs), should be made of water temperature, inlet

water quality, filtrate quality, flow rate (adjusted if necessary) and head loss using manometers or transducers. The water qualities are measured either by taking samples and analysing them externally in a laboratory instrument, or by using on-line continuous flow turbidimeters (nephelometers) and particle counters/sizers, which can also continuously record the data. The water quality will be measured in terms of the significant constituents in the filtrate. Usually this is turbidity, but could be suspended solids, particle count, algal content, bacterial numbers, residual Fe, etc., depending on the purpose of the filter and the intended use for the filtrate.

The filter run will usually be terminated either: (i) when the head loss reaches its limit causing a manometer to draw in air or the flow to fall below the desired rate, or (ii) when the filtrate quality has deteriorated to an unacceptable value, or (iii) when enough data has been collected.

Washing the filter at the conclusion of the run may be an important part of the test. Reverse flow air scour (if used) and wash water should be monitored for flow rate and duration (and possibly pressure). The degree of expansion of the bed as a function of flow rate is important, for controlling loss of media into wash water pipes and ensuring adequate release of accumulated deposits. To determine if washing is complete, it may be useful to measure the clarity of the wash water. This, however, is not a definitive test, as some experimentalists have shown that after the wash water has run clear, a shut-down and re-start of washing led to the release of further deposits into the wash water.

After washing, it is useful to run a clean water filtration test, at the proper experimental rate, and to compare the clean water head loss with that for the media when it was first installed. A higher head loss may indicate some residual deposits clogging the media; if run by run this clean water head loss progressively increases, then washing is inadequate and the cleanliness of the media is deteriorating. Trapped air can also cause an increase in head loss in pilot filter columns.

3.3.3 Analysis of data

A graph of filtrate quality (often as a ratio of the inlet quality to normalize any variations) with filter run time will appear as in Figure 3.8. The rise in filtrate concentration is sometimes referred to as the filter breakthrough, and the filter run must be terminated when this reaches an unacceptable value (0.2 in Figure 3.8). For explanations of the shape of this curve reference should be made to an appropriate textbook, for example Cleasby (1972).

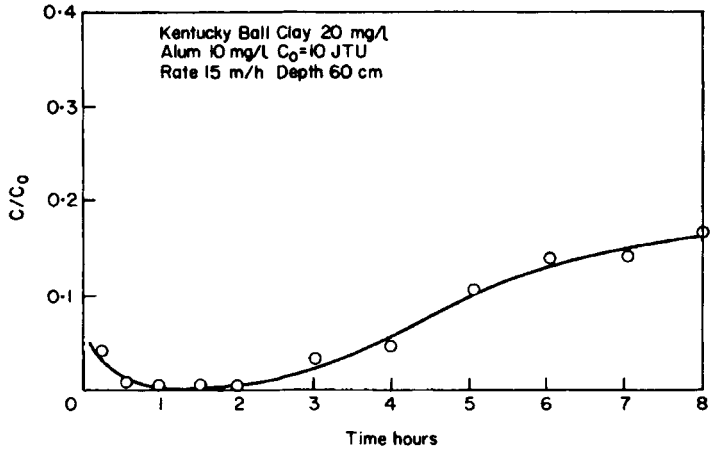


Figure 3.8 Filtrate concentration (expressed as ratio C/C_0) variation with time of filter run

If the filtrate quality is too poor, or the rise in concentration occurs too quickly, then the test conditions must be altered: better pre-treatment, or finer/deeper media, or slower filtration rate may be required.

A graph of total head loss across the filter, plotted against run time will appear as Figure 3.9. The initial head loss H_0 should be the same as the clean water head loss, at the same flow rate and temperature, and as mentioned previously it is an indication of the efficiency of the prior backwashing. An approximately linear rise in head loss indicates filtration (and, therefore, clogging) in depth. This is a desirable attribute of deep bed filtration. However, an upwardly curving line, approaching an exponential increase in head loss, indicates significant surface layer clogging. This is analogous to cake filtration and is undesirable in deep

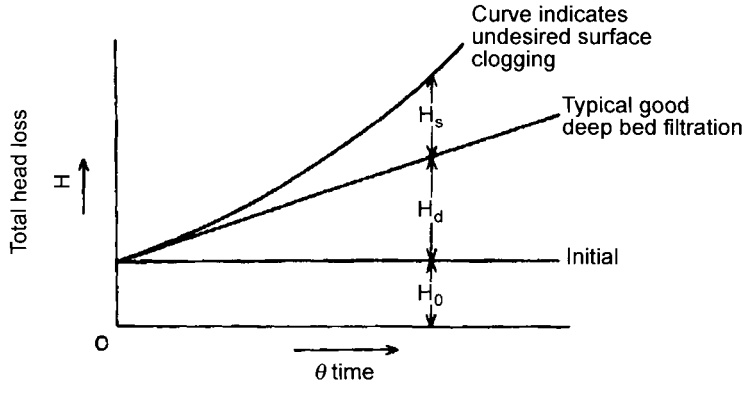


Figure 3.9 Variation of total head loss, H_L, with time. H_0 is the clean filter head loss, H_d is head loss due to depth clogging, H_s is head loss due to surface clogging

bed filtration. For further discussion of this phenomenon see Ives (1975b). This surface clogging can be reduced or eliminated either by pre-treatment (possibly settling), or by increasing the flow rate, or increasing the grain size at the filter media inlet face.

Much more insight into filter performance is provided by plotting pressure diagrams from the manometers located through the media depth. The relationship between the manometer levels and the pressure diagram is shown on Figure 3.10. Commentaries on pressure diagrams and their theoretical background are given by both Cleasby (1972) and Ives (1975b). Where the pressure line is parallel to the clean water line, little or no deposit is present in those layers of the filter. A curved part of the pressure line indicates local clogging caused by deposits in the pores. The greater the divergence between the local gradient and the clean water gradient of the pressure lines, the greater is the local clogging. Thus, the curves can indicate how the various layers are being utilised for clarifying the suspension and thereby retaining the deposits. Any changes in the clean water pressure line after washing will also reveal where residual deposits are located.

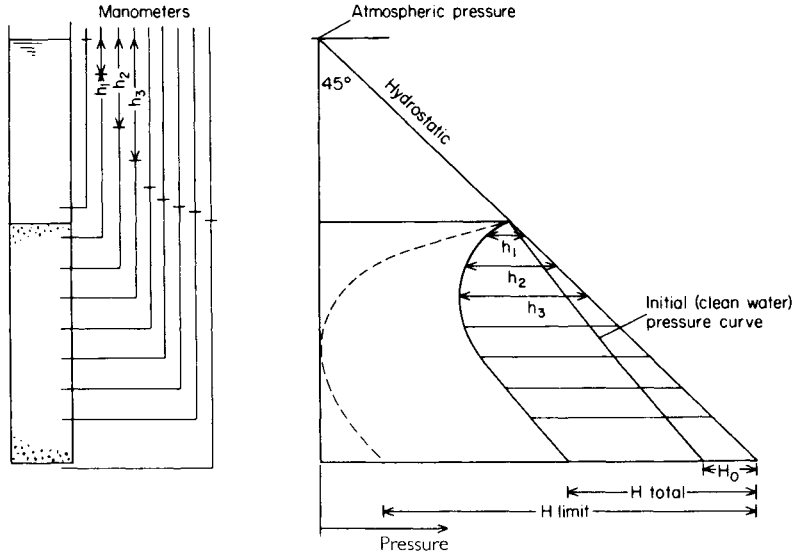


Figure 3.10 Relationship between manometer readings and pressure diagram through the depth of a filter

3.3.3.1 Use of particle counting and sizing

Since the early 1990s continuous on-line particle counters have been increasingly used to assess experimental filter performance. Their use

coincided with increased detection of Cryptosporidium oocysts in treated drinking water causing a number of cryptosporidiosis outbreaks (e.g. Milwaukee in 1993).

On-line particle counters typically count filtrate particles in a range of pre-determined size bands from 2 μm up to around 100 μm. Cryptosporidium oocysts are around 5 μm in size so are easily detected using a particle counter. Most on-line particle counters use an optical method to detect, size and count the particles (more details can be found in Colton *et al*, 1996). Other particle counter/sizers are available which use electrical resistance created by particles suspended in an electrolyte to estimate particle size. These are not generally suitable for continuous on-line monitoring. A typical output from an on-line particle counter is shown in Figure 3.11 where the counts for 2–5 μm and >5 μm particles are displayed.

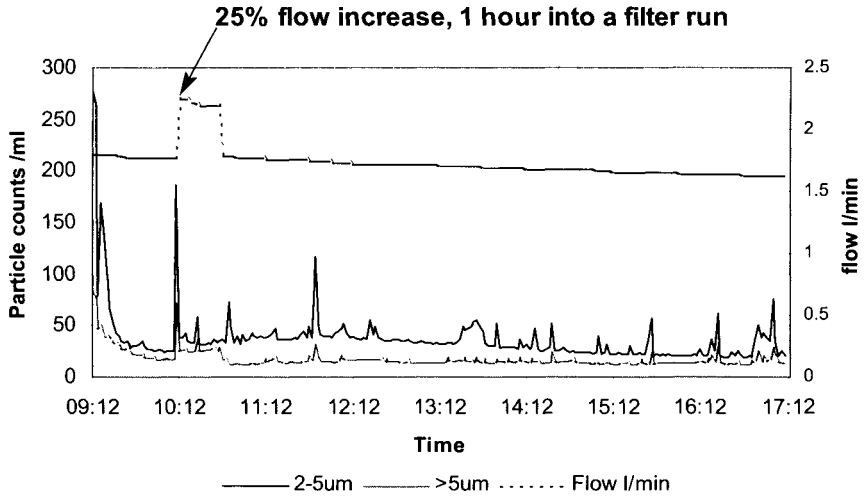


Figure 3.11 Typical output from an on-line particle counter for a laboratory column experiment investigating flow changes

3.4 Full scale filters

Tests can be made on full scale filters by varying the pre-treatment, or the rate of flow if the associated hydraulic equipment is suitable. It is more difficult to make changes in media due to the quantities involved. Consequently, tests on full-scale filters do not generally have the flexibility of model tests.

As in the case of models, observations of filtrate quality changes and total head loss during the filter run can reveal something of the filter performance. The interpretation of the data is identical to that for models. However, the observation of pressure profiles through the media is much more informative giving the same insight into the behaviour of the various media layers as with the models.

Full-scale filters are rarely equipped with manometer probes through the media depth when they are constructed. One example of this was at the Chapel Hill (North Carolina) waterworks, where tubes were incorporated in the concrete wall of a filter during construction. Probes were later inserted through the wall into the media, to which a bank of plastic manometers was connected. Similar probes were inserted through holes burned with a thermic lance through the concrete filter wall at Staines waterworks (North Surrey Water Co.). These probes were used to monitor the performance of an anthracite-sand media filter; an example of the pressure profiles obtained is in Figure 3.12. This also shows the pressure curves obtained from a model column of 15 cm diameter operating under identical conditions at the same waterworks. The close similarity of the curves proves the point that there is no scale effect in deep bed filtration providing that the media, operating regime and washing processes are identical. Similar data have been obtained from the Vereeniging (Rand Water Board) waterworks and Kempton Park Experimental Plant of Thames Water Utilities Ltd.

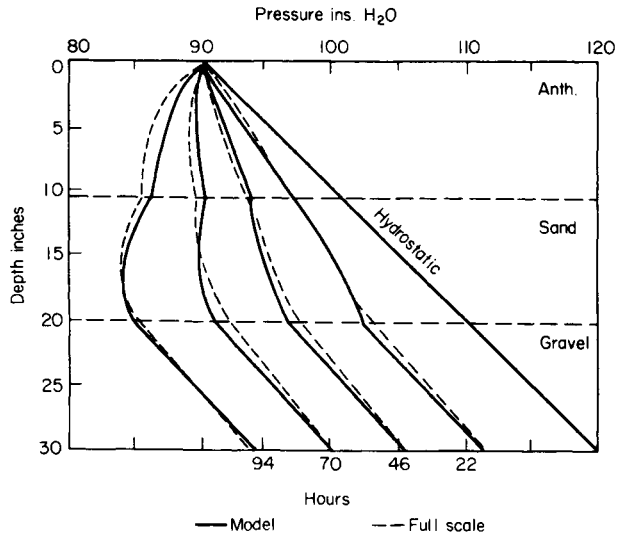


Figure 3.12 Pressure profiles through anthracite-sand-gravel media at Staines waterworks during 94 hr run. Note the close agreement between the model (15 cm diameter) and full scale readings

Currently, more and more filters now have transducers installed to record the pressure loss across individual filter beds.

Backwashing tests are more difficult to evaluate on full-scale filters due to the difficulty in observing visually the media expansion. A useful device for measuring the expansion consists of a rod with small trays (for example, 5 cm square by 1.5 cm deep) attached at 3 cm intervals. The rod is set so that the tray at its lower end rests on the undisturbed media. During backwashing, the rod is held secure in this position and media retained in the trays after backwashing will indicate the height to which expansion took place.

3.5 Filter operation

3.5.1 Flow control

3.5.1.1 Head loss and permeability

In order to maintain a steady output from a water treatment works it is usual to keep a control on the outflow of each filter. As each filter retains particles (typically fine hydroxide flocs) in the pores of its sand, or other granular media, the resistance to flow in the filter bed increases, as it loses permeability. Without some form of flow control this would lead to progressive diminution of the flow volume discharged from the filter. Consequently there is a hydraulic requirement to compensate for the loss of permeability due to the clogging of the filter media.

An exception to the requirement for constant output from each filter is the process of declining rate filtration, where filters in a group are operated at various stages of decline in output, starting from above average outflow rate. A strict sequence of backwashing maintains the output of the group to meet the requirements of the waterworks supply. A minimum of 4 filters is required, which makes model simulations more complex. A comparison of flow control systems, including declining rate filtration can be found elsewhere (Committee Report, 1984).

The clean filter media, after backwashing, offers relatively little resistance to flow – the equivalent of a small number of centimetres of head loss, H_0, which can be calculated from the well-known Kozeny-Carman equation (laminar flow in porous media):

$$\frac{H_0}{l} = k_c \frac{u_1}{g} \frac{\mu}{\rho} \frac{(1-\varepsilon_0)^2}{\varepsilon_0^3} \frac{36}{\psi^2 d^2} \tag{3}$$

where ε_0 is the clean filter porosity, μ and ρ are the water viscosity and density, d and ψ are the grain diameter and sphericity (see Section 3.6.2.2), and k_c is the Kozeny constant. There have been attempts to produce a form of the Kozeny-Carman equation for a filter bed in the process of clogging. The resulting equations are difficult and unreliable as the porosity is reducing with time of operation, by different amounts at different depths in the filter media. In addition, the filter grains are changing shape and size due to coatings of deposits which are also varying with time and depth. A third uncertainty lies in the Kozeny coefficient (k_c), usually taken as 5.0, which may vary with time and depth in an unpredictable manner.

These problems can all be solved by utilising filter models or full-scale filters equipped with manometers or pressure transducers, set at intervals throughout the media depth as described previously. The readings of head loss with time and depth are then directly applicable and there is no need to deal with ill-defined parameters.

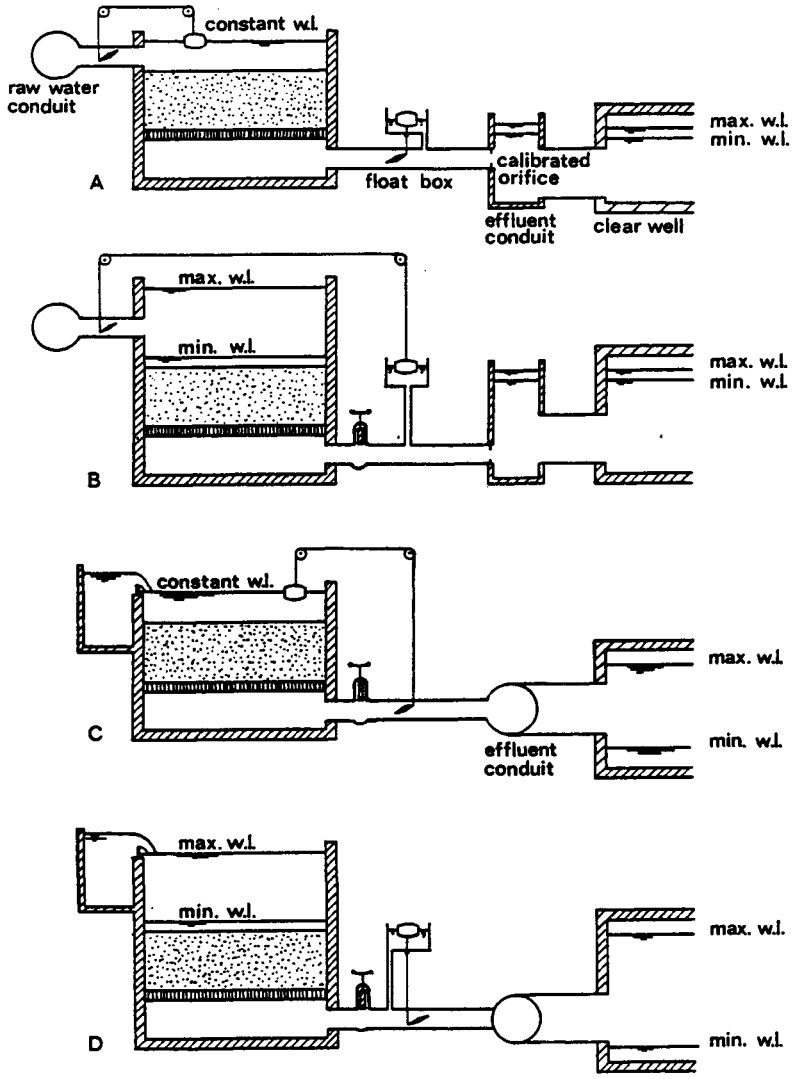
The progressive loss of permeability of the filter media leads to a decline in filter flow and so the increasing resistance of the clogging filter has to be met by a decreasing resistance in a rate controller, or an increasing inlet head to compensate. A rate controller provides an additional loss of head in the influent line (upstream control) or effluent line (downstream control). Adjusting this loss of head, usually reducing it, keeps the supply of water or discharge of filtered water at a desired constant value.

3.5.1.2 Flow controllers

The most common types of filter rate control on full-scale filters are shown diagrammatically in Figure 3.13.

As can be seen in Figure 3.13 all the systems require one or two variable control (typically butterfly) valves. It is the mechanical resistance of these valves which makes miniaturisation impractical for model-scale filters, as mentioned previously. There are several more complex alternatives to the use of butterfly valves for variable flow control: these include pump regulators, venturi devices and siphonic systems. It is unlikely that any of these would translate to the model scale; also they are more expensive. It is possible to use a laboratory peristaltic pump to maintain outlet flow on a model filter, but it needs to be appreciated that the plastic tube changes its flexibility with time. Also, if there are particles in the filtrate they may deposit over time in the flexible tube altering its characteristics.

In practical installations in water treatment works there are several rapid filters acting in parallel, with one out-of-service for backwashing,



Systems: A and B have upstream water level control, downstream rate control;
C and D have upstream rate control, downstream water level control;
A and C have constant inlet water level, variable filtered water level;
B and D have variable inlet water level, constant filtered water level.

Figure 3.13 Systems of filter control

while the others sustain the waterworks output. This involves a small increase in flow rate in the remaining filters. Although the changes are small, it is important in practice to make such transitions slowly to avoid disturbance and detachment of the deposits in the filter pores. This is not usually a significant factor in scale-up operations, although it is possible to simulate flow rate increases in a model to determine

appropriate rates of change to avoid detachment of deposits. A significant advantage in the operation of an array of rapid filters is to maintain a common inlet water inlet level in all. This is achieved by having a common inlet channel with large openings to each filter, giving a negligible resistance to flow.

Whilst most textbooks on water treatment do not contain enough detail for these practical matters of flow control to be presented, a good introduction to the subject can be found in either of the following references: AWWA (1990) or Stevenson (1997).

3.5.2 Filter washing

Granular media filters need regular backwashing to remove clogging deposits and maintain efficient operation. Backwashing of a gravity filter involves flow reversal to dislodge the deposits. There are various techniques of backwashing:

- fluidising water wash
- fluidising water wash plus surface jets
- air scour followed by a fluidising water wash
- simultaneous air and water wash followed by a fluidising water rinse.

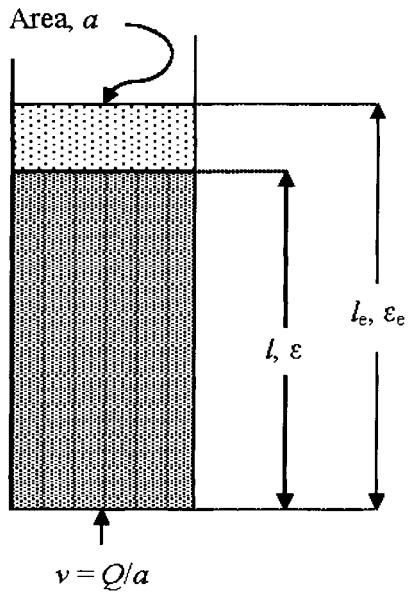
All of the above backwash procedures are conducted at varying upflow rates and bed expansions in practice. Backwashing can be applied to laboratory filters, which can be used to investigate backwash options. Backwash should be optimized to give best cleaning but to minimise water use.

If a filter is backwashed by upflowing water then the bed of granular material will become fluidised in accordance with the theory and observations to be discussed below.

3.5.2.1 Fluidised beds and fluidisation theory

A fluidised bed consists of solid particles or grains suspended by a fluidising medium, e.g. a gas or a liquid.

Consider a vessel such as a filter shell filled with a granular material, e.g. sand, of bed depth, l (see sketch below). A fluidising medium (usually water) enters at the base of the vessel and flows upwards through the bed of sand with a superficial velocity, v. The granular material has a fixed bed voidage of ε. When the bed is fluidised the particles or grains are suspended in equilibrium by the fluid drag forces exerted on them. The bed expands to a depth l_e and a consequent voidage ε_e.



When the grains are in equilibrium:

Upward force = Downward force

Upward force = Pressure difference x Area

$= \rho g h a$

where h is the loss of water head.

Downward force = Weight of the grains in water

$= a l_e (1 - \varepsilon_e)(\rho_s - \rho) g$

Equating the upward and downward forces gives:

$$\rho g h a = a l_e (1 - \varepsilon_e)(\rho_s - \rho) g$$

Rearranging gives an expression for the head loss gradient in a fluidised bed:

$$\frac{h}{l_e} = (1 - \varepsilon_e) \frac{(\rho_s - \rho)}{\rho} \tag{4}$$

The head loss, or hydraulic gradient is also given by the Kozeny-Carman equation (3) with $k_c = 5$:

$$\frac{h}{l_e} = 5 \frac{v}{g} \frac{\mu}{\rho} \frac{(1 - \varepsilon_e)^2}{\varepsilon_e^3} \frac{36}{\psi^2 d^2} \tag{5}$$

Equating equations (4) and (5) gives:

$$(1-\varepsilon_e)\frac{(\rho_s-\rho)}{\rho} = 5\frac{\nu}{g}\frac{\mu}{\rho}\frac{(1-\varepsilon_e)^2}{\varepsilon_e^3}\frac{36}{\psi^2 d^2} \tag{6}$$

which can be rearranged to give an expression for the minimum fluidising velocity, v_{mf}. At the point of incipient fluidisation, that is, as the bed just starts to expand the voidage/porosity and the bed depth are equal to those for the fixed bed, so we have an expression from which we can calculate the minimum fluidisation velocity:

$$v_{mf} = \frac{g}{180}\frac{(\rho_s-\rho)}{\mu}\frac{\varepsilon^3\psi^2 d^2}{(1-\varepsilon)} \tag{7}$$

From equation (7) it is clear that the minimum fluidisation velocity depends on media grain size, media density, media voidage and packing, and temperature (as it affects the water viscosity). Changes in any of these parameters affect bed expansion and fluidisation conditions.

If we examine a granular bed subjected to water upflow then we see that as we increase the flow rate (velocity) the head loss increases linearly with velocity while the bed remains fixed, that is, Darcy's Law is obeyed.

Once we have reached v_{mf}, then the bed starts to expand upwards with the flow and the head loss becomes constant. The head loss is constant as there is no more loss of energy as drag on the grains. All the energy is used up in supporting the grains, that is, balancing their weight.

Typical fluidisation curves are shown in Figure 3.14. This provides a simple, experimental method, using a laboratory column, for deter-

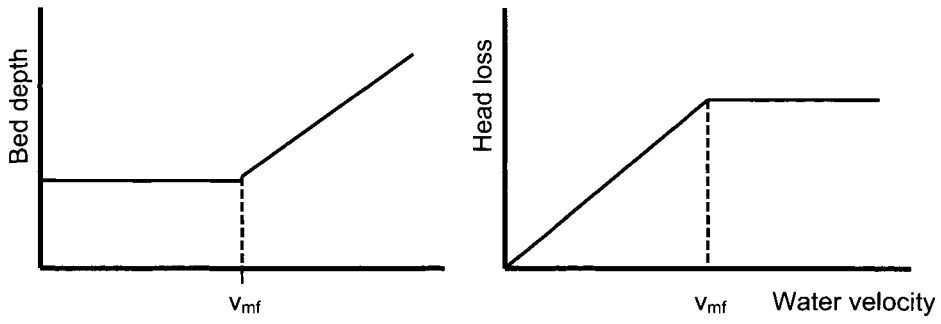


Figure 3.14 Typical fluidisation curves for an ideal monosize medium. Graded media results in a gradual change in gradient on both graphs

mining v_{mf} and the expansion characteristics for a particular granular material under specific conditions.

3.5.2.2 Calculating head loss

To begin with, consider the simplest case of a single media filter. Equation (3) gives the head loss gradient in the fluidised bed; if the backwash head loss is to be calculated then l_e and ε_e must be eliminated from the equation as they are not known and not easily measured.

During backwashing of a fluidised bed the total volume of solids is unchanged, so that:

$$al(1-\varepsilon) = al_e(1-\varepsilon_e)$$

Substitute this result into equation (4) to obtain:

$$h = (1-\varepsilon)\frac{(\rho_s - \rho)}{\rho} l \tag{8}$$

For a typical filter sand the voidage, $\varepsilon = 0.4$, and the density, $\rho_s = 2650$ kg m^{-3}; putting these values into the equation gives:

$$h = \frac{0.6 \times 1650}{1000} l \approx l$$

where the head loss (h) and the bed depth (l) are measured in metres (m) of water.

This is very useful to remember for sand filters, but it should be remembered that this only applies to sand media where the grain density is 2650 kg m^{-3}. For other media materials different values of ε and ρ_s will need to be used in equation (8).

Research has shown that water only backwash is a weak cleaning process as grain collisions are minimal in a liquid fluidised bed. The film of water flowing around the sand grains is thought to protect them from collisions as it thins (Amirtharajah, 1978). It is often inadequate for preventing mud ball formation and other filter problems if the deposits in the filter are so inclined. A minimum expansion is required in order to flush out the deposits and get the whole bed mobile. Increasing the bed expansion has not been shown to improve media cleaning.

3.5.2.3 Use of air scour

When air scour precedes the water wash, it is intended to break up and detach deposits so that they are ready to be flushed out by the fluidising water.

Research has shown that whilst air scour causes a lot of agitation to grains in the top few centimetres of the bed, there is very little agitation deep down (Amirtharajah, 1984). The agitation is caused by bubbles erupting at the surface of the media. If a filter bed retains most of its deposits at the surface then air scour alone is adequate. If, however, it acts as a deep bed filter as intended, then deeper deposits may not be dislodged. Air scour followed by a water wash is common in the UK.

3.5.2.4 Combined air and water wash

A combined wash means that the air and the water flow simultaneously up through the bed. At particular combinations of air and water flow rates for a given medium a phenomenon known as collapse-pulsing (Amirtharajah, 1993) is observed. Later research has shown this to be at flow rates around the onset of three phase fluidisation (Hemmings and Fitzpatrick, 1997). What this means in practice is that fluidised bed conditions are achieved for a much lower water rate – typically less than 0.5 v_{mf}. In addition, during collapse-pulsing there is a very high degree of bed agitation, as pockets of air form and collapse within the media.

Table 3.1 illustrates typical minimum fluidisation velocities, velocities for 20% expansion ($v_{20\%}$) and collapse-pulsing air and water rates for some common filter media. These values were obtained experimentally in the laboratory for water temperatures in the range 11–14°C. Collapse-pulsing conditions, obtained experimentally, are illustrated in Figure 3.15.

Table 3.1 Backwash parameters for selected media.

Media	v_{mf} (m h^{-1})	$v_{20\%}$ (m h^{-1})	*Collapse* v_a (m h^{-1})	*Pulsing (C-P)* v_w (m h^{-1})	*Attrition at C-P (% loss)*
F400 GAC	10	17	36	4	1.7 – 5.3
F300 GAC	16	31	36	10	4.5 – 7.3
Row.8 GAC	22	41	36	9	2.1 – 3.5
Anthracite No.2	28	50	30	21	0.5 – 3.2
Garnet (1.4–2.36)	110	170	80	80	not measured
Sand (0.5–1 mm)	18	45	40	15	not measured
Sand (1–2 mm)	52	89	24	43	0.1 – 2.3

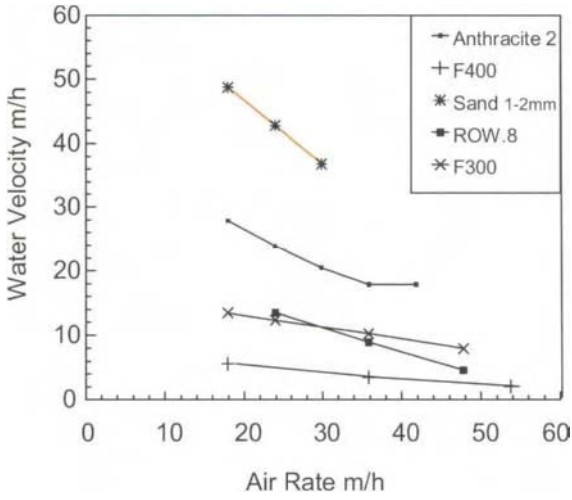


Figure 3.15 Air and water flow rates giving collapse-pulsing conditions for a range of typical UK filter media (Humby and Fitzpatrick, 1996)

3.5.2.5 Cleaning mechanisms

When a filter is being backwashed, the mechanisms that actually get the filter grains clean include fluid shear forces, grain collisions, abrasion between grains, and forces associated with the air/water interface. Cleaning mechanisms have been discussed further by Fitzpatrick (1993).

3.5.2.6 Media attrition and loss during backwash

Media loss may result from inappropriate wash rates for the type of media in use. It is usually due to the lighter grains being carried out over the wash water weir with the backwash water. Lower density materials are washed out more easily, despite lower backwash rates. This is due to their density being closer to that of water and may be exacerbated by attachment of small air bubbles.

Attrition of more friable filter media may result from a vigorous backwash, particularly if air scour is used. Table 3.1 shows the loss due to attrition of various filter media after 100 hours of combined air and water backwash. More details can be found in Humby and Fitzpatrick (1996). Further work on backwash flow rates and media attrition for biological aerated filter (BAF) media used in sewage treatment has been reported by Kent *et al* (1996).

3.5.2.7 Backwashing of dual and triple media filters

Dual and triple media filters are usually designed so that they remain stratified after backwashing. This is not usually a problem for anthracite

and sand. If garnet or ilmenite is used as a third layer the fine garnet or ilmenite may mix with the sand. This may not be detrimental to filter performance but further research is required on this subject. Research has been done by Brown *et al* (1996) on the mixing of the fine garnet and sand layers. Mixing definitely occurs between the sand and garnet combinations found in practice at treatment works.

3.6 Filter media

3.6.1 Media types

The most commonly used filter medium is silica sand. As dual and triple media filters were developed then anthracite and garnet or ilmenite were used in addition to sand. More recently granular activated carbon (GAC) has been used in combination with sand to replace anthracite. GAC performs the dual roles of filtration of particulates and adsorption of organics. Crushed recycled glass is now being marketed for filtration. This is a waste product resulting from glass recycling at bottle banks. It has been shown to have similar filtration performance to sand but its grains have more angular shapes than most filter sands. Crushed recycled glass presents a potentially viable alternative to sand in filtration but further work is required to assess the potential leaching of metals that may be present in the glass before it will be approved for use in drinking water treatment. Also, different sources of glass may have different densities which affects their performance during fluidisation.

Other media being used in drinking water filtration include crushed pumice and granular ferric hydroxide. For wastewater filtration a wider range of materials can be used, including Lytag, pulverised fuel ash (PFA), china clay products and a variety of plastic beads. Such media may be used in biological aerated filters (BAFs) as well as in conventional filters. Further details can be found in Kent *et al* (2000).

3.6.2 Media testing

Although the ultimate test of the suitability of any granular filter media will be its use in full-scale or model deep bed filters, a preliminary assessment can be made by examining certain properties of the media.

No British Standard exists but the basis of a specification has been published which allows assessment of media other than sand, for example: coal, pumice, plastics, ceramics, garnet, ilmenite, alumina

and magnetite, all of which have been used for deep bed filtration (Ives, 1975c; BEWA, 1993). Physical properties of interest are size, shape, density, durability, solubility, cleanliness and fall velocity. Typical properties of various media are given in Table 3.2 (Mohanka, 1969).

3.6.2.1 Size

It is possible to specify grain size by several methods: by microscopic measurements, settling tests, photographically, etc., but the use of sieves is the most practical both in the laboratory and on site, as well as providing a specification which relates to the suppliers' processes of size-grading. Also, sieving is a standard procedure (B.S. 1796: 1952) with sieves specified in B.S.410: 1976.

There is no single size that is desirable for deep bed filtration. It depends on the characteristics of the suspension to be filtered and the clarity required of the filtrate, and so it would be evaluated by a model filter test.

If the media contain a distribution of sizes it would be undesirable for the smaller grains to fill the pores between the larger grains. Grains that will do this have a ratio of sizes of less than 1:5, due to the geometry of pore size. Also, if multimedia filters are contemplated a similar interpenetration (not to be confused with intermixing) must be avoided, otherwise a layer may form at the interface of two media layers, which has a very low permeability. This will become excessively clogged, causing a local high head loss, and may be difficult to clean effectively. Consequently, the coarsest of the size distribution of an upper layer should not exceed 5 times the finest of the size distribution of an adjacent lower layer. This assumes that each layer will be size-graded by backwashing.

Frequently, sieve size is specified by the Hazen 'effective size', which is the ten-percentile by weight at the fine end (that is, 10% by weight is finer than that size). Originally defined by Hazen for slow sand filtration (a deep bed filtration operation where all sizes are homogeneously mixed and biological action in the top centimetre is essential), it has remained a commonly accepted measure. However, it must be realised that in modern rapid deep bed filtration it is not the equivalent size, or the weight mean size, although it does approximate the number mean size. The Hazen 'uniformity coefficient' is a common measure of the non-uniformity of grains, being the ratio of the sixty-percentile to the ten-percentile by weight ($d_{60}/d_{10} \geq 1.0$). The effective size and uniformity coefficient specify two points on the distribution curve. Although an infinite number of curves can be drawn through two such

Table 3.2 Physical characteristics of various filter media (Mohanka, 1969).

Physical Parameters	*Multilayer filter*					*Sand filter*				
	Polystyrene	*Anthracite*	*Crushed flint sand*	*Garnet*	*Magnetite*	*Crushed flint sand*	*Crushed flint sand*	*Crushed flint sand*	*Quarry sand*	*Quarry sand*
Sieve size, mm	3.175 to 2.057	1.676 to 1.405	0.853 to 0.699	0.599 to 0.500	0.500 to 0.422	0.500 to 0.422	0.599 to 0.500	0.853 to 0.699	1.676 to 1.405	2.411 to 2.057
Fall velocity, mm s^{-1}	33.9	63.5	81.0	94.0	109.5	50.0	65.4	81.0	168.5	209.0
Hydraulic diameter, mm	2.50	1.14	0.60	0.467	0.415	0.38	0.435	0.60	1.165	1.36
Sphericity	1.00	0.745	0.78	0.854	0.90	0.83	0.80	0.78	0.765	0.62
Density, kg m^{-3}	1040	1400	2650	3830	4900	2650	2650	2650	2650	2650
Porosity	0.35	0.425	0.464	0.47	0.42	0.464	0.464	0.464	0.39	0.39

points, most natural media can be thus specified as their sizes follow approximately log-normal distributions. These are illustrated in Figure 3.16.

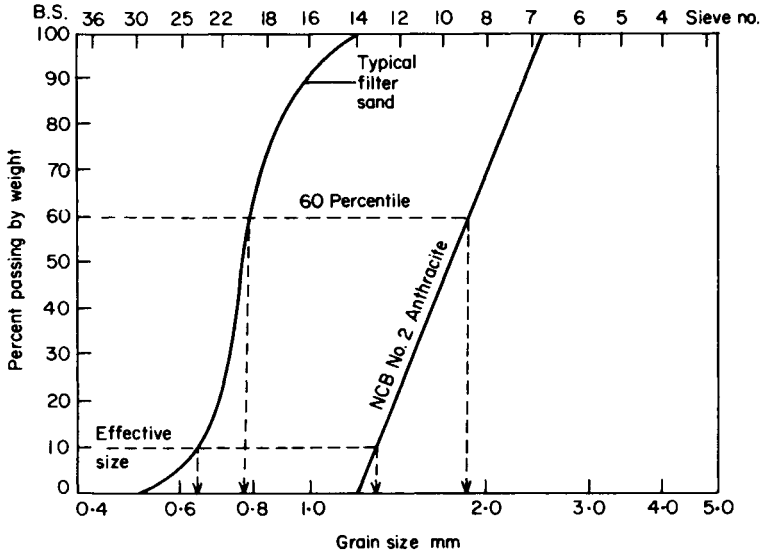


Figure 3.16 Grain size distribution of typical waterworks filter sand and of anthracite. (Note: 10-percentile is Hazen effective size; ratio of 60-percentile to effective size is Hazen uniformity coefficient)

3.6.2.2 Shape

The shape of a non-geometric grain is an elusive concept and textbooks on particle technology devote considerable discussion to the topic (see, for example, Freshwater (1975)). Very often the method of defining and measuring shape reflects the particular reason for using or investigating the grains. In the case of deep bed filtration it is a hydrodynamic interest for, within limits, the more angular grains give better filtration, due to the more tortuous flow paths allowing more effective particle transport mechanisms in the pores.

There is a limit to grain angularity, for if grains are flake-like in shape they may settle in an anisotropic manner, bedding down on each other. This would give rise to a low permeability in the major flow direction, with greater permeabilities in the horizontal plane. This would cause rapid clogging and rise in head loss.

A practical hydrodynamic definition of shape for filter grains is the sphericity, or shape factor ψ:

$$\psi = \frac{d_h}{d_s} \tag{9}$$

where d_h is the hydraulic diameter, being the size of a sphere of equal settling velocity to the grain, which is computed from fall velocity measurements; d_s is the sieve size, being the geometric mean of the openings of the passing and retaining sieves.

Using this definition, the sphericity of a sphere is 1.00; Leighton Buzzard sand is about 0.85 and Welsh anthracite is approximately 0.7. Sphericities less than 0.6 would be undesirable as the grains would be too flaky.

Fall velocity measurements, described later, will give the mean terminal settling velocity (u_p) of grains of uniform sieve size (d_s) in water at a given temperature. It is assumed that the density of the grains has been measured (ρ_s), and the density (ρ) and viscosity (μ) of the water are known from the temperature.

It is then possible to calculate the value of a dimensionless group, which is the ratio of the drag coefficient C_D to the Reynolds Number Re(= $\rho u_p d_h/\mu$) of the grain:

$$\frac{C_D}{\mathrm{Re}} = \frac{4g(\rho_s - \rho)\mu}{3\rho^2 u_p^3} \tag{10}$$

Using a graph of C_D/Re against Re, as in Figure 3.17, the value of Reynolds Number Re can be obtained. Using this value of Re, the hydraulic diameter (d_h) can be calculated from:

$$d_h = \frac{\mu \mathrm{Re}}{\rho u_p} \tag{11}$$

The value of Re usually lies between 10 and 100, indicating that the grains fall in a non-laminar flow regime. It is for this reason that the well-known Stokes Law cannot be used, as it applies to laminar conditions only. The following is an example of such a calculation giving the grain sphericity.

Media: Sand 22/25 B.S. sieve, openings 0.710 mm and 0.600 mm, geometric mean size

d_s = 0.653 mm

Density ρ_s = 2650 kg m^{-3}

Fall velocity u_p = 100 mm s^{-1} at 20°C

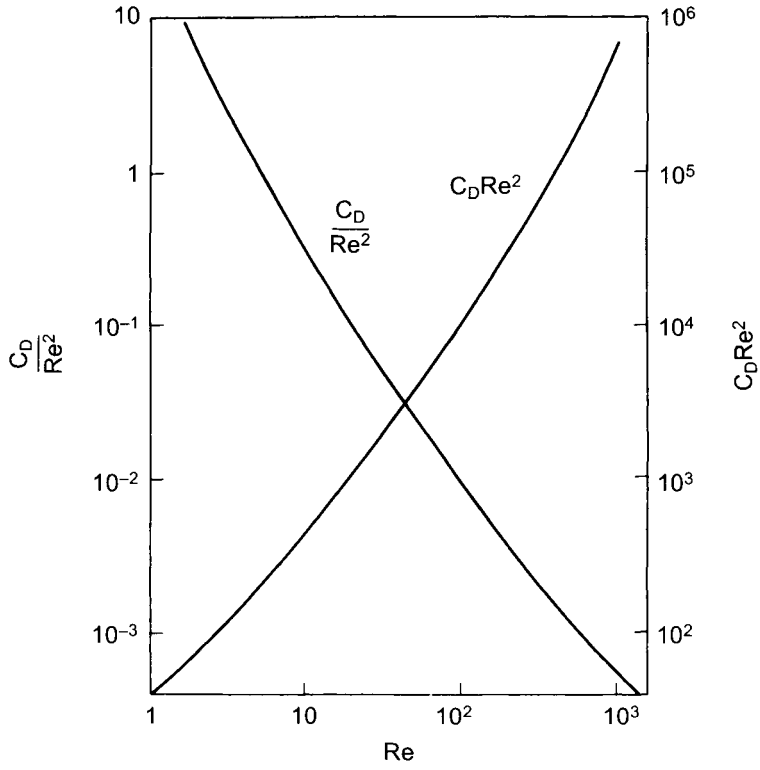


Figure 3.17 Camp curves of dimensionless groups C_D/Re and C_DRe^2 against Reynolds Number, Re, on logarithmic scales

water density ρ = 998 kg m^{-3} at 20°C
viscosity μ = 1.01 × 10^{-3} kg m^{-1} s^{-1} at 20°C

$$\frac{C_D}{\mathrm{Re}} = \frac{4\times 9.81(2650-998)\times 1.01\times 10^{-3}}{3\times 998^2\times(100\times 10^{-3})^3} = 2.19\times 10^{-2}$$

From the graph on Figure 3.17, noting that both scales are logarithmic, Re = 55.

So, the hydraulic diameter

$$d_h = \frac{1.01\times 10^{-3}\times 55}{998\times 100\times 10^{-3}}\ \mathrm{m} = 0.557\times 10^{-3}\ \mathrm{m} = 0.557\ \mathrm{mm}$$

Sphericity $\Psi = \frac{0.557}{0.653} = 0.85$

3.6.2.3 Density

The determination of density is a standard physical test requiring only a balance and a density bottle. Care must be taken that all air is excluded from the grains, and for some material such as anthracite an overnight soaking is advisable.

If the density is less than 1100 kg m^{-3} as with some plastic media, there may be difficulties in controlling backwashing so that grains are not washed away, particularly if air bubbles attach to them. Densities of coal and anthracites vary according to source from 1290 to 1600 kg m^{-3}, but sand is uniform at 2650 kg m^{-3}. One of the densest media to be used experimentally is magnetite at 4800 kg m^{-3}, but such dense materials require small grains, about 0.4 mm sieve size, so that they may be fluidized by backwashing. More dense grains are unlikely to be practicable for this reason.

3.6.2.4 Durability

The agitation to which filter grains are subjected during the washing process requires that the grains should be durable and resist attrition. Opinions differ on how this should be specified, although objectives are the same: the size of the grains should not be materially altered by fracture or attrition during a reasonable lifetime of the media.

Some specifications relate to the Moh hardness, requiring, for example anthracite to be 3.0–3.75 Moh scale. However, hardness is not necessarily associated with durability, as brittle materials may be hard without resilience, and may fracture easily. Other specifications set limits on the amount of fine material produced by impact with a hammer or a weight, with the fines measured by sedimentation or sieving. These seem unrealistic considering the likely mode of attrition and it is difficult to relate such a specification to performance in practice.

A more realistic specification is to limit the amount of fine material produced, and presumably washed away, during an accelerated washing test which simulates several years' washing.

On the assumption that washing in practice takes about 6 min, then 100 h of testing represents 1000 washings. If filters are washed every day, then 1000 washings represent about 3 years of washing operations. If losses due to attrition should not exceed 1% per annum (i.e. 1 cm in a 1 m deep filter), then losses more than 3% in a 100 h test would be unacceptable, and 1% to 3% should be viewed with suspicion. A continuous washing test of 100 h has the merit of being a Monday to Friday operation in a laboratory.

The washing apparatus should consist of an acrylic (e.g. perspex) or glass tube 1 m long, at least 3 cm internal diameter, mounted vertically. At the lower end a metal mesh should be secured (B.S.44 mesh is useful) on which the media can rest. If the tube is larger than 5 cm diameter then glass beads 5 mm to 10 mm should be placed below the mesh to ensure good distribution of wash water. A supply of clean water is connected to the bottom of the tube, with a flow measuring device (rotameter) and a fine control valve. The outlet at the top passes through a trap tank to collect any media floated over by air bubbles (see Figure 3.18).

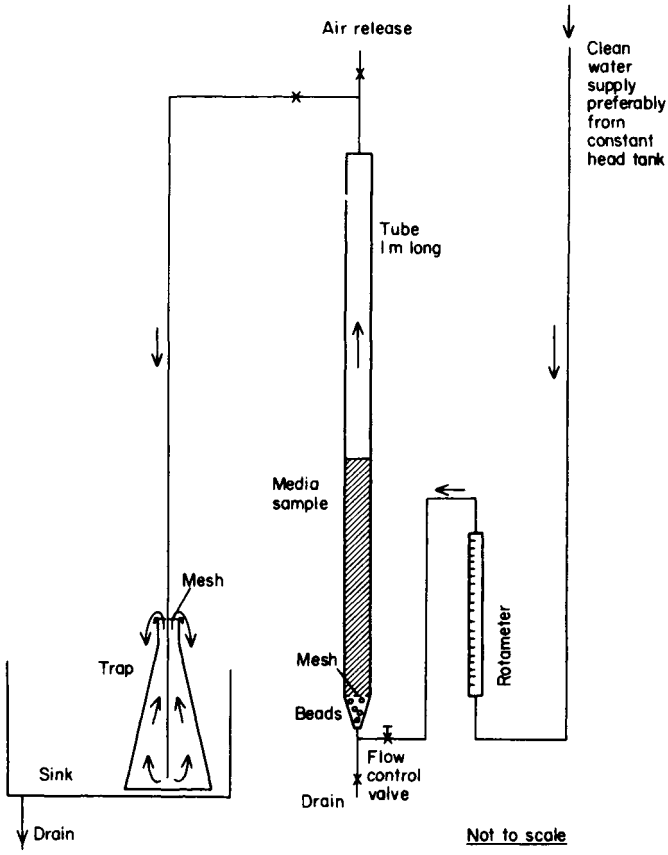


Figure 3.18 Diagram of apparatus for testing durability of granular media by hydraulic attrition

The quantity of media should form a layer about 30 cm deep; it should first be pre-weighed in the dry condition and then thoroughly soaked by complete immersion in water. For anthracite this should be for at least 24 h, with occasional manual stirring to release air. The media

should be added wet to the tube so that it falls under water, to eliminate air bubbles. The water is then turned on, with a flow just sufficient to fluidize the whole layer (this is the critical fluidization velocity), taking care not to over-expand the media and the flow rate noted. The water should be left flowing for 100 h, with the flow rate being checked as often as is convenient, for example, every 2 h during working hours and adjusted as necessary.

After 100 h the water should be turned off, the tube drained, and the media extracted carefully for thorough drying. This can be done in the air, sheltered from wind, on absorbent paper towelling. The dried media should be weighed and loss of weight noted. The trap tank should also be checked, and if it contains much media (i.e. more than 1% by weight) of size comparable with the sample, this could mean that all air was not removed from the media, and these grains were floated over. Such an amount would throw doubts on the results of the test.

Sieving of the media to check its size distribution after washing is not recommended, because dry sieving may cause considerable attrition of some media, thus rendering the measurement invalid.

Associated with this measurement of durability is a requirement that the media should comprise whole grains, and not cemented agglomerates. Although such agglomerates may appear to be stable the cemented joints may constitute planes of weakness, or the cementing material may be more soluble than the grain material, with a consequent disintegration of the agglomerated grain. A friability test has recently been developed (Humby *et al*, 1996) which is another way of assessing the strength of different filter media.

3.6.2.5 Solubility

Grains that are likely to dissolve in the filtering liquid cannot be accepted even though this may be a long term phenomenon. Usually a specification is set which is more stringent than any situation which is likely to arise in practice. Acid solution of limestone or other forms of calcium carbonate (sea shell fragments in beach sand, for example) is the usual problem. The American Water Works Association sets a limit of 5% loss of weight by solubility in 50% hydrochloric acid. At University College London 20% HCl is used with a limit of 2% loss of weight after 24 h immersion. The media should be well-rinsed with distilled water and oven dried to a constant weight at 105°C. Samples of 50 g should be transferred (at least two determinations) into evaporating dishes and immersed in the diluted HCl, with a preliminary glass rod stirring to ensure good contact. Any initial effervescence,

which would indicate the presence of calcium carbonate, should be noted. After 24 h the colour of the acid should be noted (for example, brown may indicate solution of iron compounds). The samples should be washed in distilled water to remove all acid, and oven dried and weighed to determine loss of weight.

Another aspect of solubility concerns possible toxic or undesirable compounds leaching from the grains contaminating the liquid being filtered. An example is the possibility of phenol leaching from coal media. Discussion of this and further references are to be found in Ives (1975c).

3.6.2.6 Cleanliness

It is well appreciated that filter media should be free from dirt, dust, organic matter, clay, etc. The presence of such material can readily be detected by swirling a small amount of the media with clean water in a beaker.

In addition, visual examination under a low-power microscope (about 20× magnification) can show the presence of attached dirt on the grains. Such visual examination, with experience, can confirm the shape measured by the sphericity; moreover, the mineral nature of the grains can be assessed and the presence of agglomerates detected. Both transmitted and reflected light should be used to see shape and surface texture of the grains.

3.6.2.7 Fall velocity

Fall velocity measurements require a vertical column at least 1.5 m long, by 5 cm diameter in glass or acrylic (perspex), with two marks 1 m vertically apart, the upper mark being about 15 cm below the top.

The column should be filled with water at uniform temperature (preferably 20°C), checked by lowering a thermometer through the depth. Previously sieved grains should be immersed in water in a small beaker, and picked out individually at random with tweezers. These individual grains should be released, under the water surface, and timed as each settles past the two marks 1 m apart. A minimum of 20 such grains should be timed, and the average fall velocity obtained. Grains with air bubbles attached, or falling close to the container wall, should be ignored.

The resulting information can be presented, for various media, in one graph as shown in Figure 3.19. Such a graph is the basis for selection of media for multiple layer filters (Mohanka, 1971) but selection using this method will not prevent mixing, particularly of garnet and sand

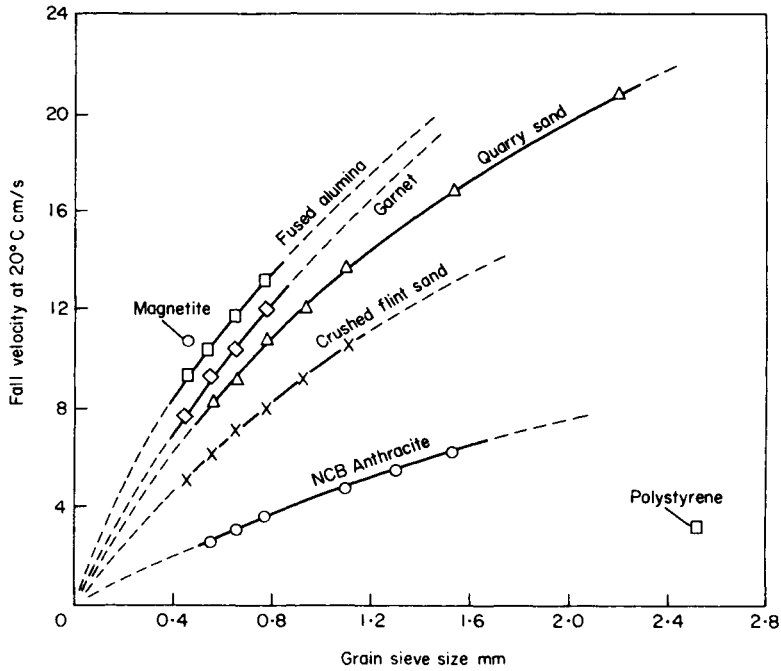


Figure 3.19 Settling curves for granular media

layers (Brown *et al*, 1996). Fall velocity is also the information required for determinations of grain shape as described previously.

3.6.2.8 SEM analysis

Detailed information about the grain shape and surface characteristics can be obtained by using scanning electron microsopy (SEM). This is also a useful tool for assessing media cleanliness and the extent of biological fouling. Kaur *et al* (2003) and Fitzpatrick *et al* (2004) give further information and examples.

3.6.3 Multilayer filters

Since the advent of filters containing more than one type of media in the 1930s, the use of dual or three-layer filters has progressed to rival the traditional sand filter. The concomitant development of granular anthracite as a filter material has led to the widespread use of the dual layer anthracite-sand design. The principle is simple: the passage of water containing fine particles in suspension should progress through sequentially finer filter media to refine the desired product of clean water. Strangely the classical sand filter resulted in the direct opposite of this principle, as the backwashing process graded the sand to have

its finest fraction at the top inlet face and its coarsest fraction at the bottom outlet of filtered water. One practical solution to this adverse effect is to filter from the bottom to the top – the upflow filter. This improves the filtration efficiency but has the disadvantage that the filtered water emerges above the sand, and is consequentially exposed to contamination if the filter structure is not covered. Such contamination could be airborne dust, dirt and faecal matter from aquatic birds, or algal growth engendered by daylight. All of these are undesirable in a drinking water supply, although they are less serious in tertiary sewage filtration, where the filtered water quality has less stringent requirements.

There are two solutions to this problem with upflow filtration for drinking water treatment. One is to use closed filter vessels such as pressure filters, the other is to draw off the filtrate from a perforated pipe laid horizontally in the topmost layer of the sand, about 150 mm below its surface. Thus water is filtered in two directions: downwards from the top through the finest sand, and upwards from the bottom through successively coarse-to-fine media. If the top and bottom water to be filtered are from the same hydraulic chamber, and the filtered water pipe is common, the head losses are equal in both directions, and the filter is in a pressure 'sandwich'. This is the biflow filter, designated AKX in Russia where it is extensively used.

3.6.3.1 Depths of filter layers

There are formal computational methods of comparing a range of different media depths of multilayer filters (Sembi and Ives, 1983), even those containing up to 10 layers. Although the coefficients and values were derived from a real experimental study, they are far from being validated for the many types of suspensions (aluminium and iron hydroxide flocs, polyelectrolytes, turbidity, colour and other organic precipitates, algae, etc) which occur in the treatment of natural waters. Therefore, such computational methods can only be indicative and are insufficiently definitive for real design. A useful conclusion that arose from that study was that as the number of layers is increased the incremental benefit from each layer becomes less and less. It was concluded that two layers are significantly better than one, three layers show some improvement over two, but four or more layers are not worth the additional complexities in managing backwashing. The extra layers bring very little benefit in filtered water quality, head loss and length of run.

There are difficulties experienced in using mathematical models with multilayer filters. The basic mathematical models assume that the

concentration of suspension entering the inlet face of the filter media is a constant. This is reasonably true for the inlet face of the top layer, but is no longer true at the interface with the next layer as this inlet concentration is the filtrate from the first layer which is variable with time. The same problem of variability will occur at each interface between adjacent layers. The consequent complexity makes mathematical analysis only possible with difficult numerical methods. All of this is taken care of, with physical models, even allowing measurement of the interface concentrations during a filter run.

The advantage of using model-scale filters to resolve the questions of how many and how deep layers should be used in a new design is that the media in a model filter is usually only a few kilograms (between 10 and 20 kg in most cases). Thus it is readily emptied and refilled within a day. A full-scale filter may contain over 20 tonnes of media requiring much more effort and time to empty and refill. Also there are many installations of 4 or 6 model filters operating in parallel from the same water source, so that side-by-side comparisons can be made in performance (Figure 3.20). Consequently the most appropriate design can be selected with a minimum of experimental effort.

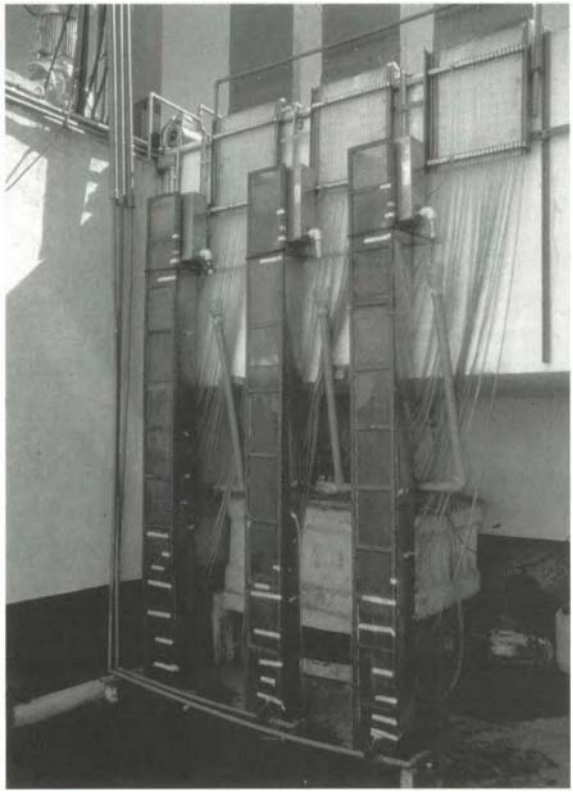


Figure 3.20 Photograph of model filters in parallel

3.6.3.2 Selection of filter media

There are two principal criteria relating to the selection of media for multilayer filters:

(i) the media should become progressively finer layer by layer as water filters through;

(ii) the media layers should remain in, or return to, their original configurations after backwashing, that is they should not become intermixed.

Criterion (i) is not difficult to achieve, although if a lower layer is too fine, it may satisfy the first but not the second criterion of no intermixing. Both criteria can be met by ensuring that the grains in a lower layer have a greater density than those in the layer above. In essence, they should settle faster so that as backwashing terminates each layer settles in place before the layer above.

An essential guide to meeting these two criteria is in Figure 3.19 which shows data for various materials, of fall velocity of individual grains in water as a function of grain sieve size. The data are for water at 20°C. The actual values would be different at different water temperatures, due to changes in water viscosity. Nevertheless, the relative values for the various grains would be unchanged. Also, in a filter the mass of grains would be falling under hindered settling conditions, not as individual grains, due to mutual hydrodynamic interference. Once again, the relative values are sufficiently accurate, in spite of the hindered fall velocities all being less than those on the graph.

The choice of appropriate materials is made by selecting the upper layer from the lowest curve towards the right hand side, for example 1.5 mm NCB Anthracite with a fall velocity of 6 cm s^{-1}. The next layer should have a higher fall velocity, but a smaller grain size, for example 0.8 mm Quarry Sand, with a fall velocity of 10 cm s^{-1}. If a third layer is required, its grain size should be smaller than that of the sand, for example 0.6 mm with a fall velocity of 11 cm s^{-1}. It should be noted that the differences in fall velocity become progressively smaller, so intermixing becomes more difficult to avoid. In making these selections in practice, the media do not comprise unisize grains, but a spread of sizes of which the size selected on the graph of Figure 3.19 is regarded as typical or average. The greater is the spread of sizes in any selection, the greater is the chance of the finer fraction becoming intermixed with the layer above, and conversely the greater chance of the coarse fraction becoming intermixed with the layer below. Consequently, sharp separations between the layers are unlikely. There

is some value in the argument that some intermixing at layer boundaries is desirable, because sharp interfaces may lead to the formation of a thin layer of an atypical cake-like deposit (as is also possible at the inlet face of the media in any deep bed filter). Such a layer would cause a high local head loss, and may be difficult to disperse during the backwashing process.

The various filter materials which are presented in Figure 3.19 are not the only ones available. In some Southern European countries pumice is used as an alternative to anthracite, because of its availability. The use of hydro-anthracite is another alternative in Europe. The granular anthracite which is available in Pennsylvania, USA, has a greater density (1600–1700 kg m^{-3}) than NCB (Welsh) anthracite (1400 kg m^{-3}). This creates a different curve of fall velocity against grain size. Also in the USA the mineral ilmenite is a preferred alternative to garnet; it has the same density as filter grade granular garnet. In Germany there is a granular product called expanded slate, and a similar product in France is made from baked clay. In Norway an artificially prepared granular material is available in two different size grades and densities, and can be directly utilised for dual-layer filtration.

No doubt other filter media will appear on the market, some of which may be suitable for multilayer filtration. If they are in granular form, they can be investigated in model-scale filters, and evaluated by the methods outlined here.

3.7 Alternative filters

3.7.1 Continuous filters

Continuous filters are a type of deep bed filter that have been applied widely in recent years, especially for the tertiary treatment of wastewaters. These filters operate in steady state, producing a constant stream of filtrate, with a virtually unchanging head loss. The two principal designs which are used in practice are the Ten-Ten inclined bed, and the Dynasand countercurrent-flow filter. The Ten-Ten inclined bed filter is illustrated in Figure 3.21, which shows the sand moving downwards through the drum-shaped container, while water flows from the surface radially outwards and downwards to a peripheral filtrate collector. More details are given by Allanson and Austin (1976). The Dynasand countercurrent filter is shown in Figure 3.22. The water flows upwards through a descending sand bed, and filtrate is collected

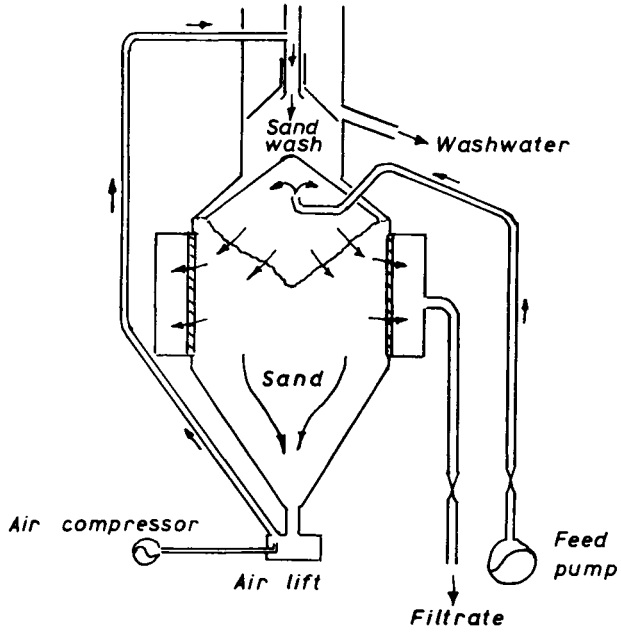


Figure 3.21 Ten-Ten inclined bed continuous filter

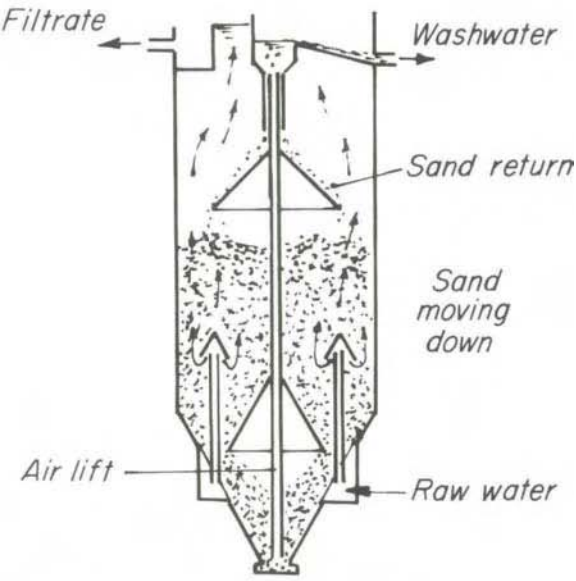
above the filter sand. Its operation and performance is described by Larsson and Hjelmer (1979), Shimokubo (1983) and Hultman *et al* (1994).

In both filters the dirty sand is removed continuously from the bottom by a continuous airlift which cleans the sand and returns it to the top of the filter bed. Dirty wash water is removed near the top of the unit. The drum-shaped designs of these continuous filters limits their upper size, although Dynasand have produced a cellular design which enables multiple units to be coupled into one structure.

3.7.1.1 Modelling

Models have been made of continuous filtration, particularly of the Dynasand countercurrent design. Like fixed deep-bed models they have reproduced the depth of the filter bed, (and the washing air-lift) at natural scale using the practical sizes of filter material. However, in plan they have been reduced, typically to about 100 to 150 mm diameter or square side. An example has been described by Fourie and Ives (1982). In most respects it is like the fixed deep bed models with sampling points and manometer connections. These have shown that steady state conditions do occur with steady pressure profiles and concentration changes through the moving filter bed depth. The sand

(a)



(b)

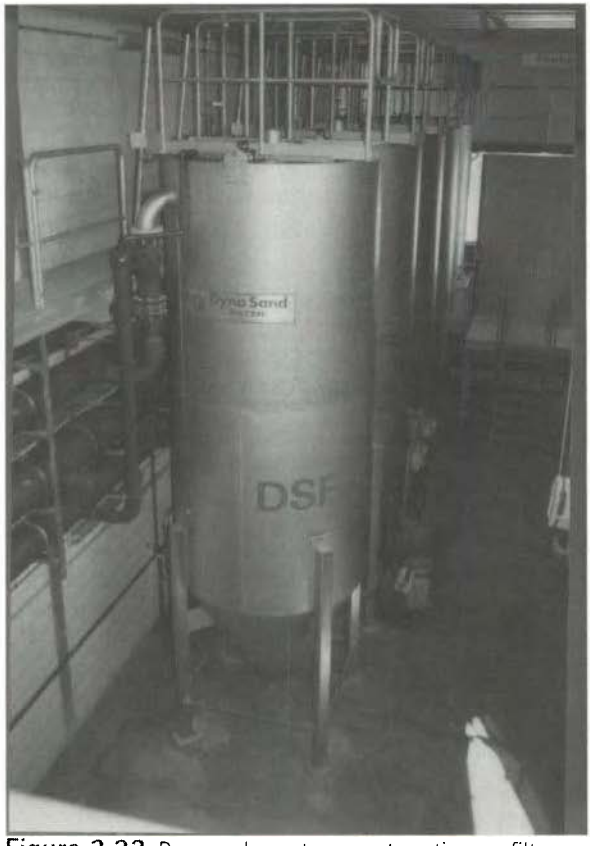


Figure 3.22 Dynasand countercurrent continuous filter. (a) schematic of filter; (b) photograph of filter

movements can be observed at the walls and velocities can be compared with sand mass balance equations.

The most significant departure from expectation with the Fourie-Ives model filter was the discovery that the sand moved downwards at different velocities across the plan section. The observed wall velocities were not typical, but were the slowest, with the sand velocities increasing towards to the centre of the 100 mm dimension (square) model. This represents a shear gradient across the sand, presumably generated by the wall drag. It is not yet clear whether such a shear gradient persists significantly across the whole plan of a full-scale filter, which may be 2 m in diameter, or whether it only affects a localised region near the wall. If it is the latter case, then a model would be unrepresentative as the majority, if not all, of the sand would be influenced by the walls.

3.7.2 Pebble matrix filters

Pebble matrix filters are based on a design which was first used in Uzbekistan, called 'skeleton-fill filters'. The skeleton, used in the structural sense, is an infilling of pebbles in the filter container. Within spaces among the skeleton, or matrix, of pebbles, is another filling of sand which is the actual filter medium. This provides routes for the filtration flow from the top inlet face to the filter underdrain. Because the flow can only take place through the sand among the pebbles, the inlet face flow rate has to proceed at a velocity of inlet face flow velocity divided by the porosity of the pebble bed. For a random packing of rounded pebbles this porosity is about 0.4, thus creating an average flow rate into the sand of 2.5 times the inlet face velocity. A schematic of the pebble matrix filter is shown in Figure 3.23.

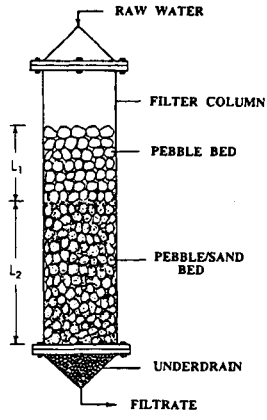


Figure 3.23 Pebble matrix filter

The unique property of pebble matrix filters is their capacity to treat suspensions of high turbidity (between 500 and 10,000 mg l^{-1}) without impossibly short filter runs due to rapid clogging. This is achieved in the filter by the presence of large areas of internal wall effect, in which the suspension flows down the pebble surfaces if the neighbouring sand has become clogged. Consequently the suspension flows from pebble to pebble until it reaches cleaner sand with a greater permeability. This results in a progressive clogging of the sand from the top downwards, while maintaining a continuing flow path for the suspension to reach the lower cleaner sand. As in conventional filters this proceeds until a limit head loss is reached, or the filtrate turbidity becomes unacceptably high. The 'wall-effect' process is enhanced by the presence of lens-shaped cavities under each pebble, where the sand has not compacted after the washing process. The suspension can pass readily through these cavities helping to transfer the flow from the surface of a pebble to the surface of the pebble below (see Figure 3.24).

Figure 3.24 Lens-like cavity underneath a pebble

The original design in Uzbekistan was for tertiary sewage filtration but no data were available on performance. It has been seen, firstly in the UK as a possible simple process for pre-filtering high turbidity waters during monsoon-type rainstorms, so that subsequent slow sand filters do not become overwhelmed by silt deposits.

In experiments using laboratory scale model filters various high (clay) turbidity waters have been applied at different rates of flow to a 300 mm diameter pebble matrix filter containing various depths of

50 mm rounded beach pebbles, infilled to various depths of different grades of filter sand. Continuous monitoring checked the filtrate turbidity to ensure that it did not exceed the suspended solids loading (20 mg l^{-1}) which is acceptable for slow sand filtration.

A slow sand filter, containing an active schmutzdecke (surface microbial layer) taken from a London water treatment works, was used as a test of the efficacy of a pebble matrix filter to allow continuous operation during a simulated 'monsoon turbidity' period. Further details are given in Rajapakse and Ives (1990).

Cleaning the pebble matrix filter is relatively simple, and appropriate to monsoon-type conditions. Draining down the filter removes about 80% of the deposits; refilling the filter from below with raw water and draining again leads to about 90% removal of the deposits, and the repetition of this filling and draining gives about 95% removal. This procedure can be repeated for 4 or 5 filter runs, after which a normal backwash fluidisation with clean water achieves a final cleaning. It is essential during this fluidisation that the sand does not emerge above the top level of the pebbles. If it does, it will not return back into the interstices of the pebbles, and the filter will be less effective.

The development of the pebble matrix filter is continuing in Papua New Guinea to determine its practicability on site. One practical problem is the filling of full scale filters with the pebbles and the sand. Two methods have failed to provide the right matrix of pebbles and sand. A filling of the filter box with pebbles, followed by addition of sand while backwashing failed to distribute the sand uniformly among the interstices of the pebbles. Much of the sand remained on the top of the pebble bed, which is undesirable. An alternative method placed the sand in the filter box, on top of which the pebbles were poured. Backwashing to fluidise the sand failed to provide a uniform sinking of the pebbles into a contiguous matrix within the sand. Enquiries to the Uzbek originators of the process revealed that sand and pebbles were placed together layer by layer, by hand. This was extremely labour intensive. Another practical problem to be studied is whether biological growths (particularly algae) will occur in the filter, and prevent proper cleaning. This is very much an on-site investigation.

Once again, the advantage of model filters, displaying no scale effects, is apparent, where changes in design can be made readily, where transparent walls of plastic filter vessels enable dynamic observation of the filtration process to take place, and where the utilisation of optical fibre endoscopy (see Section 3.8.7) reveals local effects within the filter.

3.7.3 Slow sand filters

Slow sand filters are a special form of depth filtration in which the capture and accumulation of influent particles occurs in a shallow, initial section of the sand bed, typically the top 2–5 cm. This occurs because of the very high filtration efficiency caused by the low filtration rate (~0.1 m h^{-1}) and the small grain size of the media (d_{10} ~ 0.15 mm). The advantages of the slow sand filter are that it can achieve a high filtrate quality (in terms of particles and micro-organisms) and it has a low rate of head loss development leading to a filter run of between 2 to 6 weeks. However, the actual performance of the filter depends greatly on the influent water quality and the use of any pre-treatment step. A unique and important feature of this type of filter is the presence and temporal development of substantial micro-organism populations on the filter surface and within the top layers of the sand bed. These populations, comprising mostly algae, bacteria and protozoa, have a direct influence on the behaviour of the filter in two principal ways; firstly, through the development of a surface microbial layer (the *schmutzdecke*) which acts a pre-filter, and secondly, through the accumulation of biomass within the pores of the sand media. The consequence of this is an overall enhancement of the treatment performance and, in general, an increased rate of head loss development. However, the head loss often varies through a filter run and is affected by the dynamics of the microbial interactions; in contrast, the filtrate quality invariably improves with filter run time. Thus, unlike the rapid filter process where the filter run is terminated when either the filtrate quality of limiting head loss is reached, for a slow sand filter the run is terminated only by a maximum head loss being reached.

Slow sand filtration is the oldest form of depth filtration process used in municipal water treatment and many European cities (e.g. London and Amsterdam) still have operational slow sand filters. The principal design of the filter system is very similar to that of its rapid filter counterpart and a typical section of a slow sand filter pilot plant is shown in Figure 3.25.

As is usually the case with rapid filters the flow is downwards through the sand bed and underdrains, driven by a pressure head difference between the influent water above the filter bed and the downstream collection channels. Flow rate is controlled to a set value by an automatic valve or siphon in the downstream pipe work. At the end of the filter run when the limiting headloss is reached the filter is cleaned by draining the filter and physically scraping off the *schmutzdecke* and surface layer of sand (typically 2–4 cm). After removal of the surface layer the filter is restarted, usually at a reduced flow rate for several

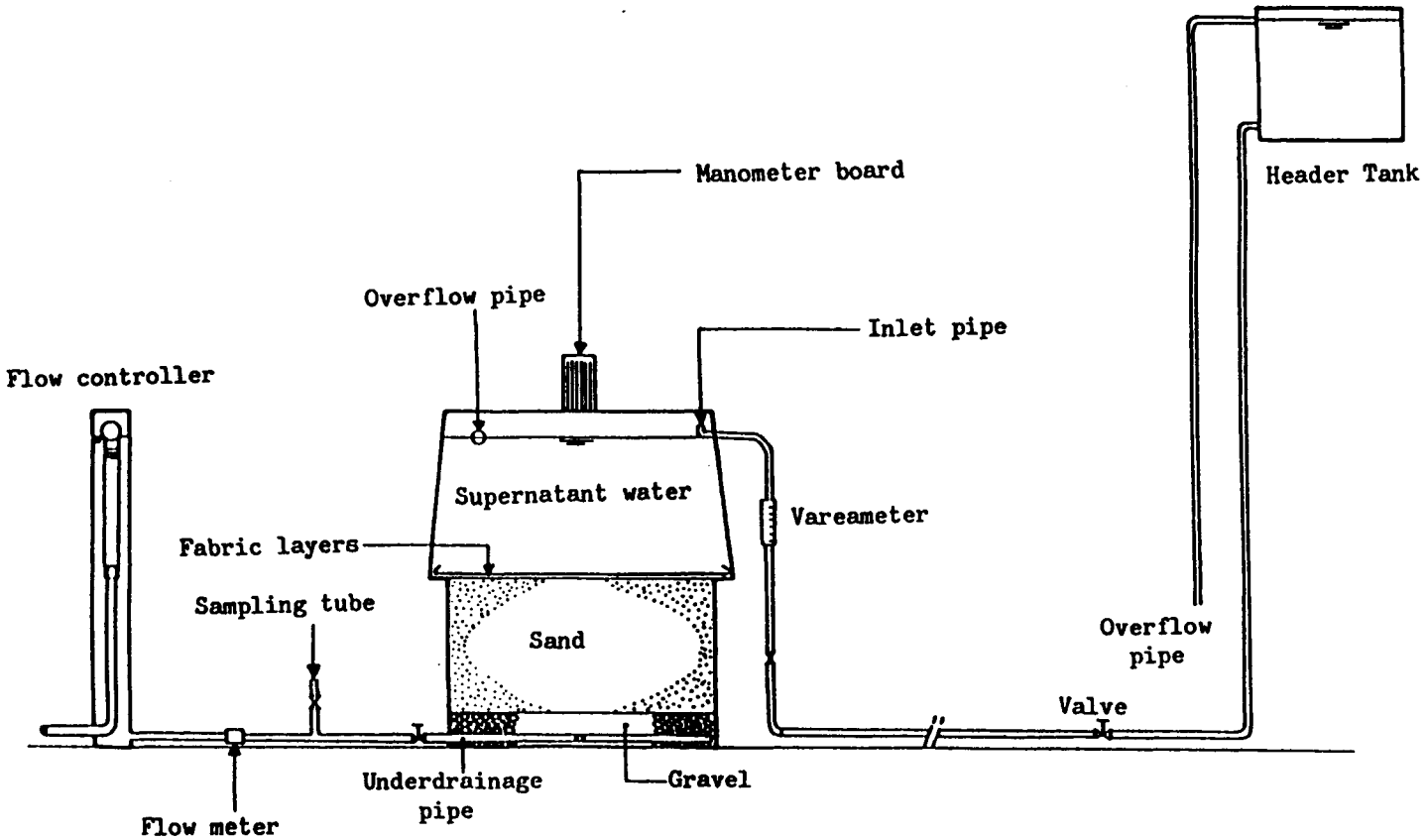


Figure 3.25 Typical cross section through a SSF pilot unit

days to allow the filter to mature. The cycle of filter operation and cleaning is repeated until the depth of the remaining sand bed is approximately 0.4–0.5 m, at which time the sand bed must be re-made with clean sand to its original depth (~1 m). Further information on the basic process and new developments can be found in the proceedings of several international conferences (Graham, 1988; Collins and Graham, 1994; Graham and Collins, 1996), professional guidance manuals (ASCE, 1991; AWWA, 1991), and literature reviews (eg. Lambert and Graham, 1995), which have been published in the last 10–15 years.

Slow sand filters made from small scale columns (~15 cm diameter) have been used in the laboratory to provide some basic information about treatment and hydraulic design, but in general these are not recommended since they fail to simulate the full scale conditions which govern the filter performance. The complexity of the slow sand filtration process requires the use of natural influent water and appropriate field conditions in order to establish representative biological populations and processes, and exposure to the normal range of external factors (for example temperature, dissolved O_2/CO_2, light intensity). Investigations into the use of slow sand filtration, and design information, can be obtained from pilot scale slow sand filters located conveniently close to the source water to be treated. Pilot filter units can be constructed as 1 m diameter columns or as slightly larger rectangular tanks (say, 2 m × 1 m) which allows for more convenient cleaning operations (Figure 3.26). Such investigations need to be

Figure 3.26 Pilot SSF units

carried out over a long time period (typically >1 year) in view of the need to complete several run cycles of filtration and filter cleaning (~1 month per cycle) to get reliable performance data, and to include the full seasonal range of influent water quality. Detailed information on the use of pilot plants for slow filter design can be found in Chapter 8 of ASCE (1991).

3.8 Specialized techniques

Various specialized techniques have been developed for use in research laboratories. Since these techniques require facilities and personnel not normally available, they are listed briefly below with reference to further information.

3.8.1 In-depth sampling

This is the withdrawal of suspension samples from various depths in the media during filtration. It should be isokinetic and continuous (Ives, 1966).

3.8.2 Multiple filter operation

This is the operation of several filters in parallel each with a different depth of media. The filtrates represent the various depth samples without the dangers of disturbance associated with in-depth sampling (Ison and Ives, 1969).

3.8.3 Radioactive labelling

By labelling the suspension particles with a radioisotope, the radioactivity emitted by accumulated deposits can be measured *in situ* with an external detector (Ives, 1962).

3.8.4 Radial flow filtration

For studying filtration in the radial direction, instead of the usual linear downward or upward flow, a radial sector model can be used (Ives and Horner, 1973).

3.8.5 Conductivity technique

The volume occupied by local deposits in the pores during filtration can be obtained by a non-disturbing technique using conductivity. The

time of passage of a slug of electrolyte injected into the filter inflow is measured between two electrodes in the media (Mints and Meltser, 1970).

3.8.6 Surface chemical properties

The surface chemical properties of media, particularly the electrokinetic potential on the filter grains, can be measured using a streaming potential cell (Gregory, 1975; Ives and Gregory, 1966).

3.8.7 Optical fibre endoscopy

A very useful technique has been developed and applied in recent years to enable areas inside filters to be viewed, photographed and video recorded with closed circuit television. The technique utilises endoscopes which can be fitted through the filter wall (model or full-scale), penetrating for several hundred millimetres into the vessel.

The endoscopes are rigid metal tubes typically 8 mm outside diameter, which contain an annular optical fibre system which conducts light to the tip, illuminating the sand and pores local to the tip. The view of this illuminated area is conveyed by a series of lenses in the axis of the tube to an eyepiece, which can be inspected by eye, camera or CCTV. Consequently, areas remote from the wall can be viewed, while the filter is in operation, and the details of the sand or suspension deposits can be recorded. An arrangement of endoscope and CCTV in a model fixed-bed filter is shown in Figure 3.27.

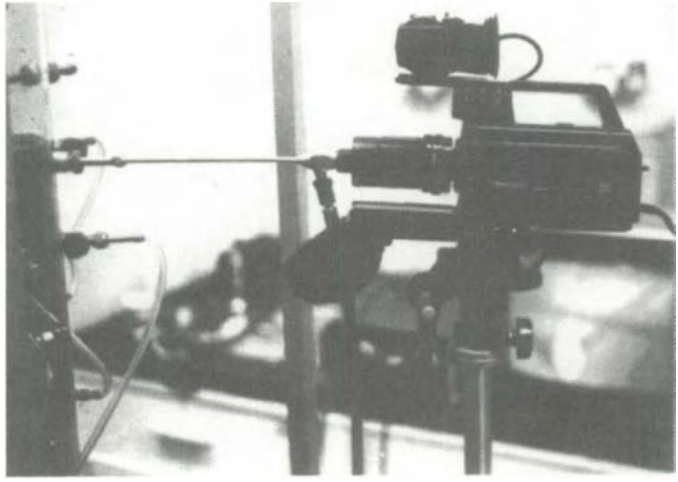


Figure 3.27 Model filter with optical fibre endoscope and CCTV camera

Endoscopes are available for direct straight-through viewing, lateral 90° and lateral 45° viewing, and are rotatable. Thus, they are able to view areas beyond the tip, and downstream or upstream. In addition to observations in the depth of the filter sand, they enable the surface of the filter to be seen, if suitably located. Typical areas of view are about 10 grains, to a distance of 1 or 2 grains. An example is shown in Figure 3.28. With the aid of a 65 cm TV monitor screen, a magnification of up to 500× is possible, allowing much fine detail of flow of suspension, and deposit structure to be seen. With video recording, freeze frame, slow motion and high speed search of the record is possible (Ives and Clough, 1985).

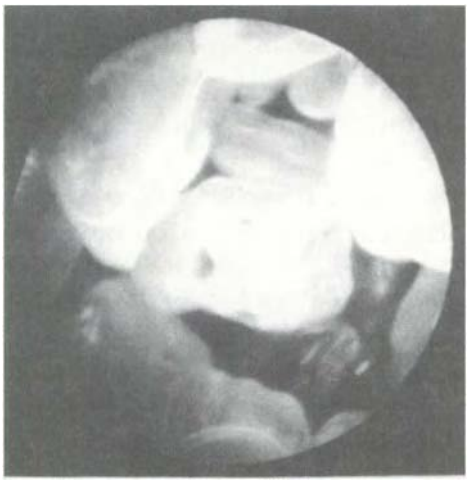


Figure 3.28 Typical view of filter grains in the depth of a filter, viewed through an endoscope

A number of observations have been made, including the unexpected shear gradient in the moving sand of a continuous filter as described in Section 3.7.1.2. Also in continuous filtration the sand grain movement was seen to be rotational and jerky, and not as a steadily moving mass. This has implications for scouring of deposits as the sand moves. Other observations, in fixed beds, have confirmed the presence of holes in surface mat deposits. In depth recordings have shown the form and instabilities of deposits, and the flow patterns through different-shaped pores.

This technique has many potentialities for detailed inspection of conditions deep inside filters, including biological growths, and washing phenomena. The magnification that was available at the time these instruments were used (1980–1990) was not sufficient to resolve individual bacteria.

3.8.8 Mathematical and computer modelling

Extrapolation of small-scale model experiments often relies on mathematical models to represent the filtration processes. Alongside physical theories of filtration, many mathematical models have developed, describing in particular the changes in filtrate quality and head loss in depth and time, for simple unisize-media filters to complex multilayer multimedia designs. Generally, the mathematical models have been too formidable for analytical solutions. Consequently, computational solutions have evolved, many based on finite-difference approximations. A comprehensive review has been presented in the book edited by Rushton (1985).

A particular result of mathematical and computer modelling has been its use for optimization. The concept of an operating optimum resulting from simultaneous filtrate quality breakthrough with reaching the head loss limit has been long known for simple unisize-media filters. Its extension to size-graded, or multilayer filters was defeated for a long time by the complexity of the problem. However, the solution for such complex filters has been presented by Sembi and Ives (1983). A commercially available filter model is included in the OTTER water treatment design and simulation software developed by WRc plc. The filter model employs either a logistic (S-shaped breakthrough curve) or finite-difference approach and incorporates the specification of different media types, backwashing arrangements and the operation of a bank of filter units. More advanced models are currently under development both for simulating filter performance (e.g. Stevenson, 1997) and backwashing performance (Hall and Fitzpatrick, 1998).

Nomenclature

C	quality of water, e.g. concentration of suspended solids
C_0	initial quality of water
C_D	drag coefficient
d_{10}, d_{60}	grain 'percentiles' (maximum size of, e.g. 10% or 60% of grains, % wt.)
d_h	hydraulic diameter (diameter of sphere of equal settling velocity)
d_s	sieve size of grains (geometric mean of openings of the passing and retaining sieves)

F	Filterability Number
g	gravitational constant
h_1, h_2, etc.	head loss between adjacent manometer points
H_L	total head loss across bed
H_{limit}	maximum allowable head loss
H_d	extra head loss, above H_0, for good deep bed filtration
H_0	head loss through bed with clean water flowing
H_s	extra head loss above $H_0 + H_d$ due to surface clogging
l	filter bed depth
M	mass of media
Re	Reynolds Number
u_1	fluid velocity
u_p	terminal settling velocity of a grain
U_g	velocity gradient of fluid
ε	bed voidage (porosity)
θ	time
θ_f	flocculation time
ψ	shape factor/sphericity
ρ	density of fluid
ρ_s	density of a grain
μ	viscosity of fluid

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4 Membrane filters – microfiltration and ultrafiltration

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Microporous membranes are used widely for the separation of two-phase mixtures. While these can be solid/liquid, solid/gas, liquid/gas or liquid/liquid systems, or combinations thereof the principal focus of this chapter will be solid/liquid separations.

These membranes are available in a variety of materials, pore sizes and constructional configurations. In the context of separation, categorisation of pore size is almost exclusively related to the size of material that will be separated. This is typically measured in microns for microfiltration (MF) and Daltons for ultrafiltration (UF). In this latter case, the molecular weight cut off is used as a measure of rating.

Membranes are physically configured in a number of ways, each designed to maximize membrane surface area. Pleated cartridges, flat sheet and hollow fibre configurations are common. Flow paths can be either direct flow where the entire process fluid passes through the porous structure or the so-called cross flow where the process fluid is split as it is incident upon the membrane, with some passing through the membrane and the remainder passing tangentially across the membrane surface. In liquid applications, the common configuration for direct flow filtration (DFF) microfiltration is the cartridge format and ratings of these types of devices are generally in the range 0.01 to 40 microns. Cross flow or tangential flow filtration (TFF) devices are preferred for UF applications and are usually of the hollow fibre or flat sheet type.

The DFF and TFF processes are illustrated diagrammatically in Figures 4.1 and 4.2.

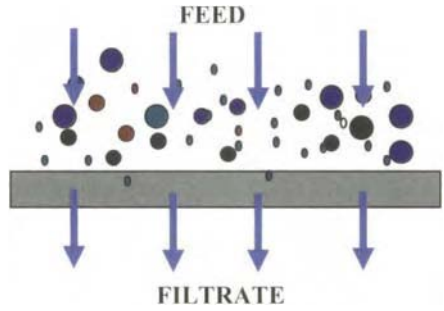

Figure 4.1 Direct flow filtration

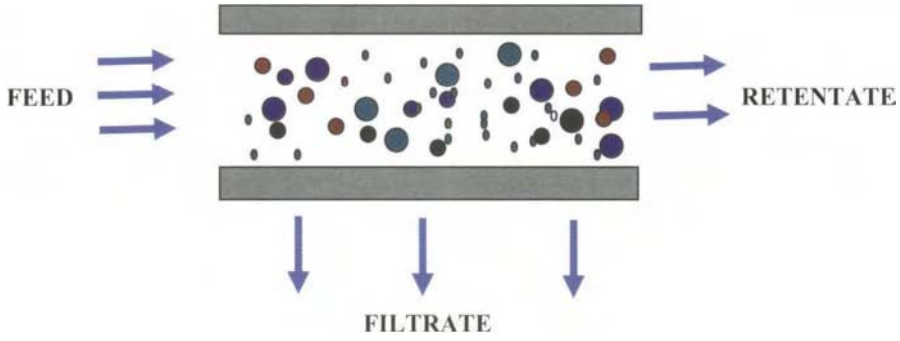

Figure 4.2 Tangential flow filtration

To select the most appropriate membrane type for a given process separation a number of factors must be considered. Firstly, the nature of the separation that is required. In the context of MF and UF, this will usually be the separation of discrete solids from a fluid stream. The required level of separation must be established and this will be discussed under the following detailed study of the respective techniques. It is, of course, a fundamental part of economic process design and scale-up.

To logically order the following discussions, we will categorise the technologies by application. Firstly, the DFF membrane microfiltration as used in the biopharmaceutical, food and beverage and finer industrial applications, then TFF microfiltration and ultrafiltration and finally, large scale membrane applications will be considered. In this way, all of the techniques for effective sizing and scale-up will be covered in such a way that any separation application could apply the given procedures irrespective of industry.

4.1 Direct flow filtration (DFF)

4.1.1 Single pass

DFF is the technique of choice for traditional sterilizing filtration stages of liquid applications as used in the pharmaceutical industry.

Scale-up testing aims to generate data on a small scale that will be used to predict the performance of a full size direct flow filtration process system. In this way, the costly consequence of under sizing or over sizing the system can be avoided. By working at a small scale the volume of fluid required is small and the data can be scaled up to the appropriate process size. In addition a range of media can be evaluated and the optimum filter sequence selected. Initial sizing can be based on representative membrane samples. Thereafter, intermediate pilot scale trials could be undertaken in order to confirm the actual sizing of the installation prior to larger operation.

At full-scale operation, fluids are processed either by pumping at a constant flow rate, or by the application of constant driving pressure. It is important to recognise this difference when undertaking scale-up testing. Both tests, whether constant flow rate or constant pressure filtration should, wherever possible, be carried out at the process system temperature. Constant flow rate filterability measures differential pressure across a filter as a function of time and volume filtered. Constant pressure tests measure flow rate, volume filtered or mass filtered as a function of time at that constant feed pressure.

Evaluation of various filter media should allow one to minimise the cost per volume, simplify the process and inventory and allow a margin of safety that ensures the entire fluid batch is filtered. This can be achieved by increasing the surface area, using pre-filters, changing the operating parameters or by modifying the product.

The base data can be gathered by using simple equipment. This would include a pump, or pressure source, a stopwatch and balance to record the increase in volume with time. The success of this technique has led to the production of fully automated bench-top systems for the development scaling-up and scaling-down of direct flow filtration processes and such equipment is commonly used by the biopharmaceutical industry.

4.1.2 Approach to media selection

Prior to commencing scale-up testing, one should consider the following in selecting the media type and grade to be evaluated for the final filter:

- Quality of filtrate required
- Compatibility of fluid with media and hardware
- Style of cartridge or capsule
- Availability of media in style and grade required
- Operating temperature and differential pressure
- Batch size and required filtration time.

In addition to evaluating final filter requirements, use of pre-filtration to make a filtration system more economical can be assessed. This is achieved by using coarser grades of media with a higher contaminant capacity for larger particles to pre-filter the fluid supplied to the final filter and, hence, prolong its service life.

The performance of a pre-filter can be assessed by using two or more discs in series and connecting the outlet of the pre-filter disc holder to the inlet of the final filter disc holder. The test is performed at a constant flow rate and the pressure drop is monitored across each disc holder. If the pore size of the pre-filter is too fine then the first stage will block prematurely. Conversely, if rating of the pre-filter is too coarse then the flow of the final filter will decay too rapidly. The successful outcome is a balance of contaminants across each stage such that the blockage, or flow reduction occurs at approximately the same rate.

4.1.3 Automated scale-up test devices

Automated filterability testers have been designed for development scale-up or scale-down of direct flow filtration processes, optimisation of direct flow filtration, and the validation or troubleshooting of existing process systems. An illustration of a device is presented in Figure 4.3. Most devices comprise disc holders, pressure transducers, a temperature probe, a load cell and a control system with on-board software and hardware. A membrane segment or disc is loaded into a special holder (or a self-contained capsule is fitted) with a pressure transducer attached. The device should be capable of running the constant pressure or constant flow tests referred to above. The constant flow test requires a pump to run at a constant rate while monitoring the increase in pressure upstream of the membrane, while in the constant pressure test, the fluid is propelled by a top pressure of gas (air or nitrogen), and the decreasing flow rate through the membrane is monitored.

Automated systems have been developed to meet these requirements, together with the sophisticated technological requirements of 21 CFR

Figure 4.3 Automated filterability tester

Part II (FDA, 1997), having three levels of user access, full electronic records and audit trails, electronic signature capability, password ageing and transfer of programmes to allow archiving of records.

The automated scale-up test device reads in a data set of temperature, pressure and fluid weight at each logging point. The device then calculates the differential pressure across each active station, the volume of fluid filtered and the flow rate.

Certain test devices can store both the raw data and the calculated values at each data point in addition to displaying graphs of time/volume (t/V) *vs* time and a graph of volume *vs* time. Some test devices are able to store user identities for security and traceability purposes and test results for ease of accessibility and provision of a full audit trail.

4.1.4 Constant pressure test

The constant pressure test measures flow decay at the given feed pressure. V_{cap} or V_{max} is the theoretical maximum volume that could pass through the membrane until the flow decays to zero. The test is based on a gradual pore blocking model that is characterised by a gradual, controlled blocking of pores as a function of the amount of filtrate passing through the filter. This mechanism of filter blocking is generally seen with controlled process applications with a constant contaminant loading.

A schematic of a typical test set-up is illustrated in Figure 4.4. Here, a top pressure of air or nitrogen is applied to a fluid in a reservoir and a

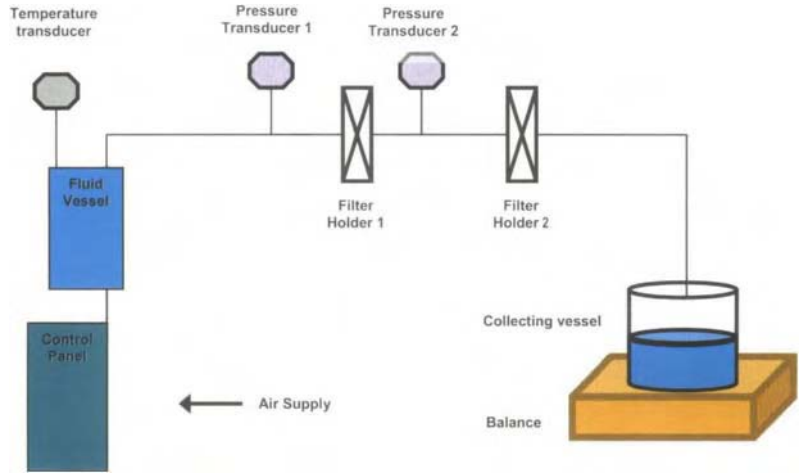

Figure 4.4 Schematic of constant pressure test set-up

pre-selected constant pressure is used to drive the flow. This flow is normally high at the start of a test, but not necessarily optimal for high throughput. The filter holder should be vented, allowing fluid and air to exit through the vent valve. Failure to fully vent all the gas from the upstream surface of the test filter can create an airlock resulting in a serious source of error due to a reduction in the effective surface area of the test filter.

The automated tester records the volume of filtrate collecting in the collecting vessel and the test ends when it reaches the operator defined endpoint. When the endpoint is reached, the system gas pressure should be isolated to the stations and vented.

Figure 4.5 illustrates the type of data collected during the constant pressure test. As the cumulative throughput increases, the time/volume increases as a function of time. The flow decreases over the same period as the filter material gradually blocks.

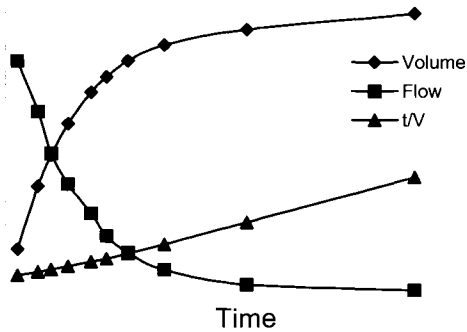

Figure 4.5 Volume, flow and t/V *vs* time

Due to its simplicity the constant pressure test provides fast comparative analyses. However, the throughput value is an extrapolation and may lose some accuracy for this reason. Also the high initial flow rates in the early stage of the tests may not be representative of actual conditions and may give a lower throughput prediction than a constant flow process.

4.1.4.1 V_{cap} data calculations

To analyse the constant pressure data, time (t) is divided by the cumulative volume (V). The quotient t/V can then be plotted against t. The slope, y-intercept, and correlation coefficient are obtained by a least squares-fit analysis of the data. Generally the last ten data points are used as they are linear and the correlation coefficient (R^2) should be >0.99. If these parameters are not met, it may suggest that filter-blocking mechanisms other than gradual pore blocking are occurring (Badminton *et al*, 1995). V_{cap}, which is the maximum volume (in appropriate units) that will pass through the test filter, equals the reciprocal of the slope.

The positive gradient of the t/V line is indicative of filter blockage as the contaminants reach the membrane surface and gradually build up on the inner walls of the pores, eventually causing blockage. The rate at which this occurs will be dependent upon the level of contaminant present and is reflected directly in the t/V gradient. Figures 4.6, 4.7 and 4.8 illustrate this point.

4.1.4.2 Sizing

Scale-up is based upon the ratio of filter disc area to that of a filter cartridge. The results can be used to give either filter size for the required throughput between change-outs or on-stream life for a required filter size. Where blockage of the filter disc is not achieved but required filtrate quality is, sizing of a suitable filter system would be based on basic sizing techniques, that is, system flow rate, maximum flow rate/cartridge or assembly pressure drop.

$$\text{Scale-up Ratio} = \frac{\text{Filtration Area of Cartridge}}{\text{Filtration Area of Disc}}$$

To calculate the total volume filterable through a cartridge, the disc throughput is multiplied by the relevant scale-up factor.

Example The fluid of interest is passed through a 47 mm test disc of a specified media and the V_{cap} value determined from the following data collected in accordance with the above discussion.

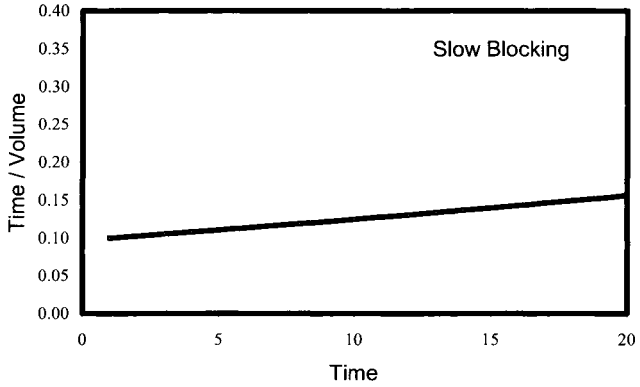

Figure 4.6 Gradual membrane blocking

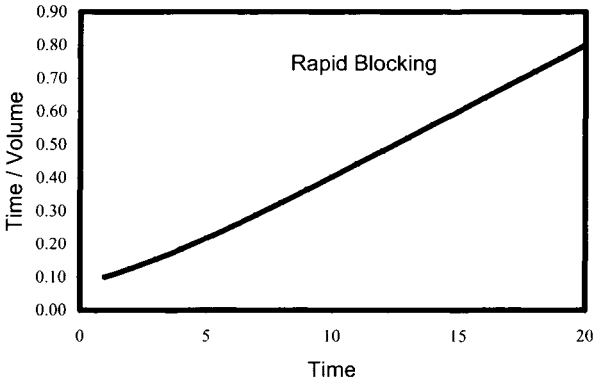

Figure 4.7 Rapid membrane blocking

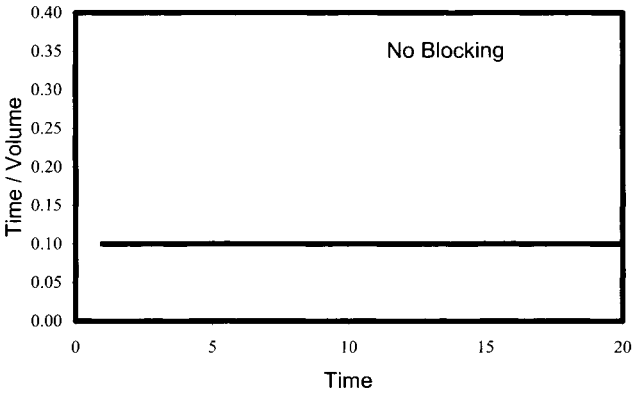

Figure 4.8 Minimal membrane blocking

From data collected in accordance with the foregoing method, a V_{cap} value of 103 ml was derived. However, in order to provide a margin of error in scale-up, a value of V_{90} is determined at a point at which the flow decays by 90% of the initial value. This has been determined to be 68% of V_{cap} (Badminton *et al*, 1995). In the case of our example, this equates to 70 ml. This value is then used in subsequent calculations.

The throughput of the fluid using a 10 inch (254 mm) cartridge of the specific media with a known filtration area can be determined as follows:

Filtration area of 10 inch cartridge filter = 6000 cm^2

Effective filtration area of disc = 14 cm^2

$$\text{Scale-up Ratio} = \frac{\text{Filtration Area of Cartridge}}{\text{Filtration Area of Disc}} = \frac{6000}{14} = 428$$

Therefore,

$$\begin{aligned}\text{Throughput} &= V_{90} \times \text{Scale-up Ratio} \\ &= 70 \times 428 \\ &= 29960 \text{ ml} \\ &\equiv 29.96 \text{ litres per 10 inch filter cartridge}\end{aligned}$$

4.1.4.3 Processing time

The data generated by the automated scale-up test device can be transferred to spreadsheet software and a trend line applied to the *t/V vs t* graph. From the resulting trend line equation the time required to process a particular volume of the test fluid through the filter media of a specific size can be established.

Example The required volume to be processed is 10 litres therefore the equivalent volume that needs to be filtered through a 14 cm^2 test disc is calculated as follows:

For a standard capsule of area of 600 cm^2:

$$\text{Process volume (ml)} \times \frac{\text{filtration area of the disc}}{\text{filtration area of capsule}} = 10000 \times \frac{14}{600} = 233 \text{ ml}$$

In this case, data collected gave the *t/V vs t* relationship presented in Figure 4.9.

The trend line equation is utilised to determine the time it will take to process the fluid through a particular sized cartridge as follows:

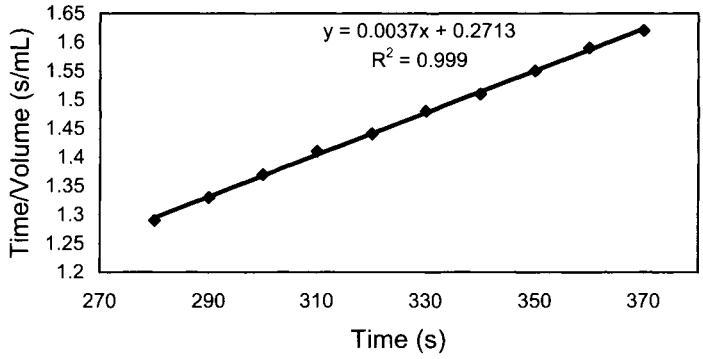

Figure 4.9 *t/V vs* time relationship

$$\frac{t}{V} = 0.0037t + 0.2713$$

Substituting for V (233 ml) to calculate t gives:

$$\frac{t}{233} = 0.0037t + 0.2731$$

$$\therefore\ t = 456 \text{ s}$$

Therefore, it will take approximately 7.5 minutes to process 10 litres of the test fluid through one capsule.

4.1.5 Constant flow test (P_{cap})

In constant flow filterability, the flow rate used is based on the process flow rate and an approximation of the size of a filtration system required. The set-up for a constant flow test is depicted in Figure 4.10.

In order to simulate the process conditions, the flow rate is scaled-down from the process flow rate as follows:

$$\text{Scale-up Ratio} = \frac{\text{Process Fow Rate}}{\text{Test Flow Rate}}$$

In the constant flow test, the selected flow rate remains constant with time whilst the differential pressure and total volume of fluid processed increase with time (Figure 4.11).

As the test is run, the test filter sample disc will begin to block and an increase in the differential pressure will occur. The end point can be readily established from the relationship of differential pressure *vs* volume. This can be set at the maximum filtrate volume achieved or by a predetermined maximum differential pressure available or desirable.

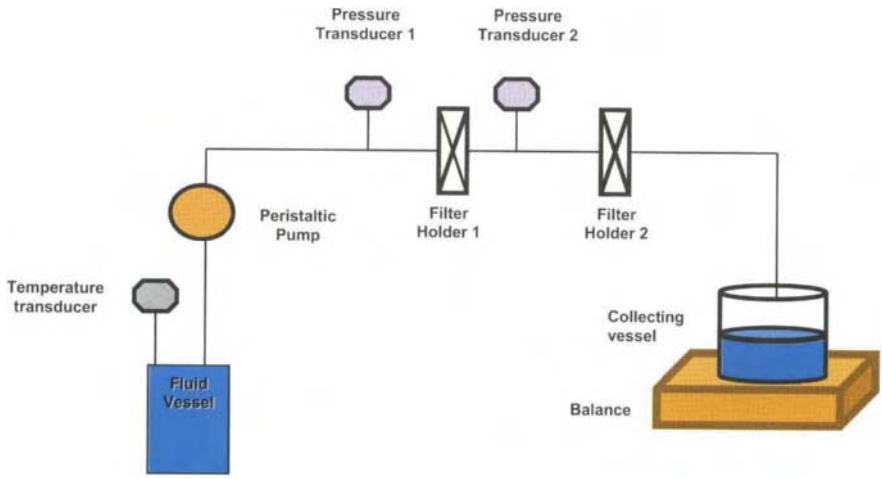

Figure 4.10 Schematic of constant flow test set-up

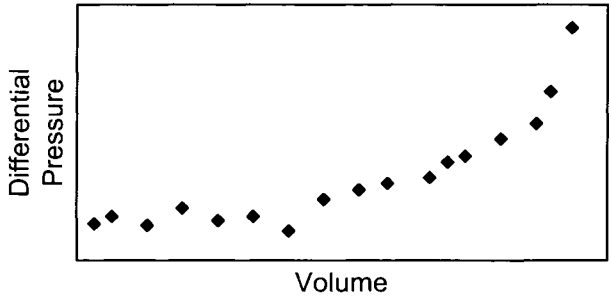

Figure 4.11 Constant flow test output

In this test, the final throughput is an actual, not an extrapolated value and can therefore be more accurate than V_{cap} for sizing purposes.

If a fluid has high contaminant loading, then only an increasing differential pressure with little throughput will be observed due to rapid blocking of the test disc. In extreme cases, complete blinding of the test disc will occur, no throughput will be seen and the differential pressure will rise rapidly. Conversely, with low contaminant loadings only minimal increase in differential pressure increase may be observed. This latter situation may arise from a naturally low level of contaminant or an inappropriately rated membrane sample.

The constant flow test allows determination of the throughput for any pressure within the measured range (Figure 4.11). From the graph or accompanying data the output volume corresponding to the differential pressure of choice can be scaled up to produce a throughput value for a particular cartridge area:

Throughput for a particular cartridge = Output to specific end point × Scale-up Ratio

The number of cartridges required can be determined as follows:

$$\text{Number of cartridges} = \frac{\text{Process volume}}{\text{Throughput for a particular cartridge}}$$

With these values, the number of cartridges required to filter a specific batch can be calculated or the life of a cartridge in the system can be determined.

4.1.6 Case study

A lipid-containing fluid required sterilisation prior to use in the pharmaceutical industry. A suitable filtration train had to be identified that would filter 350 litres of the fluid at a flow rate of 6 litres/hour.

For this application, the rating of the filter membrane was 0.2 microns to achieve sterility. Initial scale-up trials were conducted at a constant pressure of 500 mbar using filters of various media to determine the expected throughput. The results were as follows:

Filter 1:

Filtration area of 10 inch (254 mm) cartridge filter = 6000 cm^2

Effective filtration area of disc = 14 cm^2

Scale-up Ratio: 428

The following values of filtered volume were determined (Figure 4.12) using the above procedures (using part of the data):

V_{cap} = 36.3 ml

V_{90} = 24.7 ml

R^2 = 0.99

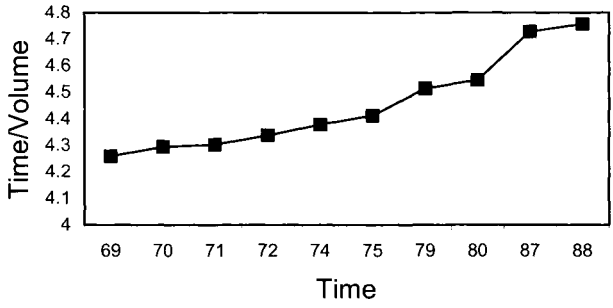

Figure 4.12 *t/V vs t* (500 mbar constant pressure test)

Throughput $= V_{90} \times$ Scale-up Ratio
$= 24.7 \times 428$
$= 10\,571$ ml
$\equiv 10.6$ litres

Filter 2:

Filtration area of 10 inch (254 mm) cartridge filter = 5500 cm^2

Effective filtration area of disc = 14 cm^2

Scale-up Ratio: 392

For this filter sample, the following capacity values were determined:

V_{cap} = 25.2 ml
V_{90} = 17.1 ml

Throughput $= V_{90} \times$ Scale-up Ratio
$= 17.1 \times 392$
$= 6703$ ml
$\equiv 6.7$ litres

Due to the higher throughput, Filter 1 was the sterilising grade filter of choice for this application.

In order to improve the throughput of Filter 1, constant pressure tests at 500 mbar were used to evaluate a selection of pre-filters in combination with Filter 1. The pre-filter can be set up in series with the final filter or the pre-filtered filtrate can be collected and passed separately through the sterilizing grade filter.

Pre-filter 1 + final filter

Filtration area of 10 inch cartridge filter = 6000 cm^2

Effective filtration area of disc = 14 cm^2

Scale-up Ratio: 428

V_{cap} = 422 ml
V_{90} = 286 ml

Throughput $= V_{90} \times$ Scale-up Ratio
$= 286 \times 428$
$= 122\,408$ ml
$\equiv 122.4$ litres per 10 inch filter cartridge

Pre-filter 2 + final filter

Filtration area of 10 inch (254 mm) cartridge filter = 6000 cm^2

Effective filtration area of disc = 14 cm^2

Scale-up Ratio: 428

V_{cap} = 252 ml

V_{90} = 171 ml

Throughput = V_{90} × Scale-up Ratio

= 171 × 428

= 73188 ml

≡ 73.2 litres per 10 inch filter cartridge

During the constant pressure tests blockage of the pre-filters did not occur and a separate V_{cap} value was not determined for the pre-filter. However, both of the pre-filter choices gave protection to the sterilising final filter. It can be seen that Pre-filter 1 gave a significant increase in throughput of the overall filter system.

In order to simulate the process conditions, constant flow tests equivalent to 6 L/hour using 47 mm discs of the media of choice were undertaken. From the output, the size of the filtration train was recommended based on the throughput to a specific differential pressure.

In some cases it may be desirable to perform pilot scale trials under process conditions in order to confirm the actual sizing of the installation and mode of operation.

A range of scaleable products for direct filtration is commonly available. These include syringe filters and assemblies, capsules and cartridges. This enables consistent materials of construction for all phases of development from R&D to pilot and full production. The range of scaleable products is capable of throughputs from a few millilitres to a few hundred litres through a variety of media.

4.2 Recirculation systems

Many solids separation applications involve recirculation of the process fluid. The most common are those in which a lubricant is recirculated, as in hydraulic, metal working and bearing lubrication systems. However, there are also many applications, such as electricity generator stator cooling circuits, electro-plating baths, gas scrubbers etc, where the fluid must be maintained in clean condition. The cleanliness

required is governed by such factors as the smallest passageway or orifice in the circuit, the life and reliability of the system, the quality of the end product (electro-plating and wash plants), and in the case of lubrication systems, the loads on the bearings.

4.2.1 Filter selection

Selecting a suitable filter rating starts with consideration of the smallest orifices in the circuit – in the case of lubrication systems, of the bearing and component clearances. Where fixed orifices are involved, with the risk that a single particle could plug the orifice, the filter rating must reflect the orifice size. Where bearing clearances are to be protected, the filter removal rating can often be significantly coarser, as the recirculation, or "multi-passing" of the fluid through the filter means that smaller particles are repeatedly presented to the filter, and have therefore a much higher probability of being captured.

A filter having a removal efficiency of say 99.5% (equating to a filter or beta ratio of 200) for particles larger than 3 μm will usually give adequate protection to bearings with running clearances of only 0.5 μm. For hydraulic and lubrication systems, the British Fluid Power Association provides much detail for filter selection (BFPA, 1999).

Having selected a suitable rating, the size of the filter in terms of flow capability and dirt capacity must be determined. The maximum permitted pressure drop across the filter must be known – this may be determined by the circuit considerations, or it could be determined solely by the design of the filter cartridge. For example, in a circuit using a centrifugal pump, there will probably be a pressure drop across the filter, above which the flow will be insufficient to maintain the system function. This then is the terminal filter pressure drop, and a suitable warning device should be fitted to indicate when it is reached. The filter should then be sized to have a pressure drop, on installation, some fraction of the terminal pressure. On occasion, the system designer will dictate both maximum "clean" and maximum "blocked" pressure drops, in which case sizing is usually simplified. However, it is important to remain within the flow capacity of the filter, both cartridge and housing, as defined by the filter supplier, and based on the viscosity, density etc. of the fluid.

Where closed-circuit systems are concerned, with minimal solid contaminant ingression rates, such as recirculating coolant designs, the filter size can often be minimised. It would be unusual to have a "clean" pressure drop as high as 1 bar, but where space is severely restricted or costs are very important this could be considered. The

disadvantage is the relatively short cartridge life, which unfortunately is not easily predictable. Further, such an aggressively sized installation will respond badly to system upsets – occasions where an unexpected high solid contaminant level occurs may require several changes of cartridges before equilibrium is restored.

Initial, or "clean", pressure drops between 100 and 500 mbar would be more usual, with even lower figures used if the circuit design demands it. The lower the "clean" pressure drop the longer will be the life of the filter, so the initial pressure drop will depend at least to some extent on the anticipated rate of contaminant ingression. Where ingression rates are likely to be high, as in electro-plating baths, rolling mill lubrication etc, sizing will be based on the required life, and prediction of life is extremely difficult without prior knowledge of similar systems. The assistance of a filtration engineer with experience of similar applications will be invaluable in these circumstances.

In the case of hydraulic systems and closed loop lubrication systems, ingression of fresh contaminant to the circuit is generally fairly low, so initial pressure drops can often be relatively high. However, components in these applications, particularly in hydraulic circuits, are subject to wear, so contaminants tend to be internally generated. Coarser filters generally have lower pressure drops, for a given cartridge size, and so would normally be expected to have long life. However, the recirculation of the larger contaminant particles can lead to higher wear rates at the bearing surfaces, with the result that finer filters can last longer due to the reduced load of wear particles, as well as providing the considerable advantage of longer component life and increased reliability.

Sizing in these applications is often based on flow rate and viscosity to give a "clean" pressure drop of 0.3 to 0.5 bar, even up to 1 bar, modifying this downwards if the environment or duty is particularly severe. One advantage of filters for these applications is that often a given design is available with different cartridge lengths, and hence effective filtration area. Again, the British Fluid Power Association document (BFPA, 1999) provides guidance in sizing filters, making allowance for their position in the circuit.

Inevitably, on a new recirculating installation, the sizing of the filter is a rather inexact science. It is rare that the contaminant ingression/generation rates can be accurately predicted, the particle size distribution will not be known until the system has reached a contaminant equilibrium, and the "dirt capacity" of a filter, where a manufacturer is able to provide it, is very dependent on the size distribution of the circulating contaminants. To compound the problem further, the

standard performance test uses a silicaceous material which is rarely typical of contaminants found in most applications, and is completed in, at most, a matter of hours. Correctly sized filters in recirculating systems rarely have lives less than a few weeks, and can often operate for a year without maintenance, implying that in-service beta ratios are likely to be quite different from the ones determined during laboratory testing. This means then, that even with the most careful consideration of the variables, and if possible leaving options such as cartridge area and rating with a degree of flexibility, on occasion a filter will not perform as anticipated.

Scale-up is rarely appropriate for recirculation applications for these reasons. Since there is no feed stream; as it is only generated once the circuit is running and in equilibrium, there is no stable platform upon which to base a test. The technique might be useful for assessing cartridge life when changing from a given filter rating to a finer one, on an existing system, but even then, it will only really be useful for predicting the life of the first set of cartridges. Any subsequent set will be presented with a completely changed particle size distribution, and will therefore have a different life.

4.3 Tangential flow filtration (TFF)

The properties of membranes render them capable of performing highly selective or specific separations as well as separation of solids from fluids with very high solids loadings. To enable such applications, TFF configurations are used. In this way, economic membrane life can be achieved.

In the context of these examples, separation of solids from liquids where high or variable contaminant loadings are present will be described as "Industrial" while applications utilising finer separation capabilities of the membranes will be considered with biopharmaceutical examples.

The first of the TFF topics will consider separations that are typically undertaken in the biopharmaceutical industry. The filtration and scale-up principles translate well into other TFF applications.

4.3.1 Biopharmaceutical MF and UF

There are numerous instances of TFF membranes in use in the biopharmaceutical industry. Examples include concentrating, desalting and performing diafiltration for buffer exchange, clarification of solutions ranging from fermenter broths to tissue extracts, depyrogenation

of aqueous streams such as antibiotic solutions and high purity water. There are also a variety of other applications that cannot easily be performed with direct flow filters.

Flow geometries are either through flat sheet devices, such as membrane cassettes and membranes that fit plate and frame type systems, tubular (polymeric hollow fibres with a number of different lumen diameters) or ceramic modules.

As described in previous sections, TFF devices contain membranes in both the MF and UF ranges. UF membranes are typically available as fine as 650 Daltons (used to remove salt from oligonucleotides for example), up to 1 million Daltons (typically used in vaccine processing); or in the microfiltration range, with ratings from 0.1 μm to 5 μm. Some typical applications for each type are listed in Tables 4.1 and 4.2.

Table 4.1 UF Membranes – typical applications.

Rating	*Configuration*	*Example application*
650 Dalton	Cassette	Desalting oligonucleotide solutions
1 kD	Cassette	Concentration of peptides
	Ceramic	Alginate (excipient) washing
3 kD	Cassette	Removal of linkers from vaccine oligosaccharides
	Hollow Fibre	Concentration of enzyme solutions
4 kD	Hollow Fibre	Desalting of hormones
5 kD	Cassette	Allergen processing
	Ceramic	Solvent based antibiotic preparation pre-crystallizer
6 kD	Hollow Fibre	Depyrogenation of high purity water and antibiotic solutions
10 kD	Cassette	Albumin concentration and ethanol removal
	Hollow Fibre	Protein removal from antibiotic solutions
13 kD	Hollow Fibre	Clarification of dialysis fluids
30 kD	Cassette	Concentration / desalting of monoclonal antibodies
50 kD	Cassette	Fractionation of vaccine oligosaccharides
	Hollow Fibre	Distillation column protection
20 nm	Ceramic	Production of polyketide antibiotics
70 kD	Cassette	Concentration of IgM
80 kD	Hollow Fibre	Pre-treatment of feed water for deionization
100 kD	Cassette	Fractionation of haemoglobin oligomers
50 nm	Ceramic	Clarification of statins
200 kD	Cassette	Oligosaccharide removal from vaccine conjugates
100 nm	Ceramic	Bacitracin production
300 kD	Cassette	Fibrinogen concentration
500 kD	Cassette	Viral vaccine concentration and protein removal
1000 kD	Cassette	Vaccine cell wall antigen fragment recovery

Table 4.2 MF Membranes – typical applications.

Rating	*Configuration*	*Application example*
0.1 μm	Cassette	Plasmid recovery after cell dissolution
	Ceramic	Continuous clarification of thermophilic bacteria
	Hollow Fibre	Washing unbound material from microcarriers
	Hollow Fibre	Water filtration for pharmaceutical applications
0.16 μm	Cassette	Vaccine toxoid clarification
0.2 μm	Cassette	Cell concentration for bacterial vaccines
	Ceramic	Clarification of penicillin acylase
	Hollow Fibre	Recovery of protein in long term mammalian perfusion cell culture
0.45 μm	Cassette	Virus recovery from cell culture
	PallSep™	Direct recovery of clavulinic acid
		Clarification of milk from transgenic animals
		Recovery of proteins from transgenic plant homogenates
0.5 μm	Ceramic	Clarification of solvent extracts from plant tissue
0.65 μm	Cassette	Batch harvesting of CHO cell culture
	Hollow Fibre	Baculovirus in insect cell culture systems
0.8 μm	Cassette	Concentration of plant cell cultures
	Ceramic	Carbon fines removal
1.2 μm	Cassette	Plankton harvesting in marine biotechnology

4.3.1.1 Cassettes

Cassettes consist of layers of membrane separated by screens and spacers that are sandwiched together as illustrated in Figure 4.13. In standard configuration all membrane layers are in parallel to each other. In cassette format, there are different types of feed channel configurations to suit different applications (generally determined by particulate or protein concentrations and viscosity). Illustrations of screen, suspended screen and open channel are given in Figure 4.14.

Cassettes are typically available in a wide range of membrane areas, in both UF and MF morphologies. They are also constructed in many different materials to suit particular applications, with the most common being polyethersulfone (PES) (many of which undergo some form of modification to improve surface properties such as hydrophilic properties) and cellulose based chemistries. Figure 4.15 shows an example of a TFF cassette.

4.3.1.2 Hollow fibres

Hollow fibre membranes can have both symmetric morphology, with uniform density throughout the depth of the membrane, and asymmetric

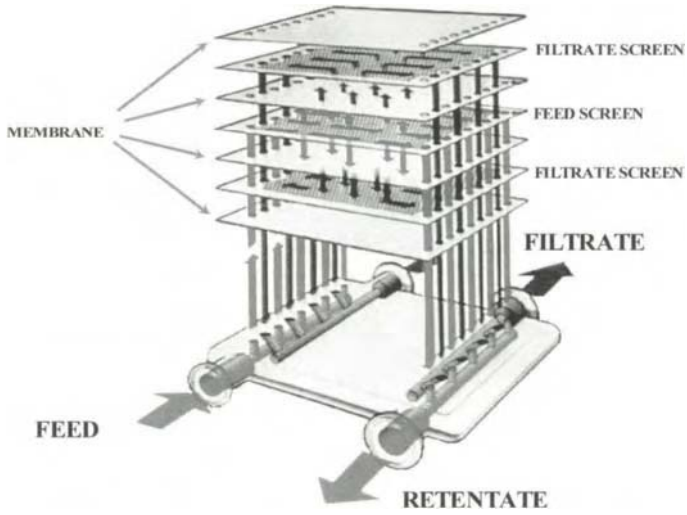

Figure 4.13 Flat sheet UF assembly

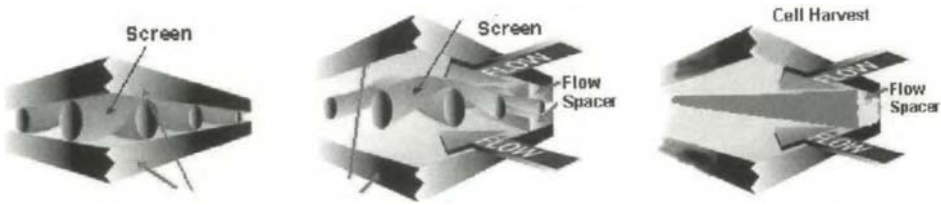

Figure 4.14 Flow channel variations

Figure 4.15 TFF cassette

morphology which exhibits a graded pore size along the permeate flow path. Hollow fibres can possess a membrane layer both on the inside, and on the outside of the fibre thereby enabling flow in either direction and the use of back flushing during filtration to recover flow performance. This is illustrated in Figure 4.16.

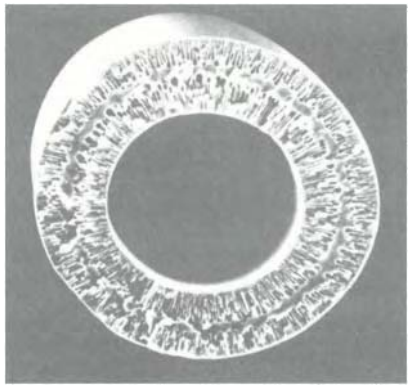

Figure 4.16 Hollow fibre

Hollow fibres, like cassettes, are typically available in a wide range of membrane areas, Molecular Weight Cut-Offs (MWCO) for UF, micron ratings for MF and different membrane chemistries to suit particular applications. The most common membrane chemistries being: Polysulphone (PS), Polyacylonitrile (PAN), Polyvinylidenedifluoride (PVDF) and Polyethylene (PE).

4.3.1.3 Ceramics

Ceramic membranes typically comprise a highly controlled surface membrane layer that is formed on the inner (feed-side) surface of a more open support layer. Typical materials available are, ultrapure α-alumina, zirconia and titania. Ceramic membranes have wide ranging chemical compatibility, are resistant to extremes of temperature and exhibit near zero non-specific adsorption of biological materials.

The ceramic elements are constructed as monoliths with usually hexagonal or round section. They are available with different diameter flow channels available to suit the specific application. Figure 4.17 shows examples of the elements. Large-scale ceramic systems contain stainless steel housings fitted with multiple ceramic elements (via a tube sheet) to provide the desired membrane area.

4.3.1.4 Systems

In general, all TFF systems are assembled and operated in a similar fashion, regardless of the particular format (cassette, hollow fibre, ceramic etc). Microfiltration usually requires some form of restriction to flow on the permeate or filtrate (downstream) side of the membrane to limit fouling (generally a valve or pump will be used). Figure 4.18 shows a typical schematic for UF and MF TFF systems. Note that the

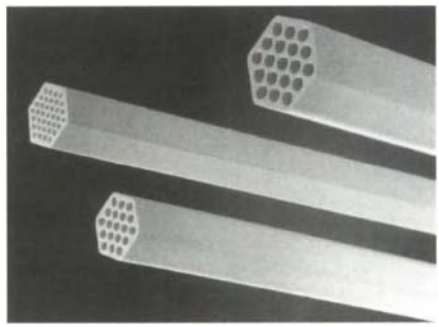

Figure 4.17 Ceramic elements

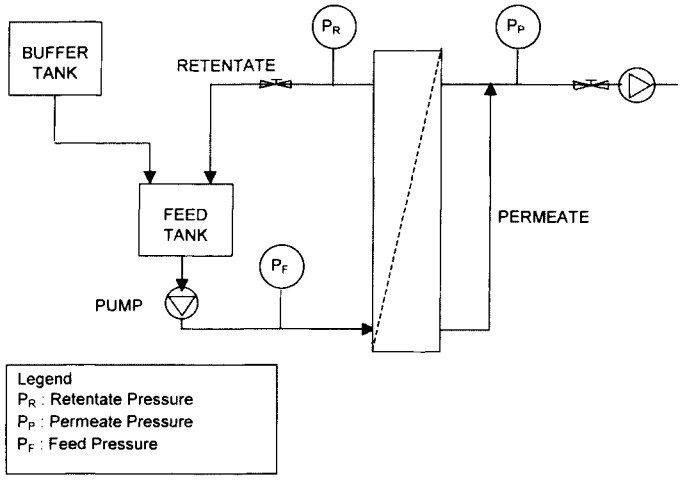

Figure 4.18 Typical MF/UF TFF set-up

pump on the permeate outlet which would normally be associated with UF rather than MF systems. This equipment will be used for a number of subsequent optimisation stages by small modifications to the pipe work. Each will be described with variations of Figure 4.18.

4.3.1.5 Sizing and scale-up for TFF UF

Small scale TFF tests are performed using reduced area formats of the production scale installations, either as a means to predict the membrane area requirement of the full scale system, or to perform scaled down studies (such as CIP) of an existing installation. Tests are performed using membrane holders or devices containing the same membrane and where possible, with the same device path length in order to achieve linear scaleability. Figure 4.19 shows a typical small scale laboratory test apparatus and Figures 4.20 to 4.22 show examples of large-scale TFF systems following scale-up.

Figure 4.19 Small scale TFF test system

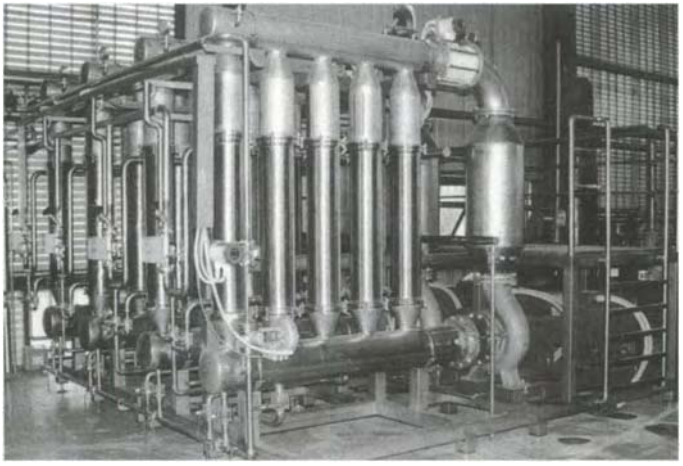

Figure 4.20 Large scale ceramic installation

The three primary objectives of TFF optimisation are to:

- Maximise product recovery;
- Maximise membrane permeability recovery;
- Maximise filtrate flux rate.

Product recovery is often the most important of these as this is driven by primary economic considerations. It is necessary to monitor the product quality and throughput during optimisation as well as the flux to establish the optimum conditions to maximise product recovery.

Adding more membrane area can reduce the processing time. It is also important to choose conditions that will allow the cassettes to be easily cleaned and reused.

Figure 4.21 Large scale cassette system

Figure 4.22 Large scale hollow fibre system

Firstly it is important to define the objectives and limitations of the process. Given the variety of applications discussed above, the important parameters will be similarly wide ranging. The following aspects will include those which are important to certain applications which should be considered prior to embarking on the evaluation:

- Volume of sample to be processed;

- Concentration and/or diafiltration of product or purification of high molecular weight or solid contaminants;
- Establish molecular weights of product and contaminants;
- Fluid concentration and viscosity;
- Target final concentration factor;
- Number of diafiltration volumes required;
- Required processing time;
- Pre-filtration requirements;
- Temperature sensitivity;
- Target product yield.

Consideration of these points will enable the appropriate technology to be selected.

4.3.1.6 Membrane selection

To concentrate in a molecule, the molecular weight must be determined and then a membrane selected that possesses a molecular weight cut off (MWCO) that is 20% to 30% of that value. For example, to concentrate a protein with a molecular weight of 100 kD, a membrane rated at 30 kD would be appropriate.

Conversely, when the objective is to pass (transmit) the protein of interest, then the MWCO should be 3–5 times greater than the molecular weight of the protein.

Next the membrane format should be determined. For example, screen cassettes should be chosen for filtered solutions to maximise the flux through the device. Suspended screen or open channel cassettes, hollow fibre or ceramics should be chosen when the process fluid contains suspended solids or the viscosity is too high to run a screen cassette. Cell harvest applications would usually require open channel or suspended screen options.

Further key considerations to selecting the most appropriate format will be the scale (process volumes) of the final process, compatibility of the device and membranes with the process fluid and cleaning and/or steam requirements.

Typically cassette technology is preferable for smaller scale processes, where the target final volumes require low system hold-up volumes. Open channel devices require higher crossflow rates per unit area, as compared to cassettes, in order to generate the required shear rates at the membrane surface. As a result, they tend to have higher hold-up volumes

per unit area, and this aspect in combination with the higher flow requirements can limit their applications in small biotech processes.

4.3.1.7 System optimisation

The primary characteristics of TFF membrane systems are flux and trans-membrane pressure (TMP). The flux is the volumetric permeate flow rate per unit area of membrane and is often measured in litres/m^2/h. It has become customary in the filtration industry to use the term (or abbreviation) LMH for flux. Derivation of the Flux *vs* TMP relationship for the system is the key to optimisation and sizing.

As discussed above, TFF enables separation of solids where contaminants exist at high concentrations. The technique also makes more complex separation stages feasible. In such cases the TFF processes include a number of steps that need to be performed. These can be broadly classified into three distinct areas: Preconditioning, Processing and Post conditioning. The flow schematic in Figure 4.23 below outlines these three areas and the key steps within each.

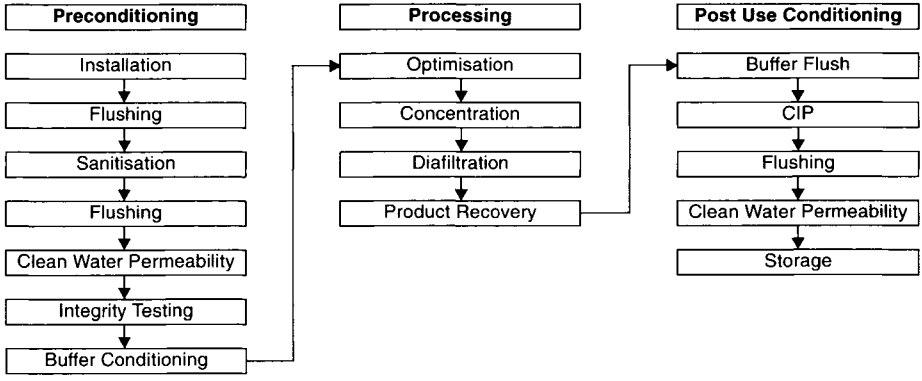

Figure 4.23 Typical TFF process steps

It is important to note that the 'Optimisation' step is performed once at the start of the process evaluation stage, to determine optimum operating parameters. Generally, optimisation tests are run to determine the TMP that gives the best flux for any given process and device combination (at a set crossflow rate). The logical process steps for the derivation of the flux *vs* TMP relationship is given in Figure 4.24. Also included in the figure is the schematic equipment layout as a variant of Figure 4.18 showing the return of the permeate to the feed tank. In this way a constant contaminant loading is presented to the membrane surface. The data collected would be tabulated as illustrated in Table 4.3.

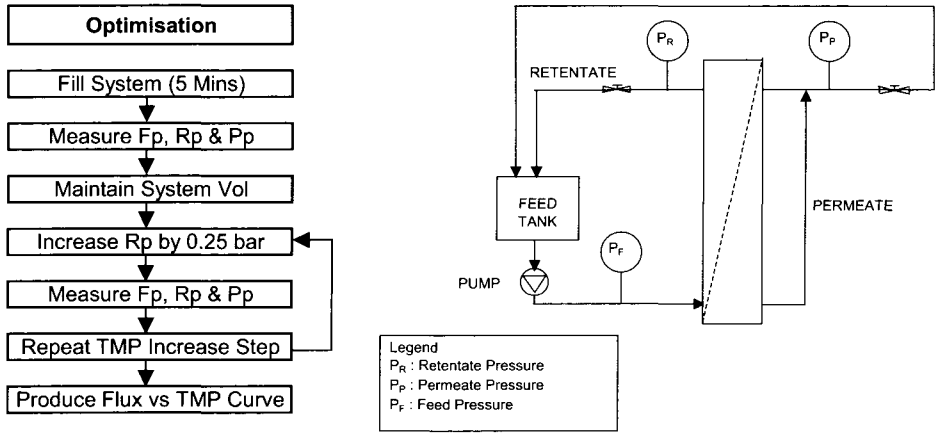

Figure 4.24 Process optimisation

Table 4.3 Typical optimisation values.

Feed pressure P_F, (bar)	*Retentate pressure P_R, (bar)*	*Permeate pressure P_P, (bar)*	*TMP (bar)*	*Retentate flow* (l/min)	*Permeate flow* (ml/min)	*Permeate flux* (lmh)
1.0	0	0	0.5	0.7	30	19.5
1.25	0.25	0	0.75	0.7	40	26
1.5	0.5	0	1	0.7	50	32.5
1.75	0.75	0	1.25	0.7	58	37.7
2.0	1	0	1.5	0.7	62	40.1
2.25	1.25	0	1.75	0.7	66	42.3

Other factors such as transmission of unwanted molecules and the number of diafiltration volumes required to achieve the desired purity, are also evaluated as necessary (see Section 4.3.1.9). It is also common to take samples of the feed and permeate streams during optimisation, and to analyse both to determine % transmission or % retention (depending upon where the product is). The Table shows an example of typical parameter values during the UF optimisation process (this data relates to tests performed with a 0.1 m^2 cassette).

During optimisation it is important to plot the relationship between flux and trans-membrane pressure as the test develops, such that the optimum TMP can be identified immediately from the graph. There is no gain in any aspect of performance by allowing the curve to plateau. Filtrate flux may decrease, percent transmission may decrease, operating costs will increase (assuming any full scale system is subsequently operated at the sub-optimal high TMP) and the membrane will be more difficult to clean.

The graphical plots in Figure 4.25 illustrate the typical curve generated during an actual optimisation run. After each setting plotting TMP against flux as discussed will develop the type of relationship shown in Figure 4.26.

At the point where the curve begins to plateau the optimum conditions for maximum flux have been achieved at that given cross flow velocity. If acceptable filtrate flux or transmission is not achieved, the membrane can be cleaned and the optimisation process repeated using a different cross flow velocity. Increasing cross flow will tend to give increased filtrate flux at the same TMP.

4.3.1.8 Determination of the maximum gel concentration (C_G)

When the optimum conditions are established as determined above, the system can be transferred into concentration mode by directing the permeate line away from the feed vessel to a separate permeate tank which may be graduated to facilitate volume measurement. This configuration is shown in Figure 4.27 as a further minor modification to the apparatus presented earlier.

At this stage the following data should be recorded:

- Time
- Pressures (P_F, P_R and P_P)
- Filtrate flow rate
- Volumes (feed and permeate).

Readings should be taken at regular intervals throughout the trial up to the end point of the test run, which is usually either the maximum concentration factor required or achievable.

This data should be used to plot filtrate flux *vs* log (volume concentration factor, VCF), Figure 4.28. The flux should decrease slowly and linearly with increasing concentration factor. If it does not then it may indicate that a component of the feed solution is changing (such as precipitation of proteins) or that the crossflow rate is not sufficient (causing excessive fouling of the membrane).

The curve can be extrapolated to the x-axis (flux = 0), the concentration factor where the curve crosses the x-axis is the maximum concentration achievable (C_G). C_G occurs when the bulk flow concentration is equal to the gel layer concentration. In reality this concentration is unachievable and the optimum point to end concentration and start diafiltration if required is at a lower concentration.

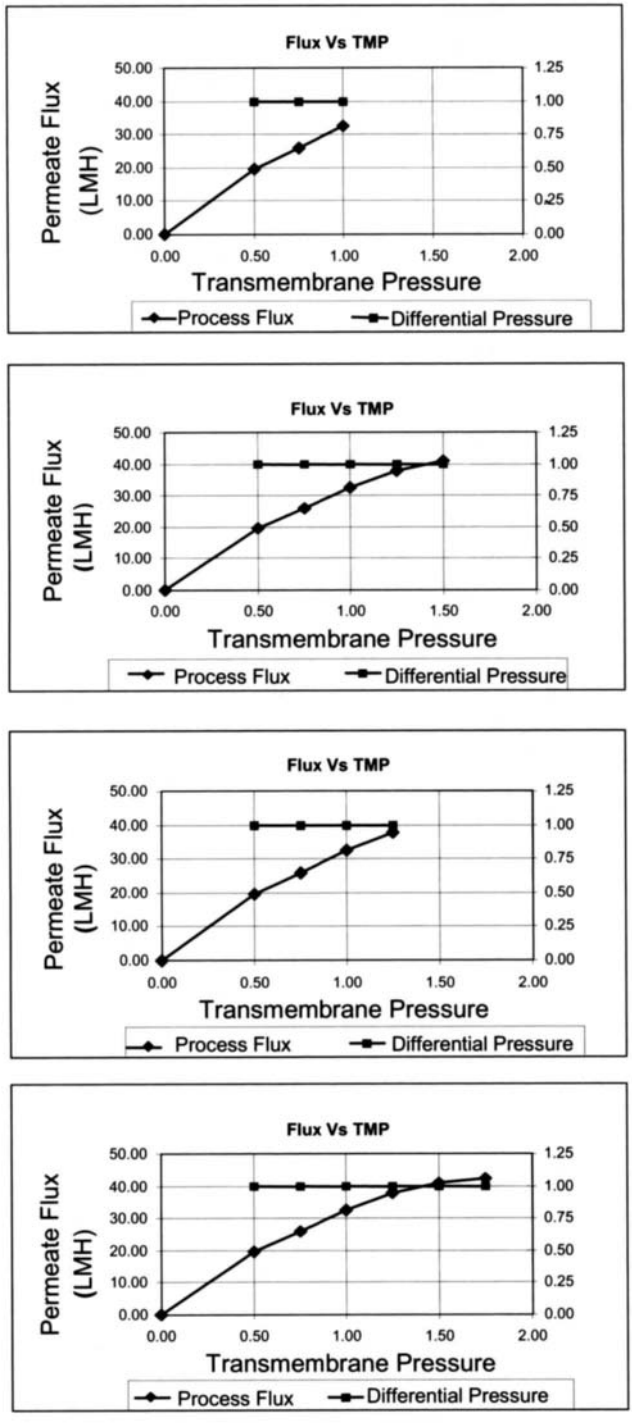

Figure 4.25 Optimisation plots

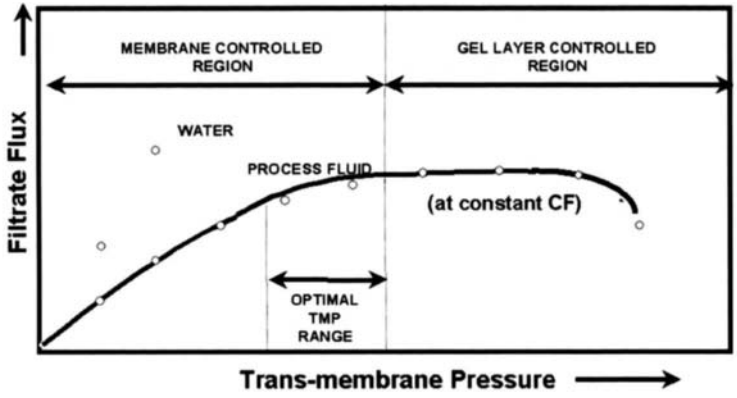

Figure 4.26 Classic overall optimisation plot

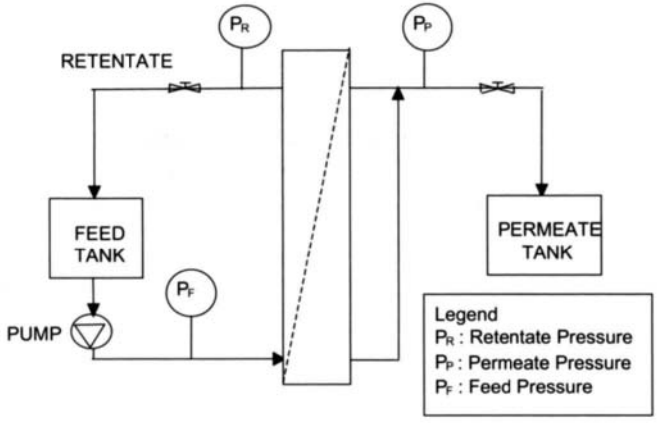

Figure 4.27 Concentration set-up

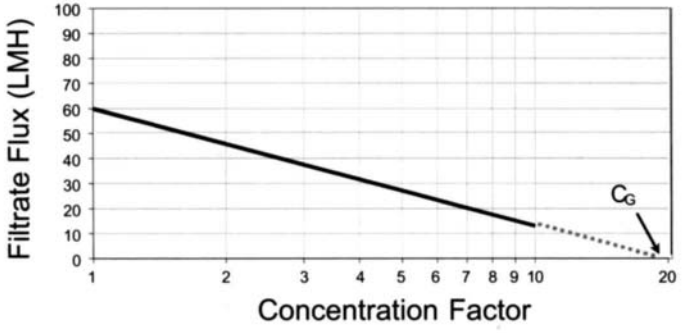

Figure 4.28 Classic plot of flux *vs* log (VCF)

4.3.1.9 Diafiltration

Diafiltration is a process used to reduce, remove or exchange salts and other small molecules from a process fluid. The process may be conducted continuously or discontinuously. In discontinuous diafiltration, the process fluid is diluted typically by a factor of 2 with the replacement fluid (buffer). It is then brought back to the starting level by concentration. This process may be repeated several times. Each subsequent step reduces the concentration of the smaller passing molecules (e.g. salts) on the upstream side of the TFF membrane device.

Continuous diafiltration requires addition of diafiltration solution at the same rate as the filtrate flow rate. The process volume remains constant. The addition of a diafiltration solution equal to the volume of the process fluid when the diafiltration started is referred to as one diafiltration volume (1 DV).

The number of diafiltration volumes required depends on how much salt or other small molecules need to be removed and the permeability of the molecule through the membrane (for either continuous or discontinuous diafiltration).

In a process that requires both concentration and diafiltration, diafiltration can be performed either before starting to concentrate, after completing concentration or part way through concentration and then completing the concentration after diafiltration.

Diafiltration, when a stage in a larger process operation, is often performed after other stages as the final volume is usually reduced. For example, if there is 10 litres of process fluid that must be concentrated 5 times and diafiltered 5 times, then if diafiltration is performed first, it will require the passing of 50 litres through the membrane but only 10 litres if diafiltration is performed after the concentration. However, the flux for each case will be different. The flux for the concentrated solution will be lower than for the starting process fluid. It is necessary to know what the fluxes are because it may take longer to diafilter the smaller volume. It should be noted that in some cases diafiltration may be done first to remove components that would cause the product to denature or precipitate when concentrated if they were not removed.

To determine the optimum concentration at which to perform diafiltration we first obtain the C_G value as discussed above. This is a theoretical maximum concentration for the fluid, and does not represent a working condition. It has been determined (Ng *et al*, 1976) that the effective optimum diafiltration concentration is given by the following equation;

$$C_D = \frac{C_G}{e} = \frac{C_G}{2.718} = 0.368C_G$$

For many applications this optimum concentration to carry out diafiltration is never reached but the value should be a target to approach.

4.3.1.10 Cleaning and storage

TFF devices and membranes can be reused many times. Manufacturers recommendations for chemicals, maximum concentrations and temperatures should be followed to recover the membranes. Typically caustic or acid solutions are used to remove organic and inorganic fouling materials from the surface of the separation media. These techniques are used widely in the high volume applications such as water processing and will be discussed later. For some devices, steam-in-place can also be considered for certain critical applications (such as virus production).

4.3.1.11 Scale-up

As with all scale-up procedures, it is vital to ensure that sample fluids are representative of the process fluids. However, with TFF systems it is important to consider the following testing conditions:

- The sample volume to membrane area ratio should be close to that expected for the final process
- Increase membrane area in direct proportion to the increase in sample volume
- Flow path lengths must be constant
- Increase volumetric flow rate in direct proportion to the increase in membrane area
- Operate the process at the TMP developed at smaller scale.

If these conditions are met, then the operating parameters developed and optimised for the process during the bench or pilot scale can be scaled directly to a full-scale operation. Filtrate flux and process time will match those determined in the small-scale test work. For devices such as hollow fibres and to some extent ceramics, where the path length tends to increase with increasing membrane area, further tests are normally required at intermediate scale to determine optimum operating conditions.

As in the case of the DFF membranes discussed earlier, to simplify scale-up, manufacturers often offer several different cassette formats. For example, the Centramate™ (area = 1ft^2) has a 17 cm path length.

This scales directly to the Centrasette™ (area = 5ft^2), which also has a path length of 17 cm.

To further demonstrate this scale-up principle, Figure 4.29 shows the Centramate™/Centrasette™ range of holders. It is possible to run trials with a 0.093 m^2 membrane area on the Centramate™ and then scale up directly to a maximum installed membrane area of 20 to 80 m^2 on the Centrasette™ and Centrastak™ holder.

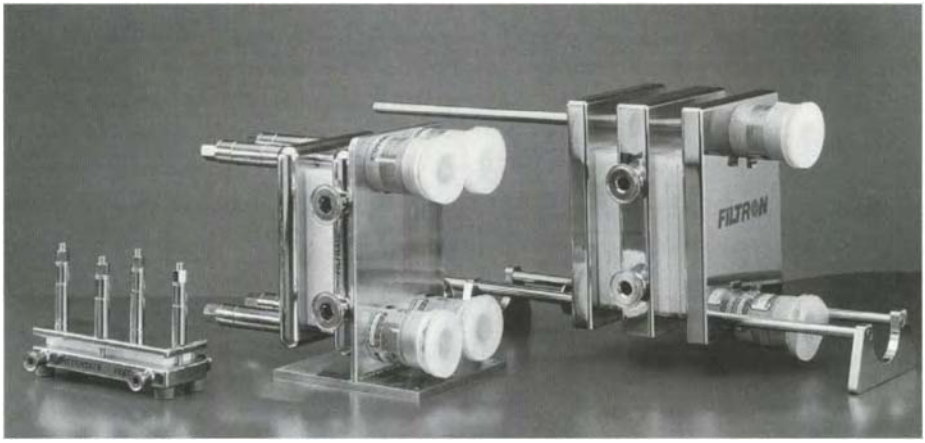

Figure 4.29 Centramate™/Centrasette™ C5/Centrasette™ C10 holders

Table 4.4 shows actual data obtained for three different volume stages of a scale-up evaluation. From the data, it can be seen that despite a membrane area increase from 0.1 m^2 to 8 m^2, the flux was directly scaleable, remaining constant at about 145 LMH.

Table 4.4 Scale-up volumes and membrane areas

Scale (l)	10	80	600
Flux (LMH)	146	144	144
Area (m^2)	0.1	1	8

4.3.1.12 Calculation of membrane surface area

Trial data as determined above can be used to calculate the required membrane area to process any volume of sample in a specified time.

$$J = \frac{V}{At}$$

where J is the filtrate flux (LMH), V is the volume filtered (litres), t is the filtration time (hours), and A is the filter area (m^2).

1. Perform optimisation on a small sample to determine average flux for each step. The example below is for a three stage process consisting of initial concentration → diafiltration → final concentration.

$$J_{step1} = \frac{V_{step1}}{t_1 A}$$

$$J_{step2} = \frac{V_{step2}}{t_2 A}$$

$$J_{step3} = \frac{V_{step3}}{t_3 A}$$

2. Calculate membrane area required based on the desired processing time:

$$A = \frac{V_i}{t_i J}$$

$$A = \frac{1}{t_T}\left(\frac{V_{step1}}{J_{step1}} + \frac{V_{step2}}{J_{step2}} + \frac{V_{step3}}{J_{step3}}\right)$$

$$t_T = t_1 + t_2 + t_3$$

Example Determine the membrane area required to process a 100 litre batch in 4 hours. The process consists of an initial concentration step (2×), followed by diafiltration (5 DVs) and then a final concentration step (2.5×) to reach the desired final VCF of 5×. Small scale tests were performed on a sample of material to determine the average fluxes below:

Small-scale test data:

Step 1 Concentrate 2× from 100 litres to 50 litres:
Filtrate Volume = 50 litres
Flux: 71 LMH

Step 2 Diafilter using 5 DV, volume increases to 50 × 5 litres:
Filtrate Volume = 250 litres
Flux: 64 LMH

Step 3 Concentrate 2.5× from 50 litres to 20 litres:

Filtrate Volume = 30 litres

Flux: 38 LMH

$$A = \frac{1}{4}\left(\frac{50}{71} + \frac{250}{64} + \frac{30}{38}\right) = 1.35 \text{ m}^2$$

Therefore the membrane area required is 1.35 m^2.

4.3.2 Industrial MF

Water-based industrial wash fluids containing detergents are used to clean components before assembly in many industries including the automotive and aerospace industries. With fresh water and waste costs increasing year by year, recycling fluids for reuse is an important process to save and reduce costs while improving the wash cleaning performance.

Tangential flow MF systems are used to recycle the water-based wash fluid by removing the contaminants from the fluid without stripping the original detergent from the process. The contaminants in the process are oil, including free and emulsified oil, bacteria, fungus and suspended solids; all these can adversely affect the wash performance.

4.3.2.1 System scale-up

The scaling up process aims to allow accurate sizing of a full-scale system to achieve control of the contaminant level in the wash fluid. The consequences of errors or inaccurate scale-up could be catastrophic. An undersized system could exhibit lower than expected permeate fluxes, high contaminant levels or increased waste fluid volumes while an oversized system would be too expensive resulting in poor cost payback.

Therefore small-scale trials that can be accurately scaled are required to assess the application and size a production unit for a water-based industrial wash system.

Initial laboratory feasibility assessment should be undertaken to determine if the fluid can be processed at a reasonable permeate flux to allow a system to be sized and evaluated for payback analysis to be completed.

The laboratory tests will also assess if any interactions exist between additives in the cleaning fluids and the microfiltration membranes. The cleaning additives should pass through the membrane and the cleaning

fluids properties should be unaffected by the tangential flow filtration system. The fluid properties that should be checked are:

- Fluid pH
- Detergent/additive concentration
- The corrosion protection properties of the fluid on components
- The foaming properties of the fluid.

The optimum membrane rating should be determined as the use of finer rated membranes such as UF can result in depletion of the surface-active additives in some cases, by either physical or chemical means. For example, anti-foams, these can be silicone, calcium or oil-based or other surface-active agents, can affect the filtration mechanisms dramatically.

Preparation for testing should include a detailed site assessment on the washes current performance and fluid usage. Then the type and volume of sample should be established. Often, a process will be sensitive to time or other operational factors. In this case a sample of fluid from the wash should be taken when the system is operational rather than static. Again, the laboratory test results will only be as accurate as the sample taken; this should be as representative as possible of the fluid quality from the wash.

The fluid, being water-based, must be transported quickly to ensure extremes of temperature do not affect the fluid quality. For example, in hot ambient conditions ~30°C bacteria and fungi can rapidly multiply, affecting the sample properties. Conversely, very cold conditions could cause the sample to freeze thereby destabilising oil emulsions.

4.3.2.2 Laboratory test equipment

The laboratory test apparatus consists of the equipment arranged in similar fashion to that described in above. The schematic layout is again broadly the same as that presented in Figure 4.18 while the apparatus is illustrated photographically in Figure 4.30.

The pump recirculates flow of the sample across the MF membrane surface, usually delivering a velocity of between 1 and 6 m s^{-1}. Typically, 1 to 3 m s^{-1} would be used for polymeric membranes and 3 to 6 m s^{-1} for inorganic or ceramic membranes.

Flow and pressure measurement can be manual at this stage of testing using measuring cylinder and stopwatch to measure permeate flows and calibrated gauges to determine pressures.

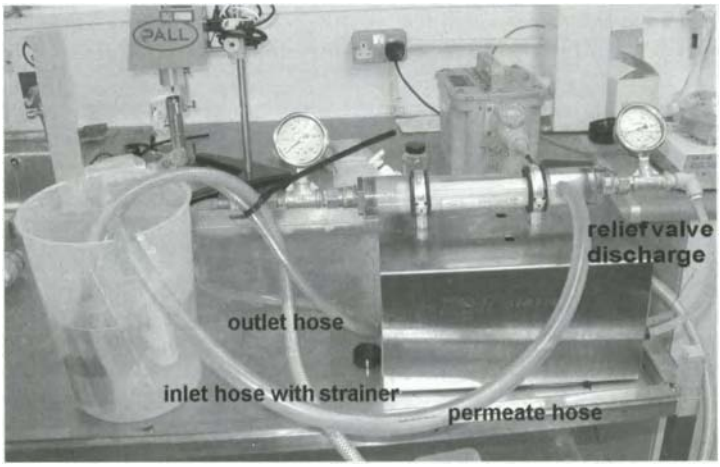

Figure 4.30 Laboratory test apparatus

The appropriate MF membrane should be chosen for the application. In the case of wash fluids, membranes with ratings in the 0.05 μm to 0.2 μm range were used. The membrane material should be compatible with the wash fluid and temperature range. Long-term compatibility tests may need to be carried out to verify this chemical stability.

4.3.2.3 Test procedure

The trial membrane should be flushed free of any preservative fluid with filtered, deionised water.

The clean water permeate flow should then be measured at various trans-membrane pressure values. The flux is then determined from a measurement of the permeate flow rate and the effective area of the upstream surface of the membrane. For the apparatus shown in Figures 4.18 and 4.30, the TMP is determined from the following expression:

$$\text{TMP} = \frac{P_F + P_R}{2} - P_P$$

TMP is usually expressed in bar.

Flux vs TMP To generate flux *vs* TMP data, the apparatus should be filled with the sample and recirculated around the retentate side of the membrane with the permeate ports closed. The sample should be pre-filtered (≤100 micron) to avoid superficial blockage or damage, as this would mask the valuable and sensitive data regarding the properties of the dynamic filter layer. The temperature should be controlled at the operating temperature of the full-scale process. Record the starting volume and any volumes added or bled later.

An initial crossflow velocity of 1 m s^{-1} should be set (corresponding to a typical average TMP of 0.2 to 0.5 bar), with all permeate and concentrate being recycled back to the feed tank. The test equipment should be maintained at a constant operating temperature.

Increase the TMP every 45 to 60 minutes, by 0.5 bar increments, and record flux versus TMP data. Observe if the flux increases with TMP. If it does not, this may indicate the formation of a gel polarisation layer or pore fouling.

Flux vs Time Run at a constant (1×) concentration by re-circulating permeate to the feed tank for 60 minutes at the optimum TMP determined above. Note any decrease in flux in 10 or 15 minute intervals to generate flux *vs* time data.

A TFF membrane is regenerable by reverse filtration. It may be possible to assess this during the initial laboratory evaluations. This being the case, a reverse permeate backwash should be carried out at a pressure >0.5 bar above P_F to assess any improvement in permeate flux.

Flux vs Concentration Concentrate the fluid in the feed tank to the desired final concentration, usually a maximum of 40% oil, holding all other conditions constant, and recording flux at various concentrations. This is achieved by retaining the permeate in a separate container rather than returning it to the feed tank.

Collect permeate and retentate samples at various concentrations to measure permeate quality.

When the desired final concentration has been obtained, the test can be progressed in continuous mode by setting permeate and concentrate control valves to give the required VCF. Fresh feed is then continuously added to the feed tank.

When testing is complete, drain the system, flush with clean water, and clean the system with the appropriate chemicals until the clean water flux is stabilised at the same conditions. The cleaning chemical used for this application would typically be an alkaline based detergent, increasing the temperature usually improves results. The stronger the alkalinity, and so higher pH, of the cleaning fluid also usually has a positive effect on cleaning. The recovered permeate flux after laboratory trials and chemical cleaning should be close to the original flux measured under the same operating conditions.

4.3.2.4 Results and significance of laboratory scale trials

As part of the laboratory evaluation, the permeate quality should be assessed for contaminant removal and detergent additives levels. Any possible fluid / chemical problems should have been highlighted. The permeate flux will be known at normal and concentrated levels of contaminants from the sample measured in litres/unit area of membrane per hour, at a known temperature. The temperature factor is very important as a 1°C change can affect the permeate flux by 3%. Figure 4.31 below provides a temperature correction factor (TCF). The recovered permeate flux after cleaning with different cleaning chemicals may also have been assessed.

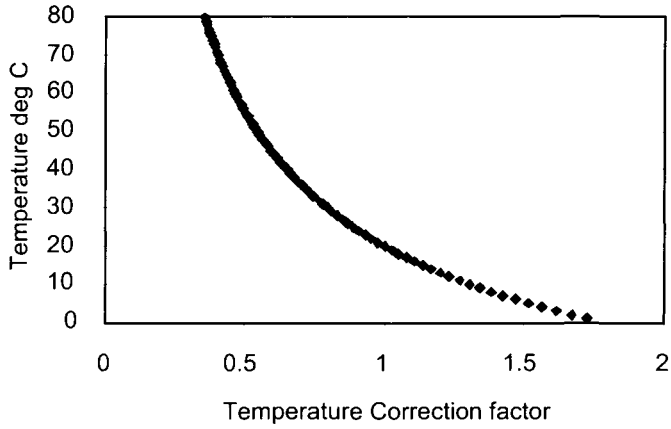

Figure 4.31 Temperature correction factor for permeate flows. The graph compares the temperature to 20°C to give the correction factor

However, many industrial applications tend to exhibit a variety of inlet conditions. This may result from many factors outside of the immediate control of the operators. Under these circumstances, the preliminary laboratory test investigation will give an indication of sizing for a filtration system unit to see if the application is viable. These results can only be scaled up to give an indication of how a full sized system would perform. The required site membrane area can be only be estimated. As with certain biopharmaceutical applications it may be prudent to undertake intermediate pilot scale trials.

For example operating a 0.05 m^2 membrane over two days of laboratory trials cannot be confidently scaled up to a 10 m^2 membrane area system that will operate for years. The scaling factor would be 200 fold on two days' work, which for certain industrial applications would represent a significant commercial risk and site based pilot scale to scale-up trials should be considered.

4.3.2.5 Pilot trials

The purpose of the pilot trial is to further investigate the application over a longer time period with a larger membrane area to more accurately size a full MF tangential flow filtration system. To ensure long-term trouble free operation of a system the pilot trial will help to give accurate data that can be scaled up. Where scale-up is significant, the laboratory tests can be seen as a feasibility study and site piloting will then give accurate sizing data.

Further advantages of a site trial over the laboratory work are that the trial will be carried out under the normal process conditions with constant ingression of contaminants and any temperature variations will be seen.

The equipment used to trial at site should ideally be in the form of a fully automated test rig. Telemetry would be advantageous, as this would enable the rig to be set up and monitored remotely over a period of weeks. The use of manual laboratory equipment will often be too time consuming and expensive to operate on site. For example, most MF systems will typically require a reverse pulse backwash every 15 minutes for long-term permeate flux maintenance.

Pilot testing is necessary to prepare the system design and configuration, to make propositions for system size and to evaluate economic payback factors.

The membrane type, crossflow velocity and rating should have been chosen from the laboratory bench test work. The trial rig system operation should be the same as the full size system with all the flux maintenance regimes operating, backwashing, chemical cleaning, and if used, reverse crossflow.

Pilot system components Each type of pilot unit can have different components, which should be chosen according to the application to be tested. An illustration of a site trial unit for industrial TFF evaluations is given in Figure 4.32 with a flow schematic diagram given in Figure 4.33. It will be noted that the design of the site pilot unit is necessarily very much more complex than the simple laboratory bench scale units discussed previously. The pilot rig often resembles full-scale equipment in that it will have temperature control, instrumentation and automation consistent with extended operating regimes.

Membrane filtration module: Modules with a membrane filtration surface area of between 0.2 and 3.0 m^2 are usually sufficient for scale pilot testing.

Figure 4.32 Pilot scale test equipment

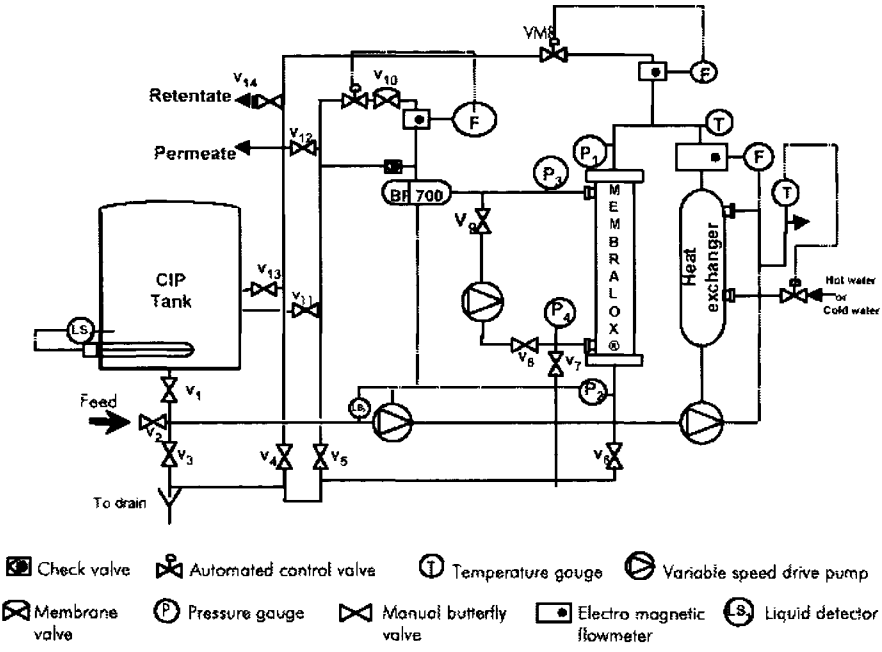

Figure 4.33 Pilot scale flow schematic

Feed and re-circulation pump: Typically, feed pumps are low flow, high pressure pumps while re-circulation pumps are high flow, low pressure pumps. The re-circulation pump in the tangential flow filtration system should provide the necessary velocity to reduce fouling, with enough pressure head to overcome the channel/fibre pressure drop. Pumps should be capable of generating the desired range of pressures and flow rates to be tested. Variable speed drives provide an added dimension of flexibility to the pilot test process.

Flowmeters: The main types of flow meters used in pilot units are electro-magnetic. These must be precise, reliable and versatile.

Flow control valves and level switches: Flow control valves are especially used for pilot tests intended to run continuously over long periods of time. At a minimum, these should allow the control of permeate flow and bleed flow (concentrate leaving the system). Level switches are needed to prevent pump failure if the tank level unexpectedly drops too low or to help maintain a certain level in the tank.

Temperature and pressure gauges: Accurate and reliable measurement of temperature and pressure values is very important in pilot testing as these two parameters may have a significant influence on the filtration performance. Glycerine-filled pressure gauges are recommended for better accuracy. Direct recording analogue gauges must be capable of accurately reading within 0.1 bar. Pressure transducers have high accuracy readings and are readily adapted to data logging.

Back pulse: The back pulse set up is usually arranged to generate a pulse 0.5 bar higher than the system trans-membrane pressure, at 10 to 30 minute intervals to ensure effective backwashing.

Feed tank: The tank should be of sufficient volume to allow for the whole range of concentrations to be evaluated or to provide sufficient residence times in situations where it is also used as a reaction vessel. The materials of construction of the feed tank and its components, usually stainless steel, must be compatible with the operating temperature, pressure and chemical nature of the process sample. The tank should have a lid and an anti-vortex device if necessary, and a sloping or conical bottom to allow more complete drainage.

Heat exchanger: A multi-tube heat exchanger is generally placed in the filtration loop to maintain the required temperature over the entire run time and during in-place cleaning/sterilizing. Small volume recirculating

systems in particular will heat up significantly with processing time. As discussed above, it is vital that the tests are undertaken at the system temperature and reflect any temperature excursions that naturally exist.

Pre-filtration: The pre-filtration of the process fluid should be set at a maximum rating of one tenth of the diameter of the microfiltration fibre or channel tube size. The other factor when considering pre-filtering for the system is the contamination sensitivity of pumps and valves used in the system which may be damaged by erosion caused by solid contaminants carried in the process fluid.

Test rig set up: The test rig should be connected up to the process line and run for a minimum of 500 hours to prove the long-term flux stability. The required volume concentration factor of the contaminants should be achieved on each batch of fluid processed.

When working with MF, it is often preferable to work at constant permeate flux, allowing TMP to increase due to membrane fouling (TMP < 2 bar). Under these conditions, back pulse cleaning can be very effective in sustaining the permeate flux.

4.3.2.6 Case study

An example of how a scale up trial was used to size a tangential flow filtration system is detailed below.

The application was an automotive industrial wash used to clean engine blocks from an internal combustion engine before assembly. Poor wash performance could result in parts being inadequately cleaned affecting production. Results from a pilot site trial are shown in Figure 4.34.

The minimum permeate flux of 42 LMH was achieved with a membrane area of 0.5 m^2 for the pilot site trial with a fluid volume concentration factor of 35%. The required batch concentration was typically achieved

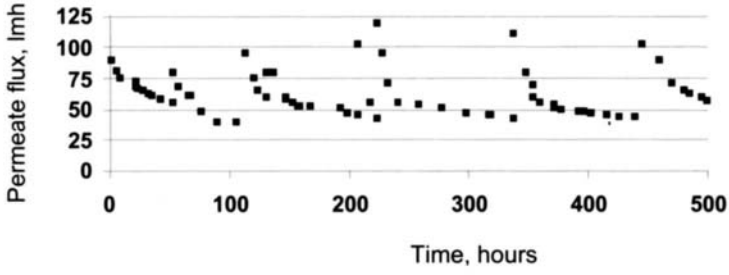

Figure 4.34 Flux maintenance. Microfiltration permeate flux from site trial of an industrial wash

in 100 hours of processing. Chemical cleaning after the trial for 8 hours restored the clean water permeate flux to 94% value of the new membrane, which was considered an acceptable recovery.

Conclusions of site trials: The trial results demonstrate that a steady permeate flux was achieved over 500 hours of operation. The peaks in permeate flows are due to the batch concentration with the lowest permeate flux being achieved at the highest concentration, which would be expected.

This trial demonstrates that a steady permeate flux has been achieved at a minimum of 42 LMH. This would allow confident selection of the most appropriate membrane, accurately scaled up to achieve the required permeate flux to maintain the wash at the low contaminant level required for acceptable component cleanliness.

Calculation example: The example wash had 10 m^3 capacity tank and the fluid required changing every two weeks, as oil level in the wash fluid reached 1%, which was known to affect component cleanliness.

A tangential flow filtration system that produced a clean permeate flow of 400 litres/hour or 9,600 litres/day would maintain the wash at maximum of 0.2% oil contamination while at full production and under normal ingression rates. Therefore the required permeate flow for the wash is 400 litres/hour of contaminant free wash fluid.

Scaling up the permeate flux from pilot site trials:

Pilot minimum permeate flux = 42 LMH

As before,

$$A = \frac{V}{Jt}$$

Therefore

$$A = \frac{400}{42} = 9.5 \text{ m}^2$$

The tangential flow membrane area = 9.5 m^2.

This represents a scale-up factor of 20 times from the pilot testing to a full size system, which is considered acceptable after operating for 500 hours.

The above system was installed on an industrial wash machine with a membrane area of 10 m^2 and gave an average permeate flux of 45 to 60 LMH over one year of operation.

Chemical cleaning of the membrane was carried out monthly to help maintain the flux. The unit successfully maintained the wash fluid at low levels of oil contamination and the fluid was kept in service for one year from the original 2 weeks fluid life that had been experienced before installing the system.

4.4 TFF/DFF applications

4.4.1 Water processing

As we have seen, membrane systems can be operated in DFF and TFF modes. There are no specific rules to follow when deciding which flow method should be used for which configuration of membrane. This is better defined by the characteristics of the fluid in question. Indeed, many hollow fibre membranes are used in DFF and vice versa.

The use of membrane systems has experienced exponential growth over the last 5 years due to the ability to deliver drinking water and wastewater within compliance limits. With a modular design and sophisticated automation, membrane plants are now being built with safety, flexibility and minimal operator intervention. Another driving force for the popularity of membranes is that their costs have never been lower, spurred by increased competition, more efficient process operation and manufacturing capacity. The single factor that dictates the membrane plant size or the number of membranes is the membrane's ability to handle fouling. A well-designed membrane, one that has high porosity and permeability, will inherently provide better flux and economics. However, to make use of those physical properties, membrane fouling must be controlled in a practical and economical manner. Pre-treatment of the feed water usually has a quantifiable benefit on the sizing of membrane systems but this obviously has to be offset against the associated additional operating cost.

Another performance improvement is the effective use of "enhanced flux maintenance" (EFM) strategies that can effectively increase the membrane flux, thereby reducing the cost of membranes installed quite significantly. Keeping the membranes clean as often and as long as possible allows the most efficient use of the membrane area and porosity.

4.4.1.1 Water quality analysis

An assessment of the required equipment and treatment options of a source water normally begins with the review of a water quality analysis. Historical data, if available, provide the best indication of seasonal changes and variations. Unfortunately, this information is not always available, so it may be necessary to procure a sample and obtain an analysis from a certified laboratory. Water quality results may also be used to determine the need for on site or laboratory pilot testing. It should be noted, however, that a single sample, while valuable, provides only a limited snapshot of the actual water constituents. Parameters that are often included in an analysis of drinking water include the following:

- Total Organic Carbon (TOC)
- Turbidity
- Total Suspended Solids (TSS)
- Iron
- Manganese
- Hardness
- UV_{254}.

Other constituents may need to be measured depending upon the water source, finished water requirements, and national and local regulatory standards.

4.4.1.2 Pilot testing

Due to variations in water quality and type, it is often advisable to conduct small-scale trials, or pilot tests, on the source. This evaluation typically lasts 2–6 months and is intended to obtain continuous data that is used to effectively size and cost the full-scale membrane system. The trial usually consists of an optimisation phase (2–4 weeks) followed by one or more design runs (4 weeks). Pilot tests are used to accomplish the following:

- Demonstrate successful operation with the specific water source
- Confirm finished water quality
- Optimise system performance and costs
- Determine full-scale design criteria –

  Flux

  Cycle duration

Operational set points

Flux maintenance protocols

- Assess power and chemical consumption
- Provide customer training and orientation.

Pilot Equipment: An automated single module test rig is typically used for pilot evaluation. The system is equipped with a single hollow fibre MF module. The physical characteristics of a PVDF hollow fibre membrane are described in Table 4.5. A schematic of the MF system is shown in Figure 4.35 with a photograph included below in Figure 4.36. There are four basic modes of operation for the membrane unit:

Forward filtration: The feed pump draws water from the feed tank and pumps through the membrane filter directly to the top of the module.

Table 4.5 Typical MF module specifications.

Outside membrane area	50 m^2
Module length	2 metres
Module diameter	6 inches (152 mm)
Nominal pore size	0.1 μm
Maximum pore size	N/A
Membrane material	PVDF
Flow direction	Outside-in
Temperature operating range	Freezing point - 40°C
Feed water turbidity operating limit	0–500 NTU

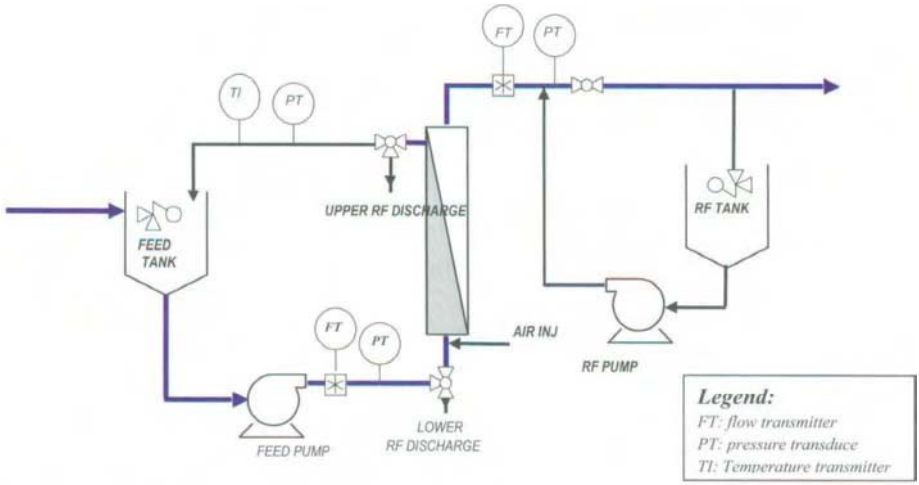

Figure 4.35 Pilot test rig schematic

Figure 4.36 Pilot test rig

Part of this flow can be recirculated to the feed tank, a process known as excess recirculation.

Simultaneous air scrubbing and reverse filtration (SASRF): SASRF is a frequent, short-duration hydraulic cleaning of the membrane to maintain optimal performance. During SASRF, air is injected into the filter module while simultaneously pumping filtrate from a storage tank into the top of the module. The combined water-air flow creates strong turbulent and shearing forces to dislodge deposits on the membrane surface. The waste is discharged through the upper discharge port of the module. A reverse flush (RF) is used after the SASRF to flush out the solids dislodged during air scrubbing.

Forward flush (FL): FL is another form of hydraulic cleaning for the membrane that follows a SASRF. The feed pump draws water stored in the feed tank and pumps the water through the membrane filter in the

same direction as that during forward filtration. The waste is discharged through the upper discharge port, flushing out the solids dislodged during the air scubbing.

Enhanced flux maintenance (EFM): EFM is a daily cleaning of membranes to maintain optimal performance. The EFM process involves circulation of a chemical cleaning solution on the feed side of the membrane at an elevated temperature for 30 minutes before returning the unit to filtration mode.

An on-board programmable logic controller (PLC) and computer controls system operation and includes supervisory control/data acquisition (SCADA) capability for real-time monitoring and data logging. The system can also be monitored and controlled remotely via modem connection.

4.4.1.3 Test operation, results and discussion

Pressurized membrane systems may be operated in an outside-in or inside-out mode with periodic regeneration of the membrane by flux maintenance protocols. In standard operation, when the TMP reaches a terminal limit, (typically 3 bar for an outside-in module or <1 bar for a vacuum membrane), the membrane is chemically cleaned in place (CIP) using a two step caustic – acid procedure. Operational parameters are often selected based upon feed water quality, removal criteria, and economic considerations.

Data that are recorded during pilot trials include, but are not limited to, the following:

- Temperature
- Flow rates (feed, excess recirculation, filtrate)
- TMP
- Turbidity (feed and filtrate)
- Particle counts (feed and filtrate) – optional
- Periodic water quality – TOC, Fe, Mn, etc.

During the initial optimization phase, system parameters are adjusted in order to determine the optimal process conditions required to achieve stable operation and meet project goals. At this point, pretreatment may be added or modified and additional water quality samples may be necessary to confirm filtrate characteristics.

After sufficient information is generated, the system is cleaned in place

and the design run started using the parameters defined in the initial phase. Typical process data are given in Figure 4.37. Note that, in this case, the membrane process yielded very stable performance with respect to TMP buildup. The daily TMP increase of 0.3 bar/day was negated with the daily EFM procedure. This resulted in peak daily TMP values averaging less than 1 bar over the entire cycle with no net daily increase, thus the general slope of the curve over the 30-day period is near zero. This suggests the potential for elongated cycles beyond 30-days or the ability to run at higher flux rates. A standard CIP procedure is normally performed following the cycle in preparation for the next design cycle or shipment to the next site.

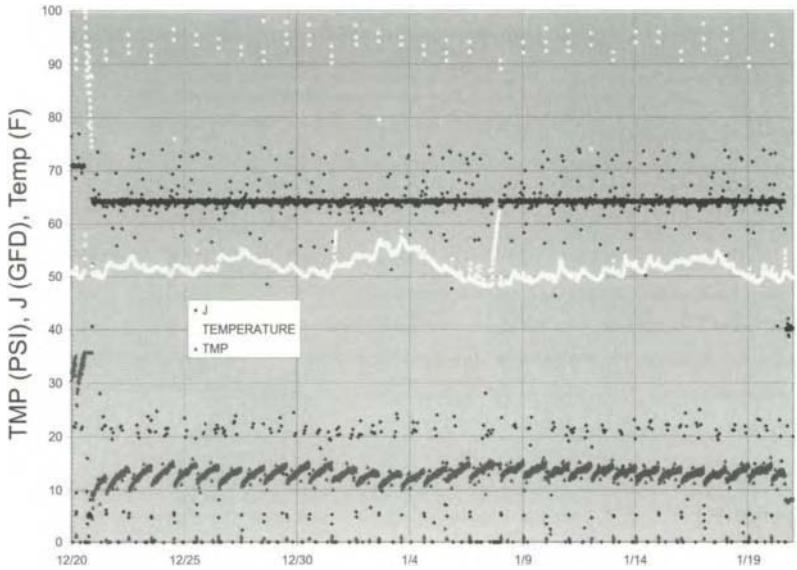

Figure 4.37 Run design process data for test rig

Turbidity: Turbidimeters are used extensively in the water industry. Information obtained with these instruments is routinely used on production systems to monitor the effectiveness of the water treatment process. Typically, white light turbidimeters have been used but more recently laser-based devices have increased sensitivity and are becoming more widely used. The pilot test rig is equipped with turbidimeters and recorded feed and filtrate turbidity results are presented in Figure 4.38. Note that the filtrate turbidity, through the MF membrane, remains stable despite variations in feed conditions.

Particle Counts: On-line particle counters have been gaining popularity in recent years. These instruments are intended to be used as

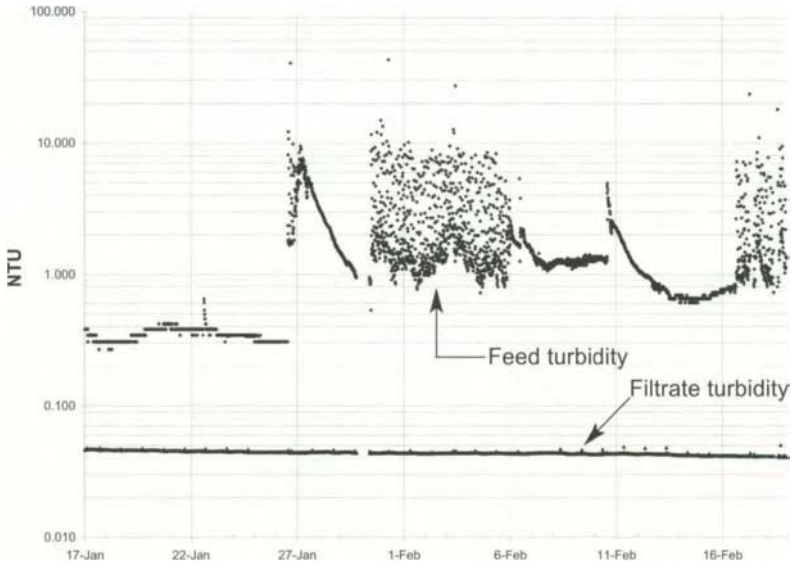

Figure 4.38 Performance data – turbidity

an "indirect" integrity test for membrane systems. However, this method does not provide the same sensitivity level for detecting a membrane breach as would a direct integrity test, such as pressure hold. In addition, the units must be properly set up and isolated during flux maintenance to prevent air bubbles and downstream contamination from creating erroneous results.

4.4.1.4 Integrity testing

Regulatory agencies typically require that membrane systems undergo direct integrity testing and continuous integrity monitoring for compliance. In general, direct integrity testing is defined as a "physical test" that is able to detect and isolate integrity breach. Based upon recent US guidance documents, a direct integrity testing must be able to identify a defect equal or less than 3 μm.

To meet the integrity testing (IT) requirements on pilot and full scale systems, a pressure-hold test is typically performed at specified frequencies as well as at the beginning of the test and at the conclusion of each run after cleaning in place. On pressure modules, one method consists of draining the filter module and pressurizing the feed side of the wetted membrane while exposing the filtrate side to atmosphere.

Integrity Test Resolution: The test pressure of >14 psi (960 mbar) is sufficient to detect a 3 μm breach based on Lin and Schaefer (2004). Using the most conservative value for this analysis (i.e. the lowest test

pressure) it can be determined (using Cantor's equation below) that the equivalent diameter of the smallest defect is 1.67 μm, which is considerably lower than the 3 μm defect for *Cryptosporidium* oocysts and the 5 μm defects for *Giardia lamblia* cysts. This situation implies that the minimum pressure to satisfy the 3 μm criterion is 13.9 psi.

$$d = \frac{4\gamma \cos\theta}{\Delta P}$$

where:

ΔP = Differential pressure applied across the membrane. The test pressure must be corrected for backpressure and any static head contribution.

γ = Surface tension at the air-liquid interface.

θ = Liquid-membrane contact angle.

d = Equivalent diameter of the smallest defect included in the test.

For the theoretical case of a perfectly hydrophilic membrane, the contact angle is zero, and assuming water at 25°C (surface tension 72 dynes/cm), the equation simplifies with d in micrometers and $\Delta P_{testmax}$ in kPa:

$$d = \frac{288}{\Delta P_{testmax}}$$

4.4.1.5 Jar testing

Jar testing is a simple and rapid bench-scale tool used to examine the effect of chemical treatment alternatives coagulants on a specific water source. This technique has been used to predict pH, oxidant dose, and detention time for iron, manganese and arsenic oxidation and determine the effects of oxidants and powdered activated carbon to remove colour and TOC.

Jar testing is usually conducted with a multiple-station stirring batch reactor having identical mixing conditions. Important factors affecting chemical reaction such as dosages, pH, and mixing intensity can be conveniently studied with this equipment.

Note that suitable safety precautions should be taken for the process fluids and materials as well as for the operation of the equipment and procedures.

4.4.1.6 Data scale up

In these cases, the scale-up factor can be very significant, and certainly considerably greater than the levels that would be unacceptable in other areas of piloting. The important feature here is that a full size module is used. It has the same path length (as called for as a desirable prerequisite in the forgoing TFF section) and is run at the same flux as the final system will. Even the cleaning regimes will accurately reflect the full-scale operation.

The pilot data can therefore be directly used for full-scale plant design. A computer program is typically used to process the pilot parameters and account for offline events such as flux maintenance and valve cycling.

Depending on the specific application, adjustments to the design may be necessary to comply with project goals such as minimizing waste. Other factors that must be considered include the following:

- Physical space
- Building type – new or existing structure
- Economic considerations (power, chemicals, operating cost, labour)
- Redundancy requirements
- Environmental factors (water quality changes, temperature)
- Regulatory issues (chemical disposal).

4.4.1.7 Case study

At one US site, groundwater had been used as the primary source of drinking water for many years. Over time, contamination by iron, manganese, arsenic and nitrates had increased. The deterioration in water quality placed an undesirable operational burden on the operators at the plant, which used conventional greensand pressure filters to remove inorganic contaminants.

Use of Surface Water To overcome these issues river water was investigated as an alternative source. The results of the investigation determined that the use of membrane technology to treat river water offered an absolute barrier to Giardia and Cryptosporidium, two key contaminants of concern in the use of surface water for drinking. The sub-micron absolute rating of the hollow fibre MF membranes also maintained effluent turbidity in the range of 0.02–0.03 NTU without any chemical addition or other pre-treatment, which may have been a requirement in a treatment scheme for the groundwater, or conventional surface water treatment.

An MF system was sized from data generated during pilot testing performed for a 75,000 MLD plant that is being constructed down river.

The modular nature of a pressure-driven MF system allows confident scaling from pilot data to full-scale system without correction factors. The pressure driven system also assures continuous operation, even during periods of increased differential pressure, which are caused by spikes in turbidity typically observed in surface water during weather related events.

The MF system was installed at the plant and it has been treating water at 580 to 1000 litres per minute with no chemical pre-treatment. An illustration of the installation is presented in Figure 4.39.

Figure 4.39 A modular MF system

The system automatically performs membrane integrity checks every day, and maintains water throughput using a reverse flow/air scrub procedure. An enhanced flux maintenance procedure using sodium hypochlorite has been performed every 14 days to prevent organic fouling. A complete CIP procedure with sodium hydroxide, sodium hypochlorite and food grade citric acid has been performed occasionally as preventative maintenance for the MF modules. The

chemical resistance of the PVDF hollow fibres in the system ensures that the membrane modules can resist fouling from organics, metals, and other contaminants, as well as chemical exposure.

The modular nature of the pressurized water treatment system allows capacity expansion by incremental addition of membrane modules or a second unit to work in tandem with the first. The existing and expanded units can share complementary equipment used for CIP chemical storage and transfer, and a CIP waste neutralization system.

Nomenclature and abbreviations

A	Membrane Area
C_D	Optimum Diafiltration Concentration
C_G	Gel Concentration
CIP	Clean in Place
DFF	Direct Flow Filtration
EFM	Enhanced Flux Maintenance
FL	Forward Flush
J	Permeate Flux
kD	Kilo Daltons
LMH	Abbreviation of flux litre/m^2/h
MF	Microfiltration
MLD	Mega Litres per Day
MWCO	Molecular Weight Cut Off
NTU	Nephelometric Turbidity Units
PAN	Polyacylonitrile
PE	Polyethylene
PLC	Programmable Logic Contrller
P_F	Feed pressure
P_P	Permeate pressure
P_R	Retentate Pressure
PS	Polysulphone
PVDF	Polyvinylidinedifluoride

RF	Reverse Filtration
SASRF	Simultaneous Air Scrubbing and RF
SCADA	Supervisory Control/Data Acquisition
TCF	Temperature Correction Factor
TFF	Tangential Flow Filtration
TMP	Transmembrane Pressure
TOC	Total Organic Carbon
TSS	Total Suspended Solids
UF	Ultrafitration
UV_{254}	Ultraviolet Absorption at 254 nm
V_{cap}/V_{max}	Max Volume Capacity
VCF	Volume Concentration Factor

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5 Pressure filters

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This chapter will describe cake filtration at pressures above atmospheric, achieved through pumping or cake compression with diaphragms (membranes). Elevated pressures are required for fine or "difficult-to-dewater" solids with high cake resistance for which vacuum filtration is ineffective.

Equipment described in this chapter includes filter presses, membrane chamber presses, tower presses and tube presses for cake filtration, but excludes "polishing" applications. This chapter will not describe hyperbaric filters that are similar in construction to vacuum disc, drum and belt filters but are enclosed in a pressurised chamber.

The chapter focuses on the practical aspects of filter selection and filter plant design. Whilst of fundamental importance to understanding pressure filtration, the use of filtration theory will be minimised as these topics are covered thoroughly in many texts (Svarovsky, 1981; Rushton *et al*, 1996; Wakeman and Tarleton, 1999).

5.1 Equipment introduction and key features

Pressure filters are used for a wide range of applications. Although the basic operating principles are simple, filters have evolved for different processes. Consequently, there are thousands of possible combinations of materials of construction, filter chamber shape, chamber orientation, operating pressure, opening mechanism, cake discharge method etc. It is not possible to cover every filter option, but this chapter will address the key features.

Figure 5.1 shows pressure filtration at its simplest, and an industrial equivalent is the batch Nutsche filter. More advanced industrial filters

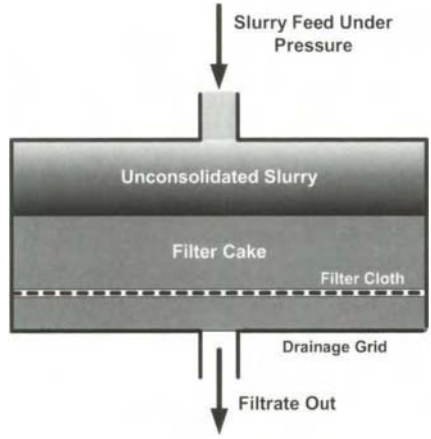

Figure 5.1 Basic pressure filtration principle

are discussed in more detail later in this section. Slurry is fed into a closed chamber under pressure. The filter chamber can be square, rectangular, circular or annular, and can be orientated horizontally or vertically. Liquid flows through the filter cloth, and via the drainage grid to the filtrate outlet. Solids accumulate on the cloth and a filter cake starts to build.

As the cake builds its resistance increases, and the rates of slurry flow into the filter and filtrate flow out of the filter decrease. For fast-filtering slurries it may be possible to completely fill the filter chamber with consolidated cake. For slow-filtering slurries, cake resistance may increase to the point where it is impossible to pump more slurry into the chamber even though the consolidated cake does not fill the chamber. As a general rule, deeper chambers (higher volume per unit area) are required for fast-filtering slurries, and *vice versa*.

When chamber filling is stopped due to the chamber being full, or through feed rate reduction, the filter chamber is opened and the cake removed. The chamber is then closed and filled again. Pressure filters are, therefore, batch units. The cycle time for each batch can vary from less than ten minutes to several hours, and depends upon the equipment, the process and the material being filtered.

A common and convenient way of expressing the filtration rate of a filter is mass of dry solids in a filter cake per unit area of filtration area per unit of time. For the simple filter in Figure 5.1, assuming the filtration area is 0.5 m^2, the time to feed the filter is 2 minutes, the time to open, empty and close the filter is 4 minutes, and the mass of dry solids contained in the filter cake is 10 kg, the filtration rate can be calculated as:

$$\text{Filtration Rate} = \frac{\text{Mass of Dry Solids in Filter Cake} \times 60 \text{ minutes/hour}}{\text{Filter Area} \times (\text{Feed Time} + \text{Cake Removal Time})}$$

$$= \frac{10 \text{ kg} \times 60 \text{ minutes/hour}}{0.5 \text{ m}^2 \times (2+4) \text{ minutes}}$$

$$= 200 \text{ kg m}^{-2} \text{ h}^{-1}$$

If 400 kg of this material has to be processed every hour it will be necessary to supply 2 m^2 filtration area. This can be provided by using four separate 0.5 m^2 filters, or by combining four 0.5 m^2 chambers into a single filter mechanism, or by using a filter with a single 2 m^2 chamber. Practical examples of dimensioning industrial filters for real applications are given later in this chapter. By necessity, industrial filters are more complex than the simple example shown in Figure 5.1, and the most commonly used types are described below.

5.1.1 Plate and frame chamber filters

The earliest and simplest pressure filters used plates and frames, with a filter cloth between each plate and frame. Filtration takes place on both sides of the chamber formed when the plates and frames are clamped together. Plate and frame filters are still used, but generally for smaller applications with manual operation.

Figure 5.2 shows a typical design of plate and frame, and how they are combined to form chambers. The sequence of alternating plates and frames is known as the plate pack. Typical plate sizes range from 0.6 m × 0.6 m to 1.5 m × 1.5 m.

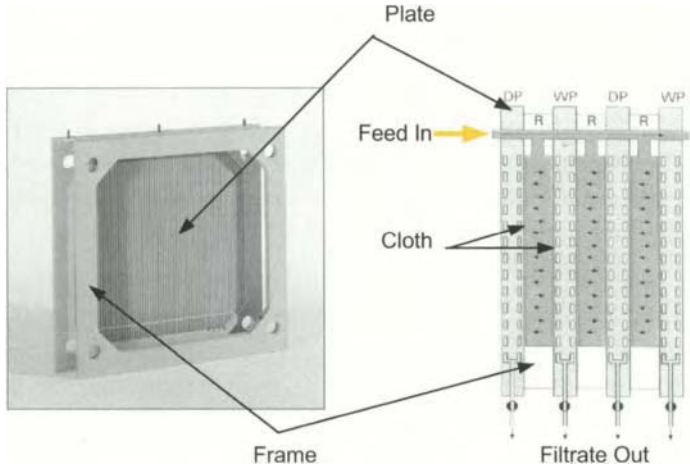

Figure 5.2 Plate and frame filter chambers (Larox Hoesch GmbH)

Feed slurry is pumped through the channels formed at the top corners of the plates and enters each chamber. Slurry pumping is often in two stages; first high flow, low pressure to fill the chambers, and then low flow, high pressure to consolidate the cake. Air may be blown through the cakes for further drying before discharge. Manual assistance is required to remove cakes from the frames.

5.1.2 Recessed plate chamber filters

Recessed plate chamber presses were developed from plate and frame filters, have similar construction, and are used for the same applications. However, the plates are recessed to form the filtration chamber when the plates are clamped together, without the need for separate frames, as shown in Figure 5.3.

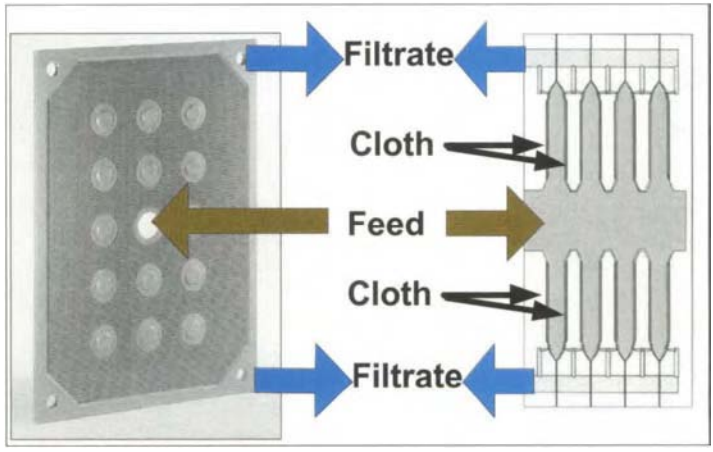

Figure 5.3 Recessed filter plates with central slurry feed and "stay bosses" to prevent plates from flexing

Filter plates can be manufactured from cast iron or polymers, and can be supported on side beams (Figure 5.4) or suspended from an overhead beam (Figure 5.5). At one end of the plate pack is a fixed head plate to which major piping connections are attached. At the other end of the plate pack is a moving pressure head. The moving head is forced towards the fixed head to close the plate pack and seal the chambers. This can be done manually, for example with a screw mechanism, or by hydraulic cylinder.

Filtration in a chamber filter is a batch process, and the following cycle is for a large unit with 1.5 m × 1.5 m plates and 80 chambers for a wastewater application:

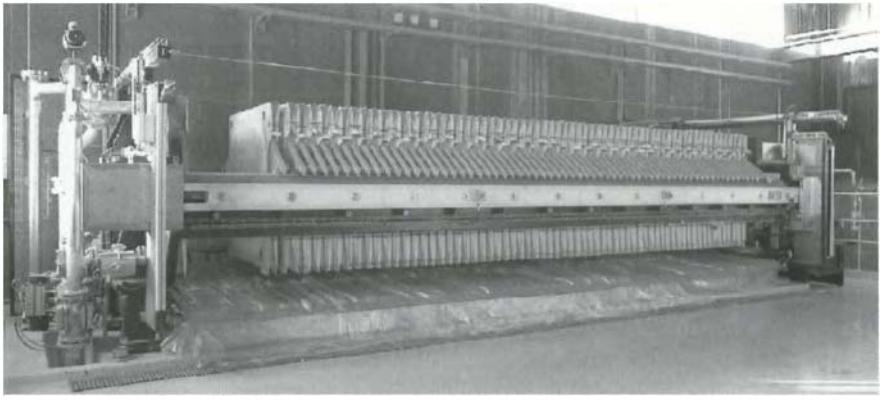

Figure 5.4 Sidebar filter press (Larox Hoesch GmbH)

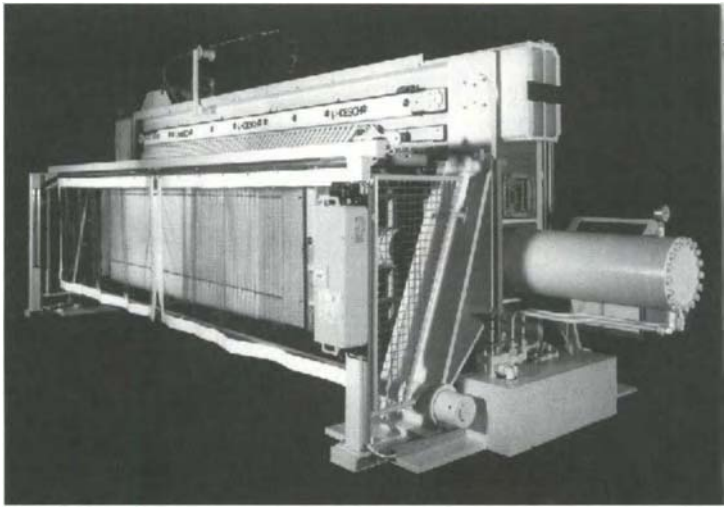

Figure 5.5 Overhead beam filter press (Larox Hoesch GmbH)

1.	Close filter press	2 minutes
2.	Fill chambers with slurry (high volume, low pressure pump)	5 minutes
3.	Filtration (low volume, higher pressure pumping)	55 minutes
4.	Blow Cake (optional further dewatering step)	2 minutes
5.	Open Filter Press (including pressure release and core wash)	2 minutes
6.	Discharge cake – mechanical plate shift and manual cake removal	30 minutes
	Total Cycle Time	*96 minutes*

5.1.3 Membrane filter presses

Membrane filter presses were first developed in the late 1940s to reduce cycle time, increase unit capacity, and produce dryer filter cakes. Filter construction is similar to recessed chamber presses, but the plate pack incorporates "membranes" (also known as "diaphragms") that are inflated at high pressure to squeeze the filter cakes. Consequently, these filters are also known as variable chamber presses.

Cake squeezing is particularly useful for compressible cakes, but also has a beneficial effect on relatively incompressible cakes by homogenising cake thickness and density. The design also makes it possible to achieve superior results with slow-filtering materials that form thin cakes.

The plate pack consists of alternate recessed plates and "membrane plates". Membrane plates have a membrane on each side that is inflated by either water or compressed air. Membranes have ribbed or dimpled surfaces to support the filter cloth and act as filtrate drainage channels. Figure 5.6 shows, from left to right, an assembled membrane plate, a membrane plate base with membranes removed, and a recessed chamber plate. Figure 5.7 shows sections through alternate recessed plates and membrane plates.

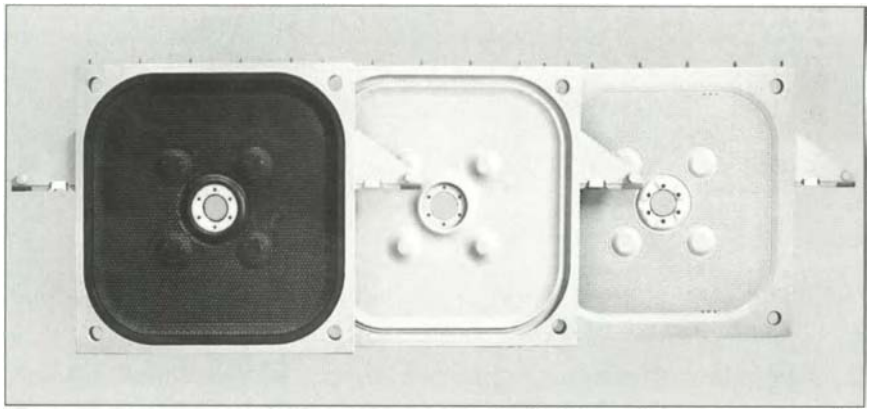

Figure 5.6 Membrane filter plate and recessed plate with centre feed

These are centre feed units, but for larger plate sizes the slurry feed port is often at the top as in Figure 5.8.

Top slurry feed ports are also required when the filter incorporates a cloth lifting or shaking mechanism to assist cake discharge. As the size of filter and unit capacity increases, the dimension of ports and

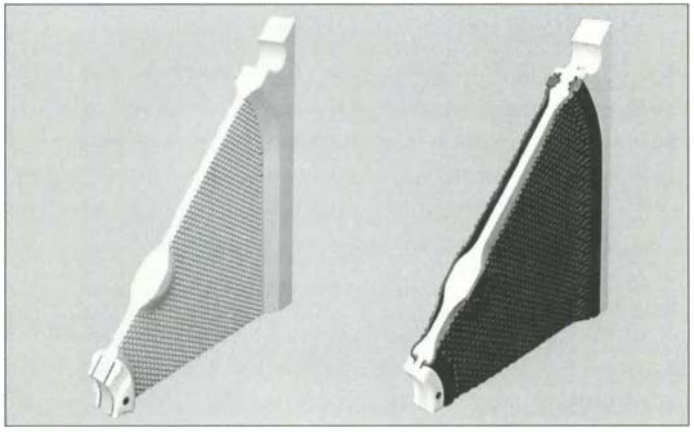

Figure 5.7 Sections through alternate recessed and membrane plates with centre feed

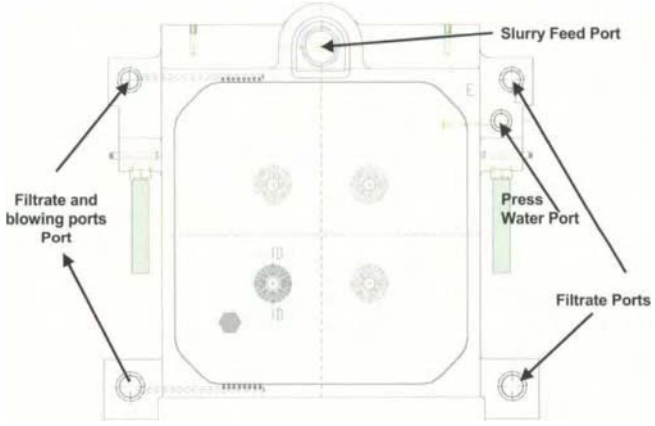

Figure 5.8 Large membrane filter plate with top slurry feed port

channels becomes increasingly important to avoid flow rate limitations or abrasion through excessive velocity.

It is possible to operate membrane filters with different cycles for dewatering only, and dewatering with cake washing. The sequence of filtration steps in the cycle is determined from experience and test work. A typical filtration cycle for dewatering only would be:

1. Slurry feeding
2. Cake squeezing by inflating the membranes
3. Blow air through the cake

4. Slurry channel (or "core") wash and/or blow
5. Cake discharge.

For dewatering and cake washing, additional steps are included:

1. Slurry feeding
2. Cake squeezing to consolidate the cake, reduce permeability and seal cracks
3. Introduce wash liquor
4. Squeeze with membranes forcing wash liquor through the cake
6. Blow air through the cake
7. Slurry channel (or "core") wash and/or blow
8. Cake discharge.

Washing stages may be repeated if required, and can use different wash liquors. Counter current washing is also feasible by storing wash filtrates and reusing them as wash liquor in subsequent cycles. Variants on cake washing are possible such as omitting the first cake squeeze, and forcing wash liquor through the cake before squeezing and blowing. Modern membrane filters are automated, and different filtration sequences can be pre-programmed and repeated automatically. A typical dewatering and cake washing sequence is shown in Figure 5.9.

The general construction principles of membrane filter presses are similar to chamber presses, but developments have increased filter capacity through using more and larger plates, shortening cake discharge time, and automation. A typical example, showing key components is shown in Figure 5.10.

Filter plates of 1.5 m by 1.5 m are typical, but 2 m by 2 m plates are increasingly common, and larger plates are being developed. Membrane filters commonly have up to 60 chambers, but designs exist for larger units.

Mechanised plate pack opening and plate shifting have reduced cake discharge time. Cake discharge has traditionally been by gravity and has relied on good cake release from the filter cloth. Cakes fall from the filter onto a collection conveyor, or directly onto a stockpile. For cakes that do not release easily, mechanisms that open one chamber at a time are suitable. These are often "semi-automatic" and allow for manual assistance if necessary. When cakes release relatively easily,

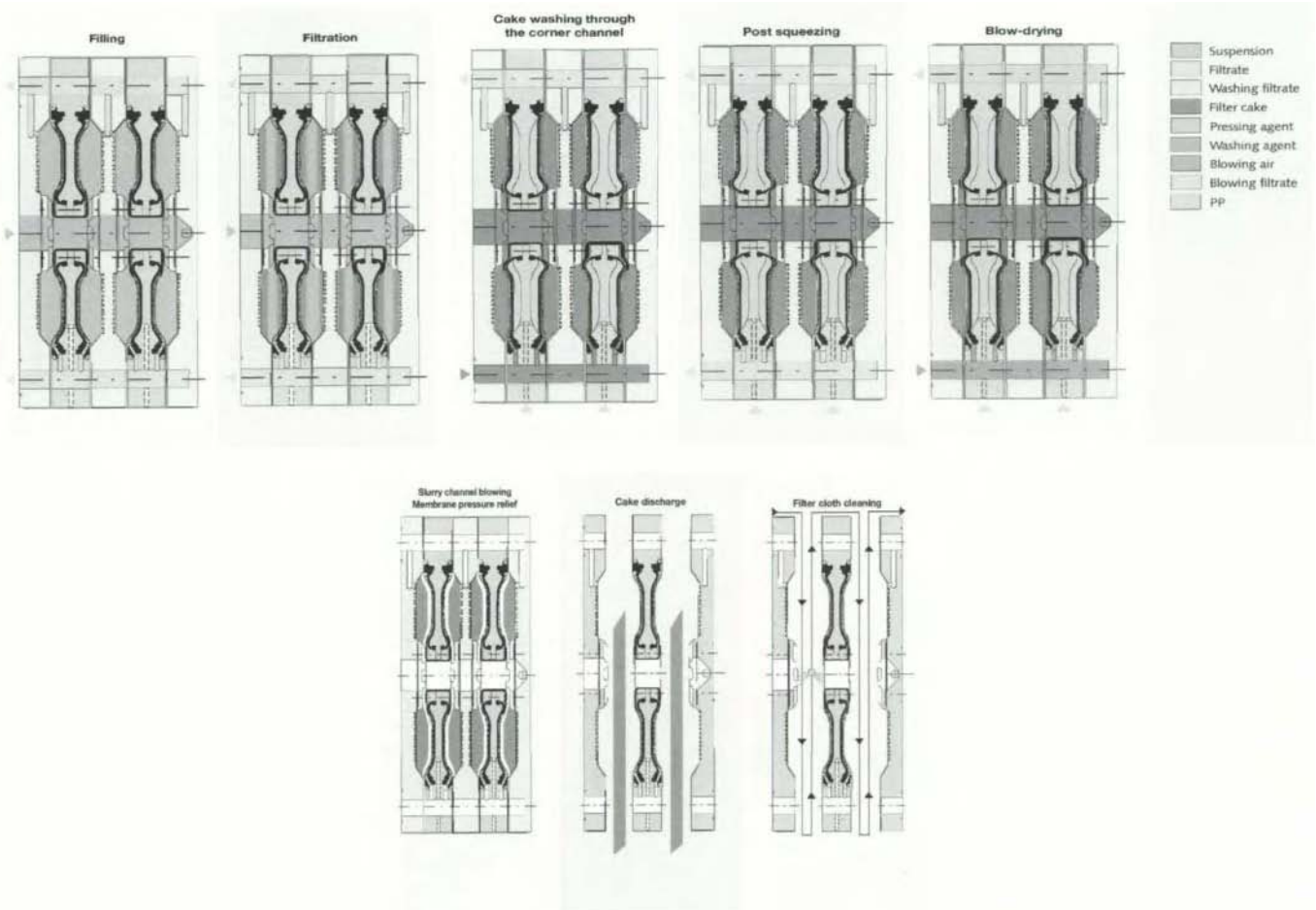

Figure 5.9 Membrane press filtration cycle with cake washing

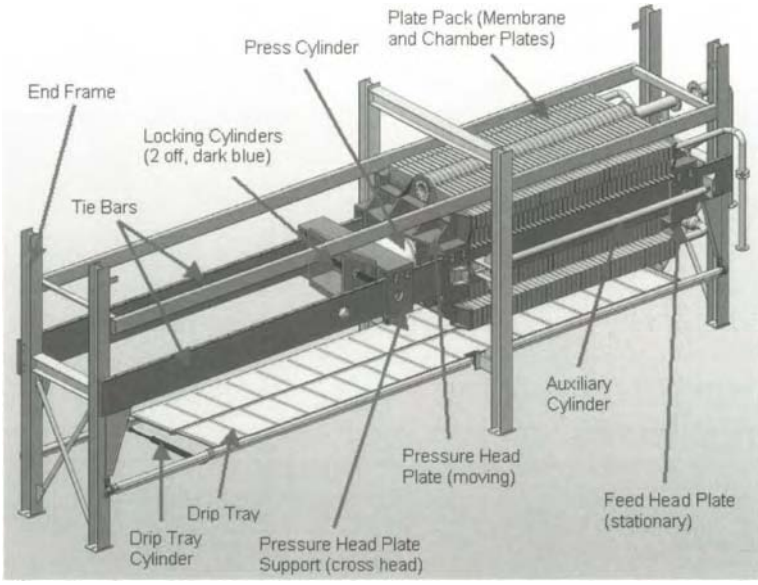

Figure 5.10 Membrane pressure filter with top feed plates (Larox Hoesch GmbH)

chambers can be opened simultaneously or in groups (to reduce instantaneous mass flow). The most recent high capacity membrane filter presses use long-travel hydraulic cylinders ("auxiliary cylinders") to move the pressure head to which plates are connected by links. Very large presses may have two moving pressure heads to further reduce opening time. Plate pack opening and closing can be completed in a few minutes.

Some membrane filter presses incorporate cloth shaking or lifting mechanisms to promote cake discharge. Filters may be placed on load cells that will indicate if the filter does not reach its tare weight, thereby warning of incomplete cake discharge. Such weighing systems can also be used for filter control, and to measure throughput.

Filter cloths will blind as particles become embedded in them, thus reducing capacity and increasing cake moisture. Most modern filters incorporate cloth flushing or washing systems to remove adhering and penetrating particles. These can range from simple spray nozzles mounted above the plates, to moving spray bars that are lowered and raised between the plates singly or in groups. When a wet cake discharge is acceptable, for example if the cake is reslurried, it is possible to use automatic moving brush systems with sprays to remove stubbornly adhering solids from the surface of the cloth. A limitation is that most cloth washing systems wash only one side of the cloth.

Additional features on membrane filter presses, especially those that are automated, include "bomb bay doors" that cover the discharge chute below the filter to prevent water entry to dry cake handling facilities, and protective screens or "light curtains" to prevent operator access.

5.1.4 Tower presses, or vertical membrane filter presses

"Tower Presses" were developed in the 1950s, but only achieved widespread use from the late 1970s. They overcome the potential membrane filter press problems of slow, incomplete cake discharge and inefficient cloth washing by using a moving filter cloth. From their inception they have been more highly automated than membrane filter presses. A typical unit used in the mining industry is shown in Figure 5.11, and construction details for a typical large tower press are shown in Figure 5.12.

Tower presses have a similar operating principle to membrane filter presses, but the filter plates lie horizontally, and are stacked vertically. Filter plates range from 0.4 to 6 m^2 each, and total filtration area per unit can be from 0.4 to 168 m^2 per filter. For dense solids such as fine iron ore, capacities of over 150 tonnes dry solid per hour per filter can be achieved.

Most tower presses have single-sided filtration, but double-sided tower presses have been developed relatively recently for slow filtering materials, difficult cake washing, and hygienic applications.

The tower press configuration permits the use of a moving cloth that zig-zags between the plates. In most cases the cloth returns outside the plate pack to form a continuous, closed loop. In an alternative design, the cloth rolls off an upper spool, passes through the plate pack onto a lower spool, and is rewound periodically. Because the cloth acts as a conveyor, its tensile strength and dimensional stability are as important as filtration characteristics in selection.

The sequence of operation for dewatering only is shown in Figure 5.13. The horizontally oriented filter plates are clamped together by hydraulic cylinders to form sealed chambers. A drainage grid at the base of the chamber supports the filter cloth. Slurry is introduced into all chambers simultaneously (Figure 5.13(1)). Filtrate flows through the filter cloth, through the grid, and into the filtrate manifold. Solids are deposited on the cloth to form a cake. Slurry feed pressure is typically 6 bar g at the end of pumping, and feeding time can be 1 to 4 minutes for most applications, but can be longer for difficult slurries.

Figure 5.11 A 144m² tower press for flotation concentrate dewatering (Larox Oyj)

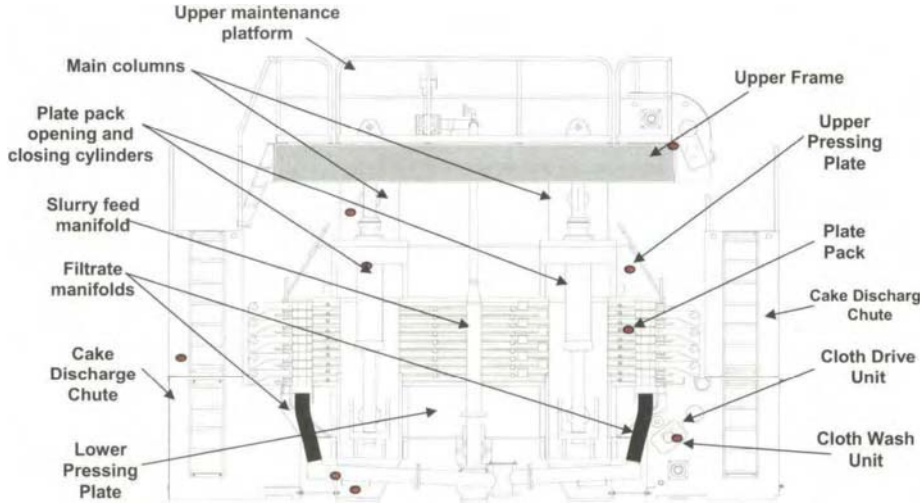

Figure 5.12 Construction details of a large tower press (Larox Oyj)

An elastomer membrane or "diaphragm" in the top of the chamber is next inflated to compress the filter cake and expel moisture (Figure 5.13(2)). Pressing produces cakes that are uniform in thickness and consistency, and avoids cake cracking. Pressing is a more energy

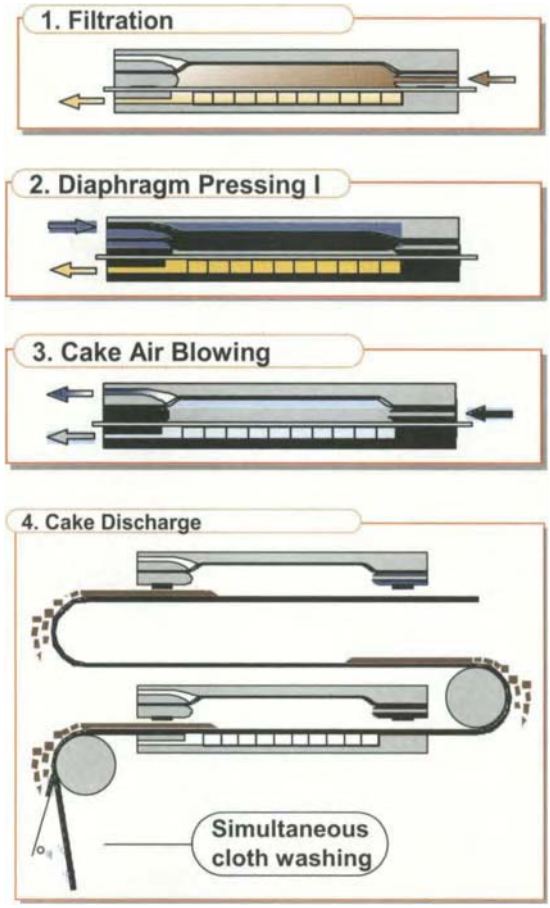

Figure 5.13 (1) Filter feed for cake formation; (2) Diaphragm pressing; (3) Cake air blowing; and (4) Cake discharge

efficient method of moisture reduction than high-pressure slurry pumping or long cake blowing with air. In smaller filters (<30 m^2 filtration area) diaphragms are usually inflated with water from a "pressing water station". In filters larger than 30 m^2, compressed air is used as diaphragm inflation and deflation is quicker. Diaphragm pressing duration is usually between 1 and 6 minutes at pressures between 8 and 16 bar g and is controlled automatically.

If cake washing is performed, wash liquid is pumped into the chambers through the feed manifold after cake pressing. As the chambers lie horizontally, and cake permeability has been reduced through compression, the wash liquid distributes evenly across the filter cakes. The diaphragms are then re-inflated to force the wash liquid through the cake to achieve displacement rather than dilution washing.

At the end of pressing, the pressure above the diaphragm is released, and compressed air at 6 to 10 bar g is introduced into the chamber through the feed manifold (Figure 5.13(3)). The diaphragm returns to the top of the chamber, and air passes through the filter cake to effect final drying to target moisture. Air blowing duration is usually between 1 and 4 minutes and is controlled automatically.

At the end of air blowing any residual pressure is released through the filter drain manifold, and hydraulic cylinders open the plate pack. Once the chambers are fully open the cloth is driven forward, discharging filter cakes into chutes at each end of the filter (Figure 5.13(4)). Cloth speed can be adjusted to meet process needs, but all cake is usually discharged in 10 to 30 seconds. As the cloth leaves the plate pack it passes through high-pressure water sprays that wash the cloth on both sides to remove adhering and embedded solids and minimise blinding. The filtration cycle is then repeated automatically.

A thorough understanding of the operating principles of an automatic pressure filter is necessary to optimise the filtration, and to ensure the correct dimensioning of ancillary equipment. These issues are discussed in later sections.

5.1.5 Tube presses

Several types of "tube press" have been manufactured with horizontal and vertical orientation. Vertical tube presses were developed for difficult-to-filter fine kaolin, but have subsequently been used for other ultra fine solids. Their key features are operation at very high pressure up to 16,000 kPa (160 bar), and an annular design to accommodate this pressure. The high pressures overcome capillary forces and surface charges associated with very fine particles.

The main components of the tube press are shown in Figure 5.14. An inflatable membrane (or "bladder") is installed inside the outer casing. The inner "candle" is surrounded by layers of backing mesh, felt, and filter cloth. The sequence of operation is similar to membrane filter presses and tower presses. Slurry is pumped into the closed tube press under pressure between the cloth and the bladder. Feed is then stopped, and the bladder inflated at up to 160 bar g. Cake forms on the cloth and filtrate drains through the cloth and backing layers exiting the tube press through holes in the candle to a drain point at the bottom of the tube. A washing stage is possible, similar to membrane and tower presses, and an air blow can be used to achieve final moisture. On completion of the filtration cycle, vacuum is applied behind the bladder to retract it. The candle is then lowered and an air pulse can be

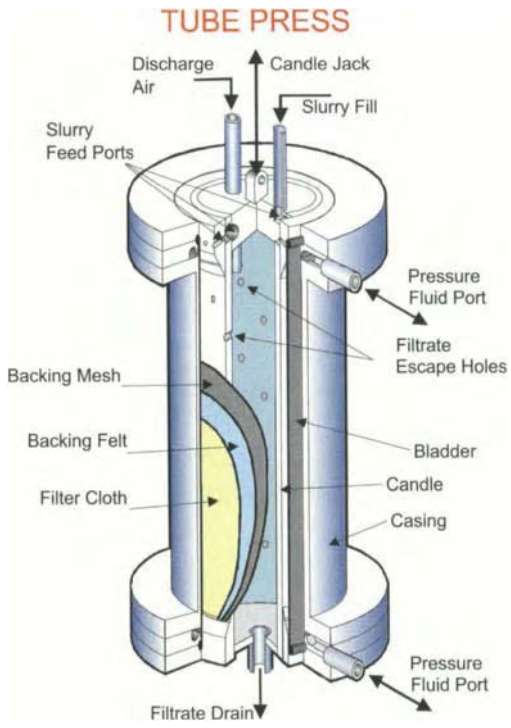

Figure 5.14 Tube press main components (Metso Minerals)

blown through it to help discharge cake from the cloth. The candle is then retracted and the cycle repeated automatically.

Tube presses achieve high filtration rates per unit area. However, as the largest units have a filtration area of 3.5 m^2, multiple units are required to process high tonnages.

5.2 Filter automation

Pressure filter automation has become increasingly sophisticated, and now includes process control in addition to sequencing the filtration steps. Current developments include data logging and reporting, remote monitoring and diagnostics (Jämsä-Jounela *et al*, 2003).

Entry level automation uses proximity sensors on the moving pressure head, pressure meters in slurry feed, wash water, air and hydraulic system pipelines, and level sensors in upstream and downstream surge vessels to control the filter cycle. A programmable logic controller (PLC) initiates each phase of the filter operation, including and starting

of ancillary equipment and valve actuation, subject to values from the field sensors being at required values. For example, the feed pump will not start if the level in the feed tank is below a minimum set point, if the proximity sensors indicate the filter is not fully closed, or if the pressure in the hydraulic system is too low for adequate plate pack sealing. The filter's PLC will give an alarm when a sensor gives a signal outside the prescribed range. Set times for filter feeding, cake pressing, cake washing and air blow can be programmed, and subroutines can be repeated. PLCs can usually accept multiple programs for different process conditions.

The next level of automation adds process control using signals from the filter's sensors. If the filter is placed on load cells its mass can be measured continuously, and can be used to control feed time for solids with higher bulk densities. Load cells will also indicate incomplete filter cake removal if a filter has not returned to its net weight after discharge. Differential pressure between pressing media pipeline and closed filter feed manifold can be used to determine the end of cake pressing. Filtrate turbidity can be measured to indicate damaged cloths, and filtrate conductivity or pH can be used to control filter cake washing. Flow meters can ratio wash liquid addition to the volume or mass of feed. Blowing air flow rate and pressure can be controlled, and blowing terminated when a set flow rate and/or pressure is reached.

Further automation options includes filter sensor data logging on a server, and display on a supervisory control and data acquisition (SCADA) system. SCADA systems can provide on-line maintenance manuals, engineering drawings, spare part lists, PLC program ladder diagrams, and troubleshooting guides. Stored data can be analysed to optimise filter operation, report production, and plan maintenance. It is possible for both the filter's PLC, and the data server to be accessed by modem or the Internet for remote support or reprogramming. Developments in artificial intelligence, fuzzy logic and neural networks are extending the use of filter sensor signals for diagnostics and optimisation.

5.3 Design considerations

The selection of new filters for a project is an iterative process. The objectives must be defined, data must be gathered from internal and external sources, conceptual designs produced and tested, and results fed back to improve the process design. The design is not restricted solely to filter selection, but must also optimise ancillary equipment

and plant layout. Overall plant design must take into account limitations imposed by factors such as infrastructure that may affect equipment and spares delivery, or power supply and altitude that will affect the selection of ancillary equipment such as compressors.

When filters are to be installed into an existing plant as part of an upgrade or expansion, historical data are available on process variations, suitable materials of construction, and safety and environmental considerations. Fresh, representative sample must be available for test work and results compared with those from existing equipment. Supplementary information may also be obtained from external sources such as filter manufacturers, other companies using filters in similar applications, and literature reviews. The potential constraints in expansions and upgrades are restricted space for installation, and the necessity to install and commission new equipment with minimal disruption to production.

For greenfield plants and new processes there are limited data and representative material may not be available for filtration testing. There are more uncertainties, but fewer constraints.

It should be fully understood how filtration integrates with ancillaries and upstream and downstream unit operations. Pressure filters are batch units and require special design considerations. Slurry feeding, cake compression, and air blowing take place during a fraction of the filtration cycle, and ancillary designs must be based on instantaneous rather than average flows. It is also necessary to provide upstream and downstream surge capacity when a batch pressure filter is integrated into a continuous process.

5.3.1 Filter project objectives

When planning a new filter project it is essential to consider the overall objectives and not just the filtration unit operation. The designer should ask, "What is the purpose of filtration in this process?" Is it to produce dry solids, to recover valuable liquid, or to remove contaminants? The range of filter performance that is acceptable in terms of cake moisture, washing efficiency and filtrate clarity should be defined, and trade-offs between filtration rate versus cake moisture, and wash efficiency versus wash liquid consumption considered. The designer should assess how filter performance will affect the overall process efficiency, reliability, safety, and costs. For example, installation of new filters may demand capital expenditure in one department, but reduce operating costs downstream. The pitfall of "departmental accounting" should be avoided.

Some of the features and design considerations for different applications and industries are summarised in Table 5.1. It is unlikely that a single filter design could meet the requirements of all applications, and this has led to design of specialised filters, and tailoring of basic designs to the needs of specific industries.

Table 5.1 Design considerations for different applications.

Application	*Objectives*	*Design Considerations*
Fermentation broth	• Solute recovery • Mycelia waste disposal	• Low density solids • Variable filterability • Hygiene
Fillers e.g. TiO_2 or PCC	• Solute removal • Product quality • Drying costs & capacity	• Materials handling • Crystal breakage • Contamination/ Colour
Hydrometallurgy – leach residues or impurity precipitation	• Solute recovery • Variable process conditions	• Corrosive slurry • Elevated temperature • Cloth blinding
Base metal flotation concentrate	• Moisture limit for transport or drying • 24/7 operation	• High tonnage of dense, fine solids • Variable mineralogy • Remote location
Fine iron ore	• Consistent, low moisture for pelletising	• Very high tonnage • High filtration rate • Dense and abrasive solids

5.3.2 Filter plant philosophy

Objectives and constraints differ between individual projects, as do the operating philosophies of companies using filters. Some of the issues to be considered include:

- What are the relevant health, safety and environmental regulations?
- What are the relevant standards and design codes?
- Is investment dictated by a limited budget or does it aim to maximise net present value of the investment?
- Will the plant have a high operating labour complement, or a high level of automation?
- Will the plant have a single production stream for economy, or multiple streams for flexibility?

- What are the constraints imposed by plant location, water quality and supply, climate, altitude and infrastructure?
- What is the availability and skill level of plant operation and maintenance staff, and how does this constrain filter selection?
- Will the plant provide standby units or allow adequate downtime for maintenance?

5.3.3 Filter plant throughput

The mass and volumetric flow rates of solid and liquid streams in the filter plant must be defined to calculate the number and size of filters required. Similarly, the operating hours per day and year, allowance for maintenance, and required availability should be known. A nominal filter feed rate can be calculated from plant capacity and operating hours. However, variations in throughput and solids concentration occur and a "design tonnage" is usually specified near the upper end of the anticipated range. The "safety" and availability factors applied to reach the design tonnage should be clearly understood by those designing the filter plant to avoid any further and unnecessary factors being applied. The filter plant design should accommodate anticipated "turn up" and "turn down" requirements. Similarly, if it is known that plant capacity will expand in the future, allowance can be made in the initial design that will simplify later upgrades. Forward planning can include selecting expandable filters, leaving space in the plant layout for additional filters, and, if appropriate, dimensioning ancillary equipment for future expansions.

5.3.4 Filter feed characterisation

Data should be readily available for expansion of existing plants, and the effects of slurry characteristics on filtration performance can be measured during testing for greenfield projects. Good data on the solid and liquid phases will help understanding of the filtration process and aid optimisation.

The solids composition will affect filtration rate through density, surface charges and compressibility. Particle size and shape will affect packing, voidage and cake resistance, thereby influencing filtration rate, final moisture and cake washing. Filter feed preparation has a significant impact on filtration rate. Higher feed solids concentration will increase filtration rate and give clearer initial filtrate.

Feed slurry temperature will affect filtration rate and cake washing through liquid viscosity. Salts may crystallise from liquids if the

temperature changes, affecting cake washing for solute removal, and may also blind cloths. Solid and especially liquid composition, and temperature, will determine materials of construction, and must be taken into account in safety and environmental studies.

5.3.5 Conceptual filter plant design

Once sufficient data have been collected and analysed, a conceptual process design for the filter plant can be made. The process can be evaluated by performing filtration tests on samples representative of the feed slurries proposed in the conceptual design. If inadequate sample is available, evaluation has to be based on modelling using filtration performance for slurries with similar characteristics.

It should be noted that filtration does not take place at a single point. There is a direct relationship between filtration rate and cake moisture, and higher filtration rates can be achieved at the expense of higher filter cake moisture. Automatic pressure filter control systems enable the operating point on the capacity/moisture curve to be selected. Similarly, higher cake washing efficiency will require a higher volume of wash liquid as the wash efficiency versus wash ratio curve becomes asymptotic. Increased wash volume extends the cycle time and decreases filtration rate, and can be evaluated during test work.

5.4 Dimensioning and scale-up

Filtration test work is conducted initially to establish the feasibility of pressure filtration for solid/liquid separation and cake washing. Further test work measures the effect of feed preparation such as flocculation and thickening, and operating parameters such as feed pressure, upon filtration rate, cake moisture, and cake washing efficiency. Multiple tests identify the optimum parameters to be used for pilot scale testing or filter plant design. Larger scale tests produce bulk samples for downstream process evaluation or trial marketing, to investigate materials handling, and to train future operators.

5.4.1 Test methods

Small-scale filtration test procedures and data interpretation are well documented in the literature, and the tests are straightforward. Nevertheless, experience improves the quality of data collected and the interpretation of the data. When generating data for the design of a large, industrial filtration plant, the services of a test laboratory

specialising in filtration, or filter manufacturers can be used unless the company has its own in-house filtration specialists.

The scale of test work is dictated by the quantity of sample available, and the purpose of the tests. "Bench Top" tests are used when only a small quantity of sample is available, and for rapid evaluation of a range of feed conditions, feed times and feed pressures. It is possible to use bench scale filtration test data to design industrial scale filters, but limitations must be recognised. Bench scale test equipment does not usually replicate exactly the operating principles of industrial scale equipment. For example, bench scale tests are "single" sided, and the data represent formation of a "half cake" in chamber presses with double sided filtration. Furthermore, there are inherent scale-up risks in using the results from a 0.002 m^2 filter to design a 100 m^2 or larger production unit.

"Manufacturer tests" use test filters that are usually designed to replicate the filtration cycle of a company's product. Manufacturers have developed their test equipment to correlate closely with their production units, and will use the results from these units for industrial scale design. They require larger samples than bench top tests, and are used to identify and confirm the optimum filtration conditions.

Pilot scale filters are small production units that are used in demonstration plants for new process development, or run in parallel to existing filters for evaluation as potential replacements. Pilot scale filters will not produce more information for filter scale up than manufacturers' test units, but when operated for an extended period will indicate potential problems with cloth blinding, materials handling or operation. They can be used for operator training, and to produce bulk samples of cake and filtrate for development of downstream processes or for trial marketing. Operation of a pilot scale filter will give additional confidence to the decision makers ultimately responsible for filter selection.

5.4.1.1 Sample acquisition and storage

For test work to be meaningful, the samples must be representative of the slurry that will be processed in the industrial scale filter. Industrial processes vary with time due to feedstock changes and operational adjustments, and the test programme should aim to cover the anticipated range of filter feed conditions.

The quantity of sample available for testing depends upon the status of the project. For a greenfield site, only small samples may be available from a development test programme in the laboratory. However, it is

possible that the company operates a similar plant elsewhere from which samples representative of the new plant can be obtained.

When obtaining material from an operating plant, careful planning of sample acquisition will ensure it is representative. Sampling theory, methods and safety considerations are well documented in the literature (Wills, 2001; Merks, 2002; Mosher and Alexander, 2002).

Some samples deteriorate with time through oxidation or biodegradation, or their particle size distribution changes through agglomeration or dissolution, affecting filtration rate and cake moisture. Storage for extended periods in vessels with excessive agitation will adversely affect flocculated slurries, and can cause particle abrasion and size distribution changes. Temperature alteration during storage can cause chemical changes, or lead to solutes crystallising, and will have an impact upon cake washing. Ideally, the test equipment should be taken to the place where the sample is acquired, and the sample should be tested immediately after acquisition. This will ensure the sample is fresh. When a sample has to be stored, for example for transport to a remote test laboratory, the storage method and conditions should protect the sample from change. Some samples, such as fermentation broths, can only be tested when fresh.

When it is not possible to acquire a sample with the correct solute for tests including cake washing, it is possible to add a chemically stable "tracer" that is simple to analyse.

5.4.1.2 Preparation for testing

Once a test programme starts it should proceed smoothly with minimal interruption to avoid potential sample changes with time. The equipment and services should be checked thoroughly, and any necessary maintenance undertaken before testing, to avoid unexpected breakdowns. Different cloth types should be prepared in advance for evaluation. Sample collection containers should be prepared, and labelled indelibly. Measuring cylinders for filtrate and wash liquid, and balances to weigh samples should be ready and checked for accuracy. A stopwatch is needed to time each phase of the filtration cycle, and to record filtrate volume against filtration time. Sometimes it is necessary to know cake moisture or filtrate assay before proceeding to the next test. The necessary assay services should be arranged in advance, and rapid moisture analysers such as thermal balances can be very helpful. Tests follow a standard format, and it is possible to produce suitable log sheets to record the test data in order. An example is reproduced in the Appendix, and spreadsheets can be created to calculate required data from measured values. Automatic data collection systems are now

readily available, and range from a load cell continuously measuring filtrate mass against time, to recording of flow rates, masses and pressures to a data-logging computer.

Typical data to be collected before, during and after filtration tests include:

- Filter equipment details – filtration area, chamber depth, cloth type etc
- Pretreatment of slurry – details of flocculation or other chemical addition etc
- Slurry solids concentration and pulp density
- Slurry temperature and pH
- Slurry viscosity
- Solid and liquid specific gravities
- Solid and liquid composition
- Particle size distribution of solids
- Time, pressure and filtrate produced during each phase of the cycle
- Cumulative volume of filtrate *vs* time for each phase of the cycle
- Wash liquid composition and density
- Air flow rate during cake blowing
- Analysis of wash filtrate for solute concentration
- Mass of wet filter cake
- Wet cake thickness
- Cake moisture
- Cake appearance – cracks, homogeneity etc
- Cake release from cloth, and evidence of cloth blinding.

A mass balance should be conducted to check the validity of the test data.

5.4.2 Test equipment

5.4.2.1 Bench top test equipment

Bench scale pressure filtration test equipment commonly uses a 50 mm diameter cloth mounted in a suitable holder beneath a chamber. The slurry sample is placed into the chamber, which is pressurised to start filtration. The chamber may be pressurised by air, or by a piston that is

forced towards the slurry sample. The "pressure bomb" shown in Figure 5.15 is constructed from a 250 mm section of 50 mm pipe. This type of filter simulates the action of a plate and frame or recessed chamber filter for both filtration and cake washing, but does not reproduce the effects of cake compression with membranes.

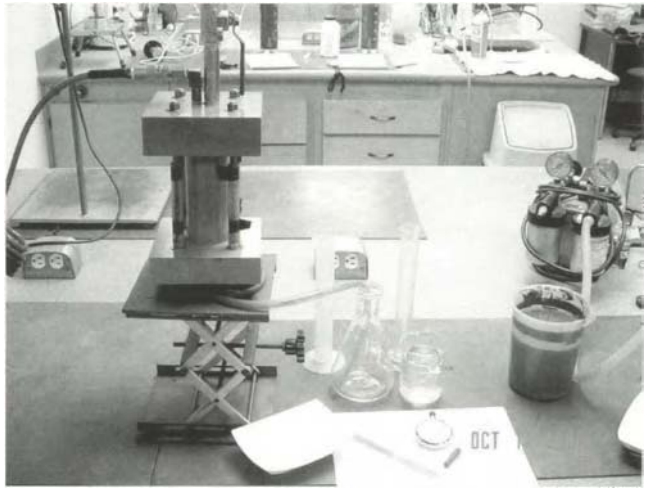

Figure 5.15 Laboratory "Pressure Bomb" filter (Pocock Inc.)

The top cap has ports for slurry and wash liquid feed, and a fitting for compressed air inlet and release. The base holds the filter cloth supported on a drainage grid, and has a central filtrate outlet port. A slurry sample is placed in the cylinder through the port, which is then closed. Compressed air regulated to the required pressure is introduced into the top of the chamber above the slurry to initiate filtration. If possible, the filtrate volume versus time should be recorded at short time intervals. This is facilitated if the filtrate can be weighed continuously on a load cell, but is difficult for manual recording with fast-filtering slurries. The end of cake formation is indicated by the end of filtrate flow and the escape of air from the filtrate port, and the time and filtrate volume should be recorded. If there is no cake washing stage, air is allowed to blow through the cake for a pre-determined length of time, after which pressure is released, and the cake removed, weighed wet, and again after drying.

If the cake is to be washed, pressure is released from the cylinder at the end of cake formation, and a known volume of wash liquid is introduced through the feed port. The cylinder is then sealed and re-pressurised. The volume of wash filtrate can be recorded against wash

time, or the end of washing noted in a similar way to cake formation time. At this scale of testing the volume of wash filtrate may be too small to analyse filtrate samples from small time increments, and analysis may have to be performed on the total wash filtrate. The cake is air blown after the end of washing.

Figure 5.16 shows a 0.0025 m^2 bench top piston press. The filter cloth and drainage grid are contained in the lower housing, and filtrate outlet is from the pipe at the front. The stainless steel cylinder, which forms the chamber, is first screwed into the lower polypropylene housing. 125 ml of slurry is poured into the cylinder, and the upper polypropylene piston is inserted into the top of the cylinder. The complete assembly is placed into the stainless steel frame, and a compressed air hose connected to a port through the piston to the chamber. The stainless steel base contains a hydraulic cylinder, which forces the chamber upwards towards the fixed piston. Typically the pressure is set at 6 bar g for cake formation, and filtrate volume is measured against time as for the "pressure bomb". If the cake is to be washed, the pressure is released and the assembly removed from the frame. The piston is retracted gently from the cylinder to avoid damaging the cake, and a known volume of wash liquor is introduced. The piston is replaced, and the assembly returned to the frame. If necessary, the pressure from the hydraulic cylinder is adjusted, and the cylinder driven towards the piston to press the wash liquor through the cake. Wash filtrate is collected and volume versus time recorded. At the end

Figure 5.16 0.0025 m^2 bench top piston press (Larox Oyj)

of cake washing, pressure from the hydraulic cylinder is increased to simulate cake compression by a membrane. Pressing filtrate volume and time are recorded. When filtrate flow stops, pressure is released from the cylinder, and compressed air is blown through the cake via the port through the piston. Air blowing time and volume of filtrate produced are recorded. Pressure is then released and the cake removed weighed and dried. Wash filtrate is analysed.

5.4.2.2 "Manufacturers'" test equipment

Most filter manufacturers have "mid size" test filters that replicate the operation of their own production-scale units. The results can be used confidently for scale-up. However, because of design differences between production filters, the results from a manufacturer's test filter may only be applicable to their product range. Manufacturers will conduct filtration tests for customers and report the number, type and size of pressure filters the customer will require for his application. Test units may also be available to rent or purchase for in-house testing.

Manufacturers' test units are designed for easy transport to a customer's site where fresh sample will be available. They should be robust, and simple to set-up on site.

A Larox test filter is shown in Figure 5.17. It integrates a 0.1 m^2 horizontal membrane chamber filter and its ancillaries into a mobile frame that can be pushed, or carried by crane or forklift. Chamber depths from 30 to 75 mm are available. Feed pressure, washing pressure, pressing pressure, air blow pressure and air flow rate can be adjusted. The complete unit is 2 m long by 1.5 m high by 0.6 m wide,

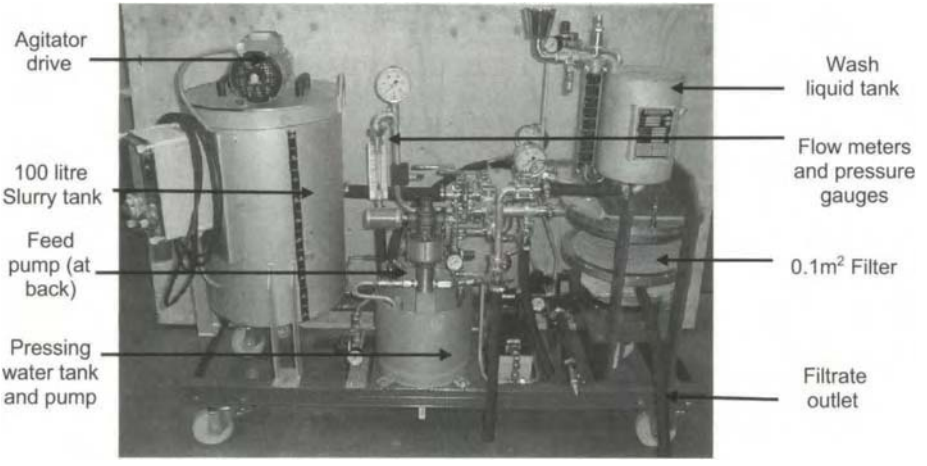

Figure 5.17 0.1 m^2 Manufacturer's Test Filter (Larox Oyj)

and all components except the variable speed agitator drive are pneumatically powered. Test units are available that can complete one complete cycle automatically, and record all operating data to computerised database. An example of data capture is given in a later section. The sequence of operation is identical to that described in the section on tower presses. Similar test units are available for chamber presses and membrane chamber presses.

5.4.2.3 Pilot plant test equipment

Filters for pilot plants are small production units incorporated into the flow sheet of a dedicated demonstration plant, and may operate at tens of kilograms to a few tonnes solids throughput per hour. Alternatively they may be units built into trucks or shipping containers for easy transport to site, for limited duration runs in parallel to existing equipment. Pilot scale filters may be fully automated, and equipped with data logging capabilities. The operating principles are similar to production chamber, membrane and tower presses described earlier. A typical containerised unit is shown in Figures 5.18a and b.

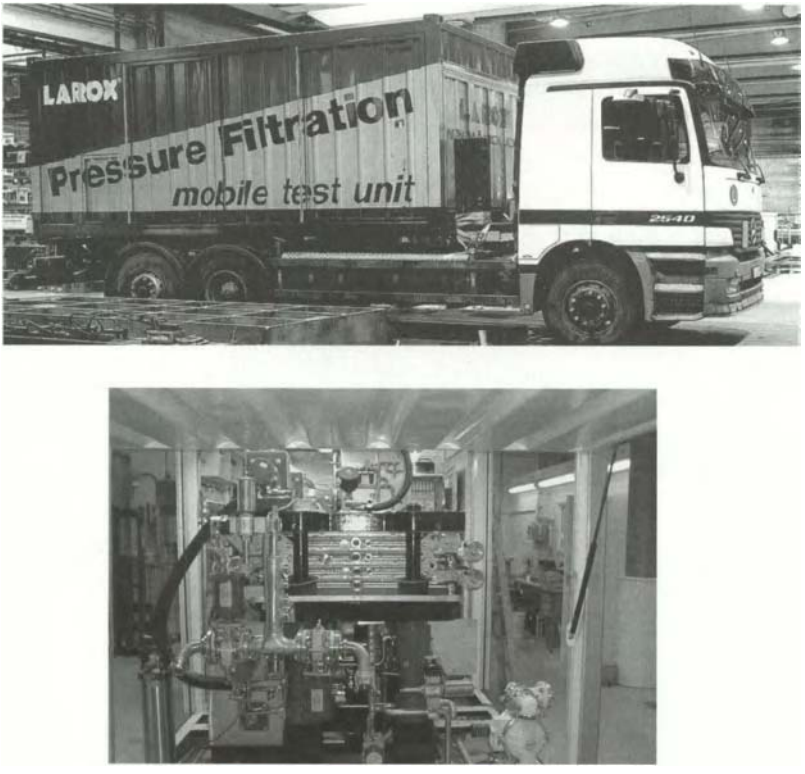

Figure 5. 18a and b Pilot scale 1.6 m^2 tower press and ancilliaries installed in a shipping container (Larox Oyj)

5.4.3 Interpreting the test data

Filtration test data can be interpreted by calculating:

- Cake and media resistance
- Mass of dry solids per unit area or volume of cake per unit of time.

The former is the classical academic approach and permits calculation of cake formation under different conditions in Nutsche, plate and frame or recessed chamber presses. It is a valuable design method for slow-filtering materials, but for relatively fast-filtering slurries in industrial processes, cake formation time may be too short for accurate filtrate volume versus time measurement. Values for particle size, filtrate viscosity and solids density are needed to complete the calculations.

Cake and media resistance data cannot be used to evaluate membrane compression of cakes and air blowing, although other mathematical methods are available for these stages of the pressure filtration cycle (Wakeman and Tarleton, 2005). Design data for membrane, tower or tube presses are often based on empirical determination of cake mass per unit area or volume per unit time. Empirical test methods measure filtration rate directly, and it is not essential to have information such as solids density, particle size or filtrate viscosity although these data are useful for full understanding.

5.4.3.1 Cake and medium resistance

Design methods based on cake specific resistance are well documented in text books (Svarovsky, 1981; Rushton *et al*, 1996; Wakeman and Tarleton, 1999) and are only summarised below. Spreadsheet programs for using cake and media resistance have also been published and are available on the Internet (Rushton *et al*, 1996). Cake and medium resistance can be calculated from all constant pressure filtration tests in which the cumulative volume of filtrate is measured against filtration time.

Example The following example is taken from "Filtration Equipment Selection Modelling and Process Simulation" by Wakeman and Tarleton (1999). "Estimate the cake specific resistance and the medium resistance from the following data obtained from a constant pressure filtration test on a calcium silicate suspension using a laboratory scale filter press".

Mean particle size		6.5 μm
Filtration area	A	0.0429 m^2

Filtration pressure	Δp	69 kPa
Filtrate viscosity	μ	0.001 Pa s
Filtrate density	ρ	1000 kg m^{-3}
Solids density	ρ_s	1950 kg m^{-3}
Mass fraction of solids in feed	s	0.0495
Ratio of mass of wet cake to dry cake	m	3.405

Table 5.2 Filtration test data

Filtration Time t (seconds)	*Filtrate Volume V* (m^3)	*t/V* ($s\ m^{-3}$)
0	0	
9	0.001	0.90×10^4
19	0.002	0.95×10^4
31	0.003	1.03×10^4
50	0.004	1.25×10^4
70	0.005	1.40×10^4
93	0.006	1.55×10^4
120	0.007	1.71×10^4
152	0.008	1.90×10^4
187	0.009	2.08×10^4
227	0.010	2.27×10^4
270	0.011	2.45×10^4

t/V is plotted against V, and ignoring the first two data points a straight line fitted through the linear part of the plot (Figure 5.19). Linear regression gives $\frac{t}{V} = 1.742 \times 10^6 V + 0.5186 \times 10^4$

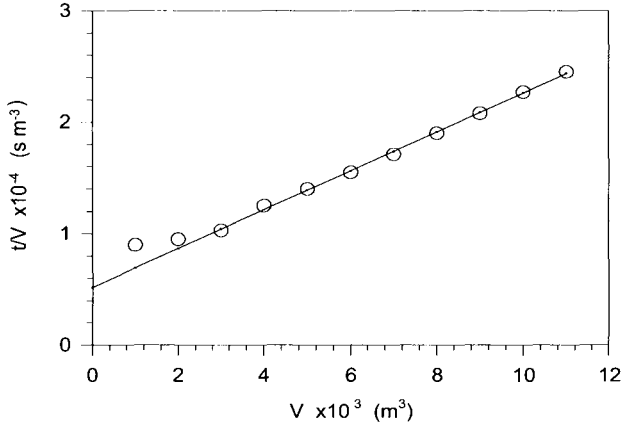

Figure 5.19 *t/V vs V* plot to determine cake specific resistance

Slope of plot = 1.742×10^{6} s m^{-6}

The mass of solids in feed slurry per unit volume of filtrate (c) can be calculated as follows:

$$c=\frac{\rho_l s}{1-m_{av}s}=\frac{1000\times0.0495}{1-3.405\times0.0495}=59.5 \text{ kg m}^{-3}$$

Then

$$\alpha_{av}=\frac{2A^2\Delta p\times\text{slope}}{c\mu}=\frac{2\times0.0429^2\times69000\times1.742\times10^{6}}{59.5\times0.001}=7.4\times10^{9} \text{ m kg}^{-1}$$

The cake specific resistance is 7.4×10^{9} m kg^{-1} at 69 kPa.

From the intercept on the t/V axis, intercept = 5186 s m^{-3}. The medium resistance is obtained as:

$$R=\frac{A\Delta p\times\text{Intercept}}{\mu}=\frac{0.0429\times69000\times5186}{0.001}=1.54\times10^{10} \text{ m}^{-1}$$

The medium resistance is 1.54×10^{10} m^{-1} at 69 kPa.

The general filtration equation is $\frac{dt}{dV}=\frac{\mu\alpha_{av}c}{2\Delta pA^2}V+\frac{\mu R}{A\Delta p}$ and can be integrated and rearranged as $\frac{\mu\alpha_{av}c}{2\Delta pA^2}V^2+\frac{\mu R}{A\Delta p}V-t=0$. This is a quadratic equation of the form $aV^2+bV-t=0$.

Known values can be substituted and solved using $V=\frac{-b+\sqrt{b^2+4at}}{2a}$ for different values of t.

Filtrate volume at different cake formation times can be used to calculate mass of dry cake deposited, mass of dry cake per unit filtration area, and cake depth at the test pressure:

Mass dry cake deposited $= cV$ kg

Mass dry cake per unit filter area $=\frac{cV}{A}$ kg m^{-2}

Cake thickness $=\frac{cV}{AV_s\rho_s}$ m

where V_s is the volume fraction of solids in the cake.

5.4.3.2 Mass per unit area from "pressure bomb" tests

Pressure bombs can produce filtrate volume versus time data that can be used to calculate cake and medium resistance as described above. Interpretation methods have also been developed for calculating the mass of dry cake formed versus time for small volumes of fast filtering mineral and metallurgical sample. Separate tests are conducted with a range of samples of known mass using the method outlined in the description of the pressure bomb, and described in greater detail in the literature (Smith and Townsend, 2002). Results are obtained for a range of cake thickness and dry weight per unit area.

An arithmetic plot of cake dry weight versus cake thickness should give a straight line. A log-log plot of dry cake weight per unit area versus cake form time should have a slope close to the theoretical value of 0.5. The two graphs may be used to calculate the form time that gives the desired cake thickness or weight per unit area. Examples of the plots are shown in Figures 5.20a and 5.20b.

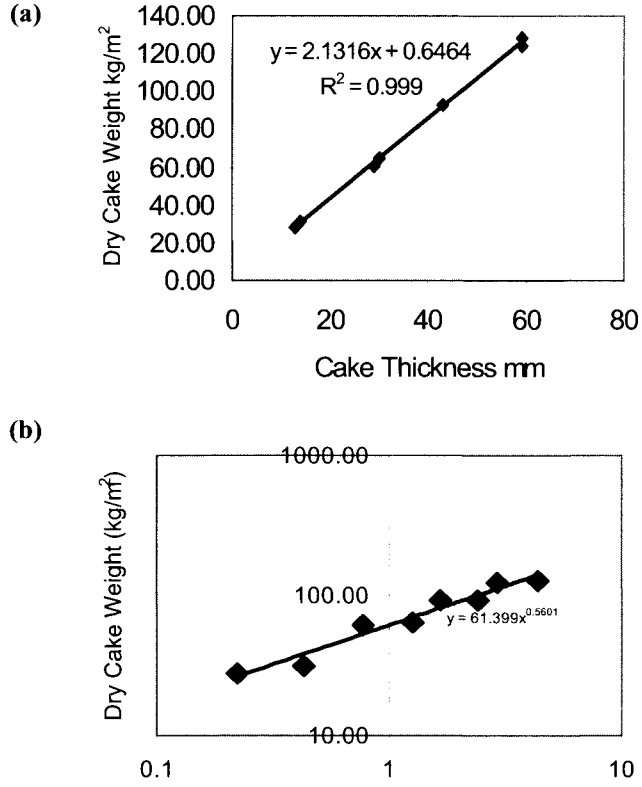

Figure 5.20 (a) Cake weight *vs* Cake thickness, and (b) Cake weight *vs* Form time

5.4.3.3 Mass per unit area or volume from manufacturers' test filters and pilot filters

As described in an earlier section, manufacturers' test filters and pilot filters replicate the filtration cycle of their specific machines. The key objectives of tests with these units are to measure mass of wet and dry cake produced under different cycle times. Filtration rate can then be determined directly without the need to measure slurry, solid and filtrate properties, although these help understand their influence on filtration.

Feed time can be varied, and different chamber depths used to produce filter cakes of different thickness. The effect of cake thickness on pressing time, cake washing time, and air blowing time can then be determined, and the feed time and hence cake thickness that produce maximum filtration rate whilst achieving required moisture and washing efficiency can be identified. If filtrate volume is measured at intervals throughout the filtration test, it is possible to calculate a mass balance, and generate a graph of filtration rate versus cake moisture.

Example An example of the minimum data required to calculate filtration rate from an empirical test of this type with a "relatively difficult" flotation concentrate is shown below.

Test filter area	A	= 0.1 m^2
Feed time	t_f	= 1.5 minutes
Pressing time	t_p	= 1.5 minutes
Air blowing time	t_b	= 4.0 minutes
Technical time	t_t	= 4.0 minutes (industrial filter opening, discharge, closing etc.)
Total cycle time	t_Σ	= 11.0 minutes
Mass of wet filter cake	M_w	= 6.6 kg
Cake moisture		= 10% w/w

$$\text{Calculated mass of dry cake} = 6.6 \times \frac{(100\% - 10\%)}{100\%} = 5.94 \text{ kg}$$

$$\text{Filtration rate} = \frac{5.94}{0.1 \times 11.0} = 5.4 \text{ kg m}^{-2} \text{ min}^{-1} = 324 \text{ kg dry solids per square metre filtration area per hour.}$$

Example The filtration rate can be used to calculate the required filtration area as follows. Assume 17.3 dry tonnes per hour flotation concentrate has to be processed, and plant availability is set at 90% to allow sufficient time for maintenance. Then the filtration area is calculated as

$$\text{Total filter area required} = \frac{17.3 \text{ tph} \times 1000 \text{ kg}}{324 \text{ kgm}^{-2}\text{h}^{-1} \times 90\%} = 59.32 \text{ m}^2$$

This area can be supplied for example, by one 60 m^2 filter or two 30 m^2 filters. The former would be the more economical, but the latter could provide greater operational flexibility. The decision will be influenced by factors including plant operating philosophy and available space.

Similar calculations can be performed using chamber volume instead of filter area to calculate kg dry solids per cubic metre (or litre) of chamber volume per hour.

The end of feeding, cake pressing and air blowing are usually indicated by a significant decrease in filtrate flow, and continuous monitoring of cumulative filtrate volume will help determine the end point. A mass balance can be calculated for any time in the filtration cycle, and from it the effective cake moisture and filtration rate at that time.

Data logging to computer can simplify analysis and understanding of the filtration process, and can produce the mass balance automatically. Figure 5.21 shows the screen of a data acquisition computer during testing of a relatively fast-filtering and incompressible solid on a 0.1 m^2 laboratory tower press. Figure 5.22 shows a data plot from for a large industrial filter dewatering a similar material and showing similar filtration characteristics.

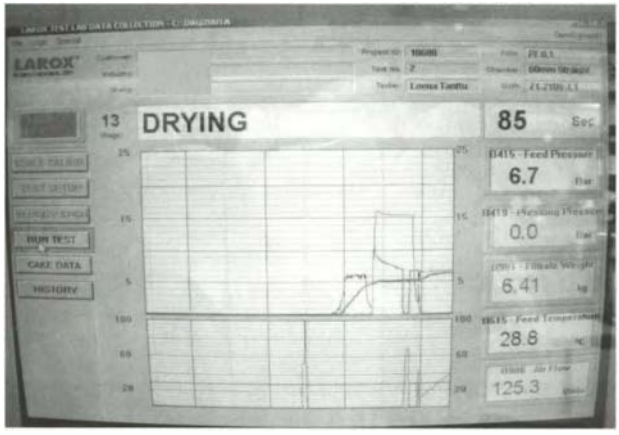

Figure 5.21 Screen of data capture computer during testing with 0.1 m^2 filter

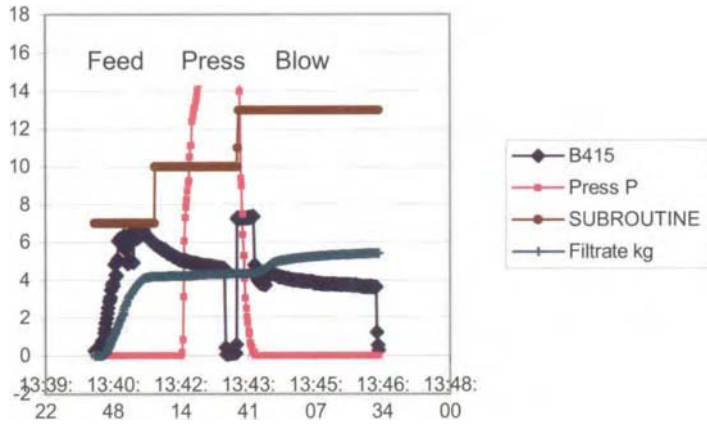

Figure 5.22 Data from an industrial filter showing similarity to filtration test

5.5 Integrating the filter into the flow sheet

Pressure filters do not usually operate in isolation. They must be integrated into a plant flow sheet, and match the upstream and downstream processes. Even if the most suitable type and correct size of filter has been selected, it will not perform effectively if it has incorrectly dimensioned ancillary equipment, or if the plant is badly laid out. The design for each plant is unique, but general guidelines are available in the literature (Coulson *et al*, 1983; Ericson and Blois, 2002; Townsend 2001). The recommended design sequence is to start with a general concept for the whole plant, ensuring good overall layout with seamless interfaces between sequential process stages. A detailed design for the filter plant can then be developed, starting with the ideal, and moving towards what is practical within the constraints of the budget. Designs should retain sufficient flexibility to accommodate (inevitable) project specification changes. The evolution of three-dimensional computer aided drafting has made it simpler to evaluate ease of access and operability of filtration plants as shown in Figure 5.23.

Figure 5.23 shows a simple plant with a lot of space, and the relative positions of key equipment is easy to see. 3D CAD is particularly helpful in obtaining the best layout when space or budget is limited.

All filter plant designs are subject to general structural, mechanical and electrical standards for the location and the industry, and equipment may have to be stamped to certify compliance. Special considerations are needed for filter plants located in seismic regions, or in plant areas designated as hazardous due to the risk of explosion. Pressure filtration plants for food or pharmaceutical products will normally have to

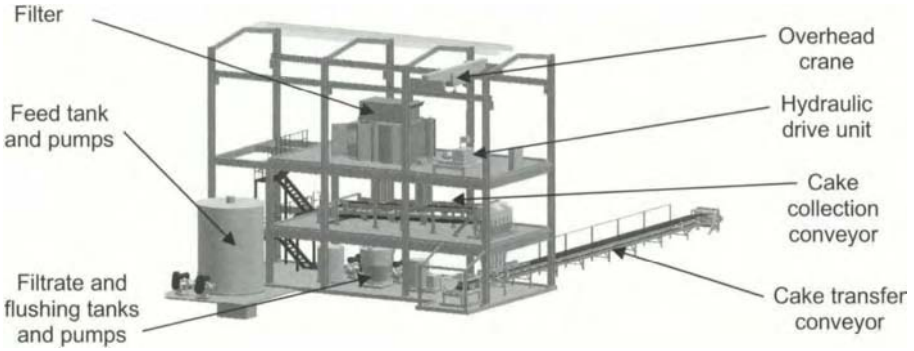

Figure 5.23 3D CAD drawing of a presure filter plant

comply with good manufacturing practice and have facilities for cleaning or sterilising-in-place (SIP) of filters, other equipment, and process pipelines.

General process engineering considerations for pressure filtration plants are discussed below.

5.5.1 Filter elevation

The elevation of the pressure filter above ancillary equipment such as slurry feed tanks and pumps has a significant impact on structural costs, ancillary selection and power consumption. Materials handling considerations usually dictate the filter elevation. Where filter cake is discharged to conveyors, every transfer point will add to elevation. If the cake is discharged to a blender or dryer, the size of that equipment, and its location relative to the filter will also influence filter elevation.

5.5.2 Feed preparation

When required, coagulant and flocculant systems should be designed for easy delivery of reagents, automatic reagent make-up where possible, accurate dosing, and adequate residence time.

Higher solids concentration in filter feed increases filtration rate and generally produces clearer initial filtrates, and filter feed slurries are often thickened. Designs should eliminate unnecessary dilution of filter feed from for example, pump gland service water.

5.5.3 Trash removal

Nuts and bolts, scaling from tank walls, cable ties, rubber gloves and other items can accidentally find their way into process streams, and

can block pipelines or filter feed ports resulting in an unfilled chamber, differential pressure between adjacent chambers, and subsequent damage. Pressure filter feed systems should therefore include screening to remove trash. A self-cleaning external screen discharging trash to a container with minimum slurry loss is ideal, and some filters have internal self-cleaning screens in their feed manifolds.

5.5.4 Surge capacity

Pressure filters are batch units, but are often incorporated into a continuous process. Surge capacity must be provided upstream to allow feed slurry to accumulate while the filter is processing a batch. The capacity of the tank should allow sufficient residence time to accommodate process fluctuations, and also to complete simple maintenance tasks without stopping the process. Feed slurry tanks are usually agitated to maintain an homogenous suspension, and the impeller should be chosen to give adequate suspension without damaging flocs or particles. The level in the surge tank can vary significantly, and the net positive suction head for the pump defines the minimum level. Feed tanks should be equipped with drain valves and an isolation valve in the suction line to permit the pump to be replaced for maintenance.

Modern pressure filters have short discharge times and high instantaneous cake discharge mass flows. When the cake is fed to a downstream process such as drying, the flow must be modulated to provide a constant feed rate to the dryer. This can be achieved through an intermediate cake storage vessel, if the materials handling properties are suitable and the cake will not bridge or otherwise block the bin. Alternatively, the cake may be discharged to a slow moving collection conveyor discharging to a variable speed conveyor controlled by a weightomer to give a constant discharge rate.

5.5.5 Filter feed pump

Plate and frame and recessed chambers may use positive displacement pumps, but membrane filter presses, tower presses, and tube presses are filled by centrifugal pumps, and rely on their membranes to generate high pressure for cake compression. This section will consider only centrifugal pumps.

The centrifugal feed pump will usually run only during filter feeding, and is started and stopped by the programmable logic controller (PLC) managing the filter's sequence of operation. In special cases the feed pump may run continuously with a recycle to the feed tank, and automatic valves to divert the slurry to feed the filter when required.

For stop/start filter feeding, the pump will only run for around 20% of the complete filtration cycle, and as a consequence, instantaneous flow rates are quite high, and larger pumps are needed than suggested by the average plant throughput.

The pump runs against increasing head as the filter cake builds, and an understanding of the system curve is important for correct pump sizing. Once the chamber is about half full the pressure will rise as the cake resistance increases, causing the operating point to move back up the pump curve. At the end of the filtration stage the pump will be operating at close to full pressure and the flow will have decreased to 10 to 40% of the initial flow rate.

At the start of pumping the uncontrolled flow rate may be too high and high velocities will cause wear when processing abrasive slurries. In such cases a variable speed drive pump should be used, starting slow and increasing pump speed as system head increases. To calculate the flow, the frictional losses for the pipeline and static head need to be known. The resultant curve should then be drawn onto the pump curve for the proposed pump. The intersection of the system curve with the pump curve at the proposed speed will then determine the maximum feed flow rate. Where large feed tanks are used, the flow rate should be checked with full and empty tank levels. With flat system curves there can be a significant change in flow with changes in tank level. Similarly, where there are significant changes in feed density, the flow with minimum and maximum densities should be checked. Once the maximum flow rate has been determined the NPSH required should be checked against the NPSH available with the proposed suction pipe and tank design.

For high capacity filtration plants with multiple filters, it is often possible to sequence the batch operation so that no filters are on the same part of the filtration cycle simultaneously. It is then possible to share ancillary equipment such as feed pumps between multiple units and reduce capital cost.

5.5.6 Feed manifold flushing and core blowing

All pressure filters incorporate manifold and core flushing and/or blowing to remove slurry from the pipelines in and around the filter, and to clear any potential blockages of feed ports into chambers. The procedure also removes residual slurry before air blowing, because solids carried at high velocity by the compressed air can be extremely abrasive. The flushing sequence varies between different types of filter and can be tailored for special conditions. Flushing water pressure should be higher than maximum feed pressure, and a dedicated

flushing water tank and pump are required. Depending on the application, process water or recycled filtrate can be used for flushing.

5.5.7 Cake compression or "pressing"

Some filters use high pressure water and others compressed air to inflate the membranes and compress the cakes.

Water pressing is commonly a closed system consisting of water storage tank, high-pressure pump, pressure regulating valve, and a three way valve to direct the water flow. During pressing water is pumped via the three-way valve to the back of the membranes to compress the cake. At the end of pressing the pump stops, and the three-way valve diverts the water back to the tank by gravity.

Air pressing systems consist of a compressor, oil filter and air receiver rated for maximum pressure, with pressure control and pressure relief safety valve(s) on the receiver discharge. Screw compressors are used for larger filters and piston compressors for smaller filters. For smaller installations a booster compressor can be used taking its feed from the plant air or other lower pressure supply. An air receiver is used because air flow is intermittent and occurs at high rate for short periods. The pressing air system is required to inflate the membranes as quickly as possible and maintain the required pressure for the duration of cake pressing. After the initial inrush, the flow of pressing air will be equal to that of the filtrate produced during cake compression. The volume of air per pressing is equal to volume of the empty space above the compressed filter cakes in all the filter chambers, and may be calculated from filtration area multiplied by (chamber thickness minus cake thickness) corrected from the pressing pressure to Nm^3. The size of compressor should be selected so that it runs almost continuously to fully charge the receiver before the next pressing. A safety factor is usually applied to compressor sizing, and special allowance must be made for operation at high altitude.

At the end of cake compression, all the pressing air that has inflated the membranes is vented to atmosphere in a few seconds. Because of the vented air's expansion and high velocity, the exhaust pipeline should be free of obstructions, may have to be lagged for sound reduction, and should discharge outside the building through a sound muffler.

5.5.8 Cake drying by air blowing

After cake compression, air typically between 6 and 10 bar g is blown through the filter cake to achieve final drying. At the start of air

blowing, a threshold pressure is required to displace residual moisture held between solid particles by capillary forces, and air flow rate is low. Once moisture has been displaced from the pores between solid particles, air flow rate increases and will increase further if cakes shrink and crack. The air blowing time and flow rate through the cake are governed by the cake properties and by earlier stages of filtration, and should be evaluated during test work. As air blowing is usually the most energy intensive phase of the filtration cycle, and should be optimised and controlled during operation.

Blowing air flow rate through the cake is measured as $Nm^3\ m^{-2}\ min^{-1}$ during testing and used to calculate air requirement for full scale operation. For small filters, drying air may possibly be taken directly from plant air supply with simple on/off control. However, a dedicated compressor and air receiver are recommended. Larger filter plants with potentially high air consumption may justify fully automatic control of blowing air pressure and flow. For some filter installations it may be possible to use a single compressor to supply both pressing and blowing air by providing a take off from the pressing air receiver via a pressure regulator to the blowing air receiver.

5.5.9 Filtrate handling

Filtrate flow rate varies greatly during filtration and pressing, from a maximum at the start of slurry feeding, to almost nil at the end of pressing. During cake blowing, filtrate is combined with the drying air exhaust in a two-phase flow, and the filtrate pipeline should be designed accordingly taking note of velocity. The filtrate can contain suspended solids at the start of feed pumping and during air blowing, and this should be taken into consideration when selecting pipe routing and piping materials to minimise abrasive wear. A cyclone air separator is used to remove filtrate from blowing air, and vent the air to atmosphere. The base of the separator cone acts as a filtrate surge tank to accommodate the variations in filtrate flow during the cycle, and permits the use of a smaller, fixed-speed pump rated for the average filtrate flow. The filtrate pipeline should be open to atmosphere, be self-draining to the filtrate separator/tank, and avoid "U" bends or other dead spots that can cause back pressure at the filter.

5.5.10 Filter cake handling

Filter presses of all types discharge cakes along the whole length of the plate pack. Single sided filtration tower presses discharge at the two ends of their plate packs, but double sided tower presses discharge only

on one side. Tube presses discharge immediately below the tube, but as installations usually have multiple tubes cake handling is designed for the full set. The method and speed of cake discharge must be taken into account in plant design.

In the simplest cases, pressure filters discharge direct to a stockpile or other storage directly below. The filter should have "bomb bay doors" or similar protection to prevent leakage during filtration, or water from routine cleaning, from falling onto the stockpile. Similarly, the operating floor should be designed to prevent any wet spillage or hose-down falling onto the stored cake.

The discharge time varies with different types of filter, from many minutes with a manually operated filter press, to a few seconds with an automatic tower press. When filters discharge to conveyors, the conveyor must be dimensioned to accommodate the instantaneous discharge rate. The conveyor must also be designed to accommodate the impact of the falling cakes, and to slope away from its direction of travel to drain any water spillage away from dry cake storage. High instantaneous cake discharge rates create air flow, and the cake handling system should be designed for dust removal and ventilation. Filter cake is broken up and fresh surfaces contact air at conveyor transfer points, resulting in further cake moisture reduction, especially in arid climates.

Variable-speed collection conveyors running beneath the filter can modulate the instantaneous cake discharge to a nearly constant continuous flow. The collection conveyor should run at high speed during filter discharge to spread the cake evenly along its length, and slow to a "crawling" speed immediately after discharge.

5.5.11 Cloth washing

Depending upon the type of filter, water can be sprayed onto the filter cloths at pressures ranging from 2 bar g to 100 bar g to remove adhering and embedded solids and salts that would otherwise accumulate and progressively blind the cloth and reduce filter performance. Cloth wash water should be of high quality with low suspended and dissolved solids that could block spray nozzles. Heated cloth wash water may be beneficial in processes where cloths may be blinded by crystallising salts.

5.6 Plant operating considerations

The most important plant operating consideration is to understand the construction and operating principles of the filter. This can be provided

through adequate training of operating and maintenance staff, and it is helpful if an enthusiastic employee becomes a "filter champion" for the plant. Knowledge of the filter and overall filter plant design will decrease the temptation to make ill-informed "improvements" that usually reduce filter efficiency and reliability. If plant capacity throughput is increased, a small expansion of the installed filter will usually prove more cost-effective than exceeding its capacity and reducing the time available for its maintenance.

Manufacturers provide manuals for their equipment and recommend maintenance schedules. It is important that these are followed, and if inadequate resources are available on site, manufacturers and contractors can provide maintenance services. Remote monitoring of filters is possible, as described in the section on automation. Maintenance is impossible without spare parts, and inventories must be managed. Accurate monitoring of spare part usage can highlight abnormal consumption, that could be overcome by operating or maintenance changes.

One of the most effective routine operating tasks is simply keeping the filter clean. A single-level concrete floor around the filter, graded to drainage points facilitates cleaning, and pipeline entry points through the operating floor should be sleeved to prevent spillage to the level below.

The plant design should provide good lighting and access to the filter for operation and maintenance. The design of the building should permit easy transfer of spare parts from the store to the filter, and removal of replaced components, some of which can be refurbished. A crane or hoist is usually needed to lift filter components and spare parts, and should have free access to all parts of the filter without interference by process pipelines or other obstructions.

Even fully automatic filters should be checked periodically for correct operation, and should be located in an accessible and frequently visited part of the plant.

Modern pressure filters are sophisticated yet robust, and when test work, plant design and filter selection are performed thoughtfully, will give good process performance and reliable, economic operation.

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Appendix Log Sheet for Recording of Test Data

TEST REPORT No.							
TEST FILTER AREA		m^2	**DATE**				
CUSTOMER			**BY**				
BUSINESS UNIT			**TEST LOCATION**				
SUSPENSION DESCRIPTION			**WASH LIQUID DESCRIPTION**				
SUSPENSION DESCRIPTION			**PARTICLE SIZE DISTRIBUTION**				
TEST NO.			1	2	3	4	5
THE TEST CYCLE FOR FILTER SIZING							
Process conditions							
Density of	slurry	kg/l					
	liquid in slurry	kg/l					
S.G of solids in	slurry	kg/dm^3					
Solids in slurry		%w/w					
Density of	wash liquid	kg/l					
Temperature of	slurry	°C					
	wash liquid	°C					
pH of	slurry						
	wash liquid						
Filtration parameters							
Duration of	pumping	min					
	pressing I	min					
	washing	min					
	pressing II	min					
	drying	min					
	technical time	min					
Calculated cycle time		min					
Measured process parameters during filtration tests							
Pressure of	slurry feed	bar					
	pressing I	bar					
	wash liquid	bar					
	pressing II	bar					
Quantity of slurry	**calculated**	l					
Quantity of filtrate during	pumping	l					
	pressing	l					
	washing	l					
	air drying	l					
	TOTAL	l					
Consumption of	wash liquid	l					
Air flow / air pressure	beginning	l/min / bar					
	at 1 min	l/min / bar					
	end	l/min / bar					
Temperature of	filtrate	°C					
	wash filtrate	°C					
pH of	filtrate						
	wash filtrate						
Process results							
Moisture in cake		% w/w					
Cake thickness		mm					
Wet cake weight		kg					
Dry cake weight	calculated	kg					
Wet cake S.G	calculated	kg/dm^3					
Filtration rate (dry solids)	calculated	kg/m^2h					
Filtration rate (filtrate)	calculated	l/m^2h					
Wash liquid consumption	calculated	m^3/ton D.S.					
Washing result							
Solids ratio in cake (vol/vol)	calculated	%					
Air in cake vs void volume	calculated	%					
Solids content in	filtrate	mg/l					
	wash filtrate	mg/l					
Filter cloth:							
Chamber thickness		mm					
Chamber type							
Remarks	Record cumulative filtrate volume versus time for each phase of filtration cycle						

6 Vacuum filters

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This chapter on vacuum filtration covers both batch and continuous discharge equipment as commonly employed in the chemical, process and minerals industry. There are many advantages in choosing vacuum filters in preference to pressure filters although, like all process filtration equipment, they do have limitations and these are discussed later in the chapter.

Batch vacuum filters, such as the tipping pan, are generally regarded as the simplest units, and are represented by an industrial version of the Buchner funnel in most laboratories. Continuous vacuum filters, for example, vacuum drum, belt or disc, can be likened to several Buchner funnels working in sequence to provide the appearance of continuous filtration. The basic test apparatus for vacuum filtration tests is therefore the Buchner funnel, or derivatives of it.

As with all scale-up work, it is important to use a test procedure and equipment that mimics the full-scale filter. Some simple, preliminary tests can be carried out to determine whether vacuum filtration is possible for the solid/liquid suspension under investigation. Following this, more extensive test work can be carried out.

The vacuum filters under discussion in this chapter will be the following types:

- single tipping pan
- rotary drum
- rotary disc
- horizontal belt
- multiple tipping pan
- table.

It will be assumed that, for all applications, cake filtration will be the controlling mechanism. The only exception to this will be the precoat rotary drum filter. This special case will be discussed later in the chapter.

Prior to undertaking any test work for vacuum filtration, it is important that some basic information on the solid/liquid suspension is available, in addition to the degree of filtration required, the mode of operation (batch or continuous), and the volumetric (or mass) flow rate of the suspension. This basic data defines the type of filter that can be used for the process and can avoid unnecessary test work and analysis. For example, if the solids within a suspension are coarse and rapid settling, then a horizontal filter would be considered. Furthermore, if the volumetric flow rate of this suspension was only 1 m^3/day, then a tipping pan filter would appear the more appropriate option, and test work can be geared accordingly towards this type of unit. Wakeman and Tarleton (1999) (see also Chapter 1) have developed software routines for equipment selection based on system and process parameters.

6.1 Advantages and limitations of vacuum filters

6.1.1 Vacuum filters – limitations

The operational and process limitations of vacuum filters should always be considered during the early stages of equipment selection, rather than establishing their unsuitability after installation. Emphasising the limitations precludes any misconceptions about performance and operation, and may redirect the investigation towards, for example, pressure filters. Vacuum filters and pressure filters are different approaches to the same problem – how to separate solids from liquids in as an efficient and economic method as possible. Both present a number of operational advantages and disadvantages. However, the criteria for selection are linked to both the filtration characteristics of the suspension and the process constraints of the system.

Vacuum filters utilise vacuum as the driving force to form and dewater a filter cake. By definition, this cannot exceed 1 atmosphere and can only be achieved by applying absolute vacuum. In practice, a vacuum of usually no more than 0.25 bar absolute is applied (i.e. –0.75 barg). As a consequence, vacuum filters are not usually employed in systems where the majority of particles are below approximately 5 microns. This is because cake bed resistance, r, is inversely proportional to the

square of the particle diameter, and 5 microns is generally considered as the lower limit for successful operation.

$$r \propto \left(\frac{1}{d_p}\right)^2 \tag{1}$$

The relationship in equation (1) is derived from the basic filtration equations and applies to ideal, single-sized spherical particles. In practice, no industrial system has such particles, but the basic relationship is generally valid and a good 'rule of thumb' when undertaking initial investigations. Particle shape and bed porosity can affect the resistance, as can many other subtle factors, so equation (1) is only applicable if other parameters remain constant. Nevertheless, equation (1) highlights the influence that particle size can have on a filtration system, and especially the difficulties that can arise where particle degradation and attrition occur.

Pressure filters can have significantly higher driving forces due to, for example, the feed pump, and are often used where the particles are sub 5 microns. In addition, the lower driving force in vacuum filters means that filtration rates are slower than for the equivalent area pressure filters.

Solvents forming part or all of the suspension liquid can often be unsuitable for use with vacuum, especially if they have low vapour pressures at the operating temperature. Similarly, solids (or liquids) that readily oxidise are not recommended for filtration by vacuum, since the majority of vacuum filters rely on an air flow to dry the filter cake. Particles that are compressible, or form compact, high resistance filter cakes are also generally unsuitable for vacuum filters. The exception to this is the precoat rotary drum filter which is unique in its ability to process ultrafine, gelatinous solids, and this is why it is used extensively in the food and pharmaceutical industries. More information on the precoat drum vacuum filter is given later in the chapter.

Filtrate discharge from pressure filters is often to atmosphere – usually directly to a filtrate collection tank. For vacuum filters, the filtrate must be separated from the entrained air prior to discharge, and this is via a suitably sized filtrate receiver (or vacuum vessel – for consistency, the term filtrate receiver will be adopted) for filtrate-air disengagement. The filtrate must be continuously or intermittently discharged from the filtrate receiver, either by a pump system or via a barometric leg.

6.1.2 Vacuum filters – advantages

Unlike pressure filters, most vacuum filters operate continuously, and require virtually no operator intervention during the normal operating cycle. There is no dead-time required for solids discharge and this, to some degree, makes up for the slower filtration rates exhibited by vacuum filters when compared with pressure filters of equivalent areas.

The lower driving force exerted by vacuum filters for solid/liquid separation can be advantageous for certain applications where particle deformation occurs. In such cases it can be beneficial to limit the force exerted onto the particles in order to maintain an open structure within the filter cake for dewatering.

With pressure filters, the filter cake forms within the confines of the unit, and the filter must be isolated and vented before the solids are discharged. If a pressure filter is installed on a continuous process then feed holding tanks will be required while the filter is discharging the solids, and this may also require a high degree of automation to ensure correct sequencing of valves. For a vacuum filter this is not the case since the filter cake is formed on the atmospheric side of the unit and cake discharge is relatively simple. In addition, it is generally easier to observe cake formation and discharge, as well as monitor the condition of the filter medium.

6.2 Descriptions of full-scale filters

6.2.1 Batch discharge filters

6.2.1.1 Single tipping pan filters

The tipping pan filter is one of the simplest types of vacuum filter available, see Figure 6.1. The filtering surface is usually of a 2:1 rectangular configuration with steep, outward tapering sides. The base of the pan consists of a drainage grid over which a filter medium is fixed. Beneath the drainage grid is a support cradle that provides rigidity to the pan, and the filtrate collection area. The cradle is fixed to a horizontal turn-over shaft with one half hollow for the removal of the filtrate. The tipping pan assembly is mounted on a support frame with a turn-over mechanism on one end and a rotary valve assembly on the other.

Tipping pan filters are particularly suited to coarse, crystalline solids that settle rapidly and/or materials which require extensive washing.

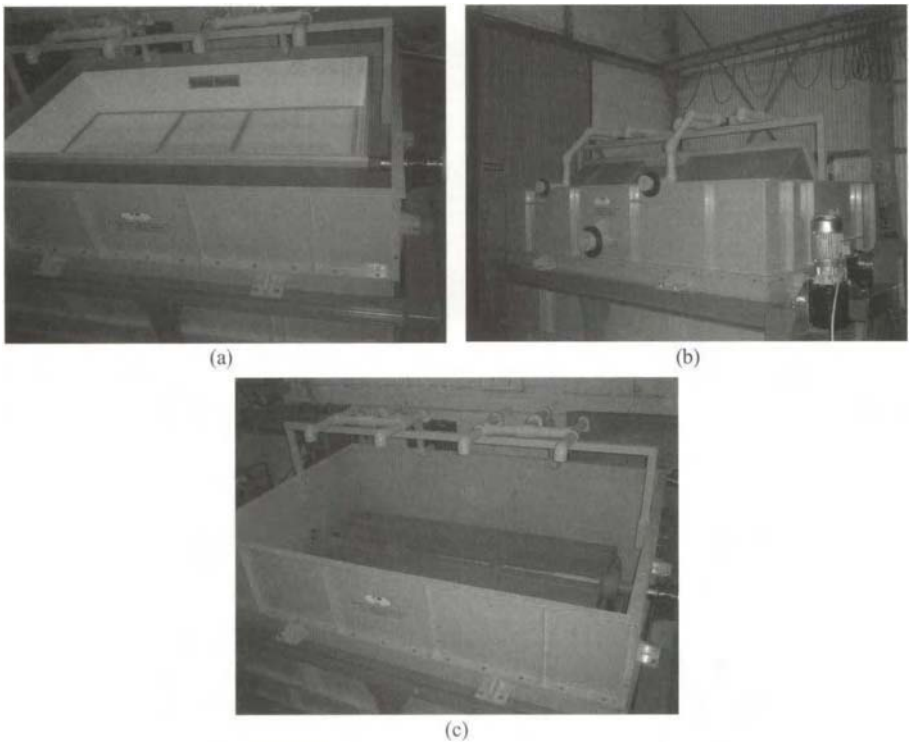

Figure 6.1 3 m² polypropylene tipping pan filter (hood removed): (a) pan in its upright position; (b) pan inverting; (c) inverted pan (Filtration Services Ltd)

This type of filter operates in batches and handles relatively small quantities of feed when compared with other vacuum filters.

With the pan in its upright position, the suspension is fed into the filtering surface via distribution pipes. At this point the vacuum can be isolated from the pan to promote solids settling, or applied to the pan for immediate filtration. When vacuum is applied to the pan, the solids are retained on the surface of the filter medium, and the filtrate is collected through the rotary valve. Once the filter cake has been formed it may be necessary to introduce a wash to displace the mother liquor. The washing and dewatering times within the process cycle can be modified to suit the process conditions.

Upon completion of the final dewatering cycle, the pan is inverted about its horizontal axis. The rotary valve isolates the vacuum source and low pressure compressed air can be introduced to the pan to aid solids discharge. The pan is then returned to its start position and the processing cycle can be repeated. The filtrate is collected and discharged from the filtrate receiver.

Single tipping pan filters can be from approximately 1 m^2 filtration area to about 3 m^2. Materials of construction range from steels through to plastics. Their simple design and operation makes them suitable for batch processing applications and process development.

The filling, washing, dewatering and discharge cycles can be automated on even the most basic unit. It is possible to sequence two or more pan filters to provide pseudo-continuous filtration. Multi-pan filters, where several pans are mounted on a carousel have been developed, although these have generally been superseded by horizontal belt filters.

6.2.2 Continuous discharge filters

6.2.2.1 Rotary drum vacuum filters

Rotary drum vacuum filters are a family of generic filter units used in a wide range of industries and applications, from food and pharmaceutical products through to general effluent treatment. The fundamental design has changed very little since 1872 when James and William Hart patented the basic drum filter. The standard drum filters are suited, in the main, to suspensions between 5 and 15% by weight and containing particles of between 5 and 200 microns. Low solids suspensions can be filtered using a precoated drum which is a special type of unit, discussed later in this section. The standard filter configuration includes a drum, trough, agitator, rotary valve and discharge mechanism. Filtration areas typically range from 1 m^2 to 80 m^2.

The drum is a hollow, horizontal-axis cylinder which is mounted on the filter trough support frame, see Figure 6.2. The drum surface is divided into a number of lateral panels that support the filter medium. For most drum configurations, these panels are connected, via drain-lines, to the rotary valve assembly – either by internal pipes (standard configuration) or pipes located at the drum ends (referred to as end-flow), and the interior of the drum is not subjected to vacuum. The drum surface is fitted with drainage grids, usually polypropylene or nylon, to allow a free flow of filtrate across the panel to the drain-lines, and is mounted on the trough frame such that approximately one third of the drum will be immersed in the solid/liquid suspension. At one end of the drum the horizontal shaft passes into the drum drive gearbox, the other end has the drain-line bundle connected to the rotary valve assembly. The drum rotational speed is variable between 0 and 2 revolutions per minute for most applications, and is dependent upon the filtration characteristics of the suspension.

Figure 6.2 3.5 m^2 scraper discharge rotary drum vacuum filter (Filtration Services Ltd)

The vast majority of drum filters are bottom feed, which means that the suspension is fed into the filter trough and filtration takes place on the underside of the drum as it rotates. This type of filtration is gravity hindered, and therefore unsuitable for dense, rapid settling solids. The suspension level in the trough is referred to as the submergence – calculated as the percentage of drum area submerged at any one time. Typically, this can vary from between 25% and 75%, although the majority of filters are configured with 35 to 40% submergence levels. The submergence level is usually fixed for a given installation, although variable submergence filters are available. The most common way of fixing the submergence level is by using a trough overflow located diametrically opposite to the feed point. Level probes and switches can be used to regulate the liquid level in the trough, and these must be located in a quiescent zone.

Top feed drum filters (Figure 6.3) are available, but are not very common. The suspension has to be loaded onto the drum approximately 30° before top dead centre. As a result, the overall filtration cycle time is very short. This special type of filter is suited to rapid filtering, dense solids where continuous discharge is required and the available floor space is limited. More suitable filters for this type of application would be horizontal belt filters or multiple tipping pans.

As the filtration for bottom fed filters is gravity hindered, it is important to gently agitate the suspension within the trough to prevent sedimentation and maintain a homogenous feed. To achieve this, a cradle

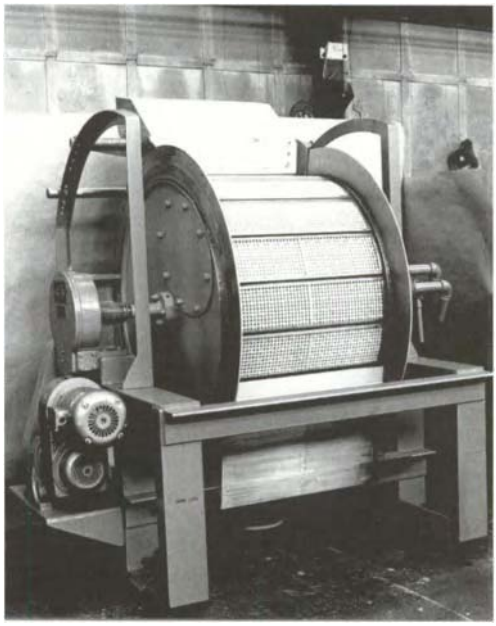

Figure 6.3 Top feed rotary drum vacuum filter (Filtration Services Ltd)

agitator is used, and it oscillates close to the base of the trough. Agitator speed is typically between 12 and 18 oscillations per minute so as not to hinder cake formation. The agitator is formed from two side arm assemblies that are joined by horizontal 'ploughs', and the agitator assembly is pivoted just above the drum shaft. The agitator assembly is driven by push rods or cranks connected to the agitator drive.

The rotary valve assembly is where the drain-lines are connected to the vacuum source. Information about the rotary valve is given in Section 6.2.3.1.

There are a number of discharge mechanisms available for drum filters, and the selection of the discharge is dependent upon the type of filter cake formed. These are:

- Scraper Knife
- Belt
- String
- Roller
- Precoat.

The most popular discharge type is the scraper knife system – a misnomer insofar as the round tipped plastic blade is to deflect the filter cake as it discharges from the drum surface, and should neither scrape the drum surface nor cut the cake away. The discharge assembly is mounted on the trough support frame and the spring-loaded blade floats against the drum surface, following the drum contours, see Figure 6.4. Scraper discharge systems are used when the filter cake is easily discharged from the filter medium. At the point of discharge the drum panel is isolated from the vacuum and low pressure air is used to both break the vacuum and partially inflate the filter medium. This is termed as the blow-back, and moves the cloth towards the discharge blade. The filter cake is then dislodged and deflected from the drum surface, thereby refreshing the filter cloth for another cycle. The pressure of the blow-back air depends on how the filter cloth is held onto the drum surface. For caulked panels, the blow-back air should be at very low pressure, and just sufficient to inflate the panel and dislodge the filter cake. Some filters use wire-winding to fit the filter cloth, and these systems can employ much higher blow-back air pressures to discharge the filter cake.

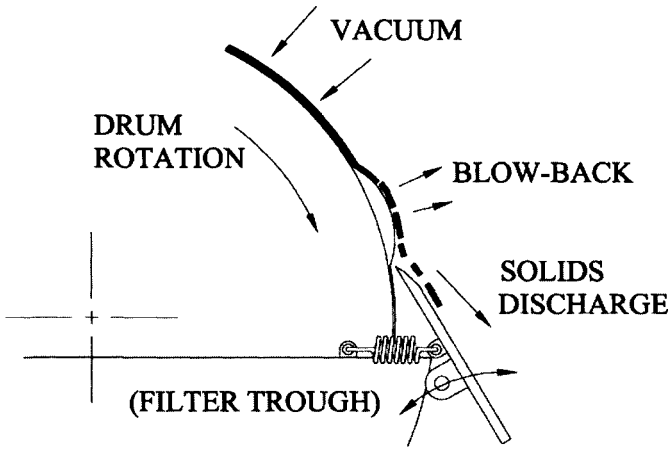

Figure 6.4 Example of a scraper discharge assembly

Belt discharge filters use a continuous filter medium that covers only part of the drum periphery. At the point of cake discharge, the belt is released from the drum surface and passes over a number of small-diameter rollers – usually three, see Figure 6.5. The filter cake is discharged at the first roller. The second and third rollers re-route the belt back to the drum surface and are used to provide belt tensioning

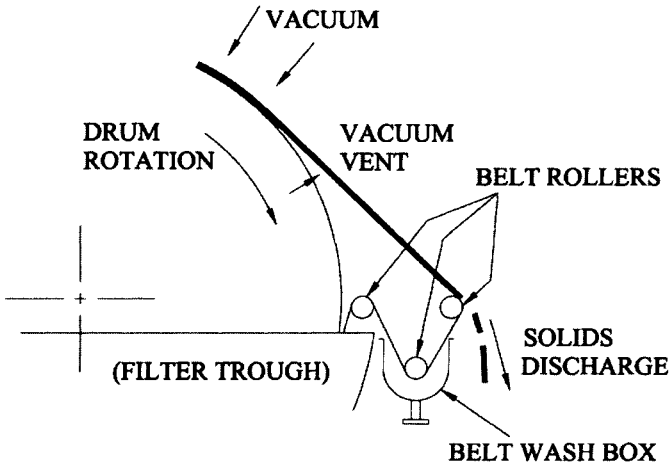

Figure 6.5 Example of a belt discharge assembly

and tracking. Intensive belt wash is usually carried out at the second roller, otherwise residual filter cake could accumulate on the surface of the third roller and affect the belt alignment. Belt filters are often used when the filter cake is thin (less than 3 mm thick) and has a tendency to adhere to the filter medium, although they can also be used to convey more easily filtered solids to a common discharge point. The other main reason for using belt filters is their facility for intense washing of the filter medium. Wash pipes are often fitted between the discharge roller and the first return roller. The primary wash pipe is located behind the filter medium, providing a back flow of clean wash liquor through the filtering surface.

String discharge is a technique used to convey the cake from the drum surface, similar to belt discharge, see Figure 6.6. The filter is fitted with a number of individually fitted strings that pass around the drum periphery. The strings are equally spaced across the drum surface, and are aligned using a discharge comb. String discharge is particularly good for mechanically strong yet sticky filter cakes.

Roll discharge is also suitable for sticky cakes and uses a driven roller mounted on the drum surface, rotating in the same contact direction, to transfer the cake from the drum surface to the discharge roller, see Figure 6.7. The roller periphery speed is usually higher than the drum speed, and this difference is used to shear the solids from the drum. Once the solids have transferred to the discharge roller, they are then continuously removed from the roller by a discharge blade.

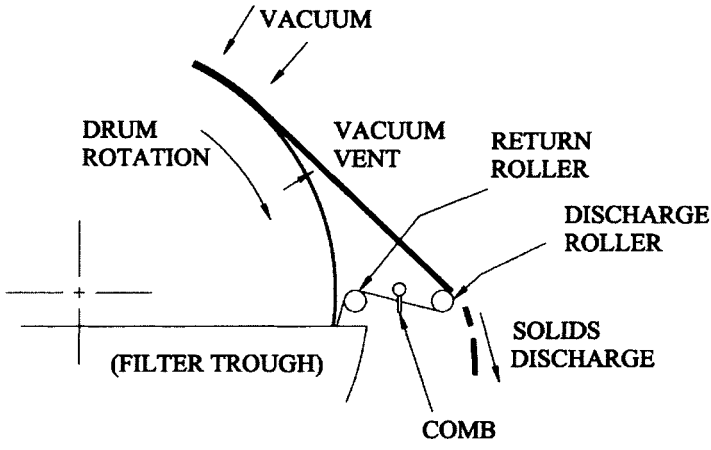

Figure 6.6 Example of a string discharge assembly

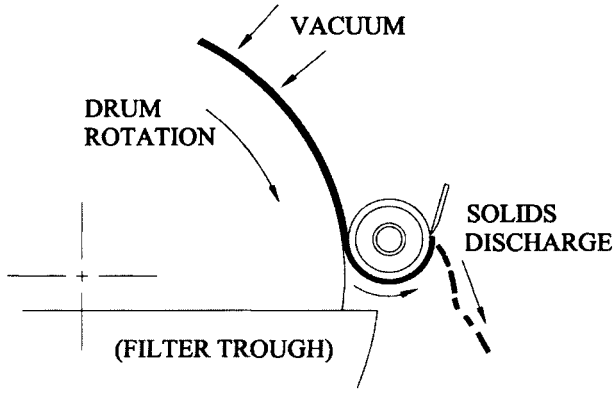

Figure 6.7 Example of a roller discharge assembly

Precoat discharge systems are unique to rotary drum vacuum filters (see Figure 6.8) and are used when one or more of the following conditions apply:

- The filtrate is the product, and its clarity is of utmost importance (as in the food and pharmaceutical industries);
- The solids are of low concentration, and do not readily form a filter cake;
- The solids are gelatinous and have a tendency to blind the filter medium;

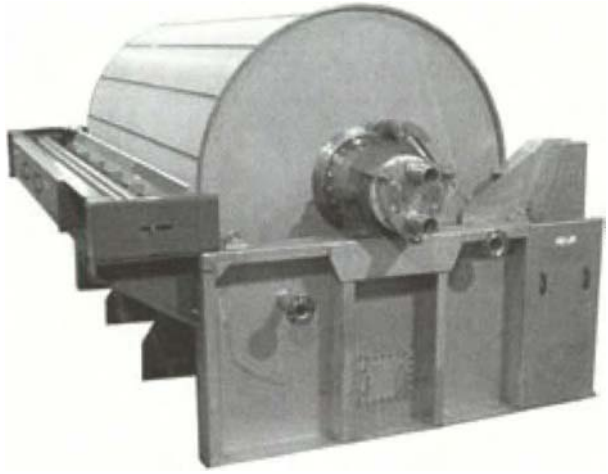

Figure 6.8 32 m² precoat discharge rotary drum vacuum filter (Filtration Services Ltd)

- The solids are typically less than 5 microns;
- The solids are not to be recovered as the product.

Prior to filtering any of the process suspension, the drum is coated with a sacrificial filter medium – the precoat, see Figure 6.9. This can be in the form of diatomaceous earth, perlite, cellulose or wood flour. A precoat suspension is made up in a separate feed tank and filtered onto a clean drum, forming an even, thick, permeable bed up to 100 mm deep. The process suspension is then filtered through the precoat, producing a bright, solids-free filtrate. The solids are retained on the

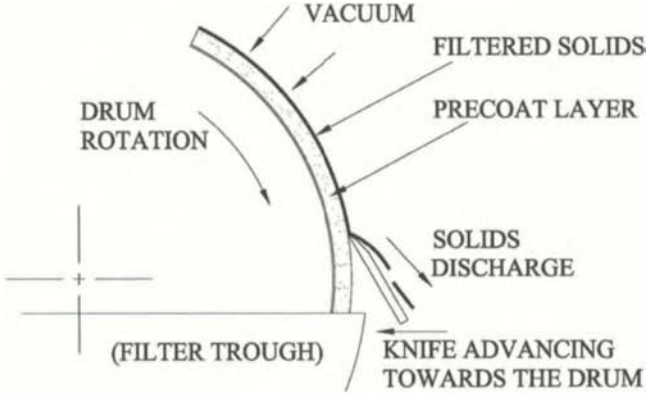

Figure 6.9 Example of precoat discharge assembly

surface of the precoat as a thin skin of contaminants. As the drum rotates, these contaminants are continuously peeled from the surface of the precoat bed by an advancing knife system. The knife removes the solids plus a very thin layer of precoat, thereby refreshing the filter medium. The knife cut depth must be sufficiently deep so as to refresh the precoat bed, yet not too deep as to waste virgin precoat. Knife advance rates are set in tens of micrometres per drum revolution, allowing the precoat bed to last a number of days between re-application. It is therefore apparent that the precoat drum filter is not a true continuous filtration unit. However, the time required to re-coat the drum is short when compared with the total operating time of the filter.

Many knife advance systems are linked to drum rotation, such that any variation in drum speed will be reflected by the knife advance rate. The synchronous system can be either by mechanical linkage or electronic signals. Either way, this type of system provides relatively efficient usage of precoat when compared to the independent drive mechanisms. In 1996 Filtration Services Ltd developed a fully synchronous automatic knife advance system (A.K.A.S.) which has been used to optimise precoat usage. The system is based on a knife drive motor receiving a predefined signal on each complete revolution of the drum. Knife advance rates can be set in increments of 5 microns per drum revolution, providing exceptional accuracy and controllability. Walker (2003) proposed a technique based on the A.K.A.S. for determining the operational parameters to optimise precoat usage based on expected bed life, precoat cost, filtration rate, drum speed and knife advance rate.

The overall filtration cycle of a drum filter can be broken down into a number of stages:

- Filtration (form)
- Initial dewatering
- Final dewatering
- Discharge
- Dead-time.

Depending on the bridge configuration within the rotary valve, the initial and final dewatering stages can be combined. The dead-time is the period between cake discharge and the beginning of the next filtration cycle. The duration of each stage can be approximated as follows:

$$\text{Form Time, } T_F = \frac{60\sigma}{R} \tag{2}$$

$$\text{Dewatering Time, } T_D = \frac{46.5}{R} - T_F \tag{3}$$

and the dewatering time can be subdivided as follows:

$$\text{Initial Dewatering Time, } T_{ID} = 0.22T_D \tag{4}$$

$$\text{Cake Wash Time, } T_W = 0.53T_D \tag{5}$$

$$\text{Final Dewatering Time, } T_{FD} = 0.25T_D \tag{6}$$

The total filtration cycle time is:

$$\text{Total Filtration Cycle Time, } T_C = \frac{T_F}{\sigma} \tag{7}$$

Equations (2) to (7) are for guidance only. The relative positions of the discharge assemblies on the drum periphery vary between filter manufacturers, so the timings within the filtration cycle are not constant for generic filter types. Furthermore, rotary valve bridge configurations can be set to provide delayed filtration (for example, bottom-dead-centre pick-up). Similarly, the sub-division of the dewatering time will vary in accordance with the dewatering characteristics of the filter cake, but cake wash should only be carried out above where the filter cake has lost the visual surface moisture and, ideally, before cake cracking occurs. This set of equations can be used to indicate the expected duration of each part of the cycle for a typical drum filter installation.

As part of the overall filtration cycle, cake washing can be used to displace residual mother liquor from the filter cake. Cake washing can take place between the point at which the filter cake first emerges from the suspension, but usually happens after the cake surface has dewatered (changing from a gloss to a matt appearance), to approximately top-dead-centre. The amount of wash water that can be applied is determined by three main factors. The first is the equivalent number of liquor displacements required (i.e. to achieve a desired final cake purity), the second is the stability of the cake whilst being washed, and finally the residual moisture content of the filter cake at discharge. Care should be taken not to apply excess wash liquor (i.e. more than can be drawn through the filter cake) since this will run back into the trough and dilute the feed suspension.

In some instances it is beneficial to add a cake compression assembly to the drum filter, but this is unsuitable for precoat types. Cake

compression has a number of benefits, for example it will reduce the residual moisture content of the filter cake, prevent excessive cake cracking (which can sometimes lead to vacuum losses), and enable more rigorous cake washing by using the compression belt to protect and stabilise the filter cake. It is not always necessary to apply heavy compression to the filter cake – sometimes the compression belt itself is sufficient. For soft, thick filter cakes it is recommended that progressive compression is applied so as not to extrude the damp cake from under the belt at the first roller. The addition of a compression assembly to a drum filter can be expensive, and regular maintenance is essential to ensure correct operation. Nevertheless, it brings significant benefits in that it can be used to produce a drier, better washed filter cake.

6.2.2.2 Rotary disc filters

Rotary disc filters, see Figure 6.10, are similar in many ways to scraper knife drum filters. Their main advantage over equivalent footprint drum filters is the significant increase in filtration area. Whereas for drum filters the filtration direction is against gravity, for disc filters it is perpendicular to gravity, i.e. sideways filtration. The filter is made up of a number of wedge-shaped segments fitted into a common manifold, and several manifolds on a horizontal, hollow shaft. Each segment is covered with a filter bag, and functions in a similar manner to the lateral panels on a drum filter. Disc filters are only bottom feed.

This type of filter is scraper discharge, and relies on the ability of the suspension to form a relatively thick filter cake that is easy to

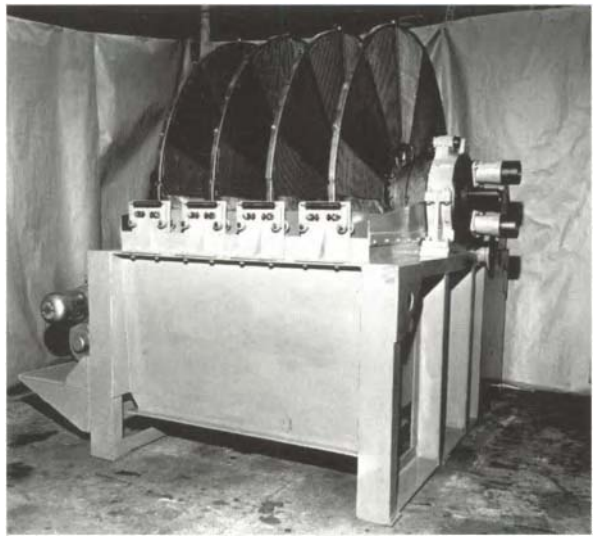

Figure 6.10 Rotary disc filter

discharge. The solids are discharged down narrow channels formed into the filter trough between the discs. Disc filters tend to be used in the minerals processing industries, and are now less frequently used in the general chemicals and process industries. Cake washing and compression are not possible on disc filters.

6.2.2.3 Horizontal belt filters

The horizontal belt filter, see Figure 6.11, is a gravity-aided, top feed filter. Whilst it has a large footprint for its working filtration area, it has several advantages over both drum and disc filters. The filter is made up of a number of horizontal vacuum chambers over which is fitted a continuous filter belt. As the belt moves over the chambers, vacuum is applied to dewater the suspension and form the filter cake. The speed of the belt can be adjusted to provide sufficient dwell time to dewater the filter cake.

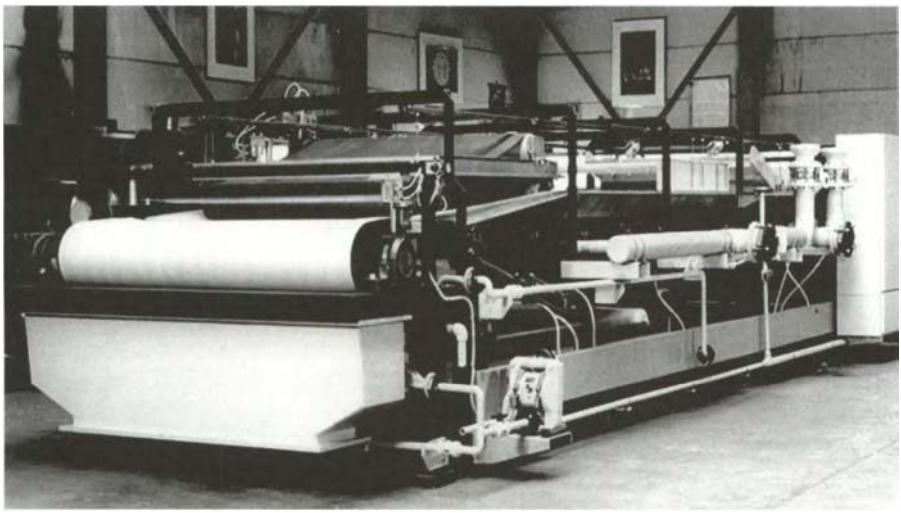

Figure 6.11 Horizontal belt vacuum filter with vacuum seal belt (VSB) (Larox Pannevis)

Two basic techniques have been developed to advance the filter belt while under vacuum. The first, and simplest, uses fixed position vacuum chambers beneath the filter belt. The belt is stationary when vacuum is applied, and it is necessary to fully vent the chambers to release the vacuum before the belt can be advanced. The belt is then indexed forward and the vacuum re-applied. In some applications, this technique can cause undue wear and tear on the underside of the filter belt.

An alternative technique is the Larox Pannevis RT vacuum filter belt introduced in 1967, which works on the principle of a continuously

moving filter belt supported on reciprocating vacuum chambers, see Figure 6.12. The filter cloth is driven forward by a variable speed motor on the drive roller. The vacuum chamber, or support tray, is drawn along with the belt by the grip created by the applied vacuum. At the end of the forward stroke a sensor is activated which isolates and vents the vacuum, thereby allowing the support tray to return to its starting position where vacuum is re-applied and the cycle repeated. The filter belt continues to advance during the return stroke of the support tray, ensuring continuous discharge of the filter cake. The return stroke time is only a few seconds, so it is not necessary to interrupt the slurry feed or wash flows. This is a mechanically complex filter when compared with the fixed chamber system, but is more suitable for larger installations where the cake weight would hinder the belt dragging system.

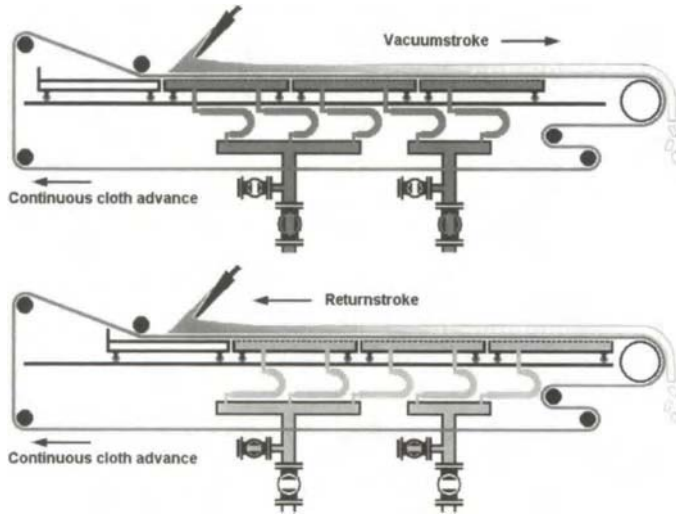

Figure 6.12 Principle of the Larox Pannevis RT belt filter

It is possible to carry out a number of process steps on a belt filter by simply increasing the machine length. These may include:

- Multi-stage cake washing
- Chemical reactions
- Cake vibration (for dewatering thixotropic cakes – thixotropic filter cakes appear solid at discharge, but become semi-fluid when stored or mechanically worked due to the entrapped liquid within the cake structure. Cake vibration on the filter surface helps to release

additional liquid and produces a drier and more stable filter cake on discharge.)

- Mechanical pressing
- Thermal drying.

Horizontal belt filters are available in many sizes, and only limited by available floor space. The arrangement of chambers makes them ideally suited for extensive cake washing, particularly counter current. Enclosed belt filters are also available for specialist applications.

The main limitation of horizontal belt filters is their inability to handle very fine, slow filtering suspensions. Cake formation and dewatering can be poor, and this leads to the filter discharging wet filter cake. Like drum and disc filters, belt filters can be used for some fine suspensions provided the belt length and/or indexing times are suitably adjusted.

Horizontal vacuum belt filters are very versatile in their ability to wash filter cakes, and there are a number of possibilities that can be used either singly or in combination:

- Co-current – the simplest displacement wash, usually with one volume of wash liquor.
- Counter-current – often a smaller volume of wash liquor passing through the cake several times in the opposite direction to the cake travel.
- Reflux – circulation of a large volume of wash liquor over the same zone with a small bleed-off and fresh wash liquor make-up.
- Reslurry – breaking up of the filter cake with sprays over a zone without vacuum, followed by a vacuum zone.

Counter-current and reflux wash conditions are shown in Figure 6.13.

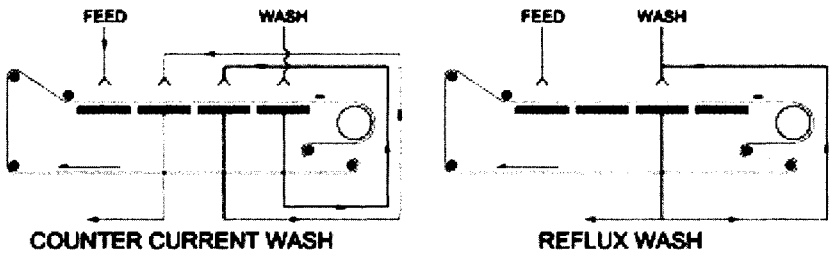

Figure 6.13 Counter-current and reflux wash on a horizontal belt filter

In order to remove additional liquid from the filter cake, compression can be applied, either by an impervious belt on the top of the filter cake – generating compression by the vacuum seal – or by rollers applying additional squeezing pressure. The Larox Pannevis VSB filter is an example of horizontal belt filter with the cake compression facility. Both of these compression techniques can be mimicked by laboratory tests.

Additional handling of the solids can sometimes be avoided by drying the filter cake while still on the filter, thereby providing a dry cake discharge. This can be achieved by thermal drying which uses hot air, up to 120°C, to produce dry cakes down to 0.1% $^{w}/_{w}$ moisture. Due to differences in latent heat it takes longer to dry aqueous based cakes than solvent based cakes. The cake structure must be open to air flow to ensure even drying throughout the depth of the cake. For low permeability filter cakes infra-red drying can be considered.

Cake discharge from horizontal filters occurs at the belt return roller. Here the vacuum is vented so that the cake is loose on the belt surface. As the belt moves around the tight wrap of the return roller, the solids fall away. For instances where there is some cake adhesion, a deflector blade can be fitted.

6.2.2.4 Multiple tipping pan filters

A simple extension to the tipping pan filter concept is the multiple tipping or tilting pan filter, Figure 6.14. In this case there are several pans mounted on a carousel. As the pans move around the central hub,

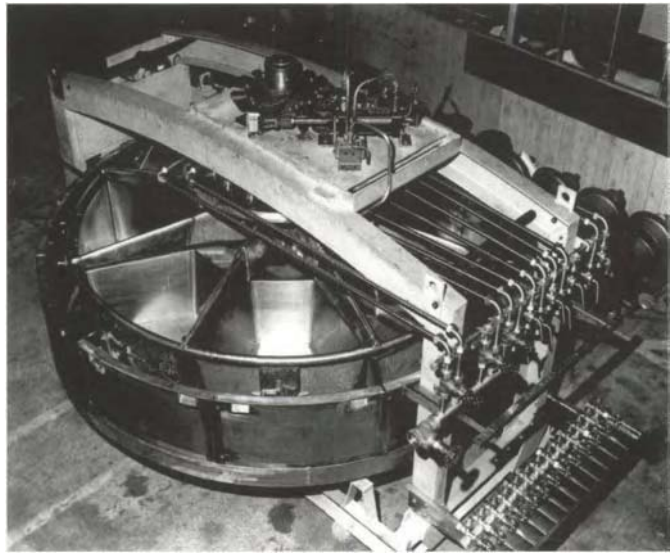

Figure 6.14 Multiple tipping pan filter (Filtration Services Ltd)

they go through the standard filtration cycle of filling, cake formation and dewatering (and washing if required). At the point of discharge, the pan inverts – usually by a mechanical cam – to discharge the solids.

In recent years this type of filter has been replaced by horizontal belt filters.

6.2.2.5 Table filters

Table filters (Figure 6.15) share many features of the multiple tipping pan filters. The table surface is made of a series of fixed segments that rotate around a central hub. Cake discharge from the table is by a screw conveyor. This type of filter is suitable for very fast settling and easily dewatered solids. Again, in recent years, this type of filter has generally been replaced by horizontal belt filters.

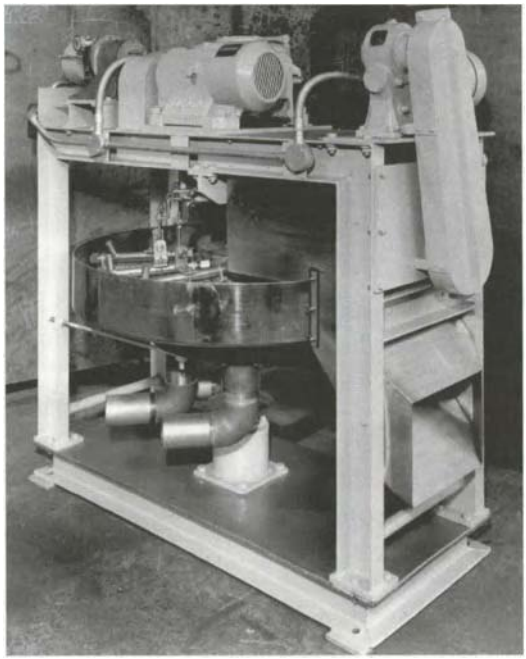

Figure 6.15 Pilot-scale table filter (Filtration Services Ltd)

6.2.3 Common components

Vacuum filters of different types and manufacture operate in essentially the same manner. Each filter is made up of generic parts. It is the assembly of these generic parts that differentiates one machine from another. These parts are described below:

- the filter medium for the physical separation of the solids from the carrying liquid;
- the filter medium support, usually a polypropylene or nylon drainage mat, with suitable drainage channels on the filtrate side;
- drain-lines for the transportation of the filtrate and entrained air from the filter to a filtrate receiver;
- a filtrate receiver to disengage the air stream from the filtrate;
- a solids discharge system to remove the solids collected on the filter medium and thereby refresh the filtering surface;
- a vacuum source to provide the driving force for solid/liquid separation;
- a filtrate discharge system to continuously or intermittently evacuate the filtrate from the filtrate receiver.

Rotary (drum and disc), horizontal belt and pan filters utilise the same basic ideas in their construction and operation. The variations in design are as a result of their operating duties. Horizontal belt and pan type filters are generally used on easily dewatering or rapid settling suspensions, or where extended washing or drying cycles are required. Rotary filters are unsuited to rapid settling suspensions, and have much shorter dewatering cycles. However, they tend to have a much smaller footprint when compared with equivalent belt filters. Information on the various types of vacuum filter will be presented later in the chapter.

6.2.3.1 Rotary valve assembly (for rotary drum and disc filters)

The rotary valve assembly is fundamental to the operation of the rotary drum and disc filters. The valve is used both to continually remove the filtrate and entrained air from the filter and, at the same time, provide control of the vacuum to the various sections of the filtering surface (i.e. filtering, dewatering and drying zones). Figure 6.16 illustrates the basic principle of the rotary valve for a drum filter. The valve body remains static while the individual filtering panels rotate about a horizontal axis. Each panel is connected via a drain-line to the terminal plate. Between the terminal plate and the valve body is a low-friction polymeric wear plate. As the filtration panels rotate, they go through the filtration, dewatering, drying and discharge zones. The zones are isolated by means of either internal bridges within the rotary valve, or via a bridge plate. Figure 6.16 shows the internal bridge option. Figure 6.17 is a more detailed illustration of a rotary valve for a vacuum drum filter with internal bridges (configured for scraper discharge). Although several different types of rotary valve assemblies are available, their

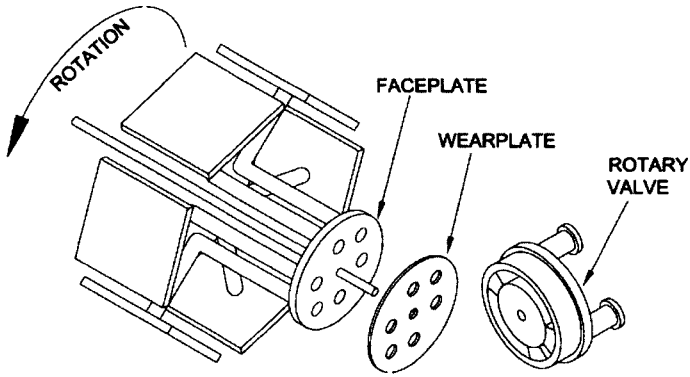

Figure 6.16 Representation of the function of a rotary valve assembly

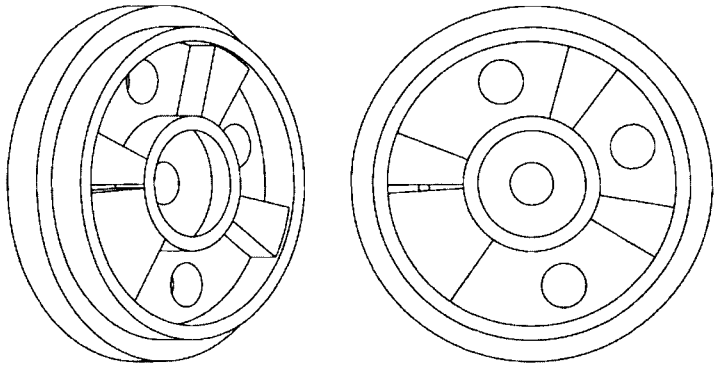

Figure 6.17 Example of a rotary valve body showing the internal bridges – used on a scraper discharge drum filter

principle of operation remains the same. The valve bridges need to be sufficiently large to provide some degree of isolation between zones, but small enough to ensure that the filter segments remain under vacuum during the transition. Alternatively, the bridges can be undercut (recessed) to allow a vacuum leak between zones and thereby maintain some vacuum to the filter segments during the transition between zones. Where fitted, the bridges allow the operator to vary the amount of applied vacuum for each zone independently and this is often required during the initial start-up of the filter. Bridges between the filtration and dewatering zones are transitional bridges.

Many precoat drum filters do not have internal bridges within the valve body, thereby ensuring that an even vacuum is applied to the entire

drum surface. When bridges are fitted to a precoat rotary valve they are used to separate the filtration zone from a common dewatering and drying zone. These transitional bridges are usually undercut to enable vacuum to leak between zones and are of primary importance during the early stages of precoating where the initial vacuum loss through the top of the drum can be controlled.

At the point of solids discharge, the valve bridges are flush to the wear plate and sufficiently wide to provide complete isolation of the filter segments from the vacuum source. Here, the vacuum can be vented, thus enabling the solids to discharge from the filter segment. For the majority of drum filters, low pressure compressed air is applied to this part of the rotary valve to provide blow-back for filter cake discharge. This reverse air flow is used to break the residual vacuum in the filter segment and partially inflate the filter medium, thereby releasing and discharging the filter cake.

Valve configurations vary between types of filter, and can usually have up to four zones – filtration, initial dewatering, drying and solids discharge. The positioning and sizes of the bridges can be adjusted to suit the filter's operating conditions and the solid/liquid suspension's filtering characteristics.

6.2.3.2 Filtrate receiver

Solids discharge from vacuum filters can be obtained, for example, by locally isolating and then breaking the vacuum, and using a discharge or doctor blade to remove the filter cake. The filtrate, however, is within the confines of the vacuum filter system and must be separated from the air flow before being extracted. To achieve this, the filtrate and air pass through the rotary valve into a filtrate receiver. The filtrate receiver is a vertical, cylindrical vessel sufficiently large so as to provide efficient disengagement of the filtrate from the air stream. Figure 6.18 is a typical example of a filtrate receiver.

Filtrate receiver configurations vary between equipment and manufacturers. The usual arrangement is for a tangential inlet close to the middle of the receiver (Figure 6.18). The top connection is for the vacuum source, and the bottom for the filtrate outlet.

Sizing a filtrate receiver, i.e. diameter and cylindrical height, depends upon two main factors. Principally, the cross-sectional area of the filtrate receiver should be matched to the air flow duty for the vacuum filter, such that the mean, vertical linear air velocity is below 1 m s^{-1}. If the filtrate receiver is too narrow then the linear upward velocity of the air will carry over some of the filtrate – as entrained droplets – into the

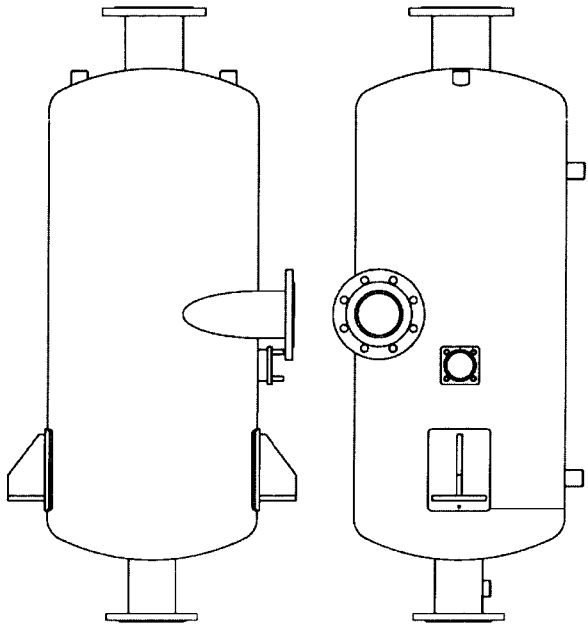

Figure 6.18 Example of a typical filtrate receiver with tangential inlet

vacuum system. In addition, the filtrate receiver's volume should be sufficiently large to prevent excessive cycling of the filtrate pump (where the receiver is fitted with a liquid level switch). The vertical length of the receiver's cylindrical body is usually twice the diameter. In some instances, a moisture trap is fitted between the top of the filtrate receiver and the vacuum system to prevent any entrained droplets carrying through with the air flow. In general, however, this moisture trap is unnecessary since the majority of applications are water based, and the vacuum system is often a liquid ring vacuum pump. Any entrained water droplets would mix with the pump's seal water without affecting the pump's performance.

Discharge from the filtrate receiver can be either via a filtrate pump, operated by a level probe or float switch located within the receiver, or a barometric leg (subject to available height), see Figure 6.19. The selection of the filtrate pump type is generally dependent upon its proximity to the receiver. If the vertical height from the base of the receiver is less than 3 m, then a positive displacement pump is usually required. For greater than 3 m, a centrifugal pump is usually sufficient. The pump supplier can advise on the correct pump selection. To ensure that the pump is fully primed, a balance line from the top of the filtrate receiver near to the inlet of the filtrate pump is recommended.

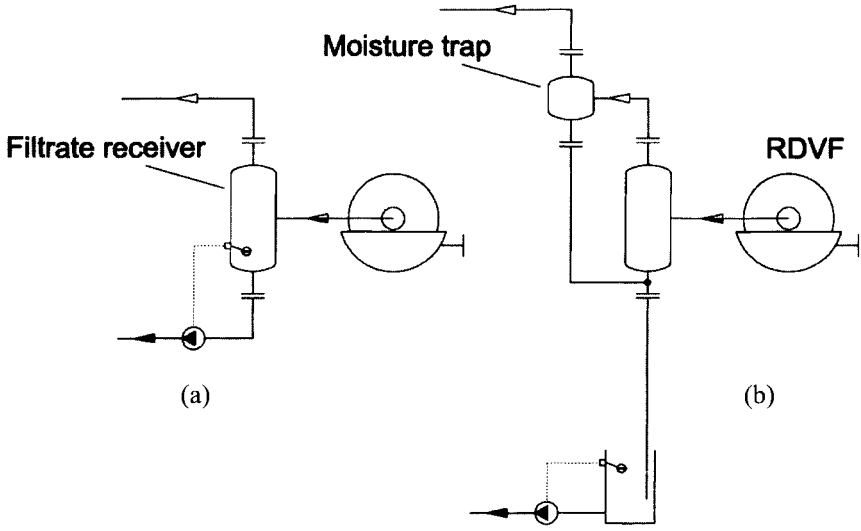

Figure 6.19 Example of typical filtrate receiver installations: (a) filtrate pump with balance line and level switch, (b) barometric leg discharge with moisture trap

For some applications, foaming within the filtrate receiver can cause operational difficulties. Foaming occurs during the filter cake de-watering phase when large volumes of air pass through the porous cake structure. Most foams are unstable and may carry through to the filtrate receiver with the air and filtrate where they collapse. If the fluid is prone to stable foaming, then the foam could fill the receiver and exit with air stream into the vacuum system. There are a number of ways to minimise the effects of foaming:

- Install an additional inlet into the filtrate receiver to minimise turbulence between the air flow and the filtrate. Use separate pipe work for the flow of air and filtrate where possible;
- Increase the filtrate receiver diameter to reduce the upward linear air velocity;
- Include an internal baffle within the filtrate receiver to deflect the foam towards the bottom filtrate outlet;

Where applicable, anti-foam agents can be used.

6.2.3.3 Filter medium

The heart of any process filter is the filter medium, whether it is a woven fabric, sintered mesh, precision screen or a non-woven needlefelt. The correct selection of filter medium can be as important

as the initial choice of filter. It is the filter medium that provides the primary layer for filtration and acts as a support for the formation of the filter cake. It should be fine enough to give the desired filtration efficiency during the initial stages of cake formation, yet remain permeable to allow the filter cake to dewater unhindered. The surface finish of the filter medium should be suitable for good solids discharge, leaving the medium 'refreshed' for the next filtration cycle. Finally, the filter medium should be robust enough to withstand to chemical attack and physical abrasion.

When difficulties arise in filtration processes, the immediate reaction is to assume that the filter medium has somehow failed. Although this can be the case, in many instances it is a change in the operating process that gives rise to the problems. The filter medium on all filters is a consumable, wearing component and should be treated as such. Regular, routine maintenance of any filter should include a change of the filter medium in line with its general performance, and this will ensure that the filter operates to its design capabilities. If the filter medium suddenly needs to be replaced on a more frequent basis then this is indicative of either a change in the process condition, a mechanical fault with the filter, or an operational fault. The selection of the correct filter medium for both the filter and process is of key importance. A filter fitted with an inappropriate filter medium can experience operational difficulties, leading to a reduction in the filter's performance and an increase in the amount of maintenance required.

There are thousands of types of filter media available from all around the world, although most are variations on a core range. Synthetics tend to be made from the more commercially available polymers, such as polypropylene, polyester and polyamide, and these cover the majority of chemical applications. Additional materials are available and advice should be sought when selecting alternative types of filter media.

6.2.3.4 Cake discharge techniques

All the vacuum filters listed in this chapter use cake formation as the filtering process, otherwise solids discharge from the filter would be impossible. Even precoated rotary drum filters use cake filtration in the formation of the precoat although the process filtration is more similar to surface filtration.

Having successfully formed and dewatered the filter cake, the filter needs to discharge the solids and thereby refresh the filter medium. If this cannot be achieved then the filter medium will blind and the filtration cycle stop. There are a number of techniques used to discharge solids from a vacuum filter, ranging from simple scraper blades on

drum filters to return rollers on horizontal belt filters. When undertaking filtration tests, the discharge characteristics of the filter cake should be carefully examined as this may influence the choice of filter for the process. The descriptions of the filters in Section 6.2 include their generic discharge mechanisms. In most cases, thicker filter cakes provide a more efficient discharge, and this results in a cleaner filter medium. Thin filter cakes tend to lack mechanical strength and have a tendency to adhere to the filter medium at the point of cake discharge.

Cake moisture also affects the discharge characteristics. Damp cakes are mechanically weaker and will shear and flow upon discharge, exhibiting thixotropic properties. Some of the cake may remain adhered to the filtering surface thereby reducing the available area for the next filtration cycle. If damp cakes are an intrinsic part of the filtration process then belt discharge drum filters, for example, could be considered since they incorporate a belt wash as part of the unit. Dahlstrom and Silverblatt (1986) tabulated typical cake thickness for effective discharge on a variety of filter units, and a summary is given in Table 6.1.

Table 6.1 Minimum cake thickness for discharge.

Generic Filter Type	*Typical Minimum Cake Thickness* (mm)
Rotary Drum	
Belt	3 - 5
Roller	1
Scraper	6
String	6
Precoat	0 - 3 (max)
Horizontal Belt	3 - 5
Table	20
Tipping Pan (single and multiple)	20 - 25
Rotary Disc	10 - 13

6.3 Basic process design considerations

6.3.1 Initial equipment selection

Vacuum filters have a number of benefits over pressure filters, the main one being that most types can provide a continuous discharge of

both solids and filtrate. However, the use of vacuum as a driving force for filtration also has its limitations. For example, vacuum filtration is generally unsuitable for volatile fluids where the vapour pressure can have an adverse effect on the applied vacuum. Vacuum filters are not just restricted by fluids with low boiling points such as solvents, the same operational problems also occur with very hot aqueous systems.

In addition, volatile fluids can give rise to mechanical and functional difficulties. The vacuum can induce localised flash drying which could cause some otherwise soluble materials to come out of solution, depositing within the structure of the filter medium or on other filter components.

The basic filter selection criteria are based on the following operating and process fundamentals:

- What is the total flow rate through the filter?
- What is the total solids handling rate of the filter?
- How quickly do the solids settle?
- Do the solids form a filter cake under normal vacuum test conditions?
- Are the solids the product?
- Is the filtrate the product?
- Is cake washing required?
- What degree of filtration is required?
- What final cake moisture is required at discharge?
- Are there any chemical compatibility restrictions?
- Are there any mechanical abrasion issues?
- Are there any installation constraints, i.e. available floor space?

Answers to the above questions will normally narrow the selection of filter type down to a maximum of two generic units. It is unlikely, for example, that drum filters and horizontal belt filters will both be considered suitable for the same application although some overlap does occur. Each generic type of filter has certain advantages and disadvantages and this makes initial selection relatively easy.

The filtration cycle, on all vacuum equipment, can be made up of a series of stages. The time available for each individual stage is determined by the type of filter. The stages can be listed as follows:

- Cake formation
- Initial dewatering
- Washing
- Final dewatering
- Drying
- Solids discharge.

Not all of these stages are incorporated within a filtration cycle. In fact, most applications, in their simplest forms, are restricted to:

- Cake formation
- Dewatering
- Solids discharge.

Table 6.2, based on equations (2) to (7), gives some typical information on cycle times for a drum filter, and this highlights the relationship between the available form times and the corresponding dewatering times. For drum and disc filters, the submergence and rotational speed set and limit the filtration, dewatering and, where applicable, the washing times. In contrast, vacuum filters, like the horizontal belt and tipping pan, do not have these constraints and the filtration, dewatering and washing times can be set as required – the only constraint is often the space available for the equipment within the process plant.

Table 6.2 Typical cycle times for a rotary drum filter (R is in rpm, T in seconds).

	σ = 25% Submergence					σ = 37.5% Submergence					σ = 50%		σ = 66⅔%	
R	T_F	T_D	T_{ID}	T_W	T_{FD}	T_F	T_D	T_{ID}	T_W	T_{FD}	T_F	T_D	T_F	T_D
1	15	32	8	17	8	23	24	6	13	6	30	17	40	7
2/3	23	47	11	25	12	34	36	8	20	9	45	25	60	10
1/2	30	63	14	34	16	45	48	11	26	12	60	33	80	13
1/3	45	95	21	51	24	68	72	16	39	18	90	50	120	20
1/4	60	126	28	67	32	90	96	22	51	24	120	66	160	26
1/5	75	158	35	84	40	113	120	27	64	30	150	83	200	33
1/6	90	189	42	101	48	135	144	32	77	36	180	99	240	39
1/7	105	221	49	118	56	158	168	37	90	42	210	116	280	46
1/8	120	252	56	134	63	180	192	43	102	48	240	132	320	52
1/10	150	315	70	167	79	225	240	53	128	60	300	165	400	65

Before undertaking any filtration tests, the processing characteristics of the suspension should be clearly defined and related to the initial choice of filter. For example, if the filter cake requires extensive and prolonged washing to remove residual soluble salts then rotary drum/disc filters are unlikely to be suitable, and tipping pan or horizontal belt filters would be the preferred option.

6.3.2 Filter cloth selection

The filter medium surface, usually a woven filter cloth, is where the initial stage of filtration occurs. The condition of the medium is critical, and when particles become embedded in its structure it begins to blind. Once blinding occurs the filter can no longer operate satisfactorily and either a rigorous cloth cleaning regime is undertaken, or the filter medium is replaced. Premature cloth blinding can result in excessive equipment maintenance and lost production. Where possible, steps should be taken to minimise the occurrence of cloth blinding and thus prolonging the operation of the filter.

Cloth blinding can be a result of several factors relating to either the condition of the feed suspension or the initial choice of filter medium. It is generally accepted that fine, dilute suspensions are more likely to cause premature cloth blinding than coarse, high concentrated suspensions. In dilute suspensions there is little particle interaction, and the particles are drawn into the structure of the filter medium by the flow of the fluid, thus becoming entrapped. In higher concentration suspensions the particles crowd around the apertures of the filter cloth, producing localised bridges and voids, thereby protecting the medium. Cloth blinding can also occur as a result of chemical attack, denaturing the yarn structure. Excessive quantities of chemical flocculants can also have a detrimental effect on the filter cloth. Free flocculant molecules can bond across the yarns and form a near impervious skin.

The choice of filter medium is important as certain types of fabric construction are more prone to cloth blinding than others, but they may offer other benefits that are more significant. The filter medium must have suitable wear characteristics as well as chemical compatibility with the process liquid. In addition, cake discharge properties and filtrate clarity will also be important factors when selecting a filter medium. General trends on yarns and weaves are given in Tables 6.3 and 6.4 (Purchas, 1981).

Yarn structure can also influence the choice of filter fabric, for example, yarn diameter, yarn twist (for multifilament and staple filament cloths) and weave density.

Table 6.3 Standard yarn types.

	Resistance to blinding	*Easiest cake discharge*	*Filtrate clarity*
Monofilament	0	0	2
Multifilament	1	1	1
Staple filament	2	2	0

0 = Best, 2 = Worst

Table 6.4 Standard weave types.

	Filtrate clarity	*Abrasion resistance*	*Easiest cake discharge*	*Least tendency to blind*
Plain Weave	0	1	2	2
Twill Weave	1	0	1	1
Satin Weave	2	2	0	0

0 = Best, 2 = Worst

Many types of filter cloth are available, some combining different yarn types with variations on the more conventional weaves listed above. The choice of filter cloth is often a compromise between longevity of performance, filtrate clarity and cloth blinding. The optimum filter cloth should provide good overall performance and not be prone to premature blinding. Generally, correct conditioning of the feed suspension will eliminate most cloth blinding problems.

If cloth blinding is inevitable, due to the sticky nature of the solids, then the precoat filter should be considered as an option. This special type of filter is used when cloth blinding normally occurs. The filter cloth is used to support a thick layer of precoat which forms a sacrificial filter medium. Further information on the precoat filter is given in Section 6.2.2.1.

6.3.3 Test suspensions

The filtration characteristics of any solid/liquid suspension can easily be established by a series of simple tests. The validity of the results, however, is dependent upon both the accuracy of the test technique and the quality of sample used in the tests. The sample should, where possible, be wholly representative of the process suspension. Ideally, the sample should be taken from the actual process stream, and be tested soon afterwards. It is accepted that this is not always possible,

especially where a new process plant is being constructed and no actual suspension will be available until the plant is commissioned. In these cases, steps should be taken to ensure that the test sample is as close as possible to the expected suspension, either taken from another plant operating the same or a similar process, or manufactured in a laboratory or pilot-plant in line with the process plant.

Where there is any ambiguity in the validity of the sample, it is worthwhile establishing the basic suspension characteristics prior to any test work. This approach defines the suspension and is an intrinsic part of the validity of the filtration test, and can be used to form the basis of the filter's performance guarantee. The basic suspension characteristics include:

- Chemical composition of the suspension (solids and liquid)
- Suspended solids
- Particle size distribution (distribution measurement method should be clearly defined)
- Temperature
- pH
- Source of sample
- Date of sampling.

Variations of these physical parameters can have a noticeable effect on the overall filtration characteristics of the suspension, and this can alter the perceived performance of the filter. Where variable feed qualities are expected, samples of each condition should be evaluated so that the total operating window of the filter can be clearly defined. This may highlight limitations in the filtration unit but is more likely to show deficiencies in the process plant and prompt the inclusion of, for example, buffer tanks to provide a more homogeneous suspension quality.

The need for a representative sample for the filtration tests is critical to ensure the validity of any results. Moreover, the sample should not be allowed to deviate from the norm as a result of sedimentation. The test sample should remain homogeneous, thereby ensuring a consistency of results and quality of filter cake and filtrate.

When handling any samples, the operator should wear suitable personal protection equipment (PPE), for example, overalls, rubber gloves, safety glasses. In addition, the sample should be provided with the appropriate COSHH (Containment of Substances Hazardous to Health) or equivalent data sheets for the liquid and solid components.

To maintain a certain degree of homogeneity, it is often necessary to pre-split the initial slurry sample into several smaller volumes, with each of these volumes suitable for two or three tests. Concentration variations within these smaller samples during the tests will be minimised by this technique.

For bottom feed tests, using an inverted leaf, the solids will sediment to the base of the sample container. To avoid this sedimentation, it is necessary to agitate the suspension sufficiently to re-suspend the particles and produce a homogeneous suspension. Excessive agitation will be detrimental to the test as it could interfere with cake formation. Ideally, the container should be no more than twice the filter leaf test area so as to allow the operator to gently agitate the slurry using the test leaf.

For top feed tests, applicable to coarse and/or rapid settling solids, it can be difficult to ensure that the suspension remains homogeneous. Often sedimentation occurs in the container as the sample is transferred to the test leaf and can give rise to inconsistent results. Sedimentation will also take place on the test leaf once the sample has been applied. It can sometimes be beneficial to delay the start of the filtration cycle until after initial sedimentation on the filter leaf has occurred. This effect can be investigated using a standard top feed test procedure.

6.3.4 Chemicals pretreatment – flocculants and coagulants

The use of chemical flocculants and coagulants as part of the chemical pre-treatment of solid/liquid suspensions is widespread in many industries (see Chapter 2). Flocculants and coagulants have the same basic effect on particles insofar as they group fine particles together, thereby encouraging them to behave as larger particles. The major benefits are that these larger particles both settle faster (ideal for thickeners and sedimentation tanks), and improve the overall permeability of the filter cake.

The terms flocculants and coagulants are used as though they are synonymous, although the main difference between the two is that flocculants are generally synthetic polymers bridging between particles, whereas coagulants are chemicals that influence the surface charge on the particles thereby enabling electrostatic adhesion. The confusion between the two terms arises because both flocculants and coagulants promote particle agglomeration. La Mer and Healy (1963) defined them as:

Coagulation *describes the phenomenon whereby very fine particles of colloidal size adhere directly to each other as a consequence of Brownian movement, once the mutually repulsive electrical surface forces have been sufficiently depressed by the addition of ions.*

Flocculation *involves the formation of more open aggregates than are formed by coagulation and depends upon high molecular weight polymers acting as bridges between separate particles; this open structure is, indeed, implicit in the name which derives from the word 'flocculus', meaning a tuft of wool.*

Most synthetic flocculants are based on polyacrylamide, which can carry either anionic or cationic charges. Flocculants are available in a wide range of molecular weights and charges, to suit a variety of solid/liquid systems. It is therefore recommended that professional advice is sought before using flocculants in order to ensure that the most appropriate grade, dose and application method is adopted.

In general, the use of high molecular weight (HMW) flocculants is reserved for settling and clarification. The HMW flocculants form large flocs that settle quickly, but they do tend to lock fluid within the structure and, if used prior to filtration, could yield high moisture content filter cakes. Low molecular weight (LMW) flocculants, on the other hand, produce smaller, tighter flocs that are more resistant to shear, making them more suitable for mechanical handling (i.e. pumping and filtration).

The method of adding a flocculant is often as important as the flocculant itself. Flocculants should be added to a system where they can be easily dispersed, for example, at a tank where the influent flow is turbulent. In addition, they should be prepared to the correct concentration and dosed into the system at a slow and steady rate. Excessive doses of flocculants can overload the system, producing a gelatinous structure that not only bonds particles to particles, but also particles to the filter medium. This can cause premature and irreversible blinding of the filter medium.

6.3.5 Solids concentration effects

The concentration of solids within a suspension can have a significant effect on the cake formation and overall filtration and solids handling rate. The basic filtration equations imply, by their mathematical development, that all particles are of equal size, are spherical (ideal) and are incompressible. The equations confirm the fundamentally acknowledged observations that larger particles produce more

permeable filter cakes, and allow for the approximate prediction in filtration performance for a similar suspension with a different particle size. This is important when particle attrition occurs as a result of excessive pumping or degradation from long term storage. No practical system has a monosized particle distribution, and the vast majority of particles are irregular and, to some degree, compressible. This however, does not invalidate the fundamental filtration equations.

In practice, it is not possible to predict accurately the effect of solids concentration on the filtration rate. It is observed that higher solids concentration suspensions produce more permeable filter cakes, and the overall filtration rates are greater. The reason for this is the way the filter cake forms. For dilute systems, the particle interaction is relatively low and the particles are given sufficient time to relax into the filter cake structure, achieving their lowest energy state. This produces a compact, dense structure with low permeability. At higher solids concentrations the degree of particle interaction increases. This hinders the siting of some of the particles, and causes internal voids and bridges within the filter cake structure. The net result of this is the overall increase in cake voidage or porosity, and a reduction in the overall permeability. At this stage, the effect of solids concentration on cake porosity cannot be predicted, but the basic principle holds true for many systems. Therefore, to improve the filtration characteristics of a suspension without adjusting the particle size the solids concentration should be increased.

6.4 Experimental test procedure

6.4.1 Filter test leaf

Where possible, the test leaf should mimic a section of the vacuum filter. For example, the test leaf should be fitted with the correct grade of filter cloth, have a similar filtrate drainage support structure, and not be subjected to edge effects that may invalidate the test results. In addition, there should be no hydraulic restriction in any of the test pipe work between the filter leaf and the filtrate collection vessel. A minimum pipe diameter of 6 mm is recommended.

In general, the test leaf is circular with a filtration area of 0.01 m^2 (approximately 0.1 ft^2). The configuration of the filter leaf will depend upon the manufacturer, and most equipment suppliers use their own designs. The basic leaf, see Figure 6.20, represents a panel on a vacuum filter and should have a recess to take a drainage grid, and a

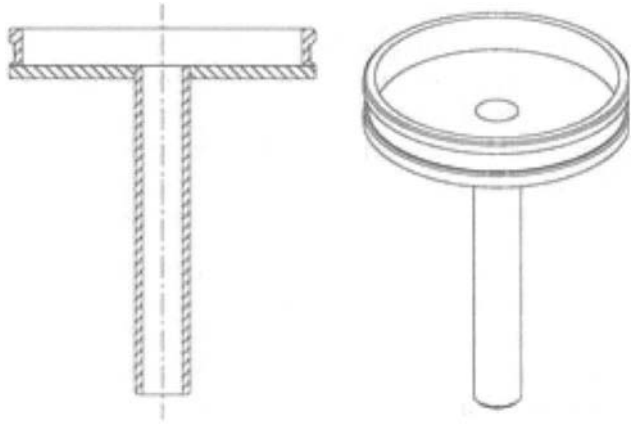

Figure 6.20 Typical filter test leaf

suitable vacuum connection. The filter cloth is fitted over the drainage grid and fixed into place using an 'O' ring seal, shim and clamping ring. The purpose of the shim is to prevent the filter cake from forming in a mushroom shape, thereby distorting the effective filtration area and test results. This is particularly important for rapid filtering materials. The depth of the shim should be greater than the expected cake thickness, but not so deep as to interfere with the cake formation – usually 5 mm excess is sufficient.

It is possible to undertake filter leaf tests with smaller area units although the edge effects (in proportion to the filtration area) will be much greater. However, this does not invalidate the results and an adjusted scale-up factor will need to be used.

In addition to the test leaf, the operator will require a flexible hose (suitable for use under vacuum without collapsing), and a vacuum vessel connected to a vacuum source. If used, the valve between the vacuum vessel and the test leaf should be full bore so as to avoid any unnecessary flow restriction.

Figure 6.21 shows a typical test assembly. The valve at the vacuum pump allows for local recirculation of air (exhaust air back in to the pump inlet), and this technique is used to regulate the applied vacuum and allow for accurate measurement of the air flow through the filter leaf. For most industrial systems, the applied vacuum is between –0.6 and –0.8 barg and this is typical for a liquid ring vacuum pump – often termed *coarse vacuum*. The test assembly shown in Figure 6.21, with the leaf inverted, is suitable for simulating bottom feed vacuum filters, i.e. drum and disc filters. For top feed tests, i.e. horizontal belt and pan

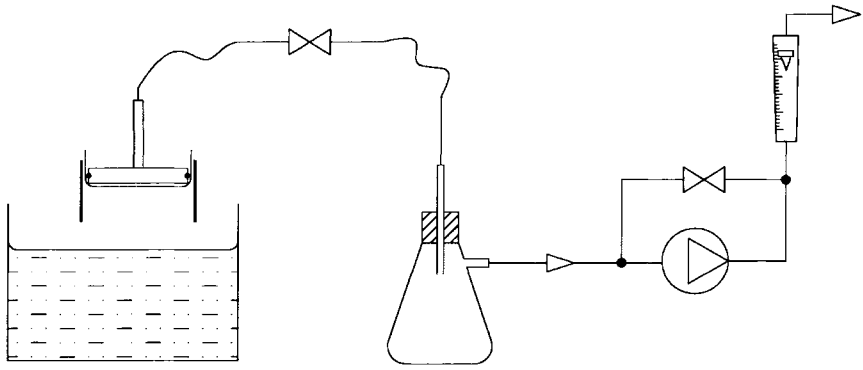

Figure 6.21 Bottom feed filter test assembly

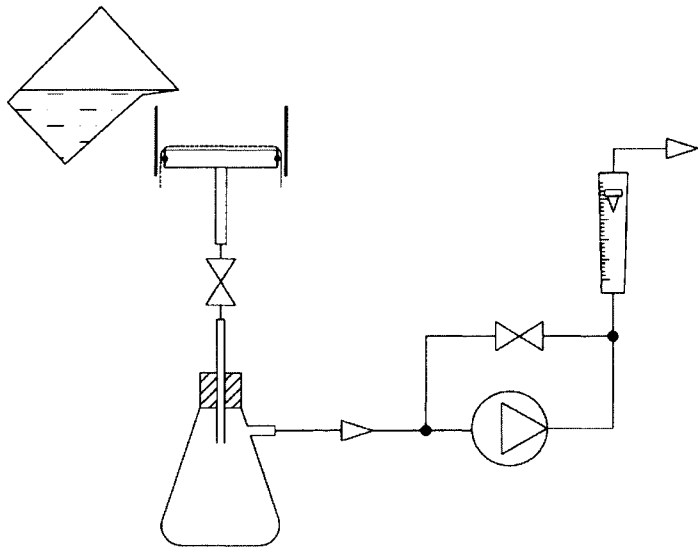

Figure 6.22 Top feed filter test assembly

filters, the filter leaf remains vertical as shown in Figure 6.22, and the feed suspension is poured on from above.

6.4.2 Filter test leaf procedures

Experience with filtration applications as well as reliable test procedures are equally important when undertaking filter leaf trials. Due to the uncertainty in initial filtration tests, it is not possible to set down a test programme prior to undertaking the first test. In fact, the first three tests are generally used to verify the cloth seal on the test

leaf, condition the filter medium (and test apparatus), and provide indicative information on the cake formation and filtration rate. The form times may vary from, for example, 30 seconds up to 4 minutes depending upon the overall filtration rate and cake formation characteristics. During the conditioning tests, it is recommended that the operator starts at approximately 30 seconds form time and evaluates the general filtration characteristics at that point. Times can be varied to suit the suspension in light of the initial findings. As the operator becomes familiar with using a filter test leaf so the process of preconditioning and initial evaluation becomes easier. Having completed the initial set of tests, the operator should be able to produce an outline test programme based on these findings. At all times, the programme should reflect the needs of the process. Testing can be carried out in a number of scenarios, for example:

- The suspension is from a chemical manufacturing process and must be filtered in its 'as received' state, and no chemical or physical additives can be introduced to the suspension.
- The suspension is a dilute effluent stream and must be processed to remove unwanted solid contaminants. No treatment process is currently in place. Flocculants would be acceptable as a pre-treatment, as would pre-thickening.
- The suspension is produced from a natural, variable feed stock and this gives rise to inconsistencies in the solids concentration and particle size distribution. It is important to determine the operating limits of the filter and the effects of the quality of the original feed stock.

The test volume must be sufficiently large to ensure that it is representative of the suspension as a whole, yet small enough to be handled in laboratory conditions. Typically, samples of 5 to 15 litres are sufficient for leaf tests.

The level of applied vacuum during the test is subjective, and experience by the operator and/or equipment manufacturer is invaluable. In general, for highly permeable, coarse, granular filter cakes (for example, diatomaceous earth) the vacuum levels may only be between –0.2 and –0.4 barg. For low permeability filter cakes the vacuum levels will be between –0.6 and –0.8 barg. Generally, this is limited by the performance of the vacuum system which usually decreases below –0.7 barg. For the majority of filter tests it is recommended that the vacuum level is limited to approximately –0.7 barg as this will be indicative of the full-scale conditions.

Figure 6.23 is a typical data sheet for recording the test conditions and results. The number of tests to be carried out will depend upon the nature of the investigation and the variability of the results. A minimum of six tests is recommended for most applications.

FILTRATION SERVICES LTD
Waters Green House, Waters Green
Macclesfield, Cheshire, SK11 6LF
Tel: 01625 616109 Fax: 01625 434678

TEST RECORD SHEET

Client:		Date:	
Address:		Contact:	
		Position:	
		Tel:	
		Fax:	

Enquiry No.:		Order No.:	

Slurry Ref:	

pH of Slurry:		Test Area (m^2):		% Cone:	
Method of Discharge:		Temperature (°C):		Cloth Ref:	

Test No.:	1	2	3	4	5
Form Time (s):					
Dewatering Time (s):					
Set Vacuum:					
Quality of Discharge:					
Clarity of Filtrate:					
Volume of Filtrate (ml):					
Mass of Wet Cake (g):					
Mass of Dry Cake (g):					
Cake Thickness (mm):					

Remarks:

Figure 6.23 Test record sheet for filter leaf tests

6.4.2.1 Bottom feed filters (drum and disc)

Bottom feed leaf tests are used to simulate the operation of rotary drum and disc filters, where filtration is not gravity assisted. During the test, the suspension will require gentle agitation in order to prevent sedimentation in the container. Vigorous agitation should be avoided as this could disturb the cake formation and, in extreme circumstances, could give rise to particle attrition. Agitation using the motion of the test leaf is usually sufficient. If the solids are rapid settling and difficult to re-suspend then top feed tests may be more appropriate (for horizontal belt or pan filters).

The bottom feed test procedure is as follows:

1. Before undertaking any test work, ensure that the operator is in possession of all the relevant safety data sheets.
2. Use personal protection equipment (PPE) during the test work. For example, safety glasses, overalls, suitable footwear and gloves.
3. Obtain a fresh suspension sample, typically 5 to 15 litres, that is representative of the process under investigation. Put the suspension into a suitable container that is large enough to take the test leaf and allow for gentle agitation.
4. Record the system's fixed parameters:
   - temperature
   - pH.
5. Note whether any pretreatment to, or chemical conditioning of, the suspension has occurred.
6. Gently agitate the bulk sample to produce a homogeneous suspension, and extract a reference sample to determine or confirm the solids concentration. If necessary, obtain a particle size analysis on the sample.
7. Select a filter cloth for the test leaf. The filter cloth should be suitable for the application. For advice on cloth selection it is recommended that the operator liaises with an approved filter cloth supplier.
8. Fit the filter cloth to the test leaf, ensuring the cloth is correctly sealed into position. The shim should be fitted to the test leaf to minimise any errors associated with edge effects.
9. Set up the test circuit as shown in Figure 6.21.

10. If used, close off the valve between the filter test leaf and the filtrate receiver, otherwise crimp the hose to isolate the test leaf from the filtrate receiver.
11. Switch on the vacuum pump and set the recirculation valve (between the vacuum pump inlet and outlet) to obtain the desired level of vacuum – typically –0.7 barg.
12. Gently agitate the suspension using a wide faced spatula to obtain a homogeneous suspension.
13. Immerse the filter leaf in the suspension and continue to agitate the suspension (using the test leaf) – a gentle up and down motion is usually sufficient.
14. Start the timer and immediately open the valve (or release the crimp) between the filter leaf and the filtrate receiver. Continue to agitate the suspension, maintaining a homogeneous sample at all times.
15. After a preset form time, remove the filter leaf from the suspension and, keeping the leaf facing downwards, allow the excess liquor to drain from the surface of the filter leaf. This is particularly important if it is a high concentration suspension.
16. Gradually rotate the filter leaf through 180°, simulating the cycle of a rotary drum or disc filter. If required, elevate the filter leaf to ensure that the filtrate drains through to the filtrate receiver.
17. After a preset dewatering / drying time, switch off the vacuum and collect the test data:
    - filtrate volume
    - filtrate quality
    - cake weight (wet)
    - cake thickness
    - discharge characteristics
    - observations (cake cracking, filtrate foaming etc).

Additional points to note:

- During the dewatering/drying time, record the exhaust air flow from the vacuum pump.
- Observe the filter cake during the dewatering/drying cycle, and record if and when cake cracking occurs.

- If cake washing is required, pour a preset volume of suitable wash water onto the filter leaf. The wash water should be added before the filter cake cracks to ensure maximum washing efficiency. The cake wash must be completed during the preset dewatering/drying time. If the wash water has not been drawn through the cake before the end of the drying time, then reduce the wash water volume and repeat the test. Alternatively, immerse the filter leaf in a beaker containing a known volume of wash liquid for a set time period, and record the volume of wash liquid drawn through the filter cake.

The tests should be repeated as necessary to obtain representative and reproducible data. The first three tests should be discarded as these should be used to condition the filter medium, validate the seal on the filter leaf and provide initial estimation of the general filtration characteristics.

Samples of representative filter cake and filtrate should be taken for further evaluation, i.e. moisture content, residual mother liquor in the filter cake (post washing), and solids concentration. Detailed analysis of the filter cake and filtrate is not usually required for most filter leaf tests. However, this work should be undertaken if it applies to the process under investigation.

A sample of the filter cake should be weighed and dried to establish the moisture content. Overnight drying is preferred, using a low oven temperature of approximately 50°C. Do not use oven temperatures high enough to cause degradation of the filter cake

At the end of the testing, remove the filter medium and examine it for signs of wear, blinding or chemical attack.

6.4.2.2 Top feed filters (pan, belt and table)

The test procedure for a top feed filter is very similar to that for a bottom feed. The main variations are:

- The filter test apparatus is as shown in Figure 6.24 (and Figure 6.22), with the face of the leaf uppermost;
- A preset volume of suspension is filtered rather than, as for the inverted leaf test, the filtration being conducted over a set time period;
- The time to form the filter cake is recorded. This corresponds to when the filter cake has no free liquor above it;
- The time to when the filter cake cracks (if applicable) is recorded.

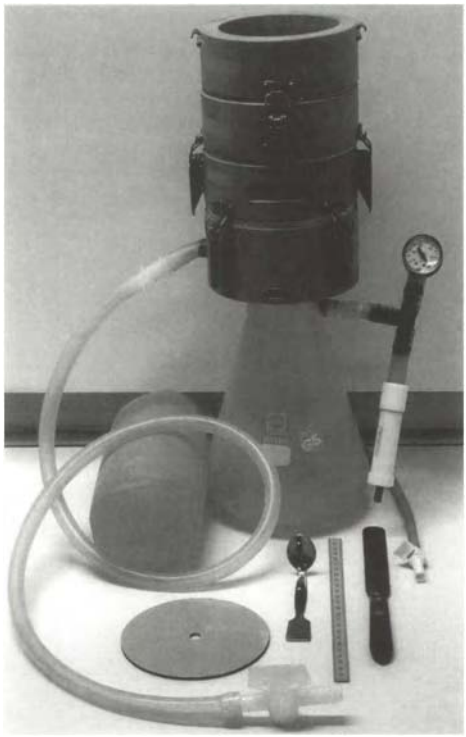

Figure 6.24 Top feed test apparatus (Larox-Pannevis)

Top feed filters are particularly suited to suspensions where the solids settle out quickly. As a consequence, the test sample will have the same tendency and this will give rise to some handling problems during the tests. In this case it is important to fully agitate the suspension, sometimes quite aggressively, before transferring it to the filter leaf. The sedimentation effect of some suspensions can be exploited as a self-forming depth filter prior to applying vacuum to the filter leaf. In fact, the application of the vacuum to the filter leaf can be delayed to encourage initial sedimentation if required. Use of this technique is unsuitable for top feed rotary drum filters, and more applicable to horizontal belt filters.

6.4.2.3 Precoat filters (drum)

The precoat filtration test, as used to simulate a rotary drum vacuum filter, is similar to the bottom feed tests covered earlier in this section. The differences are that the filter medium is used as a precoat support, and the true filtering medium is a bed of precoat in the form of diatomaceous earth, perlite, wood flour, cellulose or some other

suitable inert/compatible material. In addition, the filter leaf has a variable height shim that can be used to set a cut depth of the precoat bed. The test circuit is also different insofar as there is no isolation of the filter leaf from the vacuum source. A twin filtrate receiver system is employed to isolate the precoat filtrate from the process filtrate, see Figure 6.25.

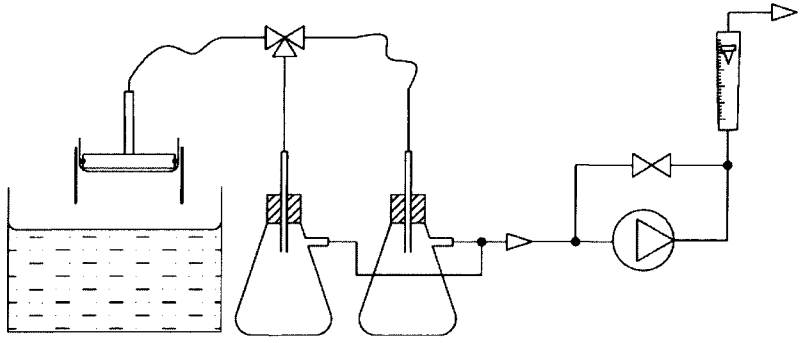

Figure 6.25 Precoat test circuit with two filtrate receivers

The choice of the precoat type and grade is similar in principle to the initial cloth selection in the bottom feed tests. The precoat should be permeable enough so as to allow the free flow of filtrate, yet sufficiently fine to prevent the penetration of solids into the precoat bed. In actual fact, there will always be some degree of particle penetration in the bed and precoat selection is a balance between its permeability and particle retention characteristics. In general, the overall flow rate through a precoat bed is not governed by the bed's permeability, but more by the retained process solids forming a near impervious layer on the precoat surface. Therefore, when selecting a precoat grade, the quality with the lowest permeability without compromising the throughput should be selected. This is also likely to reduce the amount of contaminant penetration into the surface of the precoat and therefore require a shallower cut depth to refresh the bed.

The leaf tests will provide sufficient data to determine the filtration profile and thereby size the filter. However, it is not possible to accurately determine the most efficient cut depth and optimum precoat usage. Generally, the cut depth on an industrial vacuum drum filter will be between 50 and 175 microns per drum revolution, and it is very difficult to reproduce this under laboratory conditions with a test leaf. This aspect of precoat filter testing must be conducted on either continuous laboratory filter or pilot-scale equipment.

6.4.2.4 Cake washing

Cake washing, where applicable, should be undertaken after the filter cake has been formed and has no residual surface liquid. The washing must occur during the pre-defined dewatering and drying cycle and, ideally, before any cake cracking occurs. If sufficient cake washing cannot be achieved during the allocated dewatering and drying cycle, then alternative filtration technology may be required.

It is not uncommon to re-slurry a filter cake – sometimes referred to as re-pulping – for it to be filtered on a second unit. This approach can produce very efficient multi-stage washing, and is easily simulated using the filter leaf test procedure outlined in this section.

6.5 Interpretation of test data

The accuracy of scale-up is a function of the quality and applicability of the test data. It is assumed that the sample under test is, where possible, wholly representative of the process suspension. The data can be interpreted by using some basic filtration equations, and the filtration characteristics of the suspension can be defined for scale-up. The basic filtration equations can also be used, to some degree, to predict the influence of variable system parameters such as vacuum levels, particle size and particle shape. Although not true for all filtration systems, these equations are sufficiently accurate for industrial scale vacuum filtration applications.

The derivation of the filtration equations makes a number of simplifications which, although not strictly true for all filtration systems, are sufficiently accurate for industrial scale vacuum filtration applications. Further details of the models used for filtration analysis is available elsewhere (Wakeman and Tarleton, 2005).

6.5.1 Filtration theory and applicable equations

The basic cake filtration equation is based on the work of Darcy (1856) on the permeability of limestone beds in Dijon, France. The flow velocity through a porous structure is comparable to the flow through a filter cake, and is given as:

$$u = K\frac{(-\Delta P)}{l} \tag{8}$$

Subsequent work by Kozeny (1927, 1933) and Carman (1937) established the relationship between all the parameters, and the Carman-Kozeny equation is used as the basis for many calculations on porous structures:

$$u = \frac{e^3}{K''(1-e)^2 S^2} \cdot \frac{(-\Delta P)}{\mu l} \tag{9}$$

The value of K'', the Kozeny constant, is frequently taken as 5.0, although work by Wyllie and Gregory (1955) indicates that K'' is not constant but is a function of both porosity and particle shape. Wyllie and Gregory found that, despite these variations, the value of K'' is generally around 5.0 ±10% when used for aggregates of irregular particles packed to the common experimental porosity of about 40%. For the majority of filtration models, the particles are assumed to be uniform, spherical and rigid. In these instances it is valid to assume that $K'' = 5.0$.

The bed permeability coefficient is given as:

$$b = \frac{e^3}{K''(1-e)^2 S^2} \tag{10}$$

For spherical particles, the specific surface can be expressed as:

$$S = \frac{6}{d_p} \tag{11}$$

Combining equations (10) and (11), and inserting $K'' = 5.0$ gives:

$$b = \frac{e^3 \ d_p^2}{180(1-e)^2} \tag{12}$$

Equation (12) indicates the basic influence of particle size and bed porosity on the permeability of a filter cake of monosized, spherical particles, and is illustrated in Figure 6.26. This is not strictly applicable to all filtration systems, but the general trends are valid. The bed permeability is proportional to the square of the particle diameter so, as the particle size reduces, the filter bed permeability decreases significantly. This is an important point to consider when reviewing the potential variability of a suspension and when particle reduction occurs by, for example, ageing or attrition.

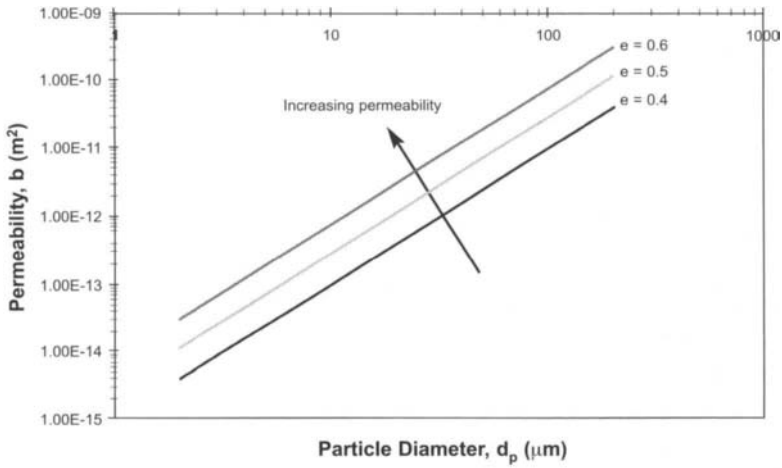

Figure 6.26 Bed permeability as a function of particle diameter and porosity

The bed resistance can be expressed as the inverse of the permeability:

$$r = \frac{1}{b} \tag{13}$$

The following filtration equations were derived by Ruth (1933; 1935) by adapting Poiseuille's equation:

$$\frac{1}{A}\frac{dV}{dt} = \frac{-\Delta P}{\mu\left[\alpha\left(\frac{W}{A}\right) + r_m\right]} \tag{14}$$

and W is related to V by the following equation:

$$W = wV \tag{15}$$

For vacuum filtration, the flow resistance of the filter medium can be assumed to be negligible in comparison with the resistance of the filter cake. This simplifies equation (14), giving:

$$\frac{1}{A}\frac{dV}{dt} = \frac{-\Delta P}{\mu\alpha\left(\frac{W}{A}\right)} \tag{16}$$

Integrating for constant differential pressure filtration, and substituting equation (15) gives:

$$\frac{V^2}{2} = \frac{A^2(-\Delta P)t}{\mu \alpha w} \tag{17}$$

or, upon rearrangement,

$$\frac{t}{V} = \frac{\mu \alpha w V}{2A^2(-\Delta P)} \tag{18}$$

Therefore, for constant differential pressure filtration, there is a linear relationship between V^2 and t, or between t/V and V. This linearity only holds true if the cloth resistance is negligible, the filter cake structure remains homogeneous and is incompressible. With negligible cloth resistance, the line passes through the origin. Example plots of filtration data are shown in Figures 6.27, 6.28, and 6.29.

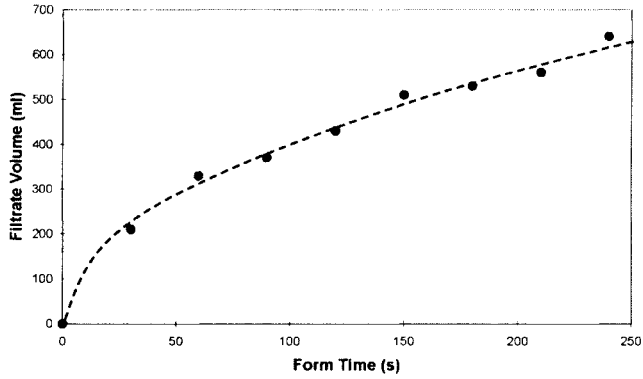

Figure 6.27 Example filtrate *vs* time profile

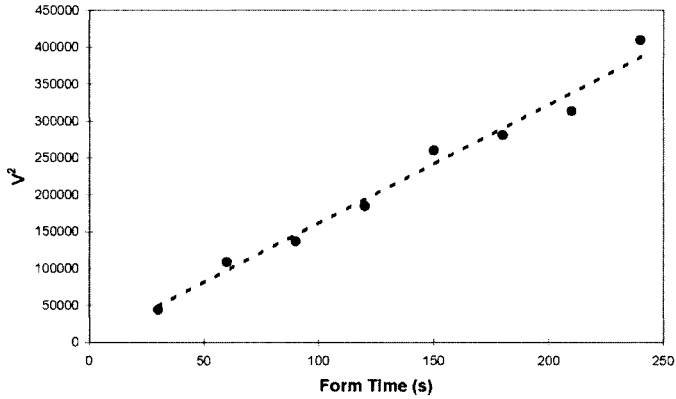

Figure 6.28 V^2 *vs* t plot (from the data used in Figure 6.26)

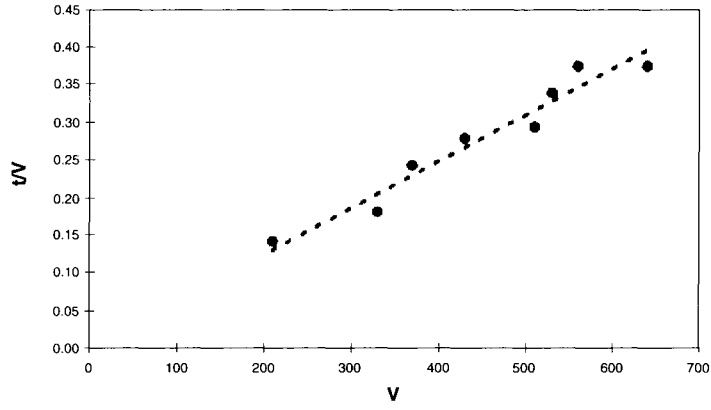

Figure 6.29 *t/V vs V* plot (from the data used in Figure 6.26)

During a filtration test, the following system parameters are assumed to be constant:

- Test area, A
- Differential pressure, $-\Delta P$
- Filtrate viscosity, μ
- Average specific cake resistance, α, and
- Mass of dry solids per unit volume of filtrate, w.

In reality, the differential pressure, cake resistance and dry solids may vary, and will be influenced by the test method and initial stages of cake formation. Where practicable, steps should be taken to avoid unnecessary variations in the differential pressure, and this will enable the operator to examine any potential variations in α, and w.

6.5.2 Filtration flux

The filtration flux can be expressed in a number of ways, and the choice depends upon the relevance of the parameters to the filtration system. For example, if the filtrate rate is important then it would be inappropriate to express the filtration flux in terms of dry solids discharge. In general, the filtration flux for a given system can be expressed as:

- Volumetric feed rate ($m^3\ m^{-2}\ h^{-1}$)
- Mass feed rate ($kg\ m^{-2}\ h^{-1}$)
- Dry solids discharge rate ($kg\ m^{-2}\ h^{-1}$)
- Filtrate discharge rate ($m^3\ m^{-2}\ h^{-1}$)

In general terms the filtration flux may be expressed as:

$$F_R = \frac{X}{1000}\frac{3600}{T_C}\frac{1}{A} \tag{19}$$

where X is the relevant measured volume or mass in either litres or grams (as appropriate), T_C is the total filtration cycle time in seconds, and A is the test area.

Figures 6.30 and 6.31 are examples of the dry solids and filtrate discharge rates as a function of the form time. On a log-log plot, these rates should be linear.

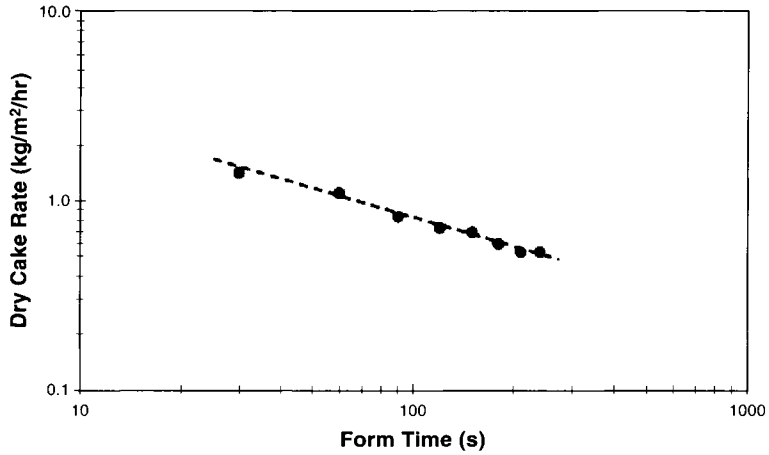

Figure 6.30 Dry cake rate as a function of form time

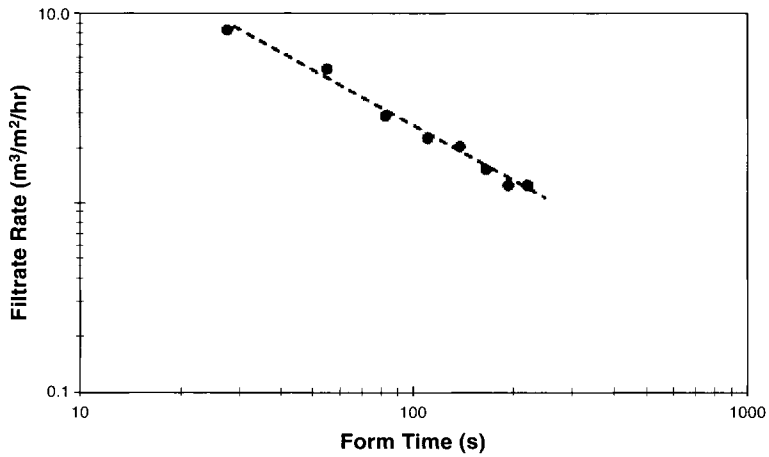

Figure 6.31 Filtrate rate as a function of form time

6.5.3 Filter cake properties

It is assumed that the mass of dry solids recovered per unit volume of filtrate is constant. This can be easily checked during the tests by measuring the cake thickness and comparing the data to the filtrate volume or, more commonly, to the dry solids rate. A plot of the dry solids weight (per unit filtration area per cycle) *vs* cake thickness should give a straight line passing through the origin. The filtrate volume (and therefore the dry cake rate) can be measured quite accurately when compared to the cake thickness, but this does not detract from the validity of the plot. Figure 6.32 is an example of the dry cake weight as a function of cake thickness.

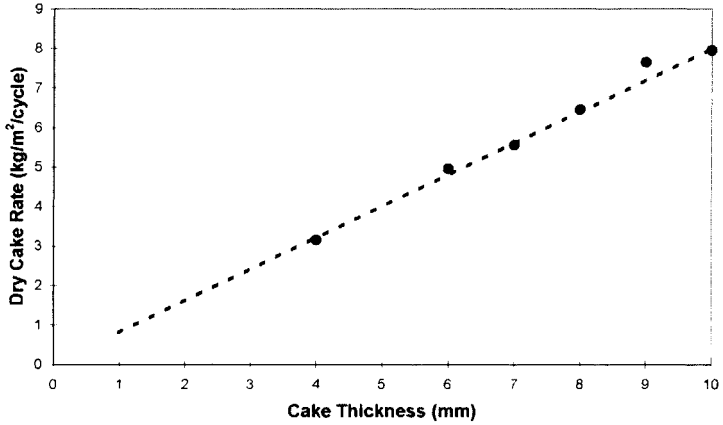

Figure 6.32 Dry cake rate as a function of cake thickness

This graphical representation provides useful information relating to the filtration characteristics of the suspension, and the suitability of certain types of filters to be able to form and discharge a filter cake. This data, in conjunction with the filtration flux rate, indicates the overall performance capabilities of a filter. If the cake is compressible, which most are to some degree, then the gradient will increase with cake thickness, signifying a rise in cake density.

The moisture content of a filter cake is a measure of the interstitial fluid within the cake structure and is a function of the drying time, overall cake permeability and particle nature. It is perfectly feasible to form good discharging filter cakes with moisture contents ranging from 15 to 70%$^{w}/_{w}$ depending upon the type and nature of solids within the filter cake, although for most applications 35 to 50%$^{w}/_{w}$ is more normal.

Cake moisture can be mechanically and thermally reduced on certain types of vacuum filter. Cake compression is available for most drum and belt filters, whereas thermal drying is more suited to belt filters due to their configuration.

Excluding any mechanical and thermal drying mechanisms, and assuming that for any given system the cake permeability and particle nature remain constant, then the only remaining variable is drying time. A plot of drying time against cake moisture will indicate the potential benefits of extending the drying time for a given cycle. Figure 6.33 is a typical cake moisture profile. The available drying time is usually an operating constraint of the filter unit, more so with a drum filter than with a horizontal belt, and so a practical approach has to be adopted. In this example, there is no significant benefit in extending the drying time beyond the knee of the graph since there is no appreciable reduction in moisture with increasing time. Cake moisture content should be low enough to ensure effective cake discharge and meet any operational and process constraints (e.g. as feed into a dryer, or 'stability' for landfill). In fact, higher cake moistures may be perfectly acceptable as part of the solids handling system, especially if the solids are to be re-slurried for further processing.

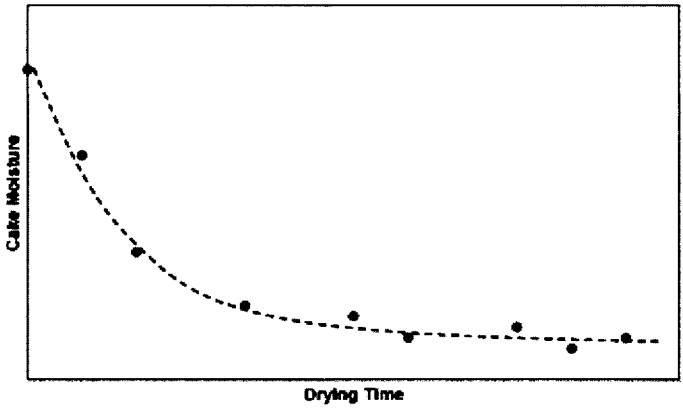

Figure 6.33 Cake moisture as a function of drying time

Cake moisture decreases rapidly during the initial stages of the drying cycle as the interstitial fluid is drawn out of the cake structure. The bulk of the fluid is displaced between the moment the cake surface just falls 'dry' and when the air flow breaks through. Thereafter, the cake moisture reduces slowly with time as more voids within the cake structure are gradually evacuated by the air flow.

6.5.4 Filter cake washing

In some applications cake washing is an integral part of the filtration process. It is particularly important when the purity of the solids needs to be maintained, or the filtrate is valuable. The quantity and application of wash liquor needs to be carefully assessed to ensure that underwashing, overwashing or ineffective washing does not occur. The filter needs to be able to remove the wash liquor and reduce the cake moisture content to an acceptable level prior to discharging the solids. This must be achieved during the normal dewatering and drying part of the overall filtration cycle.

Cake washing is the process by which the interstitial liquor within the filter cake is displaced, thereby recovering more valuable filtrate or increasing the purity of the solids. It can be assumed that displacement is by plug flow, but this assumption is only valid if no cake cracking occurs during the wash. In these instances, the wash liquor would be drawn through the cracks in the filter cake, thus bypassing the majority of solids and making the wash ineffective and wasteful of wash liquor.

The wash efficiency is a measure of the residual solute in the filter cake after washing when compared with the cake in its unwashed condition. It can be expressed as:

$$\eta_W = \frac{C_2 - C_W}{C_1 - C_W} 100 \tag{20}$$

For many applications that use cake washing, the mother liquor may contain trace salts. In such cases it is easy to measure the washing efficiency as it can be correlated to the final salt content of the filter cake. Where the filter cake is reslurried prior to another processing operation, the washing efficiency can be monitored on line by measuring the electrical conductivity of the slurry.

Effective washing is not just about determining the volume of wash liquor required to obtain a certain purity of solid or recovery of filtrate. The fundamental requirement of all cake washing applications is the even distribution of wash liquor over the filter cake thereby ensuring a good wash efficiency in the allowable time. This can be achieved by gentle wash sprays, mists or weir boxes, although in reality the washing efficiency that can be achieved under laboratory and pilot conditions is usually greater than can be achieved on full-scale filters, particularly for drum filters. Unless re-slurry washing is required, it is imperative that the wash liquor does not disturb the structure of the filter cake, especially on rotary drum filters.

Once the filter cake has formed, the solids start the dewatering and drying part of the overall filtration cycle. It is during this period that cake washing takes place. The wash cycle, where used, forms part of the dewatering and drying cycle. Application of wash liquor is normally delayed until there is no visual surface liquid on the filter cake – the cake changes from a shiny, gloss finish to matt. At this point, the filter cake has fully formed, and washing can start. It is imperative that the wash liquor is applied as soon as the cake surface falls dry in order to prevent cake cracking. The wash liquor is applied to the surface of the filter cake from where it is drawn through the cake structure by the vacuum, thereby displacing the mother liquor. The volume of wash liquor must be sufficiently great to ensure that the desired solids purity or filtrate recovery can be achieved.

After the wash liquor has been applied, the filter cake will resume the dewatering and drying process prior to discharge. The volume of wash liquor and the duration of its application must be regulated so that the solids discharge characteristics are not compromised.

On rotary drum filters, cake washing occurs up to just before top dead centre, so that the wash liquid runs back down the drum surface away from the solids discharge point. Excessive quantities of wash liquor could fall back into the filter trough and dilute the feed suspension which is detrimental to the operation of the filter. The maximum quantity of wash liquor for a filter is the volume that can be drawn through the filter cake during the allocated wash cycle.

Similarly, on horizontal belt filters, cake washing usually starts after the solids lose the surface liquid. Horizontal belt filters, by virtue of their design, can provide extensive washing cycles. On many installations, the wash cycle can be set up to provide, for example, multi-stage counter-current washing and reflux washing – this cannot be done with rotary drum filters.

Precoat drum filters need relatively small volumes of wash liquid when compared with other drum filters, and only require sufficient wash to displace the mother liquor from the outer 'skin' before it is peeled away from the precoat bed. Mist and atomising sprays are the most convenient ways to provide a gentle, even wash on precoat filters.

It is possible to estimate the theoretical wash efficiency and wash liquor volume during the scale-up tests, and this is discussed briefly in Section 6.4.2, the Test Leaf Procedures. In the majority of rotary drum filter applications, the wash efficiency is hindered by the mechanics of applying a wash liquor to a rotating, curved surface. The normal response is to increase the wash liquor flow rate but this often causes

operational difficulties. The method of application and wash rates should be carefully considered to ensure optimum washing efficiency. Conversely, washing on horizontal belt filters is very efficient and even, thorough washing can be readily achieved. For further advice on cake wash, it is recommended that the equipment supplier is consulted.

6.5.5 Time and mechanical degradation of agglomerated particles

The use of flocculants and coagulants to agglomerate fine particles is common in many solid/liquid filtration systems. The main benefit of using these additives is the increase in the apparent particle size by bonding many smaller particles together. This reduces the effective specific surface and, in turn, increases the overall cake permeability in line with equation (10). However, long-term storage of agglomerated suspensions and/or mechanical agitation and pumping can break the agglomerates back down to their constituent parts, which has an adverse effect on the filterability of the suspension.

The correct selection and use of flocculants and coagulants is usually carried out by the manufacturers – more information on this is given in Chapter 2. For filtration processes, the use of low molecular weight (LMW) polymer flocculants is preferred since the flocs have a greater resistance to shear (Moss and Dymond, 1968). High molecular weight (HMW) polymers are more suitable for sedimentation processes as they provide better flocculation and faster settling times. Flocs formed with HMW polymers also have a tendency to trap moisture within the structure, increasing the overall moisture content of the filter cake.

To assess the stability of the flocs within the system it is necessary to repeat the filtration tests at regular time intervals, although it is unlikely that laboratory conditions could fully mimic the actual full-scale process conditions and therefore these tests would provide indicative information only.

6.5.6 Air flow rate and applied vacuum

The air rate through a vacuum filter is dependent upon the permeability of the filter cake during the dewatering phase. The flow of air through the cake during its formation phase can be assumed to be zero – only liquid passes through the cake at this stage of the cycle. Once the cake has formed and begins to dewater the air displaces the moisture in the cake structure. The air flow through the cake increases from zero to an asymptotic maximum value as shown in Figure 6.34, and can be measured during a leaf test by attaching a suitable rotameter to the

exhaust side of the vacuum pump. The curve shows the difference between the dewatering and drying phases.

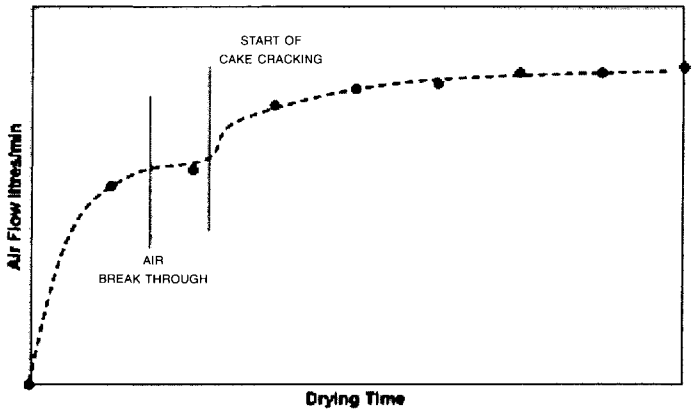

Figure 6.34 Typical air flow profile through a filter test leaf

The exact shape of the curve will be a function of the filter cake permeability, and may be influenced by cake cracking. Cake cracking is a characteristic of the solid/liquid mixture and the way in which the filter cake forms. Cracking does not always occur, but is usual during the dewatering/drying phase and the total air flow must be based on this condition. At the point of cake cracking, the air flow through the test leaf makes a step increase as the air bypasses the filter cake, taking the route of least resistance. In many instances, cake cracking coincides with the start of the drying process so no step increase in air flow is apparent. The total air flow requirement for the dewatering/drying phase of the filter cake is the mean value of the curve, although the maximum value is always the more conservative option. The air flow rate through the filter cake is usually expressed in $m^3\ m^{-2}\ h^{-1}$.

Whatever the type of filter, there are likely to be some minor air losses as a consequence of incomplete seals at either the filter medium, the rotary valve or interconnecting pipe work on vacuum connections. These potential losses must be allowed for when sizing the vacuum pump. Good maintenance on the equipment should keep these air losses to an acceptable minimum. It is not uncommon to add at least 10% to the mean air flow figure from the test work. This also allows for the attenuation of the air between the source (i.e. vacuum pump) and the filter surface, as well as the additional air volume associated with the interconnecting pipe work.

The vacuum pump should be selected in conjunction with the pump supplier, who should be made aware of anything that could influence

the choice of pump. For example, any temperature variations in the feed sample may not significantly affect filterability, but could alter the vapour pressure and therefore applied vacuum. Similarly, the location (altitude) of the proposed installation relative to the test laboratory may influence the pump size. At higher altitudes, the vacuum pump size must be increased for the same operating capacity.

The applied vacuum depends upon the filter cake structure and mean particle size. Large, crystalline solids, forming a high permeability filter cake usually have a lower applied vacuum of between –0.2 and –0.3 barg. This is also true for precoat beds formed from diatomaceous earth or perlite. For fine particle suspensions, like pigments and metal oxides/hydroxides, the applied vacuum is usually much higher at between –0.6 and –0.8 barg. For most applications the normal operating vacuum is less than –0.7 barg and this is often the basis for laboratory tests.

6.6 Laboratory and pilot scale test units

There are many benefits to conducting filter leaf tests, especially in terms of the ease of operation, flexibility when investigating the characteristics of several samples and the need for only small test volumes. The main limitation is that the tests are only a snapshot of the filtering process. In many instances, further experimentation is required to produce additional data to validate the filtration process and, under these circumstances, laboratory scale (see Figure 6.35) and pilot scale (see Figure 6.36) filtration equipment is necessary.

6.6.1 Laboratory scale trials

Laboratory scale trials are the next step up from leaf tests, and are often used to process small batches of suspension over several hours. Here, the mechanical handling of the suspension in relation to a vacuum filter can be investigated, for example, the method of feeding the suspension to the filter and the application of wash liquor. Larger volumes of filtrate and filter cake can be collected for further analysis and product validation. In addition, these extended trials can highlight any potential problems with filter medium compatibility, such as blinding. The laboratory scale filters can be set up with all the operating parameters of a full-scale unit, such as vacuum levels, cycle times and filter medium. In general, scale-up factors for laboratory equipment would be the same as for the standard filter leaf tests.

Figure 6.35 Laboratory scale rotary drum vacuum filter with scraper discharge (Filtration Services Ltd)

6.6.2 Pilot scale trials

As with laboratory scale trials, work with pilot scale filters is to further validate earlier leaf tests in a more realistic production environment. Generally, pilot scale filters are fully functional production units, often the smallest production machine within the supplier's range of equipment, and there should be no issues of scale-up between pilot-scale equipment and a full-scale production machine. Pilot scale filters are designed for operation in 'live' conditions and can be installed directly in a process plant, taking fully representative, fresh suspension as it becomes available. While the filter is on site, users have the opportunity to determine the suitability, robustness and ease of use of the equipment for the specified duty.

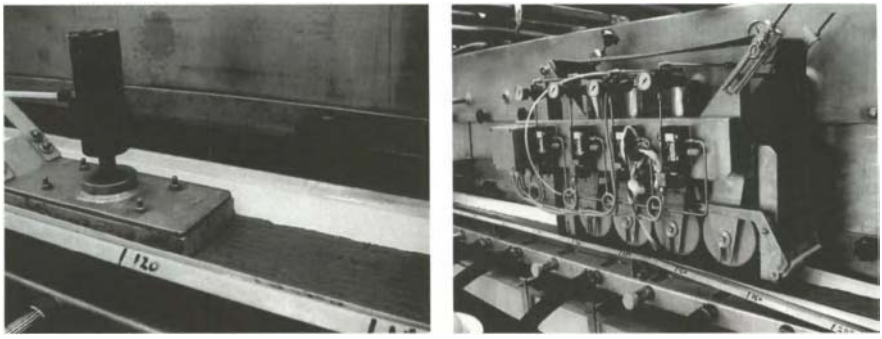

Figure 6.36 Pilot scale horizontal belt vacuum filter showing cake vibration and compression (Larox Pannevis)

6.7 Test results scale-up

Using the data from a 0.01 m^2 (or smaller) test leaf to predict the performance of a full scale filter requires the confident use of representative scale-up factors. Vacuum drum filters, for example, can have filtration areas in excess of 60 m^2 – 6,000 times the area of the test leaf. Fortunately, not all aspects of the test require scale-up. The vacuum, if set correctly on the apparatus, requires no scale-up factor, nor does the filter medium as this should be the same as that to be used on the full-scale machine. Also, it will be assumed that there were no pipe work restrictions in the test apparatus, so hydraulic losses will be taken as zero. The scale-up factors do not make any corrections for process variations, such as particle concentration, particle size distribution, particle shape etc. To estimate the effect that these variables may have on a filter installation, it is recommended that the operator refers to the basic filtration equations and, where appropriate, undertakes additional tests with other representative samples. An alternative us to use the Filter Design Software (see Chapter 1).

As a consequence, the scale-up factors can be restricted to measured rate, test area, cake discharge and air flow, and these are discussed in the following sections.

Before using the scale-up factors, it must be assumed that the tested sample was wholly representative and that the test conditions were a fair mimic of the operation of the full-scale unit.

6.7.1 Scale-up factor on measured rate, ε_R

The measured filtration rate scale-up factor is usually between 0.8 and 0.9 and mainly dependent upon the long-term operating characteristic of the filter cloth, especially with respect to blinding. The majority of vacuum installations utilise either multifilament fabrics or monofilament fabrics, or sometimes a blend of the two. Monofilament fabrics are generally less prone to blinding and so the higher scale-up factor can be employed in the calculations. Where it is suspected that the fabric will blind over time, then the more cautious figure should be used.

In addition, filters that utilise continuous filter medium washing should also adopt a higher scale-up factor. Examples include horizontal belt and belt discharge drum filters. For these units, the continuous cloth wash maintains the permeability of the filter medium and mechanical damage tends to occur before any appreciable signs of blinding.

If the operator has any uncertainty about the blinding characteristics of the filter medium then the lower scale-up factor should be adopted.

Advice on the filter medium and its expected long-term performance should be readily available from the fabric supplier.

6.7.2 Scale-up on test area, ε_A

For the majority of tests it is not necessary to include a scale-up factor for the filtration area, provided that the test area can be accurately measured and the *actual* operating area of the full-scale filter can be specified. Where the test area can only be approximated then a scale-up factor must be based on the error associated with measurement of that area. For a filter test leaf with a flexible shim, the actual test area can vary by up to 10%, and in these cases a scale-up factor of 0.9 should be used. The test area can be used to establish the filtration rate per unit area and this can be related to the operating area of a full-scale machine. The *actual* operating area of a filter should be provided by the equipment supplier, but if this information is not readily available, then the following points should be considered:

- For drum filters, the nominal filtration area is taken as the drum circumference multiplied by the face width. The drum is divided into a number of sections or panels by division strips. Similarly, the drum periphery is also fitted with a division strip to enable edge caulking of the filter medium. These division strips have no free drainage and it may be appropriate, in some instances to subtract their total area from the area of the filter drum as a whole. In most cases, however, filtration occurs at the division strips by lateral fluid flow through the filter cake, and on most installations it is not apparent where the division strips are beneath the formed cake until the point of discharge. For small drum filters (typically up to 5 m^2) the proportion of the division strips area to the whole drum will not be insignificant. Whether this has any influence on the operating area is dependent upon the characteristics of the filter cake. For the majority of drum filter installations, it can be assumed that the nominal and actual filtration areas are equal, and no scale-up factor on the area is required. For small drum installations where the division strip forms a significant proportion of the drum surface, or where the filter cake does not fully bridge the division strip then a scale-up factor of about 0.95 may be appropriate.
- For disc filters, the actual filtration area is usually significantly less than the nominal area due to the construction of the filter leaf covers. It is quite common to reinforce the edges of the disc covers with an impervious heavy duty fabric. As a result, the effective filtration area can be reduced by up to 25%. The appropriate scale-

up factor must be applied with prior knowledge of the disc cover configuration. Where no impervious edges are included, the same principle for the drum filter applies (i.e. no scale-up factor need be applied). For installations where impervious edges are used, the scale-up factor must be determined from the actual free area when compared with the nominal disc area.

- For horizontal belt filters, the nominal filtration area and the actual filtration area can be assumed to be the same, and no scale-up on area is required.

If, due to particular operating and/or process conditions, it is necessary to artificially blind portions of the filter medium (using, for example, a paint-on sealant), then this must be accounted for in the area scale-up factor.

6.7.3 Scale-up on filter cake discharge, ε_D

Ideally, the filter cake formed on the filter will fully discharge from the filter medium. Unfortunately, this is not always the case and residual solids stuck to the filter medium will have an adverse effect on the apparent filtration rate (by effectively reducing the filtration area available for subsequent filtration cycles). The full-scale machine should be designed to give 100% cake discharge, so for most installations a scale-up factor is not required. However, the discharge characteristics of the filter cake can be assessed during the filter leaf tests. If the filter cake has a tendency to adhere to the filter cloth then some estimation of the percentage discharged must be made. This figure can be taken as the scale-up factor for discharge.

6.7.4 Cumulative scale-up factor on filtration rate, E

The cumulative scale-up factor on rate is a combination of the individual rate, area and discharge scale-up figures. The filtration rate, as determined by the filter leaf test, must be corrected by each scale-up factor in turn, so as to obtain the expected filtration rate for a full-scale filter. It must be remembered that this scale-up procedure covers the mechanical operation of the filter only. It does not allow for any undue variations in process or operational conditions, and these variables should be investigated further if required.

$$E = \varepsilon_R \varepsilon_A \varepsilon_D \tag{21}$$

It has been assumed, throughout the discussion on scale-up, that the test results have been obtained from a representative sample, and that

the test work has been carried out to mimic the operation of the full-scale filter. It is particularly important that the filter medium, applied vacuum and test mode (i.e. bottom or top feed mimic) correspond to the full-scale unit.

6.7.5 Air flow requirement

The total air flow through the full-scale filter can be estimated from the air flow curve associated with the test leaf, see Figure 6.34. Since vacuum losses at a filter are generally unpredictable and unquantifiable, it is prudent to overestimate the required air flow by at least 10% (based on the expected maximum air flow through the filter cake). This approach will allow for minor losses at, for example, the rotary valve and the suction pipe work. If the air flow is underestimated then the effect will be insufficient vacuum and a significant reduction in the filtration rate.

It is good practice to overestimate the size of the vacuum pump and install a 'vacuum breaker' to protect the pump, especially if using a liquid ring vacuum pump, from cavitation. The vacuum breaker is a relief valve set to, typically, –0.7 to –0.8 barg and fitted close to the pump inlet.

6.8 Full-scale filtration equipment

As well as using the filter leaf test procedure to estimate the size of a full-scale filter, it can be used to validate the performance of an existing unit. Samples taken from an operating filter provide a direct correlation between the full-scale machine and the test leaf. This approach is particularly useful for monitoring the condition of the filter and/or troubleshooting when filtration problems occur.

Vacuum filter installations are usually much less complicated than pressure filters, requiring a lesser degree of automation and control. Figure 6.37 is a typical drum filter installation using a trough overflow system to fix the slurry level in the filter trough. Filtrate is collected into and discharged from the filtrate receiver which is normally positioned adjacent to the filter. Pipework between the filter and filtrate receiver should be either horizontal or on a slight decline to ensure free, unrestricted drainage. The discharge from the filtrate receiver can be either pumped (as shown in Figure 6.37), or via a barometric leg. The vertical height of the filtrate receiver above a filtrate pump will determine the type and duty of the pump. For a

vertical height of less than 3 m, a positive displacement filtrate pump is usually the preferred option. If the vertical height is greater than 3 m then a centrifugal filtrate pump can be considered. The pump duty must be equal to, or greater than, the filtrate collection rate in order to ensure that the filtrate receiver does not flood. For systems using level or float switches within the receivers, it is prudent to oversize the pump capacity by 20 to 50%.

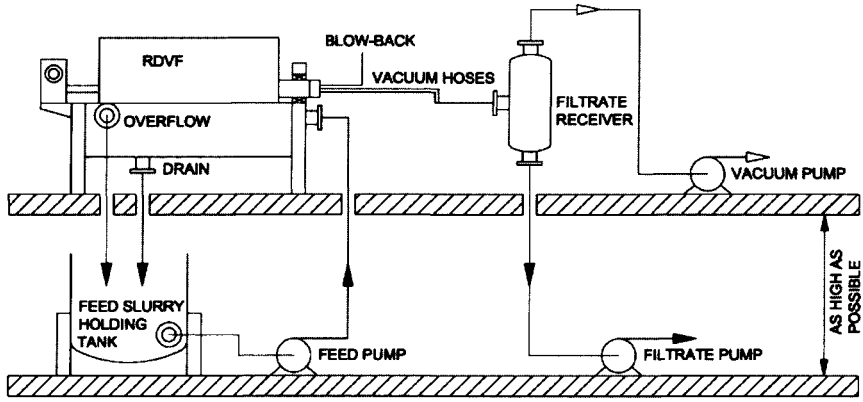

Figure 6.37 Typical vacuum filter installation

Filtrate pumps drawing from filtrate receivers (under vacuum) have to generate a suction lift to discharge the filtrate. As a consequence, the pipework around the filtrate receiver is critical to ensure that the pump remains flooded at all times. To achieve this, it is usual to connect a small bore balance line from the top of the filtrate receiver to close to the inlet of the pump. In addition, it is recommended that the discharge side of the filtrate pump is fitted with a non-return valve to prevent filtrate and/or air being drawn back through the pump when it is idle. For positive displacement pumps, if possible, it is usual to 'reverse' the pump installation by connecting the normal pump outlet to the filtrate receiver and running the pump in the reverse direction. This configuration helps to protect the pump seal from vacuum, see Figure 6.38.

The vacuum system should be positioned near to the filtrate receiver either on the same level or just below. The majority of vacuum systems are based on liquid ring vacuum pumps, providing high volume, coarse vacuum of up to –0.8 barg. This level of vacuum is normally sufficient for most vacuum filter applications. Liquid ring vacuum pumps require appreciable volumes of cool seal water to function correctly. It is

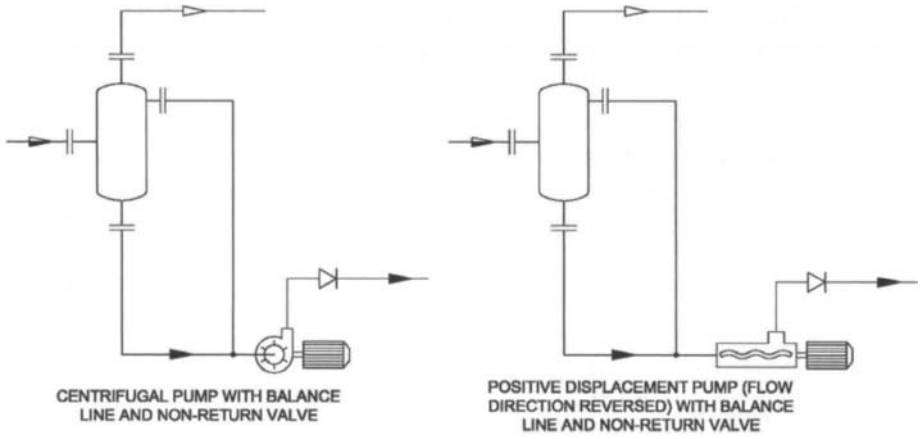

Figure 6.38 Examples of filtrate pump installation

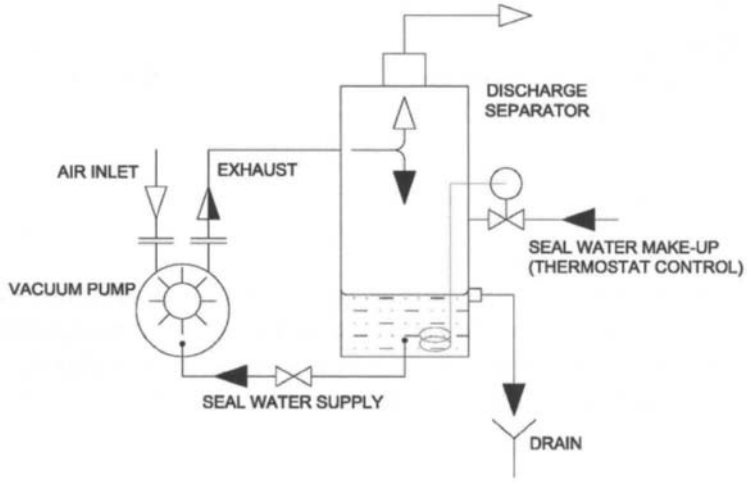

Figure 6.39 Example of vacuum pump installation

possible to reduce the seal water usage by installing a partial recirculation system for the seal water and, where possible, a thermostatic valve on the make-up water, see Figure 6.39.

6.9 Worked examples

This section covers the interpretation of test data and its application to the type of filter under investigation. Examples refer to filter sizing and

troubleshooting existing installations. This approach has been adopted to demonstrate the flexibility of the testing programme and its benefits in both qualitative and quantitative analysis.

6.9.1 Drum filter – filtration troubleshooting

A rotary drum filter is installed at an effluent treatment plant, filtering metal hydroxide waste from an acid pickling process. The installation has been in operation for a number of years, during which time the acid neutralisation process has been developed and refined. An increase in flow to the effluent plant has increased the quantity of hydroxide waste to be filtered. In addition, the filter has started to discharge a wet, thixotropic filter cake which becomes 'fluid' when mechanically worked, which is unacceptable for disposal to landfill. The plant operator is under pressure to improve the filter performance, and there is no time for a detailed investigation. The filter is generally well maintained and there are no mechanical problems with the installation.

The following example details the process by which the operator is able to determine a set of operating conditions for, and expected performance of, the filter installation.

6.9.1.1 Investigation and discussion

A sample of the filter feed is taken for evaluation in the site laboratory. An improvised filter test leaf is constructed using a 47 mm diameter split Buchner funnel, and a test circuit is set up in line with Figure 6.21. The initial observation is that the solids concentration in the filter feed is significantly higher than normal. This has occurred since the recent increase in throughput to the effluent plant.

The current drum filter operating conditions are:

- Drum rotation – 1 rpm (design maximum)
- Drum submergence – 37.5% (design overflow)
- Vacuum at the filtrate receiver –0.7 barg vacuum.

No representative samples of filter cloth are available, so paper is adopted as the filter medium for the duration of the test work.

Filter feed solids concentration, as received, is recorded as 23%$^{w}/_{w}$.

Step 1

The first stage of the investigation process is to undertake the initial filtration tests in line with the current filter set-up to establish whether

comparable results are obtained under laboratory conditions (i.e. verification of the bottom feed test procedure). This approach follows any preliminary observations noted during the test equipment conditioning.

In this example, the test equipment conditioning runs indicate that the cake formation rate is very high, and that the filter cake is excessively thick for a normal 1 rpm cycle (filter's design maximum speed). Using this initial observation as a basis for the test programme, the operator can select filtration and dewatering times to produce a more manageable filter cake thickness. As the worked example is for investigating the performance of a filter with a view to process or mechanical modifications, it is not unreasonable to conduct some of the tests outside the unit's normal operating parameters, as in Table 6.5.

Table 6.5 Setting the initial cycle times.

Test No.	*Equivalent drum rotation, R* (rpm)	*Form time,* T_F (s)	*Dewatering time.* T_D (s)
1	2	11	12
2	1	23	24
3	0.75	30	32

The first set of test results are in Table 6.6.

Table 6.6 Initial test results.

Test No.	*Filtrate volume* (ml)	*Filter cake thickness* (mm)	*Filter cake wet weight* (g)
1	54	24	46.4
2	84	38	82.0
3	118	47	101.0

Observations: The filter cake forms quickly in all three tests. The cake appears to be thick and dry, and suitable for scraper discharge. Upon discharge, however, it is apparent that the filter cake has a high moisture content and is thixotropic. This observation is in line with the full-scale unit. Test no. 2 represents the current operating conditions.

The filter cake thickness, even for a very short form time, is significantly greater than the value given in Table 6.1 for scraper discharge drum filters. The suspension is very filterable, and an attempt should be made to limit cake formation and increase the dewatering time. This can be approached in a number of ways.

To reduce the form time, and thereby limit the filter cake formation, the drum submergence can be decreased. For a vacuum drum filter, this also increases the proportion of dewatering time within the filtration cycle. Alternatively, it is possible to modify the internal bridges within the rotary valve to delay the start of the filtration cycle. Both of these options require mechanical modifications to the filter and are not a viable option at this early stage of the investigation.

It is also possible to reduce the form time by increasing the drum rotational speed. However, this also reduces the dewatering time within the filtration cycle. The drum currently operates at its maximum speed of 1 rpm, and 37.5% submergence. Test no. 1 simulated the filter operating at a higher drum speed of 2 rpm. For this test, the filter cake was thinner than for the two subsequent tests (simulating slower drum speeds), but the quality of the filter cake is comparable for all three tests. It would not be appropriate to consider increasing the drum speed beyond the filter's initial design parameters as there is no evidence to suggest that this approach would be beneficial.

Step 2

It has already been mentioned that the feed concentration is very high, and this coincides with increased throughput at the effluent treatment plant. The solids concentration may therefore be the source of the filtration problem, insofar as the filter cake is too thick and cannot sufficiently dewater within the overall filtration cycle.

If it is assumed that the drum filter is to be operated up to 1 rpm and 37.5% submergence, then the maximum filtration and dewatering times are fixed. Under these conditions, in order to reduce the thickness of the filter cake it is necessary to consider diluting the feed suspension.

Table 6.7 Setting the cycle times.

Test No.	*Solids concentration* (% $^{w}/_{w}$)	*Equivalent drum rotation, R* (rpm)	*Form time,* T_F (s)	*Dewatering time,* T_D (s)
4	10	0.50	45	48
5	6.5	0.33	68	72
6	2.5	0.20	113	120
7	2.5	0.20	113	120

The second set of test results are as follows:

Table 6.8 Test results on diluted feed samples.

Test No.	*Filtrate volume* (ml)	*Filter cake thickness* (mm)	*Filter cake wet weight* (g)
4	194	27	51.4
5	248	20	35.4
6	404	12	21.0
7	434	13	21.4

Observations: The filter cake forms well, especially at the lower feed concentrations in tests 5, 6 and 7. In all cases, the filter cake is firm to the touch and discharges as a friable solid. The driest cake discharge coincides with tests 6 and 7.

It is apparent from this set of filter leaf tests that the filter will function correctly if the suspension is maintained at a lower feed concentration. This was probably one of the fundamental design parameters of the initial installation.

Step 3

From the information in Tables 6.7 and 6.8, the filter's operating parameters can be re-set to ensure that the filter operates correctly. Tests 6 and 7 can be used as the basis for the new operating parameters, and the data used to establish the filtration rate based on these settings.

Drum rotational speed, R	0.2	rpm
Solids concentration	2.5	$\%^{w}/_{w}$
Test leaf diameter	47	mm
Test leaf area, A	1.73×10^{-3}	m^2
Filter cake wet weight	21.2	g (average value)

From equation (7), $T_C = \dfrac{60}{0.2} = 300$ seconds

From equation (19), $F_R = \dfrac{21.4}{1000}\dfrac{3600}{300}\dfrac{1}{0.0017} = 148$ kg m^{-2} h^{-1}

If a conservative scale-up factor of $\varepsilon_R = 0.8$ is adopted for the test leaf rate to allow for the potential variability in feed quality, and $\varepsilon_A = 0.9$ to allow for the small test area, then the expected overall filter cake discharge rate for the filter (based on wet cake) is 106 kg m^{-2} h^{-1}.

Therefore, to summarise:

- Drum submergence – unchanged at 37.5% and in line with the original design and operating parameters;
- Drum rotational speed – set at 0.2 rpm;
- Feed suspension – maintain at 2.5%$^{w}/_{w}$;

Expected performance:

- Filter cake thickness – approximately 12 to 13 mm;
- Filter cake discharge – good, no excess moisture in the cake;
- Wet cake discharge rate – approximately 106 kg m^{-2} h^{-1}.

6.9.2 Drum filter – sizing

A precious metals refinery plant wishes to recover solids from an equipment washing process so that the solids can be re-introduced into the refinery. The washings contain a variety of precious metal oxides and hydroxides, and are collected at a rate of 4.75 tonnes per day (7 days per week). It is the intention of the plant manager to operate a filtration unit during the normal 8 hour day shift, but not overnight nor at weekends when supervision capabilities would be limited. Cake moisture content is not critical.

6.9.2.1 Investigation and discussion

A representative sample of the washings suspension was tested using an inverted test leaf as shown in Figure 6.21. Initial tests indicated that the suspension could be processed on a rotary drum vacuum filter with scraper discharge, and the subsequent tests were based on these findings. The test results in Table 6.9 were obtained.

Preset vacuum	–0.7 barg
Filter leaf test area	0.01 m^2
Assumed drum submergence	37.5%
Feed suspension specific gravity	1.18
Discharge simulation	scraper

Using the data from Table 6.9, plot the filtrate profile as a function of form time (Figures 6.40 and 6.41).

Table 6.9 Test results.

Test No.	*Form time, T_F* (s)	*Dewatering time, T_D* (s)	*Filtrate volume* (ml)	*Mass of wet cake* (g)	*Cake thickness* (mm)
1	60	64	108	66.6	3.5
2	90	96	136	82.3	4.0
3	120	128	140	87.9	4.5
4	150	160	168	96.3	5.0
5	180	192	184	106.7	6.0
6	210	224	189	108.4	5.5
7	180	192	176	98.1	5.5
8	180	192	178	109.8	6.0
9	180	192	165	92.5	5.0

Observations: Generally good cake formation, with the clean discharge of the filter cake at ≥180 seconds form time.

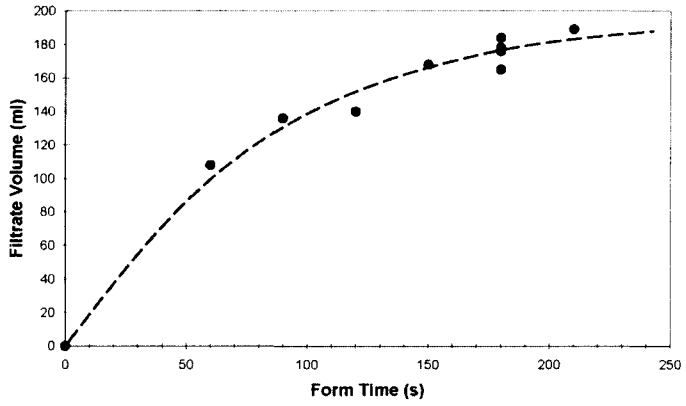

Figure 6.40 Filtrate volume *vs* form time

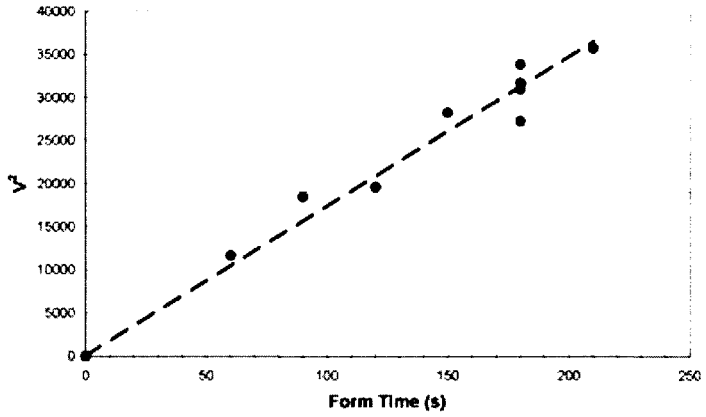

Figure 6.41 Filtrate profile as V^2 *vs* form time

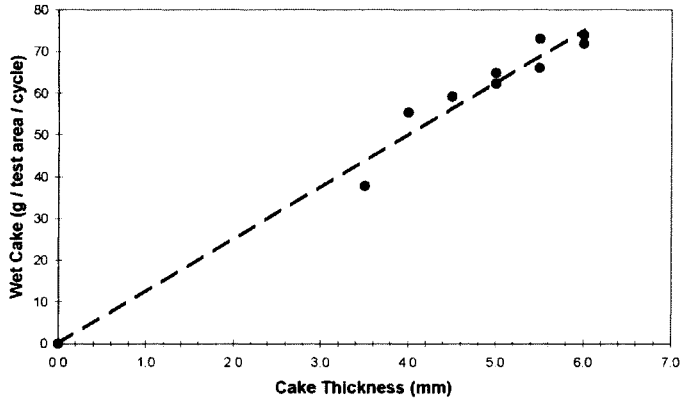

Figure 6.42 Wet cake *vs* cake thickness

The plot of the wet cake as a function of cake thickness is linear (Figure 6.42), indicating no compressibility of the filter cake.

Apply equations (7) and (19) to the data given in Table 6.9 to determine the total cycle time, T_C, and the filtration rate, F_R (based on wet cake recovery and filtrate rate). This gives the data for Table 6.10.

Table 6.10 Filtration rates.

Test No.	*Cycle time, T_C* (s)	*Wet cake rate* (kg m^{-2} h^{-1})	*Filtrate rate* (m^3 m^{-2} h^{-1})
1	160	150	0.24
2	240	123	0.20
3	320	99	0.16
4	400	87	0.15
5	480	80	0.14
6	560	70	0.12
7	480	74	0.13
8	480	82	0.13
9	480	69	0.12

The wet cake rate and filtrate rate profiles are shown in Figure 6.43 and 6.44.

Sizing calculation:

Mass feed rate	4.75 tonnes per day (initial data)
Volumetric feed rate (suspension specific gravity 1.18)	4.03 m^3 day^{-1} (7 days per week)

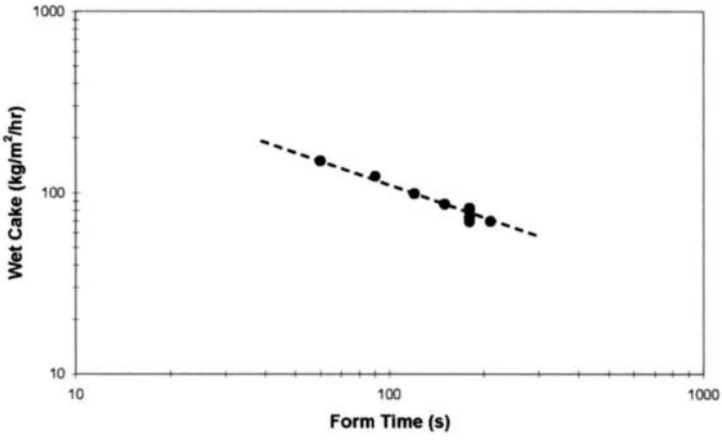

Figure 6.43 Wet cake rate profile

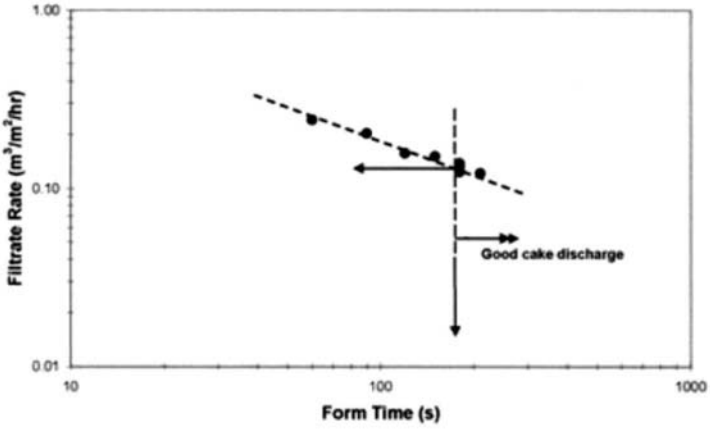

Figure 6.44 Filtrate rate profile

Daily operation	8	hours per day
Weekly operation	5	days per week
Volumetric feed rate to filter	0.70	$m^3\ h^{-1}$

Good, clean cake discharge was achieved at a form time of 180 seconds (dewatering for 192 seconds). This corresponds to an average filtrate rate of 0.13 $m^3\ m^{-2}\ h^{-1}$. Using the scale-up factors of $\varepsilon_R = 0.9$, $\varepsilon_A = 0.95$ and $\varepsilon_D = 0.98$ for the test work gives a filtration rate (based on filtrate) as 0.11 $m^3\ m^{-2}\ h^{-1}$.

Since the filtration rate based on filtrate is always less than the corresponding filtrate rate based on feed, a conservative figure for the required filtration area is:

$$\text{Filter Area} = \frac{0.70}{0.11} = 6.4 \quad \text{m}^2$$

A drum filter of 6.4 m^2 filtration area would therefore be required to process the metal oxides and hydroxides washings within the specified time period.

It should be noted that no operational correction factor (i.e. actual time 'on-line') has been included in the sizing of the filter. The specified 8 hours per day operational time may vary according to shift patterns and plant operator responsibilities. A further allowance should be made for general equipment maintenance in line with the filter manufacturer's recommendations.

Nomenclature

A	Filtration area, m^2
b	Bed permeability, m^2
c	Mass fraction of cake solids in the slurry
C_1	Solute concentration in the unwashed cake, units as appropriate
C_2	Solute concentration in the washed cake, units as appropriate
C_w	Solute concentration in the wash liquor, units as appropriate
d_p	Particle diameter, m
e	Porosity
F_R	Filtration flux, units as appropriate
K	Constant dependent upon the physical properties of the porous bed and the fluid
K''	Kozeny constant
l	Bed thickness, m
m	Mass ratio of wet cake to dry
ΔP	Pressure drop across the filter medium and cake, N m^{-2}
R	Drum rotational speed, rpm
R_w	% solute remaining after washing
r	Specific resistance, m^{-2}
r_m	Resistance of medium, m^{-1}

S Specific surface, m^{-1}

T_C Total filtration cycle time, s

T_D Dewatering time, s

T_F Form time, s

T_{FD} Final dewatering time, s

T_{ID} Initial dewatering time, s

T_W Cake wash time, s

t Time, s

u Flow velocity, m s^{-1}

V Filtrate volume, m^3

W Mass of accumulated dry solids corresponding to V, kg

w Mass of dry solids per unit volume of filtrate, kg m^{-3}

X Measured volume or mass in the laboratory tests, units as appropriate

α Average specific cake resistance, m kg^{-1}

ε_A Scale-up factor for filter test area

ε_D Scale-up factor for filter cake discharge

ε_R Scale-up factor for measured rate

E Cumulative scale-up factor

ρ Density of the filtrate, kg m^{-3}

σ Drum submergence (fraction of perimeter)

μ Viscosity of the filtrate, N s m^{-2}

η_W Wash efficiency

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7 Filtering centrifuges

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This chapter considers the equipment available to perform a solid/liquid separation by centrifugal filtration. A general description of the basic batch and continuous filtering centrifuge types is given, followed by a summary of several commercially available variants of the basic batch and continuous designs and their suitability for particular applications. Filter media and the alternative ways of classifying filtering are also considered. Basic principles of centrifugal filtration are common between all types and these are discussed with a view to giving the reader a basic understanding of the physical parameters that affect the separation performance of a centrifuge. Testing, sampling and scale-up from the laboratory or pilot plant to a full scale plant are also discussed, along with some aspects of installation and operation, and standards and safety issues.

Throughout this chapter, unless specifically stated otherwise, the following terminology is used: *Centrifuge* refers to filtering centrifuges, and *screen* refers to all forms of filter media used in filtering centrifuges. The solid/liquid mixture entering the centrifuge is referred to as *feed*. The terms *liquor* or *liquid* refer to the liquid phase of the centrifuge feed and the terms *filtrate* or *centrate* are used to represent the liquid after separation. The damp solids after filtration are referred to as *cake* with the term *crystal* used to represent an individual particle in the cake. *Moisture* or *dryness* refers to the liquid remaining in the damp cake.

7.1 The basic filtering centrifuge

There are two main categories of filtering centrifuge – batch and continuous. Batch filtering centrifuges process the solid/liquid mixture (or feed) in discrete batches whereas the continuous centrifuges

operate on a continuous flow of feed. These two general types are considered in more detail below.

7.1.1 Basic filtering batch centrifuge

Figure 7.1 shows a cutaway view of a batch centrifuge. The key component is the basket (1), or drum, which contains many perforations to allow the passage of liquid during the filtration process. The basket supports the screen onto which the batch of feed to be separated is placed by the feed pipe (2). Figure 7.2 is a cross section of a basket showing the perforations (3), screen (4) and solid/liquid mixture in the process of being separated. The cake (5) forms an annulus around the wall of the basket, with a thickness typically 10% of the basket diameter. The basket rotates at a rate sufficient to generate an angular acceleration several hundred times the acceleration due to gravity G (9.81 m s^{-2}). This acceleration (often termed high G) enhances the filtration process in much the same way as a differential pressure increases the filtration rate in a pressure filter. Typically rotation rates for large industrial scale centrifuges with a basket diameter of 1000 mm are in the range 1000 to 1600 rpm and this generates a centrifugal acceleration equivalent to 500 to 1500G (4900 to 14700 m s^{-2}). As a comparison with a pressure filter this centrifugal acceleration is equivalent to a pressure of 10 to 30 atmospheres across a 200 mm filter cake for a liquor density of 1000 kg m^{-3}.

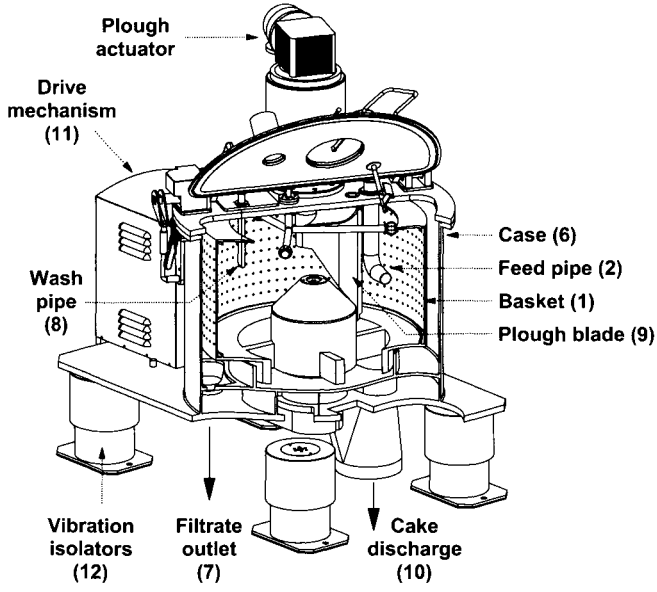

Figure 7.1 Typical filtering batch centrifuge (Broadbent Centrifuges)

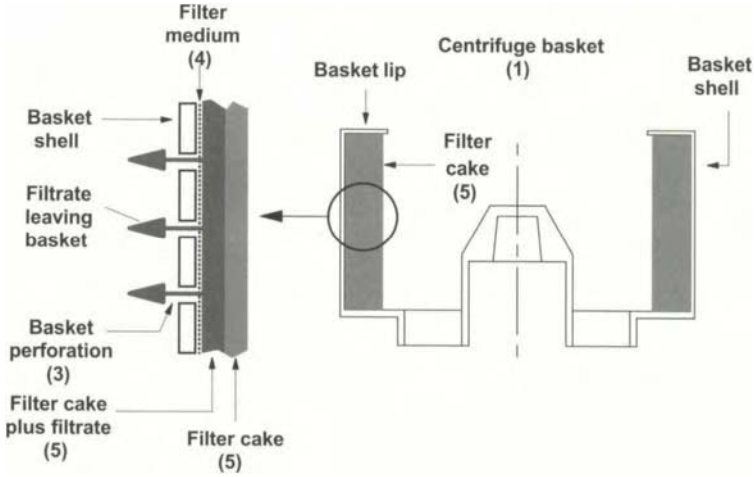

Figure 7.2 Cross section of the basket from a batch centrifuge

Whilst the basket is the key component in any centrifuge there are many other additional components necessary for a fully functional centrifuge. They are required to support, enclose, drive, fill or empty the basket. In many cases it is these additional components which distinguish one centrifuge type from another.

Figure 7.3 shows a typical process cycle for a batch centrifuge broken down into seven steps.

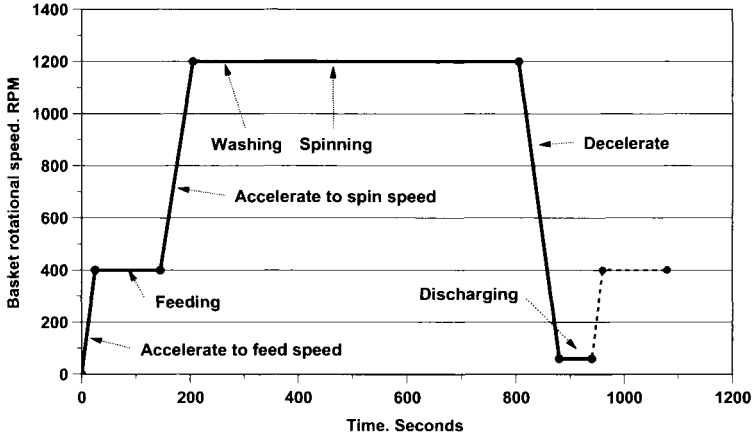

Figure 7.3 Typical batch process cycle

Taking the case of an empty stationary centrifuge as the starting point, and with reference to Figures 7.1 and 7.2, the seven basic process cycle steps are:–

- *Accelerate to feed speed.* The basket (1) is accelerated from rest to a suitable speed for feeding by the drive mechanism (11).
- *Feeding.* The slurry enters the basket and is distributed onto the basket wall to form a filter cake via feed pipe (2). Feeding continues until the feed limiter detects that a pre-set feed thickness has been reached. If the slurry is not distributed evenly within the basket then vibration at the basket rotational speed will occur. The vibration isolators (12) which form part of the centrifuge support reduce the transmission of the vibration to the surrounding structure.
- *Accelerate to spin speed.* The basket is accelerated to the required spin speed and filtration and drying proceed under the influence of a high *G*. The liquid passing through the filter cake leaves the basket via the perforations (3) and collects in the centrifuge case (6) before leaving the centrifuge via the filtrate outlet (7).
- *Washing.* Some processes require the cake to be washed by one or more liquids after initial drying. This is accomplished via the wash pipe (8).
- *Spinning.* The basket remains spinning following washing to obtain the required cake dryness. Spin times are chosen to suit the characteristics of the feed slurry and may be as short as a few tens of seconds or as long as a few hours.
- *Deceleration.* Once the required dryness has been achieved the basket is decelerated to a low speed via the drive mechanism (11).
- *Discharge.* Once the basket speed has been reduced to a speed below one *G* a discharge or plough mechanism (9) is used to scrape out the dried product which then falls through the opening in the basket bottom and exits the centrifuge though the solids discharge outlet (10).

The length of a process cycle varies dramatically depending on the slurry being processed. Some batch centrifugals process above 20 batches per hour, others require several hours to process a single batch. The differences between batch centrifuge types and the range of feed slurries are considered in more detail below.

The productive parts of the batch cycle in Figure 7.3 are feeding, washing, spinning and discharging. These are considered in more detail below.

7.1.1.1 Feeding

The method of adding feed to a centrifuge basket has an impact on both the distribution of solids in the centrifuge basket and the level of crystal

damage. It is of primary importance to get a uniform layer of material in the centrifuge basket during feeding. Failure to produce a uniform layer will result in uneven washing and drying later in the process cycle. It may also cause imbalance in the mass within the basket (that is, more centrifuge cake on one side of the basket than the other). This can cause a large out of balance force when the centrifuge accelerates to spin speed. For example, if the load in a 1250 mm diameter centrifuge basket is non-uniform to equivalent of 2.5 kg more cake on one side of the basket than the other then the resulting out of balance force is approximately 2.5 tonnes rotating at 1200 rpm (20 Hz). This will result either in significant vibration of the centrifuge or large forces transferred to the foundations – depending on the design on the centrifuge.

As the feed is added to the basket it is accelerated from zero speed up to the peripheral speed of the basket. If this is done violently then crystal damage can result, particularly with needle shaped crystals. To reduce this effect it is common to design the feed pipe (item 2, Figure 7.1) to impart a velocity to the feed in the direction of basket rotation before depositing it onto the basket wall. These steps will reduce crystal damage and reduce the power consumed by the centrifuge during the feeding process. The speed of the basket during feeding must satisfy several criteria. Firstly the feed slurry must remain fluid for a time to allow it to flow over the basket surface and give an even distribution. This process can be made simpler by having multiple feed pipes (top, middle and bottom for example) or by a more complex feed mechanism but in all cases a degree of fluidity is required once the feed has entered the basket to get good distribution. If the feed speed is too high then sufficient liquid may be filtered from the cake to allow the crystals to come into contact removing all fluidity. This problem tends only to occur with feed slurries having large crystals that filter rapidly. Examples of such fast filtering feeds are citric acid or sucrose. At the other extreme (more common in the pharmaceutical industry) is a feed containing a large proportion of liquid and small solids particles (perhaps 20 μm or less). In this case the problem is not lack of fluidity but excess liquid in the centrifuge basket. As the particles in the feed settle out and consolidate against the basket wall to form a cake the resistance to flow through the thickening layer of solids increases and if feed is added at too high a rate a significant head of liquid can form on top of the cake. For cakes that are compressible the pressure from this head of liquor compresses the cake, further reducing the channels between the crystals and increasing the resistance to flow.

In such situations the detrimental effects are twofold. Firstly the feed time is extended considerably which increases the cycle time of the

centrifuge and reduces throughput. Secondly there is the possibility of severe vibration in situations where there is a significant amount of free liquid on the surface of a centrifuge cake – particularly when the centrifuge is accelerating to spin speed. This undesirable effect is often called a liquor load or liquor surge. The full explanation is not presented here but the result is a large out of balance resulting from a circulating wave in the free surface liquor within the centrifuge basket. The resulting vibration is large and generally at a frequency significantly less than the basket rotational speed (c.f. an out of balance of solids in the cake).

To overcome such difficulties and maintain a reasonable filtration rate and centrifuge throughput it is normal to feed dilute slurries in bursts, allowing the surface liquor to drain away before adding more feed. Modern automatic batch centrifuge controls provide facilities to burst feed if required.

7.1.1.2 Washing

Some applications have a requirement to wash the centrifuge cake with one or more liquors. The uniform cake produced by the batch centrifuge allows efficient washing assuming the wash liquors are applied uniformly over the surface of the cake. Centrifuge designers go to considerable lengths to ensure that the application is uniform – particularly when the cake solids are soluble in the wash liquor(s). Wash is usually applied via a series of jets on a wash pipe. Most designs allow for several washes to be applied through one or more wash pipes as required. Valves on the filtrate discharge allow the wash liquors to be collected separately if required.

7.1.1.3 Spinning

The final dryness of the centrifuge cake is dependent on the G at spin speed and spin time together with various physical parameters of the cake and liquid. Typical values are in the range 1–20% weight for weight (w/w) depending primarily on particle size distribution. As the length of the spin time increases the centrifuge throughput reduces accordingly. There is a limiting dryness fixed by the physical parameters of the solids and liquids in the feed and the centrifuge G at spin speed.

A fuller discussion on the various phases of filtration from a centrifuge cake and a simple guidance on the effects of centrifuge and feed slurry parameters on initial filtration rate and final dryness under centrifugal filtration is given later.

7.1.1.4 Discharging

Discharging, or removal of the centrifuged cake from the basket, is an important part of the centrifuge cycle. There are various methods of cake removal depending on the type of centrifuge and these are discussed later. In most cases there is the possibility of crystal damage during the removal process. Typically (as in Figure 7.1) a mechanical blade removes the cake as the basket rotates slowly to limit the crystal damage. In many cases the screen is a woven cloth that is easily damaged or torn and it is therefore impractical to allow the mechanical blade to come into contact with the cloth and a small layer of crystals remains within the basket after the discharging operation. This layer is called the heel or residual bed and in some centrifuge types it can be as thick as 5–15 mm. Depending on the characteristics of the centrifuge cake the mechanical blade can cause a degree of smearing of the cake surface where the cake crystals are soft or alternatively cause crystal fracture with brittle crystals. In both cases the resultant effect is partly to seal the surface of the residual bed and this limits the filtration rate of subsequent batches processed in the centrifuge until the residual bed is removed.

One technique widely employed in batch centrifuges to remove the residual bed is the gas knife. A series of gas jets impinge on the residual bed and dislodge it without contacting the screen. This approach may be supplemented by a second set of gas jets fitted behind the basket which jet gas onto the back on the filter screen through the basket perforations. These techniques work well for cakes that are brittle and easily broken up. Cakes that smear (like toothpaste) are much more difficult to remove by the use of gas jets. Various alternative devices are available to remove the residual bed or prevent it from being formed in the first place.

7.1.1.5 Filtrate clarity

When feeding onto a clean screen with no residual bed the liquid in the feed, together with any crystals smaller then the openings in the screen, will flow through the screen and leave the centrifuge through the filtrate outlet. As feeding proceeds and a filter cake forms on the screen small crystals will become trapped in the newly formed filter cake and the filtrate will be virtually free of suspended solids. If the screen was covered with a residual bed prior to the start of feeding then the filtrate will be virtually clear throughout the process cycle. Generally batch centrifuges normally produce clear centrates (filtrates).

7.1.2 Basic filtering continuous centrifuge

Figure 7.4 shows a schematic of a continuous filtering centrifuge in its simplest form. The basket is a perforated cone that rotates at constant speed about its axis of symmetry.

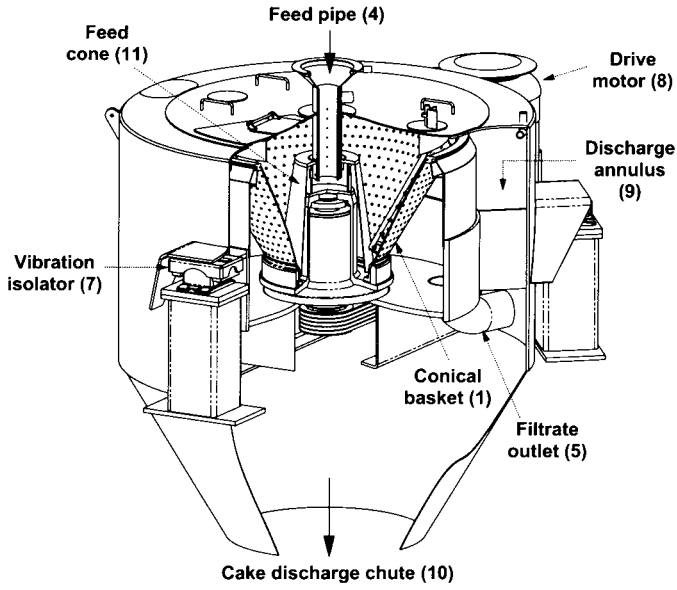

Figure 7.4 Typical continuous centrifugal filter (Broadbent Centrifuges)

Figure 7.5 shows a cross section of a conical basket (1) showing the perforations (2), filter screen (3) and solid/liquid mixture (12) in the process of being separated. After passing through the static feed pipe (4) the feed slurry is accelerated up the rotational speed of the basket in the imperforate feed cone (11) and then deposited on the filter screen at small diameter of the conical basket (1). The angle of the basket is chosen to match the friction characteristics of the feed solids to ensure that the solids will slide along the cone from the small to the large diameter as a result of the centrifugal forces acting (typical cone half angles are in the range 25°–34°). The filter cake decreases in thickness as the cake slides towards the large diameter of the cone. For this simple type of continuous centrifuge the cake thickness is in the range 5–15 mm at the small diameters of the cone and may be as low as 1 mm at the large diameter of the cone just prior to discharge.

The centrate is collected in the casing of the centrifuge and flows out via the filtrate outlet (5). The cake solids are collected round the large diameter annulus (9) between the outer casing and the large diameter

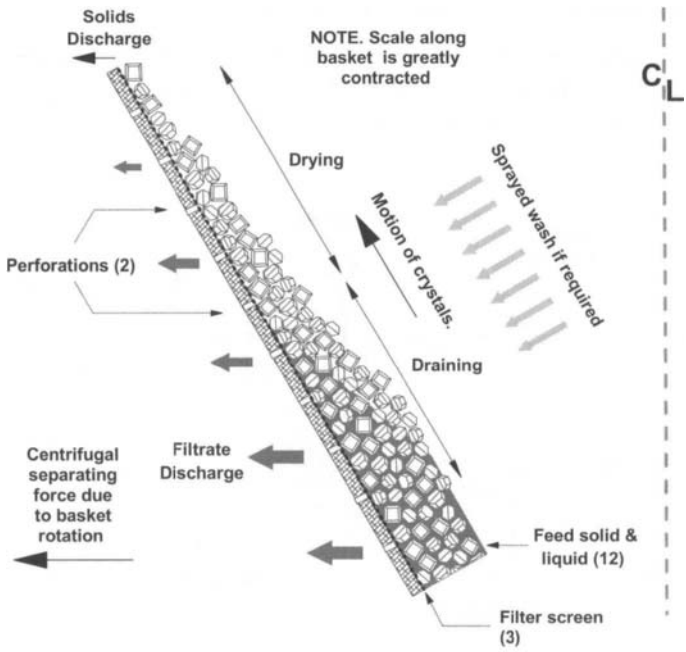

Figure 7.5 Cross section of the conical basket in a continuous filtering centrifuge

of the conical basket. The solids then fall under gravity out of the discharge chute (10). The unit is mounted on vibration isolators (7) and driven by a standard fixed speed motor (8).

It will be clear that the separation process is continuous and there is no need to feed a batch of slurry, and then accelerate, decelerate and discharge the centrifuged cake as is the case with a batch centrifuge. This inherent simplicity makes the continuous centrifuge popular, however the process performance is lower than a batch centrifuge which excludes it from many applications. Variants of the continuous centrifuge can overcome some of these difficulties but only at the expense of the main benefit of simplicity. The main variants of the continuous centrifuge are considered in more detail later.

One major difference between the batch and the continuous centrifuge is the movement of the solids relative to the screen. In the batch case the solids are stationary with respect to the screen, whereas in a continuous centrifuge the solids slide over the screen as they pass though the centrifuge. This difference has significant implications in terms of process performance and screen life.

7.1.2.1 Feeding

The feed system for a continuous conical centrifuge is generally very simple. A static pipe discharges feed into a rotating imperforate feed cone (item 11 in Figure 7.4) which is designed to perform two functions. Firstly the cone distributes the feed evenly over the conical basket and secondly it accelerates the feed slurry up to the rotational speed of the basket reducing crystal damage.

7.1.2.2 Washing and drying

The total transit time for crystals to slide along the conical basket is in the range 1 to 6 seconds depending on the characteristics of the feed material, the angle of the basket and the screen material. The time available for washing and drying is therefore short. This shortcoming is countered to a degree by the thin layer of crystals on the screen which dry more rapidly than the thick layer common in a batch centrifuge. In addition the expanding circumference of the conical basket forces the thin crystal layer to spread out as it moves towards the discharge liberating moisture that would otherwise have remained held by surface tension near the contact points of adjacent crystals. To counter the short residence high G is often used with values up to $2000G$ being common. Cake dryness is strongly dependent on the liquor viscosity and crystal size distribution in the feed with dryness in the range 1–10% for a mean crystal size of 200 μm or more. Crystal damage occurs as a result of the sliding of the crystals over the screen.

The short residence time limits the washing ability of this type of centrifuge. Large amounts of wash liquor can be used to 'flood' the fast moving cake but such an approach is often uneconomic.

7.1.2.3 Discharging

Discharged crystals leave the large diameter of the conical basket at close to the lip speed. For a 1200 mm diameter basket operating at 2000 rpm this is 125 m s^{-1} and as the energy dissipated when the crystal impacts the centrifuge casing increases as the square of the speed it can result in significant crystal damage. Lower speed operation and carefully designed centrifuge cases can reduce the damage considerably.

7.1.2.4 Filtrate clarity

During the passage of a crystal over the screen it will cross many screen openings (normally holes or slots). This presents ample opportunity for a crystal or, if breakage occurs on the screen, a fragment of a crystal to escape if it is smaller than the screen slot or

hole size. Generally the screens employed in this type of centrifuge are slotted metallic foil with a thickness of 0.3 mm and openings of 30 μm minimum and an open area of 5% or greater. Unless high screen losses and poor filtrate clarity can be tolerated the minimum slot size limits the particle size distribution that can be processed in conical centrifuges. For losses around 1% it is normal to use screens with a slot size of 20% of the mean crystal size – although actual figures depend on the crystal shape, size distribution and the ease with which crystals are damaged.

7.2 Filtering centrifuge types

There are many types of filtering centrifuge and this section considers some of the more significant examples used in the process industries. Figure 7.6 categorises filtering centrifuge types by three basic criteria:

- Batch/Continuous.
- Basket orientation : horizontal/vertical.
- Solids discharge method.

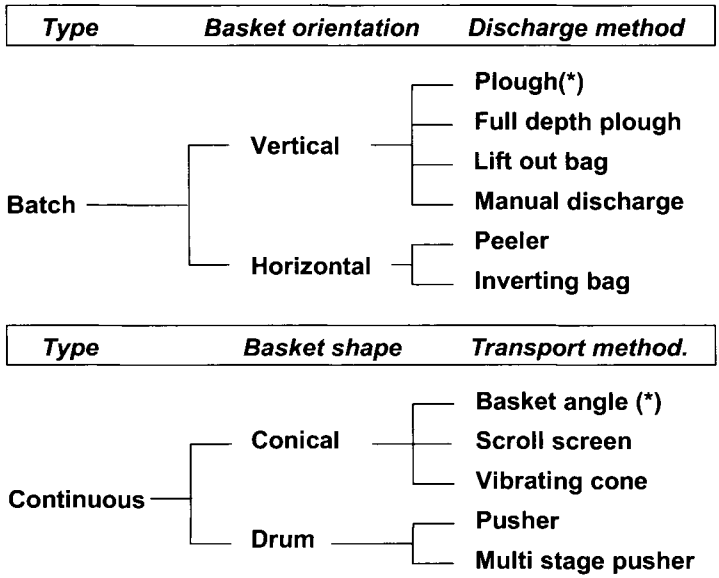

Figure 7.6 Simple classification of filtering centrifuges

The simple classification in Figure 7.6 is used to list a total of 11 variants of the filtering centrifuge. The two classifications marked by (*): 'Batch-Vertical-Plough' and 'Continuous-Conical-Basket angle' are also described the above sections. The remainder of this section highlights the major differences between each of the other variants as well as those described above.

Not all the possible filtration centrifuge types are covered by this classification – its purpose is to cover those common in the industry. For further details on additional types available in the market place see Purchas and Wakeman (1986) and Svarovsky (1990).

7.2.1 Batch filtering centrifuge types

7.2.1.1 Vertical plough discharge

Figure 7.7 shows a typical vertical plough discharge batch centrifuge. This type of centrifuge is common in the process industries and is a good general purpose unit and is available from pilot to large industrial scale. Basic information from a range of manufacturers is shown in Table 7.1.

Figure 7.7 Vertical plough discharge batch centrifuge (Broadbent Centrifuges)

Table 7.1 Commonly available vertical batch centrifuge sizes.

Basket diameter, mm	530	860	1000	1250	1250	1600	1600
Basket depth, mm	270	480	630	800	1000	1000	1275
Basket lip, mm	75	100	150	180	150	200	200
Capacity, L	30	120	250	500	620	900	1300
Maximum rpm	1850	1375	1250	950	1075	850	850
Maximum centrifugal *G*	1000	900	800	630	800	600	600

This type of centrifuge is common in general purpose process plant and is available with a wide variety of options including controls designed to cater for many differing product 'recipes', multiple wash capability and inert gas purging for use with hazardous chemicals.

Versions with the capability to overflow feed slurry over the basket lip during feeding are also manufactured as a combined classifying and filtering centrifuge. These are employed in applications where it is desirable to re-circulate fine crystals back to a crystallisation step to allow further growth. The overflow from the basket lip is collected in a trough built into the outer casing which allows the segregation of the filtrate from the overflow. The dewatering of gypsum in the wet scrubber flue gas desulphurisation process is a well known example.

Versions of this type of centrifuge are also made with imperforate baskets – however as these are sedimenting devices rather than filters they are not considered further here.

At the other end of the spectrum this type of centrifuge is also manufactured for use in processing pharmaceuticals where cleanliness and absence of cross contamination between batches is critical. These units are built with polished surfaces, clean in place system (CIP) and full access to the basket and ancillary items to allow validation of cleanliness in accordance with cGMP. Figure 7.8 shows the inside of the basket with CIP wash jets fitted to a batch centrifuge designed for pharmaceutical use.

Finally in this section there are a range of overdriven vertical batch centrifuges that are widely used in industries with high throughput requirements and very free filtering feed material, typical examples being dextrose and sodium nitrate – see Figure 7.9. Cycling rates are generally in the range 5–20 cycles per hour with basket working volumes up to 1.5 m^3 operating at 1100–1400*G* on feeds with a crystal size distribution of 150 μm or above. These designs are optimised for free filtering high throughput applications and are not suitable for general purpose use, inert gas purging or pharmaceutical levels of cleanliness.

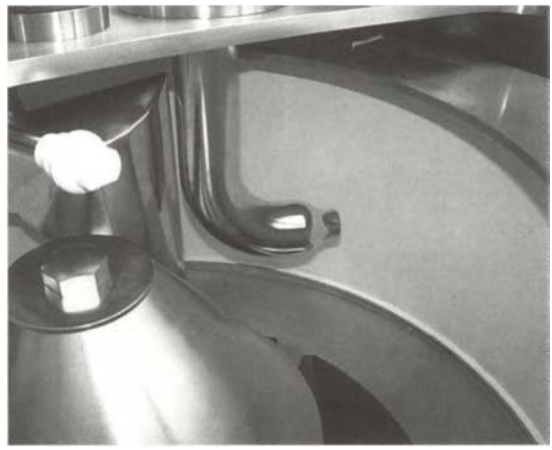

Figure 7.8 Batch centrifuge for pharmaceutical use (Broadbent Centrifuges)

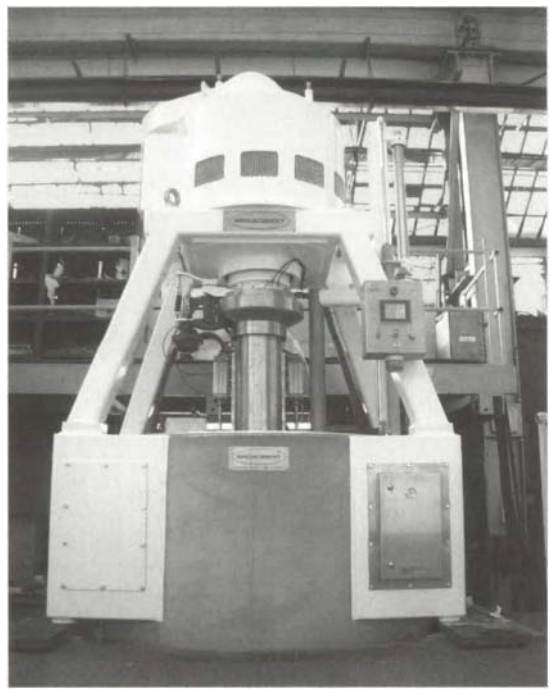

Figure 7.9 Overdriven batch centrifuge (Broadbent Centrifuges)

7.2.1.2 Vertical full depth plough

One variant of the vertical batch centrifuge uses a full depth plough where the up and down axis of movement is replaced with a plough blade that spans the whole depth of the basket. The headroom

requirements and mechanical complexity of the centrifuge are reduced. For brittle or friable cakes the full depth plough can reduce the cycle time and therefore increase the throughput of the centrifuge. For cakes that are hard or otherwise difficult to plough a smaller blade that traverses down the basket is preferred. Figure 7.10 shows a batch centrifuge fitted with a full depth plough.

Figure 7.10 Batch centrifuge with full depth plough (Broadbent Centrifuges)

7.2.1.3 Vertical lift out bag

Lift out bag centrifuges have a screen in the form of a bag made from woven filter material which is tailored to fit over the bottom, circumferential shell and top lip of the basket. The resulting bag acts as the screen during feed and spinning etc. in the normal way. To discharge, the basket is stopped and the bag is lifted out through the basket top. In some cases the bag is lifted out together with a removable top of the basket. This operation usually requires a small hoist. One approach is shown in Figure 7.11 where the discharge is made simpler by forming the bag from a cylinder of woven screen material. The advantages of the lift out bag discharge system are that the centrifuge is mechanically simple, the filter screen is easy to clean with no residual bed problems and crystal damage is near zero. However, the disadvantages are significant. Of particular significance is the requirement for the operator to come into close contact with the contents of the basket. This is generally unacceptable especially with corrosive, toxic or inflammable materials etc. A secondary disadvantage is the long time necessary to discharge the cake and refit a bag for the next batch.

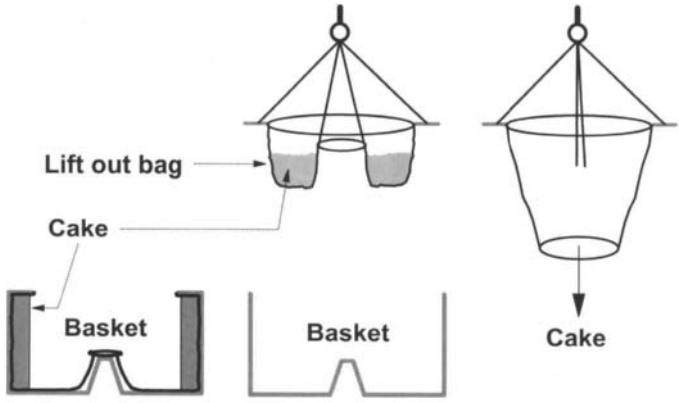

Figure 7.11 Lift out bag discharge

Lift out bag centrifuges are available with diameters 500–1250 mm with capacities of 0.03 – 0.3 m^3. Lift out bag centrifuges are generally only used for low cost non toxic materials or small scale production or pilot plants.

7.2.1.4 Vertical manual discharge

The manual discharge or dig-out centrifuge is the most basic unit available. Its only advantage is mechanical simplicity and low cost. The method of discharge requires that the operator comes into contact with the contents of the basket which raises important safety and exposure issues when processing hazardous material.

It is generally only used in laboratory work and pilot plant trials. The size range available reflects this limited use. Typical diameters are 200–600 mm with capacities 5–50 litres. The maximum G available is generally high with 1500G being quite common. Figure 7.12 shows a 900 mm unit.

7.2.1.5 Horizontal peeler

Peelers are available in two versions, firstly as a heavy duty version for use in the bulk chemical industry and secondly as a cGMP (current Good Manufacturing Practice) design suitable for use in the pharmaceutical industry. The significant difference between a peeler and the vertical batch centrifuge is the horizontal axis of the basket and the possibility of feeding and discharging whilst the basket is rotating at high speed. A full depth blade is used to 'peel' out the cake at high speed (see Figure 7.13). The feed pipe is mounted horizontally and arranged to pour the feed directly onto the basket wall through a slot running the whole depth of the basket. Where the feed material being

Figure 7.12 900 mm diameter simple manual discharge centrifuge (Broadbent Centrifuges)

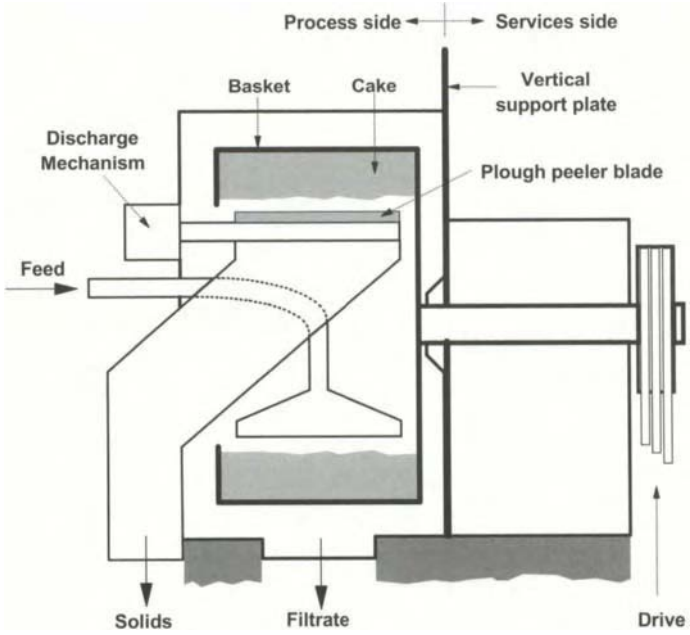

Figure 7.13 Schematic of a peeler centrifuge

processed is suitable then feeding and discharging can take place at a high basket speed which avoids the acceleration and deceleration delays present with a vertical batch centrifuge. This gives the peeler throughput advantages.

Large forces can act during the peeling operation and the discharge mechanism, basket and bearing support etc. must be sufficiently rigid. Peelers are therefore heavy and bulky and tend to be more expensive than the lighter vertical basket centrifuges. Figure 7.13 shows these key features schematically, and Figure 7.14 shows a typical peeler suitable for pharmaceutical use.

Figure 7.14 Peeler for pharmaceutical use (Broadbent UK & Riera Nadeu S.A.)

Figure 7.13 shows the discharge chute for the solids and the peeler blade in the parked position. The blade is designed to direct the solids directly down the chute. In an alternative arrangement the solids are not directed down the chute but removed by a screw conveyor in the basket which transports the solids through the case front. The positive transport of the solids permits the use of deeper baskets where the angle of a chute would be too shallow for reliable discharge. Chute discharge is simpler and easier to clean than screw discharge making it better suited to pharmaceutical use. Screw discharge is more common on high throughput chemical applications such as starch.

Peelers are normally built with all process related areas grouped together on one side of a large vertical support plate and all drive, bearings and other ancillary services on the other. The segregation of process related areas on one side of the vertical support plate (see

Figures 7.13 and 7.14) allows the centrifuge to be installed with the support plate forming part of a wall such that the process areas are located in a clean room. Feeding and discharging are from the front of the basket which avoids solids build up near the spindle at the back of the basket. Cleanliness is further enhanced by the full opening case providing excellent access to the basket, feed pipe and discharge mechanism making the peeler well suited to use on products where containment and the avoidance of cross contamination are important. Table 7.2 shows a selection of sizes available. Some models are available with deeper baskets when using screw discharge.

Table 7.2 Commonly available peeler centrifuge sizes.

Basket diameter, mm	650	800	1000	1250	1400	1600	1800
Basket depth, mm	300	400	500	625	700	800	1000
Basket lip, mm	100	115	150	190	210	230	280
Capacity, L	50	100	200	400	550	820	1350
Maximum rpm	2200	1900	1500	1200	1100	950	900
Maximum centrifugal *G*	1750	1600	1250	1000	940	800	800

Washing and residual bed removal techniques are similar to those used with a vertical batch centrifuge. The high speed feed and discharge will lead to greater crystal damage than a vertical batch centrifuge and fragments can become embedded in the surface of the residual bed significantly increasing the resistance to flow and leading to feeding and drying problems in subsequent batches. Alternatively, cakes liable to smearing will glaze over leading to the same problems necessitating the removal of the residual bed, normally via a gas jet applied behind the basket or along the peeler blade. This technique does not work with all cakes but is most successful with friable cakes. Bed removal also requires that the basket speed is reduced to below one G – or centrifugal force will maintain the residual bed in contact with the basket. The need to reduce the speed may remove much of the high throughput advantage; however peelers are used in some applications where slow speed discharge is necessary. For slurries with a low filtration rate or fragile crystals prone to smearing or glazing the vertical batch or inverting bag centrifuge may be preferable.

7.2.1.6 Horizontal inverting bag

The inverting bag centrifuge has a horizontal axis basket similar to the peeler however the method of cake discharge automates the approach used in the manual lift out bag design. Figure 7.15 shows a schematic of a typical inverting bag discharge mechanism.

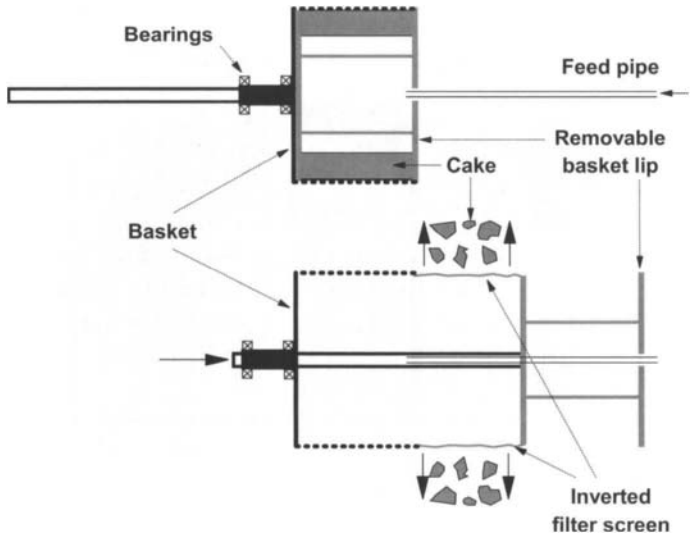

Figure 7.15 Schematic of inverting bag mechanism

Discharge occurs at a low basket speed by inverting the filter bag as illustrated in Figure 7.15. No plough blade is needed and crystal damage is minimised. The inversion of the bag breaks up any residual bed on the screen (filter bag) allowing the next batch to be fed onto a clean cloth. Typical centrifuge sizes are shown in Table 7.3. Feeding, washing and drying are similar to the peeler centrifuge. The dragging of the cake out of the basket during the inversion process is an arduous duty and filter bag life is less than the screen life in other batch centrifuge types; however the ability to remove the residual bed is an important benefit. Figure 7.16 shows an inverting bag centrifuge for use in the pharmaceutical industry. The positive discharge mechanism and residual bed removal allows the inverting filter centrifuge to be used on feed materials with a fine particle size distribution – down to less than 5 μm, however cycle times are long. Inspection of the area behind the filter bag necessitates the removal of the bag to obtain full access. Relative to their basket capacity inverting bag centrifuges require significantly more floor area than a vertical batch centrifuge.

Table 7.3 Inverting bag centrifuge sizes.

Basket diameter, mm	300	450	600	800	1000	1300
Capacity, L	6.5	26	52	120	200	350
Maximum rpm	3000	2300	1940	1600	1270	1000
Maximum centrifugal *G*	1500	1320	1250	1140	900	720

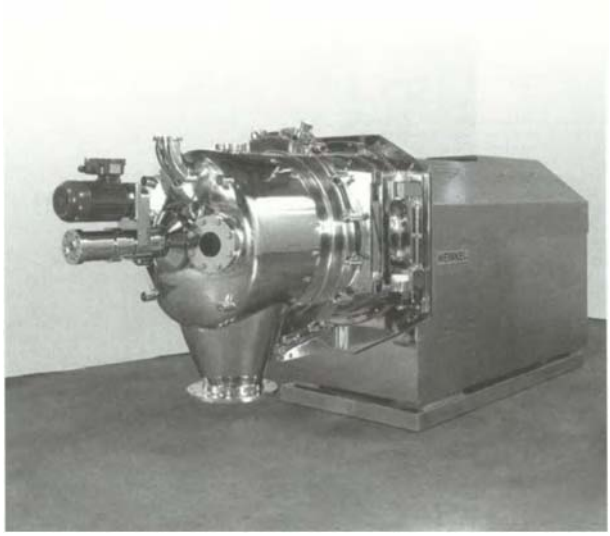

Figure 7.16 Inverting bag centrifuge for pharmaceutical use (Heinkel Filtration UK)

An interesting variant of the inverting bag centrifuge is produced by Heinkel (PAC® System). After centrifugation is complete the feed pipe (see Figure 7.15) is used to introduce a hot gas which flows out through the cake, drying it as it does so. Once the process is complete the cake is discharged by inverting the bag. This arrangement requires a rotating seal between the static feed connection and the rotating basket to support the pressure of the hot gas during the drying phase. With suitable feed materials very low dryness can be achieved.

7.2.2 Continuous filtering centrifuge types

Where suitable for the application continuous centrifuges are generally preferred. Normally they do not need a buffer storage upstream/downstream to cater for the uneven flows inherent in a batch centrifuge. For many continuous centrifuges the controls are simpler as there are no multi-step process cycles. Other benefits may include reduced power consumption and mechanical simplicity.

The disadvantages are generally a reduced process performance, greater losses of solids with the filtrate and screen wear. The balance between the advantages and disadvantages depends upon the particular application and the type of continuous centrifuge selected. The sections below consider the advantages and disadvantages of several common types of continuous centrifuge.

7.2.2.1 Conical basket

The key features of the conical basket centrifuge are described in Section 7.1.2. The sweetener industry is a large user of this type of centrifuge, for example see Figure 7.17.

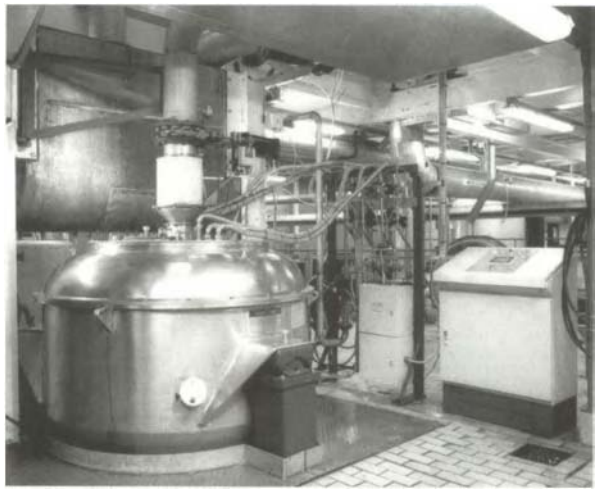

Figure 7.17 Conical basket centrifuge as used in the sugar industry (Broadbent Centrifuges)

The centrifuge shown in Figure 7.17 has a dual angle basket. The lower section is at 15° with 25° on the upper section. The 15° portion is designed to remove as much liquid from the feed as possible whilst still maintaining its fluidity. Before the cake loses its fluidity it has progressed to the 25° section where the steeper angle keeps the cake in motion along the remainder of the basket. Discharging the liquid at a small radius has two benefits, firstly less kinetic energy is lost with the liquid which reduces power consumption and secondly a larger portion of the basket area is available for washing and drying the solids.

The example in Figure 7.17 is fitted with a domed top to reduce crystal damage by limiting the impact angle between the crystals leaving the basket lip and hitting the centrifuge casing. Careful design of the case allows the impact angle to be reduced from approximately 45° found in simple cylindrical cases to 6–7°. This, coupled with lower operating speeds on some applications, greatly reduces the crystal damage caused by these conical basket centrifuges.

The short residence time (1 to 6 seconds) necessitates that concentrated feeds are used with more than 25% solids w/w. Screens are generally metal foil with etched slots in the range 20–120 μm. The sliding action

of the solids over the screen causes both screen wear and crystal damage. Etched foil screens typically last 1 to 3 months of continuous operation, however wedge wire and laser cut stainless steel foil screens are now in use which last significantly longer. In addition to sucrose, alternative uses for conical centrifuges include lactic acid, starch, salt and low protein fibre from grain.

The simple design of the centrifuge makes it easy to clean and inspect, however they are not generally used in the pharmaceutical industry due in part to their limited washing ability and high screen losses when processing feeds containing crystals under 150 μm.

Table 7.4 A selection of conical centrifuge sizes.

Basket diameter, mm	1070	1220	1425	1575
Maximum rpm	2200	2000	1800	1600
Maximum centrifugal *G* at basket lip	2890	2730	2580	2250
Maximum throughput, t h^{-1}	20	25	40	50

7.2.2.2 Scroll screen

The scroll screen centrifuge is mechanically similar to the conical basket centrifuge of Section 7.2.2.1. The main difference is the addition of a helical conveyor that has a small differential speed relative to the conical basket. The helical conveyor is used to control the transport of the cake allowing the residence time of the solids in the basket to be increased giving improved process performance.

Figure 7.18 shows a section of a scroll screen centrifuge. The conveyor and basket cone angle are designed such that the conveyor drags the solids along the cone towards the discharge. The solids do not form a uniform layer on the screen but form piles of triangular section in front of the blades of the conveyor. The residence time within the centrifuge, typically 4 to 15 seconds, is longer than the simpler conical basket centrifuge allowing a reasonable contact time between any wash liquor and the cake. However the non-uniform layer of solids on the screen limits the efficiency of the wash. The basket may comprise sections with two or more angles, 10°, 15° and 20° being common.

As can be seen from Figure 7.18, scroll screen centrifuges are relatively complex and difficult to clean although units are available which provide clean in place facilities. In addition sealed units incorporating inert gas purging are also available, however the scroll screen centrifuge is not widely used in applications where cleanliness is paramount.

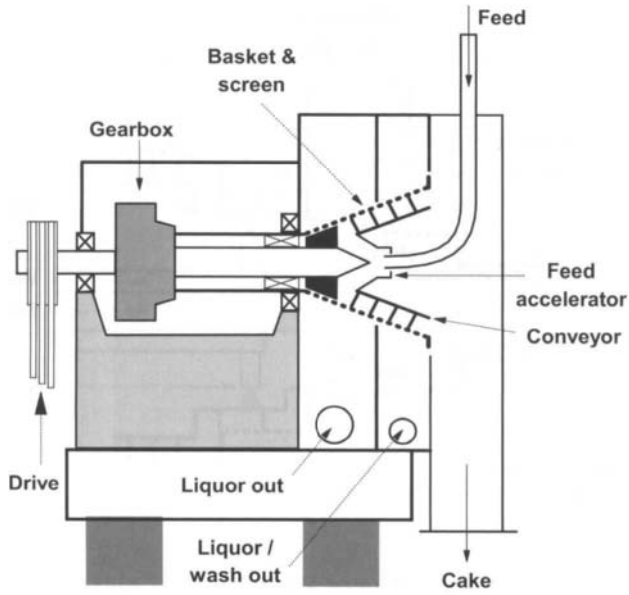

Figure 7.18 Schematic of a scroll screen centrifuge

For good throughputs the scroll screen centrifuge requires a high solids content in the feed – typically greater than 15% and up to 60% w/w. Screens are generally metallic foil or wedge wire and are available in a variety of materials. Slot and hole sizes vary depending on the application but are typically in the range 40–200 μm with open areas from 5–15%.

As with the conical centrifuge screen wear is inevitable and crystal damage occurs both on the screen and during discharge. There is a clearance between the screen and the conveyor blades, normally between 0.3 and 1 mm depending on the basket size and application, which can be adjusted to allow some compensation for wear on the blade tips. A small additional element of crystal damage is caused through the conveyor blades grinding the crystals which pass under the blade tip.

Scroll screen centrifuges are available with up to four separate stages. The first stage is used to de-liquor the feed and this can be followed by a wash stage, with the last stage being used for final drying. For a four stage unit two separate washes can be applied with the possibility of segregating the wash liquors. Figure 7.19 shows the arrangement of a three stage scroll screen centrifuge with washing on the middle stage and segregation of the wash liquors. Figure 7.20 shows a complete machine and Table 7.5 shows some typical sizes.

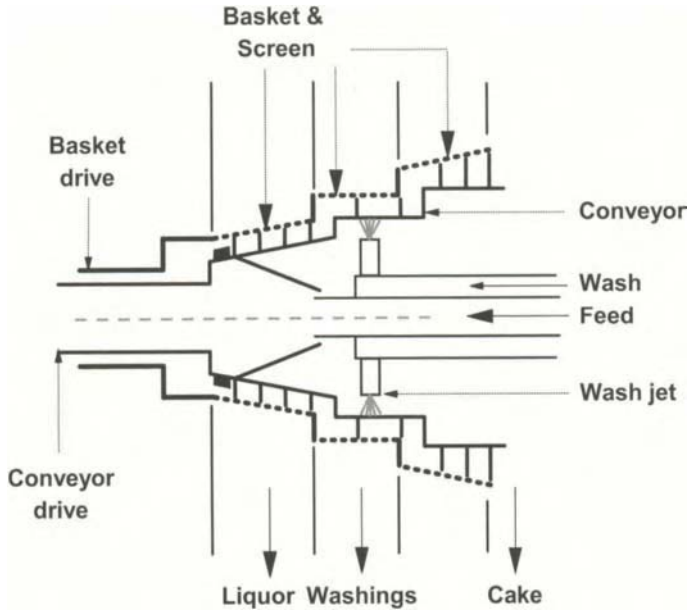

Figure 7.19 Schematic of three stage scroll screen centrifuge

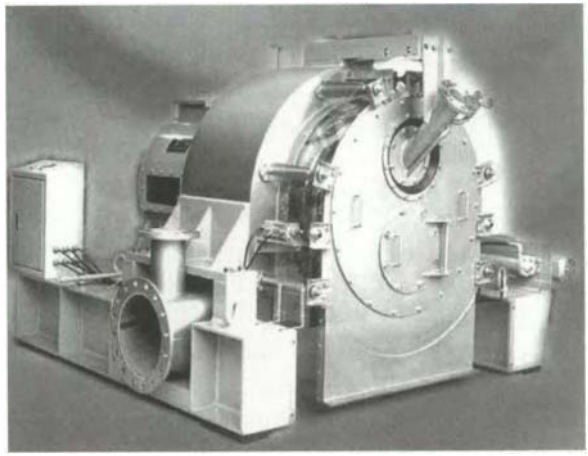

Figure 7.20 Typical scroll screen centrifuge (Broadbent UK & Tanabe Willtec Inc. Japan)

Uses for the scroll screen centrifuge include ABS, BPA, gypsum, sucrose, citric acid, copper sulphate and polystyrene. Mean feed crystal sizes of 100 μm or above are suitable for the scroll screen centrifuge. Final liquor content depends on the application but is usually in the range 2–20% w/w for crystalline solids.

Table 7.5 A selection of scroll screen centrifuge sizes.

Basket diameter, mm	250	400	550	800	1000
Maximum rpm	3800	3000	2000	1500	1200
Maximum centrifugal *G* at basket lip	2000	2000	1500	1000	800
Maximum throughput, t h^{-1}	3.5	10	25	50	70

7.2.2.3 Conical vibrating basket

Conical vibrating basket centrifuges are widely used in the mineral and mining industries. They have a low operating speed and very high throughputs of up to 350 t h^{-1}. Figure 7.21 shows the principle of operation. The conical basket angle is in the range 13° to 15° less than that required to make the crystals of the feed slip along the cone. To transport the crystals an additional axial vibration is imposed on the basket which acts in a similar manner to a shaking screen causing the crystals to move towards the large diameter. A degree of control over the solids residence time within the basket is possible by altering the magnitude of the imposed axial vibration.

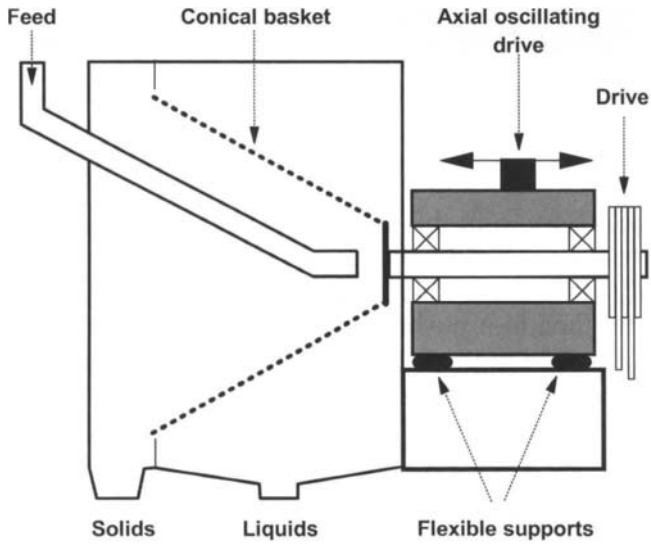

Figure 7.21 Schematic of a conical basket vibrating screen centrifuge

Screens must be robust and are manufactured from wedge wire with slots in the range 300–600 μm depending on the application. Mechanical considerations limit the maximum *G* that can be produced with the vibrating screen centrifuge and most operate in the range 75–150 *G*. The centrifuge is designed for large solids particles of between

0.5 and 50 mm and discharges solids with a surface liquid content of 5–10% w/w depending on the particular application. High capacities are possible with these centrifuges with up to 350 t h^{-1} for a unit fitted with a 1500 mm diameter basket. To achieve this high throughput feed solid concentrations in excess of 50% are needed. These centrifuges are commonly used for dewatering coal, Figure 7.22 shows an example.

Figure 7.22 Conical basket vibrating screen centrifuge (Ludowici Pty.)

7.2.2.4 Pusher

The pusher differs from the continuous centrifuges described above in two key respects. Firstly the basic pusher basket is predominantly cylindrical rather than conical and resembles the basket of a peeler and secondly the feed to a pusher is continuous, however the discharge is discontinuous. The motive force to discharge the solids from a pusher is a reciprocating pusher plate fitted within the basket. Figure 7.23 shows the basic arrangement schematically.

A cycle of the pusher mechanism starts with the plate shown in Figure 7.23 extending by moving towards the right pushing the previously formed cake towards the open end of the cylindrical basket. The plate then retracts to the left leaving a space between the remaining cake and the pusher plate which is filled by the incoming feed which drains leaving fresh filter cake. The cycle then repeats and the cake moves in steps along the basket cylindrical surface until discharged. The plate is shown extended in the top half of Figure 7.23 and retracted in the bottom. Pushers work well with freely filtering cakes that are sufficiently strong not to buckle under the force of the pushing plate.

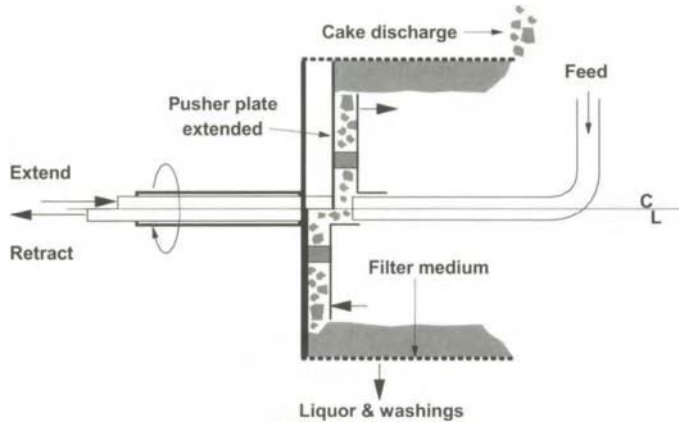

Figure 7.23 Schematic of a pusher centrifuge basket

Typically the reciprocating cycle repeats 10–100 per minute with a stroke of 20–80 mm.

Table 7.6 shows typical data for a range of pusher centrifuge sizes with Figure 7.24 showing a typical unit for use in the chemical industry.

Table 7.6 Typical pusher centrifuge data.

Basket diameter, mm	250	315	400	500	630	800	1000	1250
Maximum rpm	2400	2100	1900	1700	1500	1300	1200	1070
Max centrifugal *G*	800	800	800	800	800	800	800	800
Solids capacity, t h^{-1}	3.5	5.5	10	14	20	33	60	90

Figure 7.24 Pusher centrifuge for use in the chemical industry (Krauss Maffei Process Technology Ltd.)

For best results pushers require a feed concentration between 25% and 75% w/w on feeds with a mean crystal size in the range 100–5000 μm.

The liquor content of the discharged solids depends on the application but can be as low as 1% w/w with larger crystalline products. Pushers are used on many applications including ABS, BPA, sodium chloride, citric acid and DMT.

The motive power for the pusher plate is a mechanical linkage or eccentric drive for smaller units; larger units use a hydraulic actuator. Screens are wedge wire with the slot aligned with the direction of motion of the crystals. Slots are in the range 40–200 μm as required by the application. The sliding action causes a degree of crystal damage and wear of the screen. The solids are discharged at the rotational speed of the basket and further damage is caused by the impact of the crystal with the centrifuge casing although this can be reduced by the fitting of a static deceleration ring which lessens large angle impacts on discharge. The sliding of the cake over the screen slots allows some of the smaller crystals and any fragments created by the sliding action to escape giving higher solids in the filtrate than a batch centrifuge. Solids recoveries of 98% can be achieved with larger crystals not prone to damage.

The uniformity of the cake within a pusher coupled with a reasonable residence time (6–20 seconds) provides good conditions for washing. Multiple washes are possible and wash liquors can be segregated within the machine. To achieve good drying and washing the cake must be able to support the forces exerted by the pusher plate without buckling. The load the cake must support during the pushing action depends on the length of the pusher basket and the characteristics of the cake itself such as the friction between the screen and the cake and the lubrication effects of any liquor present. For materials which form weak cakes the length of the basket must be limited and for paste like cakes the transport mechanism may fail with the cake tending to pile up at the feed end of the basket.

If the cake buckles then cake drying and washing performance falls off substantially. In an effort to overcome such difficulties the multistage pusher was developed.

7.2.2.5 Multistage pusher

The principle of the pusher centrifuge can be extended by splitting the basket into several stages and employing a separate pusher plate for each stage. Figure 7.25 shows this approach schematically for a two stage pusher.

Up to 4 stages are used with the basket diameter increasing at each stage. Multistage pushers confer several advantages. The use of several pusher plates allows weaker cakes to be transported successfully and

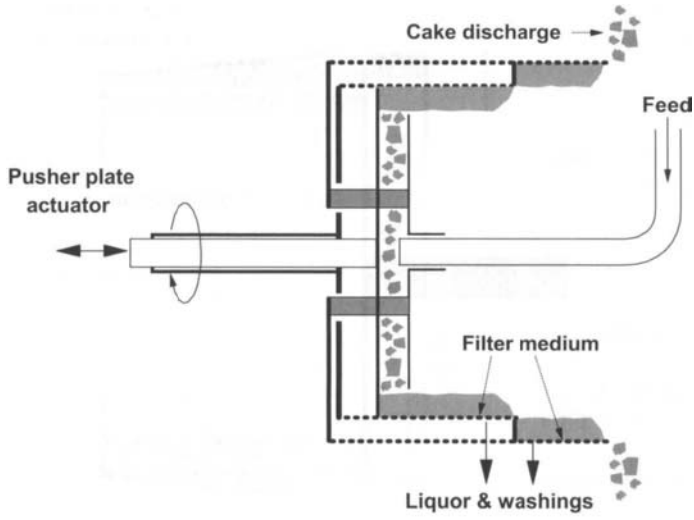

Figure 7.25 Schematic of a multistage pusher centrifuge basket

the transfer from one basket to the next breaks up the cake, which helps to liberate liquor trapped between the crystals improving washing and drying. The separate stages are also a convenient method of segregating multiple washes.

The main disadvantage is complexity leading to higher purchasing and maintenance costs. Multistage pushers tend to have a higher level of solids in the filtrate. A proportion of the finer crystals that were within the main body of the cake and remote from a screen opening on the first stage will end up adjacent to the screen after transfer to the second stage. These finer crystals may then be lost through the screen whereas in a single stage pusher they would remain trapped in the cake.

7.2.3 Syphonic basket

The syphonic basket, developed by Krauss Maffei, is an interesting variant on the batch centrifuge basket design applicable to peelers and vertical batch centrifuges. The principle of operation involves retaining a proportion of the filtrate within a additional chamber which forms part of the basket. A difference in liquor level between the chamber and liquor draining through the cake produces an additional driving pressure that aids filtration – see Figure 7.26.

The driving force for filtration caused by the centrifugal action is given by the product of the liquid density, the centrifugal acceleration and the liquid thickness within the cake h, and this can be thought of as a

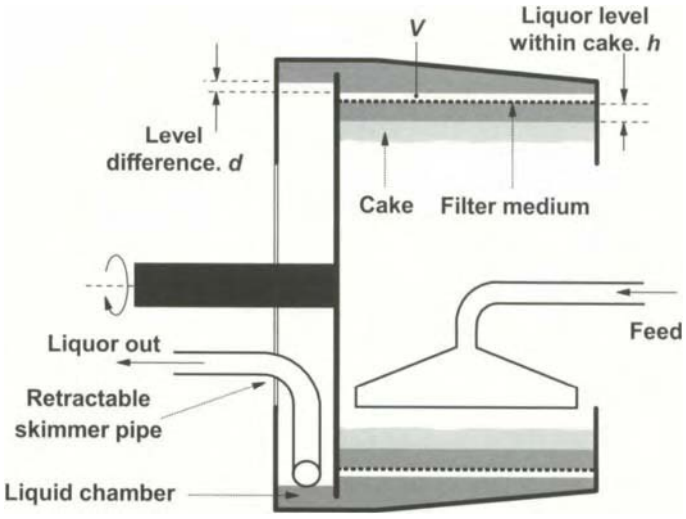

Figure 7.26 Schematic of a syphonic basket

centrifugal pressure. In the syphonic basket there is a further pressure differential of slightly less than 1 atmosphere produced by the difference in level d produced when the skimmer pipe, acting as a simple centrifugal pump, is used to skim liquor out of the liquid chamber. The pressure in volume V then approximates to a vacuum (in practice it will be at least the partial pressure of the liquor being separated). For thick liquid levels (i.e. large h) the effect of this additional pressure differential on the drying performance of the centrifuge is slight as the centrifugal pressure is significantly larger then 1 atmosphere. However as filtration proceeds h and the centrifugal pressure reduce whilst the syphonic effect remains unchanged. It is useful to calculate the liquor level h for which the syphonic pressure effect equals the centrifugal pressure. Taking an example of a cake containing liquor of density 1000 kg m^{-3} in a centrifuge operating at $1000G$ then, for a cake thickness that is small relative to the basket diameter, the maximum syphonic pressure (1 atmosphere) equals the centrifugal pressure when the liquor level h within the cake is approximately 10 mm. In other words, the syphonic pressure is the larger force driving drainage for liquor depths within 10 mm of the basket wall. Clearly for a total cake thickness of perhaps 100–150 mm this is a small effect when considering the deliquoring of the entire cake. A syphonic basket always has a larger outside diameter than a standard basket of the same internal dimensions due to the external liquor chamber. An alternative comparison would be to compare a syphonic basket to a standard basket of the same outside diameter and the same volumetric capacity. The standard basket would

then have a thinner cake and higher G than the syphonic basket of the same outside diameter.

The main benefit of the syphonic basket is not improved drying but the ability to retain the liquor in contact with the cake. Normally wash liquor leaves the basket after passing through the cake however with a syphonic basket wash liquor can be kept in contact with the cake by retracting the skimmer pipe prior to washing. This is an advantage for applications requiring a long contact time between the cake and the wash liquor. In certain applications the syphonic basket can be used to rejuvenate the residual bed by back washing and dissolving fine crystals blocking the bed.

7.2.4 The screen

All batch centrifuges use a screen as an inner lining to the basket and the type and fixing varies according to the separation duty. There are four general types: woven screens, etched slotted screens, punched plates and wedge wire screens. Some fabric screens are held in place using a caulking groove in the basket top and bottom. The groove is filled with a caulking material (e.g. PTFE rope) which traps the filter cloth in the groove. Another method employs expanding metal rings which are fitted at the top and bottom of the basket and expand to trap the screen material, this technique works well for both cloth filters and thin metallic screens. A further method employs industrial 'velcro' strip sewn into the edges of a filter cloth. Matching velcro strips are mechanically fixed to the top and bottom of the basket using pegs fitted through the basket perforations. The filter cloth is then pressed in place. This technique, whilst convenient, requires that the velcro strip materials are compatible with the process and the cleanliness requirements are not compromised by the inevitable gaps and crevices formed by the strips.

Thick metallic screen such as punched plate used in overdriven batch centrifuges (Section 7.2.1.1) can be self supporting. Wedge wire screens in pushers (Section 7.2.2.4) and foil metallic screens in continuous conical centrifuges (Section 7.2.2.1) are fixed with mechanical clamps that retain the screen and resist the frictional forces transferred to the screen by the moving solids.

If the screen is fitted directly onto the basket wall (or shell) then only that area of the screen covering a basket perforation will be able to discharge liquor – all other areas will be blinded by the basket wall. To overcome this it is normal to use a coarse backing mesh between the screen and the basket wall to act as a manifold to allow drainage from

the entire surface area of the screen to the basket perforations. The backing mesh can be woven wire, plastic or similar material capable of resisting the crushing forces of the filter cake at spin speed. Some screens (e.g. wedge wire) are constructed such that the backing mesh forms part of the screen itself.

There are many types and manufacturers of screen. Table 7.7 shows the range of some important parameters for a selection of screen types.

Table 7.7 Typical screen parameters.

Screen type	*Slot or hole size* (μm)	*Open area* (%)	*Material of construction*
Wedge wire	35–500	10–30	Special section rolled stainless steel or alloy wire.
Perforated plate	40–500	10–30	Stainless steel sheet 0.15–0.5 mm thick.
Foil screen	20–150	3–15	Nickel or stainless steel foil 0.15–0.3 mm thick.
Woven wire screen	5 –100	1–5	Fine stainless or alloy wire.
Cloth screen	5–60	See note 1	Various natural or man made fibres.

Note 1: For cloth screens it is normal to define air permeability rather than open area. Typical values are 10–50 l/dm^2/min at 20 mbar

7.2.5 Materials of construction

All batch centrifuges considered in this section and many of the continuous centrifuges are available in a wide range of materials of construction including all types of stainless steel. Those intended for the chemical and pharmaceutical industries are also available in titanium, Hastelloy®, nickel, Inconel and proprietary inert coatings such as Halar or Fluoroshield. Some designs use thin linings of expensive alloys on mild steel or stainless steel to reduce the costs of components such as the centrifuge case or the base of the basket. Careful design is required to ensure that differential thermal expansion between the lining and base material does not lead to tearing. Periodic inspection of lined or coated components is recommended to ensure that they are undamaged and no corrosion is resulting from a tear or perforation. This is particularly important for baskets with inert coatings.

7.2.6 Basket size limitations

There is a trend towards large process plant and therefore larger centrifuges. The currently available material for the construction of centrifuge baskets places a limit on the size of practical units. The relationship between centrifugal acceleration G, basket revolutions per minute N and diameter D is:

$$G = \frac{D}{2}\left(\frac{2\pi N}{60}\right)^2 \tag{1}$$

The hoop stress σ_h in the basket material is approximately proportional to:

$$\sigma_h \propto D^2 N^2 \tag{2}$$

The centrifugal acceleration G is proportional to D but the hoop stress in the material of the basket is proportional to D^2. This is important because the maximum G that can be produced with a given material of construction decreases in proportion to the diameter of the basket – so larger centrifuges are limited in the G they are able to support. This effect can be seen in the tables of centrifuge basket sizes and G presented earlier in this section for some centrifuge types.

Consider an example of a basket with a capacity of 1000 kg of material of density 1000 kg m^{-3}. The basket operates at a G of 1000 and a cake thickness of 150 mm to produce the required cake dryness. Figure 7.27 shows the effect of differing basket diameters on the basket shell material self stress and weight. The self stress is the hoop stress in the shell of an empty basket spinning at the speed necessary to generate 1000G. In Figure 7.27 the self stress is shown as the percentage of the maximum allowable material stress – with anything left over being available to support the centrifuge cake. Figure 7.27 also shows the total weight of the basket excluding the 1000 kg of feed material.

It is clear in this example that at a diameter of approx 3500 mm all the basket shell material strength is used simply to support its own weight against the centrifugal G. It is also clear that as the diameter rises towards 3500 mm the mass of the basket rises sharply approximately as the square of the diameter. This suggests that it is more efficient to have a small diameter higher speed basket as the basket mass is reduced and more of the shell strength is available to support the load of the cake etc. This is the reason the highest G centrifuges are in the form of a very small diameter cylinder rotating about its long axis. For

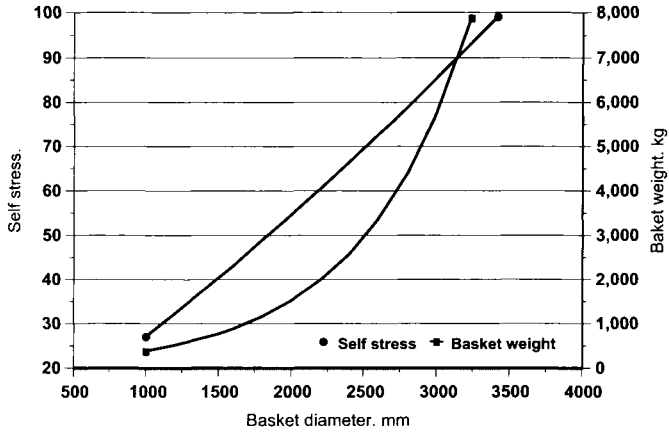

Figure 7.27 Effect of basket diameter on basket weight and self stress

practical applications small diameter long centrifuges are unsuitable because access for washing, feeding and discharge is too restricted and it can be difficult to get the product to form a cake of even thickness. The competing requirements of access and efficient use of material and lower weight (therefore cost) makes the basket size in the range 1000 to 2000 mm the most efficient.

In practice this means that the largest centrifuges available are usually only used for freely filtering materials that require lower G to produce an acceptable separation and dryness. The highest G is limited to the smaller centrifuge basket diameters. These comments apply to all centrifuges, not just filtering centrifuges.

7.2.7 Alternative classifications

In Section 7.2 a simple classification of centrifuge types was employed based on three criteria: batch/continuous; basket orientation (vertical / horizontal); and discharge method. This classification is largely arbitrary and was chosen to provide a logical framework to describe common types of filtering centrifuges. Alternative classifications are presented below based on feed mean crystal size (Figure 7.28), feed solids concentration (Figure 7.29) and the ability to wash the cake (Table 7.8). These classifications can assist in the centrifuge selection process (Anon, 2001).

The classifications on mean crystal size and feed solids content show the normal range of operation of each of the centrifuge types described above together with an extended region where reduced performance can be expected. The reduction in performance will manifest itself in one

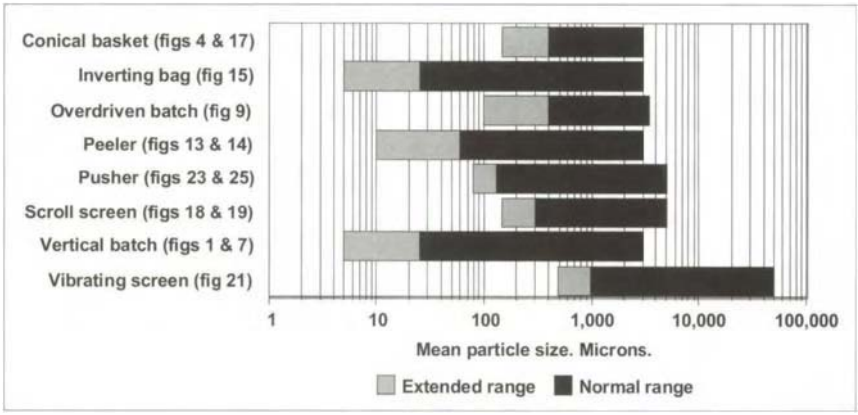

Figure 7.28 Simple centrifuge classification based on feed mean crystal size

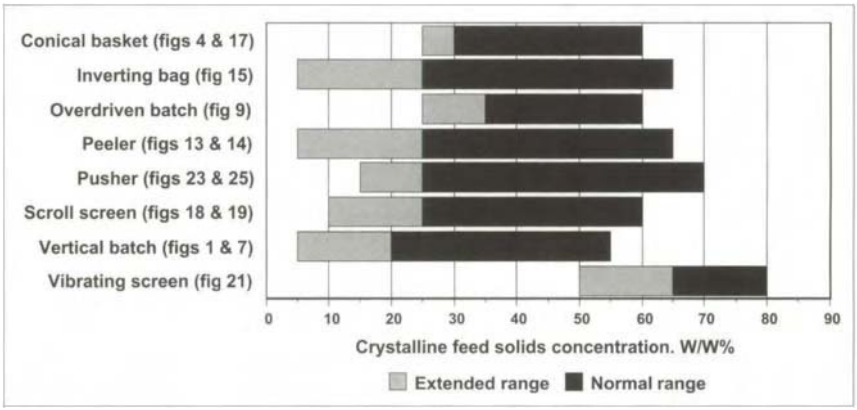

Figure 7.29 Simple centrifuge classification based on feed solids content

Table 7.8 Summary of washing performance.

Centrifuge type	*Washing ability*	*Multiple washes possible*	*Segregation of wash liquors*
Conical basket (Figures 7.4 & 7.17)	Limited	No	-
Inverting bag (Figure 7.15)	Very good	Yes	Very good
Overdriven batch (Figure 7.9)	Very good	Yes	Very good
Peeler (Figures 7.13 & 7.14)	Very good	Yes	Very good
Pusher (Figures 7.23 & 7.25)	Good	Yes	Good
Scroll screen (Figures 7.18 & 7.19)	Good	Yes	Good
Vertical basket (Figures 7.1 & 7.7)	Very good	Yes	Very good
Vibrating screen (Figure 7.21)	Limited	No	-

or more ways including reduced throughput, difficulties in feeding, cakes with higher liquid content and, for continuous centrifuges, greater loss of crystals to the filtrate. The classifications are general and depend on parameters not discussed in detail here such as crystal shape, spread of crystal sizes either side of the mean, liquor viscosities, etc.

7.3 Pilot scale tests and scale up

Section 7.2 provides information to assist with the selection of a centrifuge type likely to be suitable for a particular duty. However the estimation of process performance is of equal or greater importance and the integration of the centrifuge into the upstream and downstream process plant requires information on many facets of the centrifuge performance such as cake dryness, feed rate, wash consumption, solids recovery and centrifuge throughput.

The better the understanding of the centrifugal filtration process the simpler and more accurate is the prediction of performance. Unfortunately the complexity and uncertainty of some key parameters, such as the crystal size and shape distribution, makes a purely analytical approach impractical or impossible in most cases outside a research environment. The common methodology is to perform tests on representative samples of the feed material in a laboratory or on a pilot plant scale. From such tests the performance of a plant scale unit can be predicted with reasonable accuracy.

Whatever the method of predicting separation performance a basic understanding of the general centrifugal filtration process is useful. Section 7.3.1 attempts to provides such a basic understanding.

7.3.1 The physical basis for centrifugal filtration

Filtration is the separation of a liquid from a solid by means of a screen which allows the passage of liquid whilst retaining the solids. Centrifugal filtration increases the forces on both the solids and liquids by spinning them at high rotational speed in a perforated cylinder or cone which supports the screen. The centrifugal force acting on the liquid increases the filtration rate in much the same way as the applied pressure in a static pressure filter.

From the point at which the feed comprising solids and liquids enters the centrifuge the process of separation can be considered as four discrete phases – the first generally being sedimentation followed by three filtration phases.

The initial sedimentary phase occurs when the feed material enters the centrifuge and, assuming the solid is denser than the liquid, the solids sediment through the liquid and onto the screen to produce a cake under the influence of the centrifugal G. Phase 2 occurs when the cake is formed and all the solids are fully immersed in the liquid and the liquor is draining through the interstices between the crystals. This phase continues until the liquor level drops to the level of the cake surface whereupon the third phase commences.

In applications where the solids are less dense than the liquid then phase 1 is not sedimentation as the crystals will float on the liquid. A cake is formed once the excess liquid has drained through the screen, whereupon phase 2 of the separation starts.

As the third phase proceeds the liquid level recedes through the cake and the interstices between the crystals are no longer all filled with liquid. The crystals thus exposed are covered in a thin layer of liquid. The drainage of this thin film of liquid is phase 4 of the filtration.

The sedimentary phase 1 normally proceeds rapidly and is replaced by phase 2. Phase 2 continues until the liquid level drops below the cake surface whereupon phase 3 and 4 start at virtually the same time; however phase 3 predominates until close to the end of filtration. Finally, when phase 3 is complete, all that remains is phase 4, plus non filtration effects such as evaporation. Ignoring non filtration effects phase 4 fixes the final solids dryness.

Clearly filtration is a complex physical process with many variables. Section 7.3.2 attempts to give an overview of some of the theoretical considerations governing the filtration behaviour of phases 3 and 4. This overview is necessarily superficial and is presented to provide an understanding of the main physical mechanisms involved that will assist in understanding the characteristics of the various centrifuges discussed in this chapter.

7.3.2 Centrifugal filtration – a simple analysis

A more comprehensive treatment of centrifugal filtration can be found in Purchas and Wakeman (1986), Svarovsky (1990) and Coulson *et al* (1991). The approach adopted here is presented to give a basic physical understanding of the main parameters that govern centrifugal filtration.

It is possible to idealize the flow of liquor through a simple incompressible crystalline filter cake by considering the flow down a thin pipe. The flow rate Q down a capillary was derived by Poiseuille in

1840 when investigating the flow of blood in the arteries of horses (Tabor, 1969):

$$Q = \frac{\pi r^4 P}{8\mu l} \tag{3}$$

where Q is the flow rate through a capillary of radius r and length l, P is the pressure difference across the capillary, and μ is the dynamic viscosity of the fluid.

The flow rate Q can also be expressed as:

$$Q = \frac{V}{t} \tag{4}$$

where V is the volume which flows in time t. The pressure P at the outlet of a vertical capillary filled with liquid of density ρ resulting from the acceleration due to gravity g is:

$$P = \rho g l \tag{5}$$

The volume V flowing out of the capillary in time t is obtained by combining equations (3) to (5) to give:

$$V = \frac{\pi r^4 \rho g t}{8\mu} \tag{6}$$

The volume of the capillary is $\pi r^2 l$ and the volume of liquor expressed as a proportion of the capillary volume that flows out in time t is:

$$\frac{V}{\pi r^2 l} = \frac{r^4 \rho g t}{8\mu\pi r^2 l} \propto \frac{r^2 \rho g t}{\mu l} \tag{7}$$

The liquor density can be taken as constant giving a flow out of the cake over time t proportional to $r^2 g t/\mu l$.

This expression can now be used to compare the amount of liquor flowing from capillaries of different lengths and radii filled with liquor of different viscosities in different times etc. The use of this expression for centrifuges comes from associating the various parts of the formula to features of the cake in a centrifuge. To make these associations, firstly consider the small gaps which exist between the individual crystals of the solids in the centrifuge basket. These gaps or interstices form a channel (or capillary) through which the liquid flows. It is reasonable to

assume that the radius of the channel through the cake is roughly proportional to the average size of the crystals forming the channel. Secondly, the length of the capillary is proportional to the depth of liquid in the cake. Finally, the acceleration due to gravity, g, is replaced by the centripetal acceleration G generated in the centrifuge, t is the spin time and μ the viscosity of the mother liquor being filtered from the cake.

Using these idealised associations it is possible to express the initial filtration rate, F_i, for phase 3:

$$F_i \propto \frac{(\text{Average Crystal Size})^2 \times (\text{Centrifugal Acceleration}, G) \times (\text{Spin Time})}{(\text{Liquid Viscosity}) \times (\text{Cake Thickness})} \quad (8)$$

Equation (8) ignores potentially important factors such as the spread of crystal size either side of the average, crystal shape and the compressibility of the cake formed within the centrifuge. It relates solely to an idealised view of filtration of liquid through the cake – phase 3.

This simple expression has two uses. Firstly, it indicates the main properties of the feed material and the centrifuge that affect the initial filtration rate. Secondly, it allows a basic comparison to be made between one centrifuge and another. For example, one centrifuge may have a higher G, a thicker cake and a shorter spin time than another. If the numeric value of equation (8) is calculated for both cases then a better initial purging rate would be expected from the centrifuge with the higher value. Alternatively the effects of having a larger crystal size can be gauged in terms of a corresponding reduction in spin time and/or a decrease in centrifugal G.

Once filtration nears completion the gaps between the crystals are no longer full of liquid and all that remains is a thin layer of liquid on the surface of the crystals and in the interstices between adjacent crystals held in place by surface tension. The amount of liquid retained in this thin layer defines the final dryness that can be achieved in a centrifuge. A full analysis of the purging process in phase 4 is extremely difficult, however an analogy similar to that used to for equation (8) can be adapted to give a simple physical understanding of the properties of the centrifuge and feed which govern the final dryness.

Consider an isolated crystal as a simple sphere of radius r covered in a thin layer of liquid of thickness ∂r held in place against the centrifugal force by a surface tension force or the equivalent surface energy γ (Tabor, 1969). The limiting thickness of the liquid coating on the crystal (that is, the thickness after a long spin time) can be estimated for a given set of centrifuge conditions by using the principle of virtual

work. Under the influence of the centrifugal force the liquid around the crystal elongates in the direction of the applied G force – see Figure 7.30. If this distorted shape is assumed to be a spheroid (that is, the shape of a three dimensional ellipse) then the work done by the centrifugal force can be calculated as the centrifugal force multiplied by the average distance moved by the liquid layer. This work done on the liquid must be counterbalanced by an increase in the surface energy of the liquid surface. If this is not the case then some liquid will escape and the liquid layer thickness ∂r will reduce. This simple energy balance approach allows the limiting liquor thickness ∂r to be estimated. Figure 7.30 shows this arrangement.

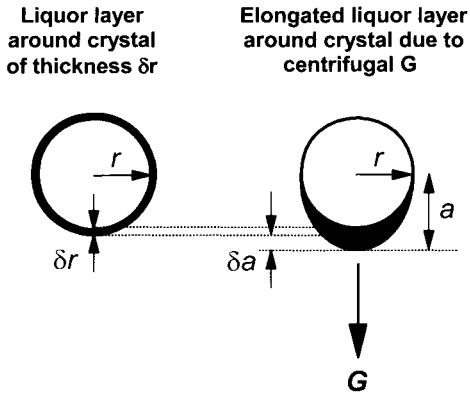

Figure 7.30 Schematic of moisture on a single crystal

Assuming that the major and minor axes of the spheroid in Figure 7.30 are such that a is only slightly larger than r then the surface area of the lower half of the spheroid can be approximated to $(\pi r^2 + \pi ar)$ and the increase in the surface area caused by displacing the liquid layer by a distance ∂a is $\pi r \partial a$. The increase in energy is the product of the liquid surface tension γ and the increase in the surface area of the liquid $\gamma \pi r \partial a$. The work done by distorting the liquid layer from a sphere to a spheroid is given by the product of the mass of liquid and the average distance the liquid moves. For a liquid of density ρ and a centrifugal acceleration of G the work done is $4k\pi r^2 \partial r \rho G \partial a$, where k is a constant relating to the geometry. Equating the work done by displacement to the increase in surface energy gives:

$$\partial r = \frac{\gamma}{4kr\rho G} \tag{9}$$

In this simple model the liquor layer of thickness ∂r is assumed to be present around each crystal. The total volume of liquid remaining in the cake is the product of the number of crystals and the volume of liquid surrounding a single crystal. With k' as the packing factor for crystals in the cake the liquor volume as a proportion of the total cake volume in the centrifuge, the moisture M is:

$$M = \frac{3\gamma}{4\pi r^3 \rho G k k'} \tag{10}$$

On the basis of this approximate analysis the final limiting moisture, M_i, can be expressed as:

$$M_i \propto \frac{\text{(Surface Tension)}}{\text{(Centrifugal Acceleration } G) \times \text{(Average Crystal Size)}^2} \tag{11}$$

Equations (8) and (11) show some of the basic physical aspects of the centrifuge and feed material which affect the separation performance. The centrifugal force and average crystal size both appear in equations (8) and (11), whereas other aspects such as cake thickness and spin time appear only in equation (8) and consequently have a lesser effect on overall separation performance. Whilst spin time and cake thickness undoubtedly affect separation performance the stronger dependence on G and particularly crystal size suggested by equations (8) and (11) is found in practice.

The simplistic analysis presented in this section excludes the important effects of cake compressibility or a variation in crystal size either side of the mean and other drainage mechanisms but it should be clear that anything that restricts the flow path through the cake will have a similar marked effect on separation performance as a smaller mean crystal size. As a general rule users of centrifuges are interested in the mass of cake discharged, whereas the centrifuge is processing the surface area of the crystals and it is the crystal size distribution that links the cake mass to the cake surface area. The importance of the crystal size distribution in governing separation performance cannot be overemphasised.

7.3.3 Scale-up – general considerations

It is necessary to define each element of the process cycle to specify fully a batch centrifuge application and therefore small scale tests used as the basis of a scale-up to a plant size unit must consider all the elements of the cycle; feeding, washing, drying and discharging. In

addition as most applications are likely to involve trade-offs between competing process parameters it is useful to have a clear idea which are the critical performance requirements in order that they are given due importance in the scale-up. Scale-up is an inexact science that requires a mix of experience and suitable test equipment to achieve success. Generally speaking centrifuge manufacturers have both the experience and equipment, in addition a number of larger centrifuge users have laboratories capable of conducting test work.

7.3.3.1 Test samples for scale-up

Central to any scale-up is the sample of material to be tested. Two aspects are of primary importance, firstly the test sample should be chemically and physically representative of the feed from the full scale plant and secondly sufficient sample material should be available to perform adequate tests. On occasion a full scale plant is already in existence so there is little or no limitation on the quantity of test material that can be provided. In such circumstances it may be possible to install a full scale trial centrifuge, which gives complete test results without the need to scale-up, however this situation is rare unless a new separation technique is being considered for an existing plant.

As a general rule the larger the sample the better (Pearson, 2002; 2003). On rare occasions scale-up is required from samples of less than 100 ml to a full scale centrifuge with a basket capacity of perhaps 1000 litres or more. Such scale-up is possible but as might be expected the margin for error grows in inverse proportion to the sample size. More common is a small initial sample that can be used to gauge the general suitability for centrifugal filtration, which is then followed by a sample of perhaps 20 to 200 litres which is sufficient for small scale centrifuge tests.

There are many pitfalls to avoid when providing a sample for a scale-up test. The sample is usually taken from one of three sources: an existing full scale plant; a pilot scale plant; or from laboratory trials. Samples taken from a full scale plant are to be preferred; however these can still be unrepresentative. Samples from smaller scale plant or laboratory trials introduce further opportunities for error. Some of the issues to consider when providing test samples are summarised below:

- **Time delays between sampling and testing**
  For tests conducted remotely from the sample point the effects of any transport delays should be considered. For material taken from a crystalliser the crystallisation process may continue after sampling if the liquor is supersaturated. Alternatively small crystals in the

sample may dissolve in unsaturated liquor or other chemical, biological or physical changes such as agglomeration may take place prior to testing. Such effects change the crystal size distribution and can therefore have a large effect on separation characteristics.

- **Temperature changes**
  For materials produced above ambient temperature the sample is usually cooled after collection and later re-heated prior to testing. Recrystallisation or precipitation of the solids may occur resulting in a change to the crystal size distribution of the solids in the sample.

- **Reconstituted test material**
  The solids and liquid are sometimes sampled separately and then re-mixed prior to testing. Segregating the solids and liquid avoids problems of recrystallisation and transport delays but can introduce its own problems. In some cases the solids are taken from a dryer and the liquids from the output of an existing centrifuge or filter. The mechanical action of the dryer and its associated conveyors may cause crystal breakage and the thermal action of the dryer may cause agglomerations or alter the surface of the crystals. Cake taken from a dryer can be difficult to wet-out when reconstituting the sample and in extreme cases it may be necessary to add a surfactant. For solids with a porous surface or a significant level of inherent moisture – that is, moisture bound to the crystal rather than simply present as a film of the crystal surface – drying may alter the solids such that a reconstituted feed is a poor representation of the original and give erroneous moisture levels after centrifuging. In these circumstances it is generally preferable to take damp solids from the input to the dryer.

  Separating solids and liquids is often a convenient way to collect and transport test materials, particularly from existing large scale plants, however it is important to check that reconstituted feed is an adequate representation of the original.

  Whilst not recommended it is sometimes useful to conserve sample material by using one sample batch for several test runs. A degree of crystal breakage will occur due to handling, feeding and discharging that occurs with each test. It is important to check that the crystal size distribution has not changed significantly before relying on the results from samples that have been processed several times.

- **Sampling techniques**
  Taking a small sample from a large volume of material is simple, ensuring that it is representative of the whole is not. Much has been written on general good sampling procedures – see for example, Perry and Green (1984) – and such guidelines should be followed

when collecting a test sample for centrifuge scale-up. The basic ground rules are that samples should be taken from a well mixed process stream without any obvious bias in terms of the ratio of solids to liquids or crystal size distribution at the sampling point. An example of bias is a sample point on the outside radius of a pipe bend. As larger crystals tend to migrate towards the outer radius of the bend the sample would be likely to have a larger mean crystal size than the bulk material. A further example is a tank with a sampling point in a corner that is remote from any agitation and in a dead zone where solids tend to settle. The sample would be likely to have both a low solids content and a mean crystal size below that of the bulk material.

- **Effects of scale**
  Not all test samples come from a full scale plant, many come from a pilot scale or perhaps a laboratory scale plant. On the laboratory scale it is common for the design of the equipment to be fundamentally different from that used on the final full scale plant. For example batch crystallisers are normally used on the laboratory scale whereas the full scale plant may use continuous crystallisation. It is very likely these will produce differing crystal size distributions. Any scale-up based on the laboratory sample may therefore not accurately reflect the full scale plant.

  If a pilot plant is constructed with the same design of process equipment (e.g. continuous crystallisers etc.) the scale-up will be more representative, however the simple change in scale may have a noticeable effect. The surface area to volume ratios, mixing (kW per m^3), circulation and static head all differ between a small and large crystalliser of the same design and this can introduce changes in crystal size distributions even if the plant and laboratory scale crystallisers are operated in the same way. In practice economic considerations often dictate that plant scale crystallisers are operated at higher supersaturation levels and cleaned less often than a laboratory unit making agglomeration or spontaneous nucleation more likely in the plant scale unit.

- **Feed systems**
  The feed system selected for the full scale plant may modify the crystal size distribution from that of the pilot plant. Consider the example of a small pilot plant where a representative sample of material is collected in a drum prior to testing in a centrifuge. The full scale plant may use a pumped ring main system to feed the centrifuge(s) and it is likely that the circulation prior to drawing off the feed to the centrifuge will damage the crystals and modify the

size distribution, particularly if a centrifugal pump or high circulation rates are used in the ring main.

Few individual samples used for centrifuge scale-up are susceptible to more than one or two of the potential pitfalls listed above and in most cases reasonable care during sampling will help ensure the samples are representative and the scale-up results are accurate. If a shortcoming in the sampling or scale-up, such as those discussed above, is known then allowances can be made in the final centrifuge selection to address the particular issue.

Lastly it is important that any sample sent for testing is accompanied by the necessary safety data sheets and certification as required by local regulations and good safety procedures.

7.3.3.2 Preliminary tests on sample

Basic physical data on the sample is required before any estimation of separation performance can be made. Some basic physical data is normally provided with the sample or is available in the published literature. Additional tests are usually required to give a more complete understanding of the feed material. Most of the information, with the exception of crystal size distribution, can be measured using fairly standard laboratory equipment on small sample volumes. A typical set of preliminary information required for a batch centrifuge filter application is listed below with brief comments on possible methods of measurement.

- **Sample pH**
  Measurement using a standard laboratory pH meter.
- **Density of packed saturated cake**
  For very small samples less than 500 ml and where the density of the solids exceeds the liquor density the measurement can be made by sedimenting the solids into a cake in a test tube centrifuge and decanting the supernatant liquor. The density of a dry packed cake can also be estimated if the solid density of the crystals is known. If the sample is sufficiently large it is always preferable to use a small batch centrifuge test to measure the cake densities.
- **Density of feed material and liquor at the operating temperature**
  Measurement by weighing a known volume.
- **Solids concentration, w/w**
  A typical measurement uses thermal drying of a known mass of feed material to obtain the weight of solids.

- **Viscosity of liquor**
  For small samples and where the viscosity cannot be obtained from the extensive published data measurement may be made using a standard Brookfield® viscometer or similar. For larger samples where small scale centrifuge tests can be conducted a viscosity measurement is not normally required.
- **Crystal size distribution**
  A variety of techniques are available for measuring crystal size. The most common is sieve analysis which is able to give a size distribution with the finest fraction being that below 45 μm. A more detailed crystal size distribution is to be preferred particularly where the fraction below 45 μm exceeds 10% by weight. Common techniques to measure the distribution down to 1–2 μm include the Coulter Counter® which measures crystal size distribution by monitoring the conductivity of an electrolyte solution containing the crystals as it flows down a thin channel. A second approach uses laser diffraction to calculate the size distribution from the scattering of laser light passing through the sample. Another technique uses optical imaging to measure the size and shape of a large number of particles thereby constructing the distribution. This method is capable of producing both a size and a shape distribution which can be an advantage when investigating samples containing needle crystals.

For small samples this preliminary data may be the limit of the testing that can be undertaken. When combined with experience of the separation of other materials with similar characteristics the information from these preliminary tests is sufficient to make a basic assessment of performance. A more complete and accurate assessment of separation performance requires an investigation of cake blinding during ploughing, cake compressibility, cake filtration rates over time and perhaps washing characteristics etc. This can only be achieved with larger samples that allow tests on a small scale centrifuge.

7.3.3.3 Small scale batch centrifuge testing

A typical laboratory batch centrifuge has a diameter of 250 mm, a maximum cake thickness of 40 mm and a basket working volume of 2.5–3.5 litres. Sample volumes in excess of 10 litres with a reasonable solids concentration normally allows a good series of tests to be conducted in such a unit. Smaller laboratory basket centrifuges (e.g. 100 mm diameter, volume 200 ml) and a lesser sample volume can be used but the small cake thickness (10–12 mm) limits the ability to

Figure 7.31 Laboratory scale batch centrifuge – 260 mm diameter (Broadbent Centrifuges)

scale-up to large centrifuges. Figure 7.31 shows a typical mobile laboratory scale batch filtering centrifuge.

- **Cake density measurement**
  The density of the cake within a centrifuge basket is required when calculating throughput, basket acceleration/deceleration rates and basket stress levels. The formation of the cake in the centrifuge basket is a complex process with larger crystals sedimenting to form a filter cake most rapidly followed by the finer fractions. The presence of voids means the density of the cake within the basket is significantly lower than the theoretical crystal density, however the cake density is higher than that of material stored in a silo or bin. Small scale centrifuge tests allow accurate measurement of the average density and void volume at a particular centrifuge *G*. Measurements are made by forming a cake then removing the basket containing the cake from the test centrifuge, measuring the diameter of the cake surface and weighing the complete basket and contents. With known dimensions and weights for the empty centrifuge basket and screen it is a simple matter to calculate the cake density. If the moisture content of the cake is obtained through thermal drying then the density of dry cake within the basket is obtained. If the theoretical density of the cake material and the liquor is know then the percentage of voids within the cake and the density of a saturated cake can also be calculated.

- **Cake compressibility**
  Materials are often classified as compressible or incompressible whereas in practice all cakes are compressible to some degree. The centrifugal pressure on the crystals in the cake pushes the crystals closer together causing deformation at the contact points between adjacent crystals. This reduces the cross sectional area of the pathways for the liquor through the cake and therefore reduces the filtration rate. The centrifugal pressure on both the crystals and any liquid head above the cake contribute to this effect. Small scale centrifuge tests incorporate the effects of cake compressibility directly in their results.

  Equation (8) suggests that filtration rate will increase linearly with G for a given cake thickness; however for compressible cakes the increase is less than this. Extreme cases where the filtration rate falls with increasing G are mentioned in the literature (Dahlstrom *et al*, 1966) but are rare in practice.

- **The effects of cake thickness**
  Centrifuge testing automatically includes the complex effects of cake compressibility and cake formation in their results. However if only small scale tests are possible then the effects of the thicker cakes on a full scale centrifuge must be included as a scale-up factor. To aid scale up to larger lips, laboratory test centrifuges can be provided with baskets that have an oversized lip, however the scale up is not exact as the G at the surface of an 80 mm cake in a 260 mm diameter basket is just 38% of the G at the basket wall whereas the same cake thickness in a basket 800 mm diameter the figure is 80%. Nonetheless the deeper lip is a useful tool that reduces the need for scale-up based on cake thickness.

  An alternative approach relies on experience of other separation duties where the full scale process results from cakes with similar compressibilities and crystal size distributions are known. Generally many plots of cake moisture as a function of spin time follow a similar pattern and data for a variety of other materials is an invaluable tool for performing a scale-up on a new material.

  With reference to the basic description of filtration in Section 7.3.1 the time required for phase 3 of the filtration process to be replaced by phase 4 scales roughly as the square root of the cake thickness (Dahlstrom *et al*, 1966). This is a rough guideline at best and it is important to note that the limiting dryness is dependent on centrifuge G and largely independent of cake thickness – see equation (11) – however a longer time is required to reach final dryness (end of phase 4) the thicker the cake.

Cake thickness scale-up is generally performed by the judicious use of one or more of these techniques.

- **Filtration rate measurements**
  Measurement of the filtration rate through the cake per unit area of screen provides information for scale-up of the feed and drying phases of the centrifuge cycle. Correct feeding of a batch centrifuge requires that the rate of addition of the feed liquor is approximately balanced by the feed liquor filtration rate through the cake at the centrifugal *G* used for feeding. If the rate of liquor addition is lower than the filtration rate then the feed does not distribute evenly in the basket and the full capacity of the centrifuge is not realised. If the liquor is added too fast then the basket is being 'overfed' and surface liquor will collect above the cake. This may be acceptable to a limited degree but is liable to cause high levels of vibration during feeding in certain circumstances. See Section 7.1.1.1 for a fuller discussion on this topic. A good feed rate for scale-up purposes is one where the liquor addition rate matches the filtration rate. Tests are conducted at various *G* levels to measure the filtration rate (see Figure 7.32) and to ensure that the material is distributed evenly within the basket during feeding.

  Once the filtration rate per unit area of screen is known, scale-up to a full sized basket is made by adjusting the feed rate for the larger screen area and thicker cake of the full scale centrifuge.

  Figure 7.32 shows the filtration rate against time for a 98 mm thick cake with a mean crystal size of 110 μm at 500, 700 and 900*G*. The initial filtration rate is measured with a saturated cake and scales

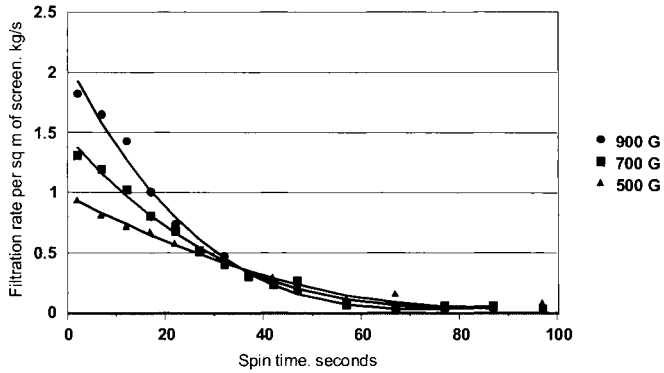

Figure 7.32 Variation of filtration rate with spin time and centrifugal *G*

approximately with G indicating that the cake is largely incompressible.

Filtration rate measurements can be made in several ways. One method that provides extensive information requires the segregation of the solids and liquid in the feed. Initially a normal feed (solids and liquid) is used to form a uniform cake of known thickness in the test centrifuge basket. The feed liquor is then added onto the cake in increasing quantities until free liquor appears on the surface of the cake. The liquor flow rate that maintains this condition is the filtration rate at that particular G. Enhancements to this approach include placing the centrifuge on load cells to continuously monitor the mass in the basket. Monitoring the reducing weight of the basket after the liquor feed has been turned off gives the drying rate of the cake during spinning. Figure 7.33 shows the cake moisture against spin time for various levels of G measured by this technique.

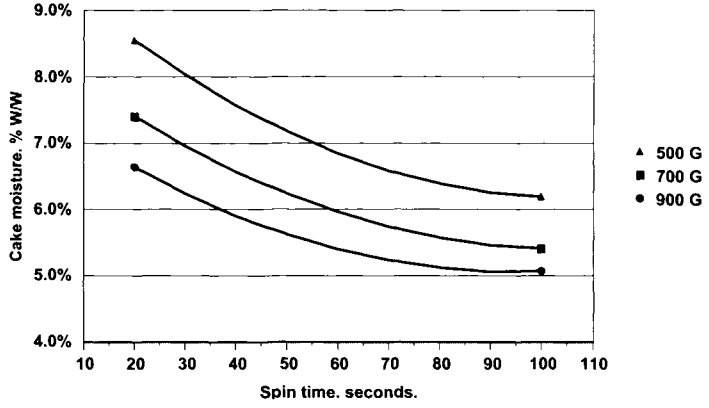

Figure 7.33 Variation of cake moisture with spin time and centrifugal G

A simpler but less versatile approach involves measuring the liquor filtration rate during feeding. Assuming that the cake remains saturated and that cake formation is uniform the liquor addition rate in the feed is the same as the filtration rate for the conditions at the time of the measurement.

- **Wash quantities and time of application**
  The quantity of wash liquor required is fixed by the process requirements and the time needed to apply the wash is fixed by the filtration rate of the wash liquor through the cake. Quantities of wash liquor are generally specified by the end user in terms of kgs

of wash per kg of cake to achieve a given condition (e.g. removal of a certain impurity) however if this data is unavailable a test centrifuge can be used to perform wash trials to find the minimum wash required to meet the needs of the process. For a given cake thickness the wash time is calculated from the wash quantity, the filtration rate through the cake per unit screen area and the total screen area on the full sized centrifuge. If the physical characteristics of the wash liquor, particularly viscosity, differ significantly from the feed liquor it will be necessary to repeat the filtration rate test for the wash to get an accurate wash time. If the wash quantity is small then an estimated filtration rate will often be adequate.

Batch filtering centrifuges provide a good environment for washing. The normal approach is to apply wash after the feed liquor has receded below the surface of the cake (i.e. during phase 3). This ensures that the wash and feed liquor do not mix other than at an interface within the cake. Some cakes shrink as the feed liquor drains resulting in cracking on the cake surface. If washing is applied after cracks have appeared the wash liquor flows preferentially down the cracks and washing becomes ineffective. These effects can be easily detected during small scale centrifuge tests and countered by early application of the wash liquor prior to the formation of cracks.

- **Ploughing tests**
  Small scale test centrifuges with manual discharge systems are well suited to investigating the ploughing characteristics of centrifuge cakes. After spinning to the required dryness level the cake is ploughed out and the forces required to operate the plough and the form of the discharged cake are noted and compared with the ploughing characteristics of known successful applications. Listed below are some general qualitative comments on the characteristics of the material after ploughing. Similar tests can be conducted on full depth and peeler ploughs – see Figure 7.34.

  Solids come off as a ribbon with toothpaste like consistency. Material possibly thixotropic and may block discharge chutes. Try to dry further before ploughing by lengthening spin time or increasing spin G.

  Solids come off as flakes, but tend to be sticky and ball up. Material possibly dilatant. Regular cleaning of chutes and basket may be required. Try to dry further before ploughing by lengthening spin time or increasing spin G.

  Solids fall like dry power with little force applied by the plough mechanism. The ideal situation for easy ploughing.

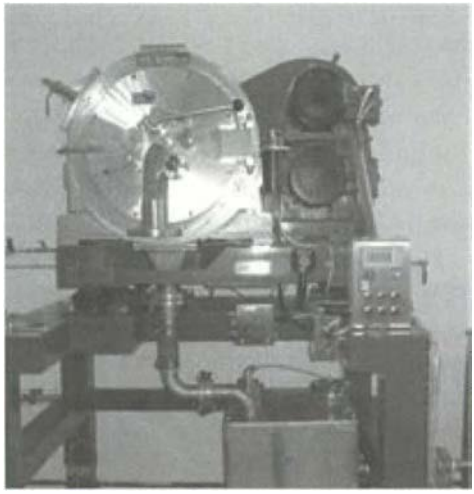

Figure 7.34 350 mm diameter test peeler with manually operated plough (Broadbent Centrifuges)

Solids fall in small pieces and larger plough forces are required. Solids have the appearance of being machined off the cake. Reliable ploughing should be possible. A sharp multi-blade plough may be necessary, or a slower plough traverse rate.

Solids are very hard. The plough has difficulty entering or cutting the cake. Ploughing may not be possible. Try a stiff narrow plough blade and/or slow the plough traverse rate. Use a lower spin time or G to produce a softer cake.

- **Residual bed permeability tests**
  A residual bed is left on the screen after ploughing in the test centrifuge in just the same way as a full scale unit. Some residual beds will become impervious over time either by a smearing action or crystal fracture caused by the plough blade or by some other effect such as recrystallisation (see Section 7.1.1.4). The test centrifuge allows the fall off in filtration rate through the residual bed to be assessed by running repeated batches through the test centrifuge with the same residual bed. This gives a clear indication of the number of batches that can be processed before the residual bed must be removed and reformed. In addition trials can be conducted within the test centrifuge to investigate the best residual bed removal strategy which can have a strong bearing on the most suitable type of centrifuge for use at full scale – inverting bag, peeler or vertical batch centrifuge.

- **Filter screen selection**
  There are few scale-up issues with screens. If a screen is used successfully in a test at small scale the same screen with the same slot or hole size and percentage open area can be used on a full scale unit. The total area of the screen scales up with the basket shell area but the slot dimensions and number of slots per unit area remain the same. Initial screen selection is often based on small scale centrifuge tests and these tests will generally accurately represent screen performance at large scale.

  Various screen types, open areas and slot/hole size can be tested in the trial centrifuge as required. Collection of the initial filtrate from the test will show the highest level of crystal loss through the filter screen. Once the cake has started to form the crystal loss usually drops to near zero. Trials in a small scale centrifuge will expose a screen that is prone to blinding.

7.3.3.4 Small scale continuous centrifuge tests

All the points raised in Section 7.3.3.1 and many of those in Section 7.3.3.3 apply to continuous centrifuges, particularly those that bear a close resemblance to a batch centrifuge such as the pusher. However there are several important differences in the scale-up for batch and continuous centrifuges. The cake transport mechanism and the losses through the screen resulting from the movement of the cake apply only to continuous centrifuges. Consider the case of the continuous conical centrifuge (Section 7.1.2 and 7.2.2.1). The speed of cake movement is fixed by the characteristics of the feed material, screen, the angle of the basket and G. It is important to have the same transport mechanism operating in both the test and the full scale centrifuge and this requires that the same basket geometry, screen type and G is used in both units.

For conical centrifuges the mass flow rate is generally considered to scale-up approximately as the product of the G and the screen area, so for the same discharge G on both the test and full size centrifuge the scale up is by the relative screen areas (Hugot, 1972). Figure 7.35 shows a laboratory scale conical centrifuge used for scale-up tests.

Screen slots size and open area have a significant impact on the performance of a continuous centrifuge. Testing to assess screen losses and investigate blinding by crystals close to the slot size or any fibrous material present is an important part of selecting the optimum screen for a new application.

All continuous centrifuges cause a degree of crystal damage as the cake moves along the screen and further damage as the cake is

Figure 7.35 Simple laboratory conical centrifuge – 440 mm exit diameter (Broadbent Centrifuges)

discharged. Estimates of the level of damage expected at full scale may be required for some applications and this is difficult to predict from small scale test results. Experience of similar applications at plant scale is invaluable in making such estimates.

Finally the quantities of test material required for continuous centrifuge tests may be large for two reasons. Firstly any test requires that a stable flow pattern is established before representative results can be expected, and secondly crystal damage will occur during the trials severely limiting the extent to which test samples can be reprocessed in the test centrifuge.

7.3.3.5 Summary

The main issues to be considered as part of any scale-up investigation are:

- Use large samples.
- Take great care to obtain representative samples – particularly with regard to the crystal size distribution.
- Where it is known that samples will not be fully representative due to pilot plant scale etc. highlight the issue to all involved and investigate the likely effects on the scale-up.
- Perform the test as soon after sampling as practical.
- Use the largest test centrifuge possible in the circumstances.

- Perform tests to investigate feeding, washing, drying, ploughing and the effects of residual bed blinding.
- Do not reuse feed material many times as breakage during feeding and ploughing will change the crystal size distribution.

The costs of such a testing regime can be high, but the costs to the end user and the centrifuge supplier of getting it wrong are almost always higher.

7.4 Installation and operational considerations

There are numerous applications that use one of the many types of filtering centrifuge, each with specific installation requirements. This section provides some guidelines to assist with planning, installation and the avoidance of some of the more common pitfalls.

7.4.1 Process

For centrifuges where crystal losses in the filtrate are expected (e.g. continuous centrifuges) provision must be made to handle these solids. Depending on the application it may be a simple matter to recirculate the filtrate back into the process without penalty. It is not uncommon to find that the solids in the filtrate are not chemically identical to the feed solids, for example the impurities in the feed may be present as very small crystals that preferentially report to the filtrate. In this case recycling the filtrate back into the process will lead to a build up of impurities over time and cause difficulties. Even if the fine crystals are not rich in impurities the build-up may still become a problem. It can take several days of continuous operation before the effect on the overall crystal size distribution becomes noticeable in the process. Such problems are extremely difficult to reproduce and detect in small scale laboratory testing and may only occur in pilot plant operated for a prolonged period. If the filtrate cannot be recirculated then it may have to be treated as an effluent with additional process plant requirements and associated costs.

The performance of centrifuges that leave a residual bed on the screen after discharge (e.g. vertical batch and peeler types) may reduce over a period of time as the bed becomes impervious (see Section 7.1.1.4). Periodically the bed will have to be removed and one common way is by the use of a gas knife. The bed solids removed by a gas knife are usually discharged after the normal plough step. The form of the bed

solids dictates what route they take in the overall process. Dry solids are often routed with the normal cake solids; wet or impure bed solids are normally routed as waste. If some form of liquid washing is required to remove the residual bed then alternative arrangements have to be made to route the washings away from the normal dry cake discharge. This is normally accomplished by a diverter chute under the solids discharge of the centrifuge. A similar arrangement is also used to remove liquor used in clean in place (CIP) operations.

Many applications require inert gas purging of all operations in the plant. Clearly there is a need to seal the solids and liquid discharge system of the centrifuge to maintain a small over pressure of the inert gas during processing. One method used is a simple liquid seal or trap in the filtrate line. For centrifuges where there is a likelihood of crystals in the filtrate liquor (e.g. continuous centrifuges) the liquid seal needs to be self cleaning or be fitted with a wash connection to avoid the possibility of blocking by the accumulation of solids.

To improve cleanliness and appearance, some horizontal centrifuges (e.g. peeler or pusher) are mounted in a 'through wall' configuration with the process related elements such as the basket, discharge, feed etc. located in a clean area and the drive, bearings and other systems on the other side of the wall in a non-clean area. In these cases the vertical support plate (e.g. see Figures 7.13 and 7.14) forms part of the wall dividing the two areas. Due to the movement of the centrifuge during operation caused by out of balance loads etc. this plate must include a flexible membrane which allows a degree of movement whilst maintaining the seal between the clean and non-clean areas.

7.4.2 Mechanical and electrical installation

- **Structural support**
  Small centrifuges can be rigidly fixed to the floor or a suitable support structure but large industrial centrifuges are generally mounted on low natural frequency flexible supports (vibration isolators). Some large centrifuges have heavy inertia blocks attached to further reduce the transmission of vibration to the building. The loads imposed by the centrifuge on the building structure are made up of two components. The first is the simple static weight of the centrifuge plus basket load etc., the second relates to the dynamic loads imposed by residual out of balance forces that are not completely eliminated by the vibration isolators. To allow for dynamic effects a simple rule is to design the support for double the static loads. However a support structure adequate for all static and

dynamic loads does not guarantee trouble free operation as structural resonances may occur in the supports beneath the centrifuge if the stiffness is too low. Such a resonance may disrupt the operation of the centrifuge by causing high vibration trips. The ideal installation has a support structure with a stiffness such that all resonances in the structure are at least 30% above the maximum spin speed of the centrifuge.

For centrifuges without a large attached inertia block this stiffness condition imposes more onerous requirements on the design of the support structure than static or dynamic loads. In practice the structural strength needs to be perhaps four times the strength required to support the centrifuge static weight in order that the resonant frequencies are above the maximum spin speed.

An appropriate design standard and the individual documentation for the specific centrifuge should be used to ensure the support structure is able to carry the loads imposed.

- **Electrical and piping installation**
  When an out of balance is present a centrifuge will vibrate at the running speed. For centrifuges mounted on flexible supports the amount of movement increases as the speed reduces from spin speed before finally falling to nothing as the centrifuge approaches zero speed. The maximum displacement (typically in the range 150–250 rpm) may reach a few tens of millimetres peak to peak. As would be expected the centrifuge vibration isolators are designed for such movement and it is important to ensure that other connections to the centrifuge are also designed to allow sufficient movement without damage or fracture. Such connections include electrical cabling, particularly to the main drive motor, and pipe work such as feed, wash, filtrate and cake connections. An additional allowance for movement needs to be provided for any process or electrical connections that attach to a hinged cover or door on the centrifuge that may be opened during normal operation.
- **Maintenance requirements**
  Centrifuges typically have a life span of 20 years, therefore when planning an installation it is sensible to consider the maintenance and repair requirements likely to occur over this period. Removal of the basket for inspection or periodic bearing changes will require lifting equipment and reasonable headroom above or adjacent to the centrifuge. Adequate access allows maintenance operations to be completed more rapidly and reduces the possibility of delicate components being damaged during handling. To further assist with minimising the effects of unplanned maintenance it is useful to be

able to isolate the centrifuge from the rest of the process plant. Good access and the ability to isolate the centrifuge are also essential for the safety of maintenance operations.

7.5 Safety, standards and risk analysis

Centrifuges have the potential to be hazardous. This is fundamental to their design. Similar comments apply to many items of equipment used in the process industry such as pressure vessels, dryers, pumps etc. For a typical 1.2 m batch centrifuge basket spinning at 1200 rpm the energy stored in the rotating basket is about 4 MJ and the peripheral speed of the basket is 270 km h^{-1}. The stored energy is the same as that of an average family car travelling at 280 km h^{-1} (175 mph) or a 2.5 m^3 vessel pressurised with gas to 16 bar.

With speeds and energies such as these, perhaps together with a corrosive, toxic or inflammable chemical present, there is the potential for hazardous situations to develop. The safety of a centrifuge depends on the controls, design and maintenance of the machine and operating procedures reducing this risk to an acceptable level.

The key European standard for industrial centrifuges is EN12547:1999 'Centrifuges – Common safety requirements'. This standard covers a wide range of issues but it is not exhaustive. A major safety concern for any centrifuge is the long term integrity of the basket. Failure of the basket whilst rotating at high speed will destroy the centrifuge and possibly anything or anybody near it. Some users of large industrial centrifuges assume that the casing is designed to act as a containment device in the event of basket rupture. The energy stored in a centrifuge basket rotating at high speed is considerable and most casings will not contain a ruptured basket. For example, a casing made of mild steel would need to be at least 50 mm thick to contain a rupture of a 1.2 m diameter basket rotating at 1200 rpm. Most centrifuge cases are typically 6–12 mm thick, depending on the application.

Most batch centrifuges operate in a regime where fatigue is important with 5000 to 50000 basket stress cycles per year and any failure would be a major hazard. Whilst baskets are normally designed for an infinite or extremely long fatigue life the unlikely possibility of a fatigue failure must be considered. For this reason, periodic examination of the condition of the basket is recommended by most manufacturers. If for any unexpected reason a fatigue crack appears the day after an inspection it should not grow to a point where failure will occur before the next inspection. Fatigue crack growth is a complex phenomenon

and for a component that will eventually fail by fatigue the majority of the life is taken up initiating the crack, and only a small proportion of the time (typically 10%) is needed to grow the crack from a point where it is visible to the point where the component fails. For this reason it is most important that regular basket inspections (normally every year) are undertaken in accordance with the manufacturers recommendations throughout the life of the basket.

Control systems are a key part of the centrifuge overall safety system. Whilst it ought to go without saying that changes to the controls should be made with care and in discussion with the originators of the control system, experience shows that this is not always the case. Most control systems (software, hardwired trips, interlocks etc.) are associated with avoiding hazardous events and checking the operation of the centrifuge and ancillary equipment. Only a small part is employed to control the actual steps in a normal centrifuge cycle. Designing a good control system requires more of an understanding of the hazards and what may go wrong rather than a clear understanding of what should happen when things are operating normally.

It is good practice, and a legislative requirement in the EU, for the user to undertake a risk assessment prior to installing or operating an item of process equipment such as a centrifuge. In the EU the user has a responsibility to carry out a risk assessment, select only equipment suitable for the application and provide an unambiguous technical specification to the supplier. In addition the user also has a responsibility to check that the equipment is safe, complies with relevant regulations, and is maintained and used correctly. The supplier has matching responsibilities which for centrifuges are covered primarily by the EU standard EN12547:1999. The EU standard for risk assessment (EN1050) provides a framework for identifying hazards but it is important that the centrifuge supplier and user consult with one another on areas where the centrifuge interfaces with the remainder of the process plant. An example where such dialogue is necessary is the recent ATEX requirements for the user (99/92/EC) and the supplier (94/9/EC) where a formal exchange of data on the potential explosive hazards and limits is required.

The regulations mentioned above apply only for new equipment installed in selected European states; however they provide, along with the regulations of other industrialised countries, a sensible framework and a consistent approach to plant and equipment safety. See The Institution of Chemical Engineers (1987) and Grimwood (2002) for additional information on centrifuge safety issues and associated standards. Centrifuges are versatile items of process equipment and

with care in the installation, operation and maintenance they will give many years of safe and reliable operation as demonstrated by the many thousands of units in operation in the world's process industries.

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8 Sedimenting centrifuges

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8.1 Introduction

Sedimenting centrifuges are commonly used to separate solids from a solid/liquid mixture when the solids have a density difference compared to the suspending liquid phase (Leung, 1997a, b, 1998b; Stahl, 2004). In this chapter, centrifugal acceleration is discussed followed by a brief description of different types of sedimenting centrifuges and how they work. This is followed by a discussion of the scale-up of the equipment, testing and securing the appropriate data for scale-up, and implementation of the equipment in flow sheets, and any other special considerations that are required.

8.1.1 Centrifugal acceleration

High centrifugal acceleration, G, is generated in the bowl of a rotating centrifuge to separate a mixture of suspended solids from a liquid. Centrifugal acceleration can be thousands of times greater than gravitational acceleration g (9.81 m s^{-2}) to effect separation, especially with difficult-to-separate suspensions. The latter pertains to suspensions in which there is a small density difference between the solid and liquid, high viscosity of the liquid, and small sized solid particles. The relative centrifugal acceleration or relative centrifugal force G/g is related to the rotation speed by:

$$\frac{G}{g} = \frac{\Omega^2 R}{g} \tag{1}$$

In equation (1), Ω is the angular rotation speed and R is the radius at which G is evaluated. By default, G is evaluated at the maximum

radius of the centrifuge bowl wall. For engineering calculations equation (1) can be expressed as $^{G}/_{g} = 0.0056(\text{rpm})^2 D$ where D is the diameter of the bowl in mm and rpm is the rotor speed in revolutions per minute. Figure 8.1 shows a graph of G/g versus rpm for a range of bowl diameters expressed in mm.

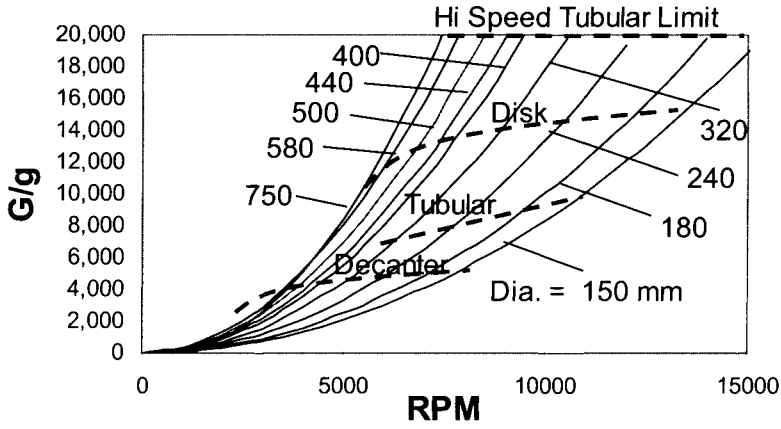

Figure 8.1 Relative centrifugal gravity for different rotation speeds and bowl diameters

While it is desirable to have high G for separation, there are several limiting factors for high G, one of which is the peripheral speed that is dictated by the mechanical yield stress of the material making up the bowl. Other limiting factors are the critical speed of the rotating assembly which in turn depends on the support and design of the rotor, and maximum speed and temperature of the bushings or bearings, seals, and gearbox (for decanters). As a general rule, smaller centrifuges can be operated at higher speed and G to address difficult-to-separate suspensions, however they process at a lower throughput; vice versa, larger centrifuges are operated at relatively slower speed and consequently lower G, yet they can be used to process higher throughputs.

Figure 8.1 delineates the typical sizes and G force of the three common types of centrifuges – decanter, disk and tubular, and these centrifuges will be discussed in greater detail later. The dashed curves show the maximum G-force that these machines typically can attain. However, it is not always necessary for centrifuges to operate at their maximum speed and G depending on the applications.

8.2 Centrifuge types

8.2.1 Sedimenting and filtering centrifuges

There are two types of centrifuges – sedimenting and filtering centrifuges as shown, respectively, in Figures 8.2a and 8.2b. For sedimenting centrifuges, the heavier phase (presumed to be the solids) settles under centrifugal gravity to the solid bowl wall to form a cake, while the lighter phase (presumed to be the liquid, but can be the solid phase) is displaced toward the slurry pool surface. In order to separate a solid phase from a liquid phase, there should be a difference in density between the two phases, although this could be small. For filtering centrifuges, there is no need for a density difference between solids and liquid. Both liquid and solid migrate toward the perforate basket wall under centrifugal acceleration. Solids are retained to form a cake on the filter medium while liquid permeates through the basket. Only sedimenting centrifuges will be dealt with here.

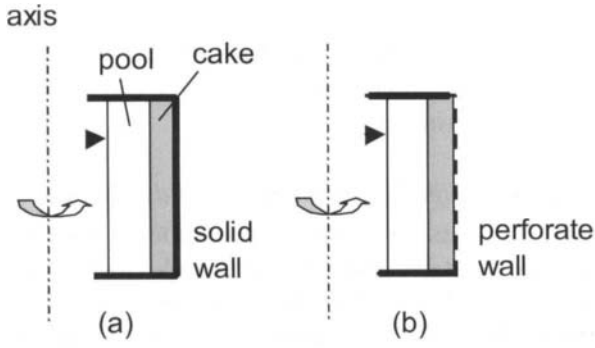

Figure 8.2a & b (a) Schematic of solid-wall sedimenting centrifuge. (b) Schematic of perforate-wall filtering centrifuge

8.2.2 Sedimenting centrifuges

8.2.2.1 Discontinuous and continuous feed and solids removal

Feeding can be continuous or discontinuous. Likewise sediment or cake solids removal can also be discontinuous or continuous. For continuous feed, especially at high feed rate, it is important to accelerate the continuous feed stream properly (Leung and Shapiro, 1996a, b, 2001). For continuous sediment discharge, a mechanism has

to be in place to discharge the sediment continuously. Depending on the rheology or conveyability of the sediment for a given application, special design considerations may be necessary to ensure continuously smooth operation. For discontinuous cake removal, or intermittent solids discharge, additional space in the centrifuge bowl is necessary to temporarily store the sediment prior to solids discharge. Obviously this takes up volume in the bowl that could have been used for sedimentation and clarification. Also, when the solids storage space is filled, the clarified liquid can turn turbid over time it entrains by the sediment. At that point, the cake needs to be discharged and feeding may also have to be interrupted for solid removal, depending on the centrifuge operation.

Continuous-feed, continuous-solids-removal centrifuges are particularly suitable for use in process applications for high production, easy separation and convenient handling while smaller batches of slurries such as used in pharmaceutical applications tend to favour continuous-feed, discontinuous-solids-removal or even discontinuous-feed, discontinuous-solids-removal taking advantage of more flexible operation to accommodate feed-stock variations during feeding and separation, possibly washing, and deliquoring. Also, sedimenting centrifuges are often employed to process batch samples in a continuous-feed, continuous-solids removal operation until the feed slurry is completely consumed.

8.2.2.2 Discontinuous-feed, discontinuous-solids-removal centrifuges

This category includes laboratory spin tube types (see Figure 8.2c) with various horizontally mounted or angle mounted configurations and assortments tailored for the application; 200000–500000 g ultracentrifuges; and zonal centrifuges for fine classification of solids with different densities (Leung 1998b).

8.2.2.3 Continuous-feed, discontinuous-solids-removal centrifuges

These include the manual low-*G* (5000–10000 g) and automatic high-*G* tubular (20000 g) centrifuges for bioreactors and fermenters for protein/cell biotech separations, and the multi-bowl and slow-speed solid-wall basket centrifuges. The latter is superseded by the continuous feed, continuous cake removal centrifuges.

Tubular centrifuges are used to process small amounts of feed samples in which there is low amount of solid in the feed. The rotor is long and slender with bowl length larger than the bowl diameter. The diameter of the bowl can be as small as 150 mm and as large as 600 mm. All tubular centrifuges are driven and supported from the top, overhanging

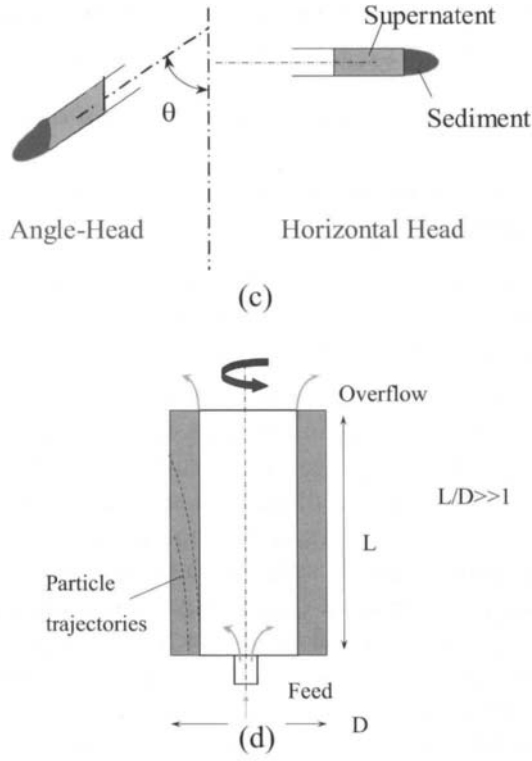

Figure 8.2c & d (c) Spin-tube centrifuges (angled-head in left schematic, and horizontal swing-out typed in right schematic). (d) Schematic of tubular centrifuge

vertically similar to a suspended top. Feed is introduced to the centrifuge bowl after acceleration to speed. In Figure 8.2d, it is shown that the feed is introduced from the bottom while clarified liquid overflows at the weir at the top, and in recent designs feed suspension is introduced from the top while clarified liquid overflows the weir located at the bottom of the bowl. Solids settle in the bowl until they build up to a point beyond which they get entrained by the high-velocity clarified liquid. When this happens the liquid turns turbid and feeding is stopped. The liquid pool is drained or siphoned at a low *G*-force and the cake is removed manually or with automatic plough or knife at lower *G* force. In automatic ploughing, a system of valves are opened, or closed, to facilitate discharge of respective liquid and solids to prevent mixing. Subsequently, the tubular centrifuge is cleaned before feeding again. The down time can be significant compared to the feeding time.

The manual disk centrifuge operates similarly to the tubular centrifuge in having continuous feed but discontinuous solids removal. It is

discussed in the next section along with the general disk stack centrifuge.

8.2.2.4 Continuous-feed, continuous-solids removal sedimenting centrifuges

There are two major classes of continuous sedimenting centrifuges – disk and decanter.

Figure 8.3a shows a schematic of a disk centrifuge. Feed slurry is introduced at the axis through a stationary feed tube to the core of the disk centrifuge wherein it is accelerated to speed.

Subsequently the feed is directed radially outward to the periphery of the disk stack. The conical disks in the stack are spaced between 0.3–2 mm apart depending on the application, particle size, and feed solids concentration etc. For a dilute feed, the disk spacing used is typically below 1 mm. While feed slurry flows up the conical channel formed between adjacent disks, heavier solids settle to the underside of the conical disk displacing the lighter clarified liquid moving radially inwards along the topside of the adjacent conical disk. The longitudinal component of the centrifugal force drives the heavier solids at the underside of the disk to the solids holding area located at the large diameter of the centrifuge bowl. The lighter clarified liquid collected near the centre exits the centrifuge at a weir dam located at the top of the centrifuge. The weir also sets the appropriate pool level for separation and clarification. In certain applications, a stationary centripetal pump, also known as a pairing disk, is used to skim the rotating clarified liquid converting its kinetic energy to hydrostatic pressure. This allows the liquid to be pumped to other locations without the need of additional pumping and at the same time avoids foaming when the liquid contains dissolved protein. When the holding area is filled with solids, the overflow/effluent becomes turbid and dirty, the operation is temporarily stopped and the disk stack is removed to allow access to removing the accumulated solids. The "manual" discharge disk centrifuge is suitable only for low feed solid concentration. Depending on the process application and the size of the centrifuge as many as 50 to 200 disks are used to increase the separation area. The high centrifugal gravity (4000–15000 g) together with the increased separation area render the disk stack ideal for fine solids separation and where feed concentration is dilute. It can be used to separate two immiscible liquid phases such as oil and water. For liquid-liquid separation, the feed to the disk stack takes the path of the feed holes located in the interior of the disk stack located at appropriate diameters in the disk stack, and clean liquid phases are discharged, respectively, at different diameters via weirs or centripetal pumps.

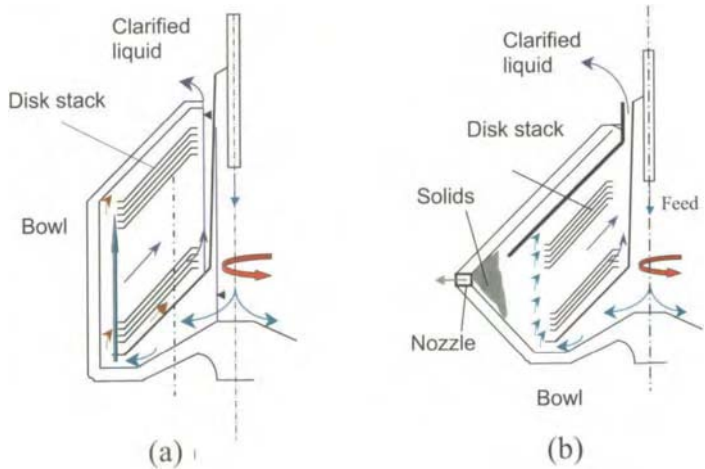

Figure 8.3a & b Disk centrifuges (a) manual disk, and (b) nozzle disk

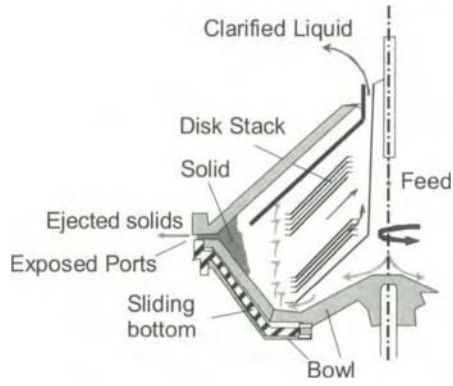

Figure 8.3c (c) Dropping bottom disk centrifuge showing bottom fully dropped exposing the solid discharge ports

As is well known, disk centrifuges are commonly employed in dairy separation of milk and cream. When the cake solid is flowable, a nozzle disk can be used wherein the geometry of the centrifuge bowl takes on a double cone with a series of nozzles distributed around the periphery of the bowl, see Figure 8.3b. The flowable cake is discharged continuously through the nozzles. For non-flowable cakes the bottom part of the "dropping bottom" or "intermittent-discharge" disk centrifuge opens periodically to discharge the accumulated solids and the opening/closing mechanism is controlled by hydraulics, or pneumatics together with a mechanical spring. This is shown in Figure 8.3c for a hydraulic mechanism operating on a sliding sleeve/bottom.

The period which the bowl is required to open for cake discharge depends on the solids loading rate and the size of the solids holding volume in the disk stack, which is typically between 40–50% of the total volume of the disk bowl.

For higher feed solids, the disk centrifuge is not suitable and decanter centrifuges should be used instead.

A schematic of the countercurrent flow decanter or solid bowl centrifuge is shown in Figure 8.4. After being accelerated in the rotating feed compartment or accelerator, feed slurry is introduced to the annular pool. Under high centrifugal force, the heavier solids migrate radially outward toward the bowl displacing the lighter liquid to the pool surface at a smaller radius. Solids are compacted against the bowl wall to form a cake by the centrifugal force. The cake is subsequently conveyed to the small diameter solid discharge end of the conical beach by the screw conveyor rotating at a differential rotational speed relative to the bowl. The cake is lifted above the annular pool in the "dry beach" and liquid from the cake further drains back to the pool, resulting in the discharge of drier cake.

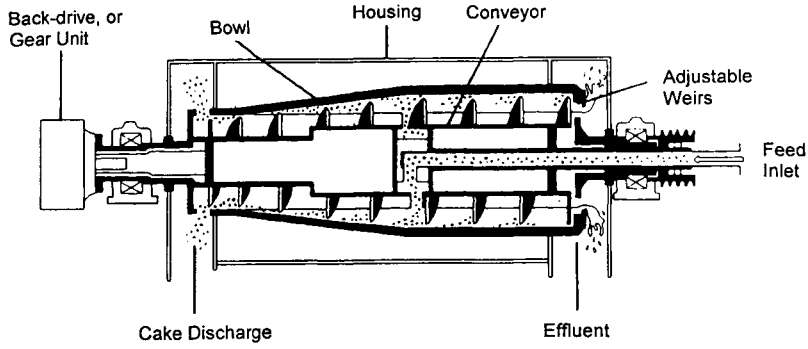

Figure 8.4 Solid-bowl or decanter centrifuge schematic

The gear unit and/or conveyor-drive controls the differential speed between the bowl and conveyor changing, as necessary, the solids retention time in the machine. The clarified liquid overflows the weirs located at the opposite end (large diameter end) of the machine. The pool depth is controlled by the discharge diameter of the weirs. The performance of the centrifuge depends on various operating variables such as the feed rate, pool depth, rotation speed or *G*-force, and differential speed and they should be optimized for a given process.

(a) Feed rate. The residence time of the slurry in the bowl affects centrate clarity. Decreasing the feed rate increases the liquid residence time and allows more efficient settling of suspended solids. With dilute suspensions (when the solids concentration is <1%), gravity or cyclonic thickening upstream of the centrifuge is recommended to concentrate and reduce the total volume of feed slurry or liquid handled. Hydraulic loading affects the main drive motor requirement from the point of accelerating the feed stream (Leung and Shapiro, 1996a, b, 2001) while solids loading affects the conveyor torque load.

(b) Pool depth. The proper pool depth depends on the settling characteristics of the solids in the feed slurry. By reducing the pool depth, a drier cake is normally obtained because a longer dry beach is available for cake drainage before discharge. The pool level should not be lowered to a point where centrate clarity suffers or solids conveyability is hindered. When the pool depth is increased the length of the drying beach is reduced. This generally results in higher cake moisture. A deeper pool improves centrate clarity, since liquid retention time is increased providing lighter and smaller particles more time to settle. Increasing the pool depth also eases transport of the cake due to liquid buoyancy, resulting in improved cake conveyability.

(c) Rotation speed and *G*-force. Higher rotation speed produces a greater centrifugal force acting on the solids in the cake and improves settling rate. The consequence is lower cake moisture and/or a clear centrate. However, this does not necessarily always hold. Some solids, especially the finer size fraction, have density very close to that of the liquid (i.e. nearly neutrally buoyant) due to adhesion of contaminants or bubbles to the solid surfaces. They do not settle regardless of large centrifugal force. Some cakes drain more readily under lower centrifugal force in which larger voids exist with higher cake permeability or lower specific cake resistance. While solids that form a compactible cake tend to pack tightly under high centrifugal force, increasing G beyond a certain point does not increase cake dryness for the compactible cake due to increasing cake resistance to deliquoring. For optimal operation with maximum centrate clarity, cake dryness and least power consumption, the centrifuge should be operated at the "lowest possible" speed compatible with the material characteristics and performance requirements. It is good practice during the initial start-up period to compare cake dryness and centrate clarity at different rotation speeds and Gs with the appropriate size driver or driven sheaves, or better still, with the machine speed controlled by variable frequency/speed drive to provide the capability of speed/G change. This allows selection of the optimum centrifugal forces for the specific application.

(d) Differential speed. By lowering the differential speed between the conveyor and the bowl, the solids residence time is increased. This usually causes an increase in cake depth piling against the bowl wall with increasing compaction stress and consequential higher cake dryness. This is often accompanied by increasing conveyance torque. Lower conveyor differential provides less turbulence and less re-suspension of solids. However, low conveyor differential may have the opposite effect that the incoming feed solids rate is higher than the transport rate offered by the conveyor in which un-transported solids build up in the bowl, get entrained by the high velocity clarified liquid, and eventually overflow with the centrate liquid. This imbalance in solids feed-to-transport rate is often accompanied by a conveyance torque gradually escalating over time. Based on the foregoing discussion, there should be a balance between solids input and solids removal to prevent loss of the centrate clarity and more seriously solids build up causing plugging of the centrifuge.

Differential speed $\Delta\Omega$ may be changed by changing the gear ratio r if it is available for the gearbox, or changing to a different gear box (with different r) altogether. The differential speed is related to the bowl speed Ω_b and the speed Ω_p of the pinion shaft (i.e. shaft protruding from the input end of the gearbox opposite to the conveyor) by the following:

$$\Delta\Omega = \frac{\Omega_b - \Omega_p}{r} \tag{2}$$

The kinematic relationship between $\Delta\Omega$ and Ω_p is shown in Figure 8.5a for a bowl (attached to gearbox housing) rotating at 3000 rpm for different gear ratios r = 20, 40, 60, 80 and 100 respectively. Gearboxes with larger gear ratios (r >100) are rated at higher maximum rotation speeds because the differential speed is much lower generating less friction and heat, and less dependent on lubrication to dissipate the excess heat generated as compared to gearbox with lower ratio. Consider Ω_b = 3000 rpm and the pinion shaft locked stationary (Ω_p = 0), with a gear ratio r = 80:1 this gives a differential speed of 37.5 rpm. An alternative is to provide an electric back-drive where the pinion is driven by a DC motor, or an AC motor, which can be controlled by a variable frequency drive (VFD). A hydraulic motor-and-pump system is also used as a back-drive for centrifuges. The hydraulic motor drives the bowl wall while the hydraulic forces cause the conveyor scroll to rotate relative to the bowl at lower speed and high torque. By controlling the hydraulic flow rate of the oil, or tuning the frequency of

the AC motor, the pinion speed is adjusted thus changing the differential speed $\Delta\Omega$ while the centrifuge is running. For example, when the pinion rotates in the same direction as the bowl (positive pinion speed in Figure 8.5a), respectively 1000 rpm and 2840 rpm, the differential speed becomes 25 rpm and 2 rpm, both of which are smaller than the $\Delta\Omega$ = 37.5 rpm when the pinion is locked $\Omega_p = 0$. The small differential speed allows longer retention time, which facilitates cake deliquoring to higher dryness. This can be easily accomplished by the conveyor back-drive (hydraulic or electric) that acts as a brake, slowing the pinion down without which the pinion would have rotated at the same speed as the bowl. The AC back-drive can further regenerate power back to the main drive as needed.

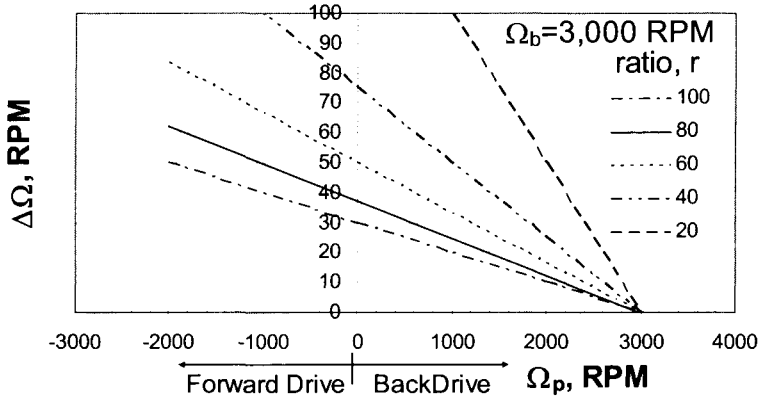

Figure 8.5a Differential speed versus pinion speed for various gear ratios

On the other hand, when there is a higher solids loading the differential speed needs to be increased to transport the cake at a faster differential rate. The electric drive and motor can drive the pinion opposite to the rotation of the bowl (i.e. $\Omega_p < 0$). Obviously this requires power input into the system. For example, with r = 80 and the pinion rotating at −1000 rpm, $\Delta\Omega$ = (3000– (–1000))/80=50 rpm. This increases the $\Delta\Omega$ above to beyond the nominal value of 37.5 rpm when the pinion is locked. This forward conveyor drive finds an important yet interesting application for processing plastic dewatering when stick-and-slip (due to momentarily increased or reduced friction) occurs between the conveyor-cake-bowl system. There is a maximum differential speed for a given gearbox design (not shown in Figure 8.5a) due to increasing heat dissipation at high differential speed. Figure 8.5b shows the trend with $\Delta\Omega$ plotted against Ω_p for three different bowl speeds. Based on

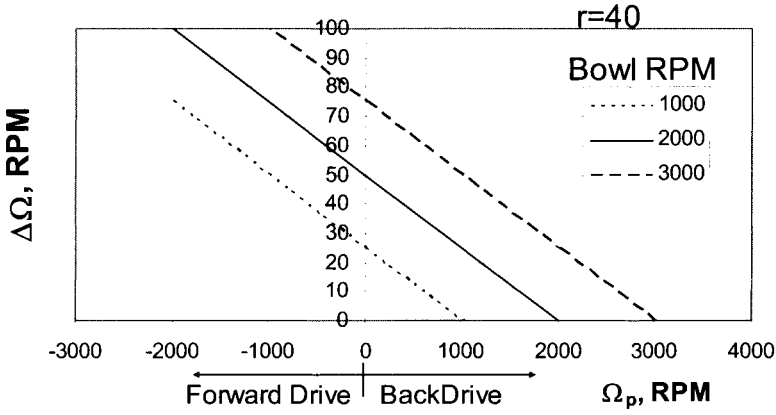

Figure 8.5b Differential speed versus pinion speed for various bowl speeds

equation (2), for a given pinion speed (including locked pinion at Ω_p = 0) higher bowl speed yields higher $\Delta\Omega$. Again for a bowl speed, the conveyor can be forward driven (Ω_p <0) with higher $\Delta\Omega$, or back-driven (Ω_p >0) with lower $\Delta\Omega$ depending on the process application.

The centrifuge can be over-torqued due to plugging with unconveyed solids accumulating in the bowl. If temporary reducing or stopping of feed to the machine while continuously maintaining the differential speed between the conveyor and bowl does not clear the machine, the rotation speed and thus the counteracting centrifugal force is reduced to facilitate cake transport. Unfortunately, the differential speed also reduces in lieu of equation (2). A centrifuge equipped with an electric or hydraulic back-drive has an advantage in that it allows the machine to adjust to the maximum differential speed to transport cake out of the machine despite the reduced bowl speed, or when the bowl stops rotating.

Flocculant and/or coagulant are frequently added to the feed slurry to agglomerate fine particles and improve the centrate clarity. This is frequently adopted for wastewater treatment in which polymer dissolved in the liquid stream is of lesser concern.

8.3 Dimensioning and scaling-up of sedimenting centrifuges

The scale-up of spin tube, tubular, disk and decanter will be discussed in the following sections.

8.3.1 Spin tube centrifuges

Spin tube, bottle, or test tube centrifuges are commonly used in the laboratory to determine whether a solid/liquid mixture is separable. Figure 8.6 shows a spin tube after centrifugation with the lighter phase separated from the heavier phase.

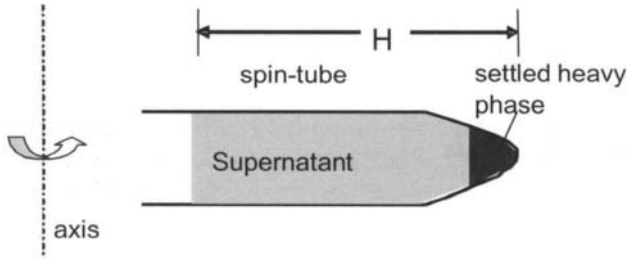

Figure 8.6 Schematic of a spin-tube

A systematic methodology has been developed to scale-up separation and classification of a suspension using spin tube (Leung, 2004a). The analysis requires a new dimensionless number *Le* for the spin tube that is defined as:

$$Le \equiv \sqrt{\frac{2\pi\mu H}{\Delta\rho\lambda(\phi)\eta G t x_0^2}} = \sqrt{\frac{2\pi\mu' H}{\Delta\rho G t x_0^2}} \tag{3}$$

In the above equation, the hindered settling factor λ is given by the Richardson and Zaki correlation $\lambda(\phi)=(1-\phi)^{4.65}$, where ϕ is the solids volume fraction in the feed. μ is the suspension viscosity, H is the initial height measured from the air-suspension interface to the spin tube large radius (see Figure 8.6), $\Delta\rho$ is the density difference between solids and suspension, η is a geometric factor accounting for non-radial oriented spin tube walls and it also accounts for acceleration efficiency, G is the centrifugal acceleration, t is the spin time, and x_0 is the characteristic particle size taken conveniently at 1 micron.

The cut size, x_c, of separation is defined as the maximum particle size in the supernatant (or centrate) or the smallest particle size in the sediment (settled solids). It is directly proportional to the *Le* number:

$$\frac{x_c}{x_0} = \frac{3}{\sqrt{\pi}} Le = 1.693 Le \tag{4}$$

The amount of solids that settles depends on the time and G as well as other parameters through *Le*, as evident in equation (3). The solids recovery R_s is defined as the solids settled at the bottom of the tube to the total solids initially suspended in the tube prior to centrifugation. It has been shown (Leung, 2004a) that R_s can be calculated from the following relationships:

$$R_s(Le) = \frac{1}{x_c^2}\int_0^{x_c} f_f(x)x^2dx + \int_{x_c}^{\infty} f_f(x)dx = \frac{I(x_c)}{x_c^2} + 1 - F_f(x_c)$$

$$F_f(x) = \int_0^x f_f(x)dx$$

$$I(x) = \int_0^x x^2 f_f(x)dx = \int_0^x x^2 dF_f \tag{5a–d}$$

$$I(x_n) \approx \frac{1}{4}\sum_{k=1}^{n}\left(x_{k-1} + x_k\right)^2\left(F\left(x_k\right) - F\left(x_{k-1}\right)\right)$$

The solids recovery R_s as given by equation (5a) depends on the cut size, which is a function of *Le*. $F_f(x)$ is the cumulative undersize distribution and $f_f(x)$ is the frequency distribution both of the feed suspension, and they are related in equation (5b). $I(x)$ is defined in equation (5c) and it can be approximated by the finite sum given by equation (5d).

In centrifugal classification using the spin tube centrifuge, when particles have the same density but different sizes, oversized particles are removed in the sediment residing at the bottom of the tube while the product containing the undersized unsettled particles are left in the supernatant (see Figure 8.6). The cut size as given by equation (4) can be changed by operating the centrifuge with appropriate *Le*.

The total solids suspended in the supernatant R_e by classification is the complement of the total solids settled R_s, thus

$$R_e(Le) = 1 - R_s = F_f(x_c) - \frac{I(x_c)}{x_c^2} \tag{6}$$

The particle size x_k in the supernatant can be determined from the cumulative size distribution F_e:

$$F_e(x_k, Le) = \frac{F_f(x_k) - I(x_k)/x_c^2}{R_e(Le)} \tag{7}$$

This shows theoretically how a feed suspension can be classified to obtain a finer sized product. Further, for classification the recovery of a given particle size (size recovery denoted by *SR*) in the product is given by:

$$SR(x_k, Le) = \frac{\int_0^{x_k} f_f(x)\left[1 - \left(\frac{x}{x_c}\right)^2\right]dx}{\int_0^{x_c} f_f(x)dx} = 1 - \frac{I(x_k)}{x_c^2 F_f(x_k)} \tag{8}$$

The index *SR* tracks particles of a given size in the centrate product and compare to the same size population in the feed. These relationships will be used later to demonstrate interpretation of bench testing and performance prediction.

8.3.2 Disk centrifuge

8.3.2.1 Le approach

For continuous centrifugation, the *Le* approach can be used to scale-up disk centrifuges for particle sedimentation. The dimensionless *Le* number is given by:

$$Le = \frac{\sqrt{\frac{Q}{L_p}\frac{\mu}{\Delta\rho}}}{\Omega R' x_0 \eta}$$

$$R' = R_1\left(1 + 0.5536\left(\frac{R_2}{R_1} - 1\right)\right) \tag{9a–c}$$

$$L_p = nL\cos\theta$$

Q is the volumetric feed rate to the disk centrifuge, θ is the half angle between the inclined disks and the vertical axis, L is the slant length of

each disk stack, L_p is the projected length of the disk stack, R_1 and R_2 are the inner and outer disk stack radii respectively, and the effective radius R′ is defined in terms of R_1/R_2. μ and $\Delta\rho$ are, respectively, the viscosity and density difference between the suspended solids and suspension, Ω the angular rotation speed, and η the efficiency of acceleration and feed distribution uniformity to each of the conical channel. The cut size for a disk centrifuge is also given by equation (4). For scale-up:

$$Le_1 = Le_2 \tag{9d}$$

In other words, if machine 1 can achieve 95% solids recovery, by maintaining the equality of equation (9d), machine 2 with different feed rate and size can also achieve the same recovery. The *Le* approach can be further used to predict performance of machines.

8.3.2.2 Sigma factor

Traditionally, the Σ factor has been used in scale-up in particle separation for similar design machines (Leung, 1998a, 1997a, b). The Σ factor is related to the equivalent sedimentation area required for 50% particle capture of the feed.

$$\Sigma = \frac{2\pi n\Omega^2}{3g\tan\theta}\left(R_2^3 - R_1^3\right) \tag{10}$$

The required Σ factor for 100% capture is higher than that of 50% capture (Leung, 1997b, 1998a).

For scale-up between similar design centrifuges, the feed rate for Q_1 with Σ_1 is related to the feed rate Q_2 feeding a machine with Σ_2. Thus,

$$\frac{\Sigma_1}{Q_1} = \frac{\Sigma_2}{Q_2} \tag{11}$$

In other words, if machine 1 attains 95% solids recovery, by maintaining the equality in equation (11) a machine 2 of similar design but with different feed rate and size can also achieve the same recovery according to the model.

8.3.3 Decanter and tubular centrifuges

8.3.3.1 Le approach

A model has been developed and used for separation, classification and degritting (Leung, 1998a, 2004b). The *Le* model accounts for the

complicated flow pattern that exist in rotating flow as found in a centrifuge. In some decanter designs and mostly with tubular bowl, the feed is confined to a thin surface layer. Based on the *Le* model, the scale-up for tubular bowl and decanter centrifuges are given by (Leung, 2004b):

$$Le = \frac{\sqrt{Q/L}\sqrt{\mu/\Delta\rho}}{QR_p x_0 \eta_a} \tag{12}$$

L is the clarifying length of the bowl, R_p the radius at the air-suspension interface (i.e. radius of the pool surface), and η_a the acceleration efficiency of the feed suspension.

Despite the fact that the flow pattern and *Le* number differ between the rotating test tube and the continuous feed tubular and decanter centrifuges, all the essential results remain the same (Leung, 2004a, b). The cut size for both the decanter and the tubular centrifuges is also given by equation (4), the solids recovery R_s together with the ancillary equations is given by equations (5a–d), the solids recovery in the centrate R_e is given by equation (6), the solids size distribution in the centrate is given by equation (7), and the size recovery is given by equation (8).

In some decanter designs, the flow in the bowl can be approximated as plug flow in which case a second dimensionless parameter also becomes important. The second parameter r_p is the ratio of the pool surface radius to that of the bowl radius R_b:

$$r_p = \frac{R_p}{R_b} \tag{13}$$

It can be shown that the plug flow model can be approximated by the surface flow model with mathematical transformation using a power law $(r_p)^n$. Furthermore, the surface flow model result can be used to cover both cases (surface flow and plug flow) with the adjustment all lumped in the efficiency factor η_a in equation (12), which includes the acceleration efficiency, flow pattern (surface or plug flow), and viscosity deviation from actual measurement.

8.3.3.2 Sigma approach

The equivalent Σ factor for 50% for the elongated bowl which is applicable to both decanter and tubular centrifuges is given by the approximation:

$$\Sigma \approx \frac{2\pi\Omega^2 L}{g}\left(\frac{3R_b^2 + R_p^2}{4}\right) \tag{14}$$

The sizing rule follows that of equation (9d). The Σ factor has pitfalls in that for tubular and decanter centrifuges the flow may not be plug flow as assumed in the Σ model. In fact, in some designs for both tubular and decanter (especially for the axial flow with thin ribbon blade) it has been found that the flow sweeps through a thin surface layer bypassing the rest of the pool liquid (Leung, 2004b).

8.3.3.3 G volume approach

Another empirical approach that has limited merits is the *G–V* approach. *V* is the clarification volume of a given centrifuge. This is especially for suspensions containing poorly defined solids (particle size and density unknown, such as flocculated solids). For similar designs of centrifuge:

$$\frac{G_1 V_1}{Q_1} = \frac{G_2 V_2}{Q_2} \tag{15}$$

The above approach stems from the equality of *Gt*, i.e. $G_1 t_1 = G_2 t_2$ (see equation (3) for the spin tube), given that *t* the retention time for an ideal plug flow is equal to *V*/*Q*. Obviously the pitfall as with the Sigma approach is that the flow pattern may not be a plug flow uniformly sweeping the entire bowl volume.

8.3.3.4 Deliquoring

The maximum cake solids attained by a continuous decanter is affected by the product *Gt*/*h* or more precisely t_d (as defined later) for drainage of granular cakes where *t* is the retention time of the cake solids in the centrifuge, and *h* is the cake height. Note the retention time for cake solids is much greater than the liquid in the bowl.

For process applications involving a compactible cake, setting aside the transient associated with liquid expression as a result of slow compaction of solids under centrifugal force, the cake solids is a function of the compaction pressure p_s and time.

8.3.3.5 Conveyance torque

Since there is continuous cake transport the scale-up of conveyance torque is equally important. The conveyance torque for a decanter is the torque required to drive the spline shaft of the conveyor maintaining a differential speed for the conveyor with respect to the

rotating bowl. Obviously if there is no differential speed the conveyance torque is zero. The conveyance torque T has two contributions, equation (16): (a) torque under loading T_L when there is continuous feed and continuous transport of cake from the cylindrical bowl to the cake discharge of the decanter, and (b) torque under no-load (i.e. no feed and no cake transport) T_{NL} as a result of (i) build-up of the cake heel/residual trapped in the space between blade tips and the inner bowl surface, and (ii) friction from bearings between the conveyor and bowl. Thus, at a given cake solids throughput m_{cs} the total torque is a sum of the two components as illustrated in Figure 8.7a:

$$T = T_{NL} + T_L \tag{16}$$

The no-load torque has to be determined from testing. On the other hand, predicting the torque under loading T_L is equally difficult. An approximate prediction of T_L is given by the torque required to transport cake in the cylindrical bowl (against resistance from friction between the conveyor blade and the cake) and the torque required to transport cake in the adjoining conical bowl (against frictional resistance from the blade and lifting the cake against centrifugal acceleration). While the cake is always fully submerged in the liquid pool in the cylinder, the cake in the conical bowl initially is submerged in the liquid pool but subsequently it is transported out of the pool in the dry beach. This is depicted in Figure 8.7b. The effective density of the cake is modified accordingly to account for the buoyancy of the liquid in different regions.

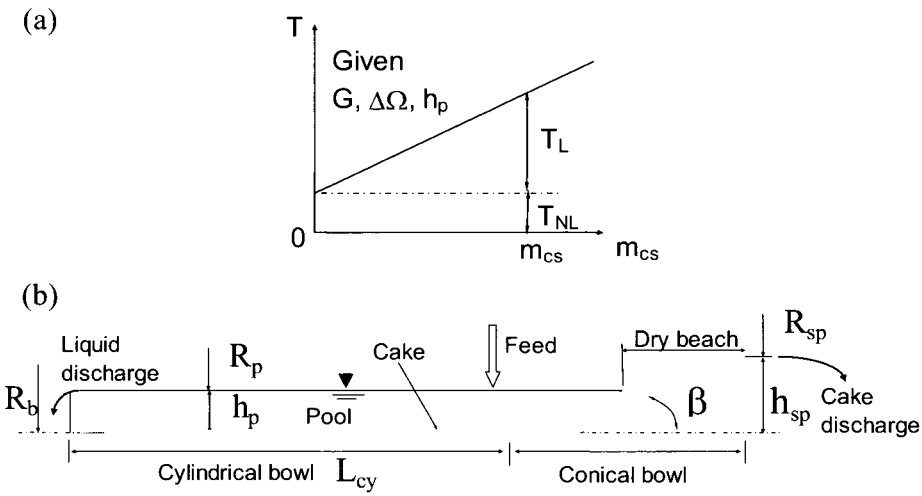

Figure 8.7 (a) Schematic of conveyance torque at the spline shaft of the conveyor in a decanter as a function of dry solids throughput. (b) Schematic of a decanter (conveyor omitted for clarity)

The approximate torque under loading is given by:

$$T_L = \frac{m_{cs}G}{\Delta\Omega}\left\{\left[\frac{\Delta\rho}{\rho_c}\left(\frac{R_p + R_b}{2R_b}\right)h_p + \left(\frac{R_p + R_{sp}}{2R_b}\right)\left(h_{sp} - h_p\right)\right]\right.$$
$$\left.\left[1 + C_f \cot\beta\right] + \frac{1}{2}C_f L_{cy}\frac{\Delta\rho}{\rho_c}\right\} \qquad (17)$$

As always, G is evaluated at the bowl wall, i.e. $G = \Omega^2 R_b$. $R_p = R_b - h_p$ where h_p is the pool depth, and $R_{sp} = R_b - h_{sp}$ where h_{sp} is the spillover height or maximum pool depth. C_f is the effective frictional coefficient between the cake and the face of the conveyor blade, ρ_c is the cake density and $\Delta\rho$ is the density difference between the cake and the pool liquid. Angle β is the conical beach angle with respect to the axis of the machine. C_f is determined from testing. High conveyance torque can be generated from any of the following scenarios separately, or in combination: (a) transport of high solids throughput with thick cake height; (b) high G; (c) cake or reject containing coarse solids fraction as in mineral classification; and (d) low differential speed.

For deliquoring of environmental solids (Leung, 1992) high torque is generated from a thick cake height, low differential speed and high G; the cylindrical bowl is largely occupied with cake solids and the conveyor can be treated as a coaxial rheometer with respect to the rotating bowl (Leung, 1998b). The differential speed is analogous to the rheometer rotation. The rotation of the bowl simply generates a G-field acting on the cake. The torque in lifting the cake in the conical beach against G-force can be neglected as compared to the conveyance torque in the entire length of the bowl. Furthermore, the cake is largely submerged in a deep pool in the entire bowl (i.e. no dry beach) taking advantage of the buoyancy force to facilitate cake transport. In this case, the torque as given by equation (17) can be further approximated by (Leung, 1998b):

$$T_L = \frac{\Delta\rho}{\rho_c}C_f L_{eff}\frac{m_{cs}G}{\Delta\Omega} \qquad (18a)$$

Furthermore, the ratio $(\Delta\rho/\rho_c)C_f$ is defined as:

$$\frac{\Delta\rho}{\rho_c}C_f = \frac{T_L\Delta\Omega}{m_{cs}GL_{eff}} \qquad (18b)$$

L_{eff} is the effective length for transport of cake (typically approximated as $^2/_3$ of the entire length of the bowl). This can be modified depending on the design of the centrifuge.

8.4 Testing

Testing can be divided into bench testing, pilot testing and production testing. These tests are usually conducted in this sequence depending on the circumstance and need. Bench tests are best suited for feasibility studies on a new process application or an existing application in which it is desired to confirm that the suspension behaves identically, or in similar manner, to what has been known in the past on the same suspension. Once bench tests show the separation process is feasible and promising, it is recommended to go for piloting, provided time, larger quantities of representative test materials, budget and a scaleable pilot test machine are all available for the venture. Also it is the intent to determine the impact of dynamic effects, if any, on the final scale-up for the production unit. After the production has been initiated based on successful bench and pilot testing, it is not uncommon during the course of production over time that representative samples are bench tested in the laboratory with the objectives of possible process or product modification, or as diagnostic checks especially when the product quality from the production units (centrate suspended solids or cake moisture) seems to drift from meeting specification. Another valid occasion for testing after a production-size centrifuge has been installed is to test available new technologies on a bench or a pilot scale.

8.4.1 Process objectives

The process objectives need to be established prior to any test work being conducted. Bench tests should be carried out on the feasibility of whether the process objectives can be met with the full size centrifuge, bearing in mind the quantitative measure of the process objectives. Below are some typical process objectives.

8.4.1.1 Separation

A common example is the two-phase solid/liquid separation. Again the solid phase refers to suspended solids and not dissolved solids in solution. For example, it is desired to achieve 95% recovery of the solids in the sediment. For a three-phase separation, this can be a

liquid/liquid/solid such as 50% oil- 20% water- 10% organic/biological solids with a 20% oil-water emulsion. As an example, the aim is to get the water phase with less than 1% oil, cake solids with 30–40% w/w, and an oil phase with emulsion mixture. Another three-phase separation may involve a liquid/solid/solid suspension such as polypropylene (PP) with specific gravity of 0.9, polyvinyl chloride (PVC) with specific gravity of 1.4 and water with a specific gravity of 1. The objective is to split these three phases with both the PP and PVC separated solids to contain less than 30% water and minimal cross contamination of solids.

8.4.1.2 Clarification

The objective is to get a clear liquid free from suspended solids. For example, the quality of the separated liquid is measured by having less than 0.2% suspended solids. Another measure is the solids recovery in the sediment or cake, for example it is desired to have solids recovery of 95–99% leaving 1–5% solids in the supernatant/effluent/centrate.

8.4.1.3 Classification

This is to fractionate the feed suspension into a suspension with finer particle sizes and remaining suspension with coarser particle size by centrifugation. In contrast with clarification, it is undesirable to have 100% capture or removal of solids from the suspension. Several measures have been practised. If a certain fine size such as 2 microns is required for enrichment, the feed may contain say 65% <2 microns and after centrifugation 85% <2 microns is required in the product. Centrifugation is carried out with coarser particles settling out of suspension until this process condition is met. It is also common to stipulate that the size recovery of the <2 microns needs to be at least greater than 85–90%. This may sometimes be replaced by another requirement that the total solids of all sizes recovered from fractionation needs to exceed 70%, i.e. 30% of solids are lost in the reject sediment. Another common measure in fractionation is to monitor the median size of the feed and product. For example, the feed may have median size of 45 microns and the product should have 5–10 microns, etc.

8.4.1.4 Degritting

In degritting, oversize particles such as particles with size greater than 25 microns, or 45 microns, need to be removed. For example, a measure may be <1% of the oversize particles present in an acceptable product. Also foreign particles (such as grinding media or sand) should be removed to meet a tight specified concentration in parts per million.

8.4.1.5 Concentration or thickening

The objective is to remove liquid so that the product (concentrated phase) meets a thickened consistency. The specification would typically be on the product solids concentration together with minimal solids in the separated liquid phase.

8.4.1.6 Washing

Often in mineral and chemical processing, the solids purity of the product is important and it should have minimal byproducts from upstream processing. Washing with pure liquid (with minimal contaminant) is needed. For example, one goal may be to reduce the chloride content of a bicarbonate salt to less than 1% by washing, and another goal may be to reduce the amount of wash liquid as measured by a wash ratio.

8.4.1.7 Deliquoring or dewatering

The object is to remove by mechanical centrifugation as much liquid as possible from the cake. A typical measure is the % solids, or % moisture, in the cake.

8.4.2 Bench testing

There are several key bench scale tests that can be performed to provide a feasibility study along some set objectives, only the spin tube and bucket tests will be discussed. The batch bench-scale solid bowl and basket centrifuge, which require a larger test sample, are also frequently conducted as one level beyond the spin tube and bucket tests to reassure that separation can be achieved, the details of which can be found elsewhere (Leung, 1998a).

8.4.2.1 Spin tube testing

A spin tube as illustrated in Figures 8.2c and 8.6 is perhaps the most common centrifugal test that is widely performed as the test unit is readily available commercially and the basic unit is inexpensive.

8.4.2.1.1 Settleability

A series of tests is carried out to determine whether a suspension is separable under reasonable centrifugal gravity. If indeed the suspension is separable what would be the G and time duration that are required to make a separation? To that end, a trial involving several G's and several t's are typically carried out. Both G and t should cover a wide range of values in the tests. If necessary, chemicals such as

coagulant and flocculant can be added in small dosage to agglomerate the solids to enhance separation. The type and the dosage (denoted as D) of added chemical are to be determined from the tests. Note there are applications in which the product is the liquid phase in which the application prohibits addition of chemicals which may be soluble in the liquid. This constraint needs to be known upfront. Also another variable of interest is the effect of feed solid concentration by weight W_f. Testing should start out with the nominal or process specification value of W_f, and if sample quantity permits testing should also be done with more concentrated feed (higher W_f), or diluted feed (lower W_f) to evaluate the impact of feed solids concentration. In each case, qualitative observation can be made on the supernatant (relatively cloudy, turbidity, clarity, etc.) from each test and more quantitative measures should be taken such as the solids recovery in the sediment.

The solids recovery by centrifugation in the spin tube from a material balance is given by:

$$R_s = \frac{1-\frac{W_e}{W_f}}{1-\frac{W_e}{W_s}} \approx 1-\frac{W_e}{W_f} \qquad \text{for } \frac{W_e}{W_s} \ll 1 \tag{19}$$

In the above, W_i is the weight fraction of suspended solids with subscript i equal to f (feed), s (cake) and e (effluent or supernatant). Note it is difficult to measure accurately the sediment solid concentration as it is present in such a small quantity. So the above approximation is routinely adopted. Another point is that the solids compaction in the spin tube does not reflect what is to be expected from a centrifuge anyway (Leung, 2004a).

The solids recovery R_s depends in general on G, t, W_f, and if a chemical is used it depends on the type and dosage D of the chemical. Figure 8.8a shows a schematic on the solids recovery as a function of t for different levels of G. The arrow points to curves with increasing G. Figure 8.8b shows a schematic involving replotting the same data but cast in form of the function of G for different t's with the arrow pointing along increasing time. On the other hand, Figure 8.8c shows a schematic with the data replotted as a function of the product of Gt, and finally Figure 8.8d shows a schematic recovery versus Gt and with chemical dosage D as the parameter, or with feed concentration as the parameter. If there is an improvement in solid recovery for diluted feed this is due to reduced hindered settling from the original feed concentration.

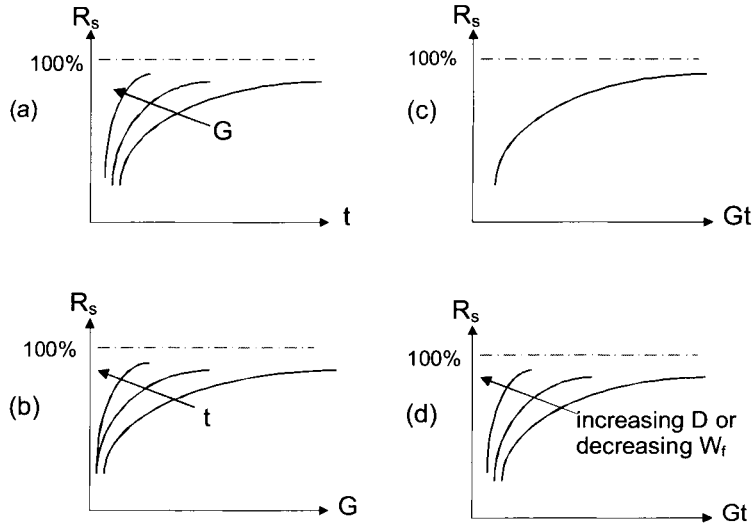

Figure 8.8 (a-d) Solids recovery (R_s) as function of t, G, Gt with and without polymer

t can be considered as the retention time of the suspension in the separation zone. A high feed volumetric rate to a centrifuge represents reduced retention time and vice versa. The appropriate correlation of Gt can be seen (Greenspan, 1983; Sambuichi *et al*, 1987; Leung, 2004a). The product Gt is referred to as G-seconds implying the time duration and G-force for effecting the separation. In theory, if a spin tube centrifuge can provide a given solids recovery under a given Gt, then another spin tube centrifuge of similar geometry having the same Gt should provide the same performance, i.e. same solids recovery. While this all holds for a batch spin tube, extending the approach to a continuous-feed centrifuge leads to $Gt = G(V/Q)$ which requires caution. This implies that the centrifuge is treated as a "batch tank" subject to plug flow which may not hold due to the complicated flow dynamics in a rotating bowl with or without a conveyor screw, and with continuous feed and continuous/discontinuous sediment removal.

In the following example, we will illustrate how test data are analyzed and interpreted for a spin tube centrifuge.

Example 1 A flue-gas-desulfurisation (FGD) sludge from a scrubber is obtained in which the particle size distribution measured from a Coulter particle size analyser is shown in Figure 8.9a. The median particle size in the slurry is about 17.3 microns and 10% of the population size is below 4 microns.

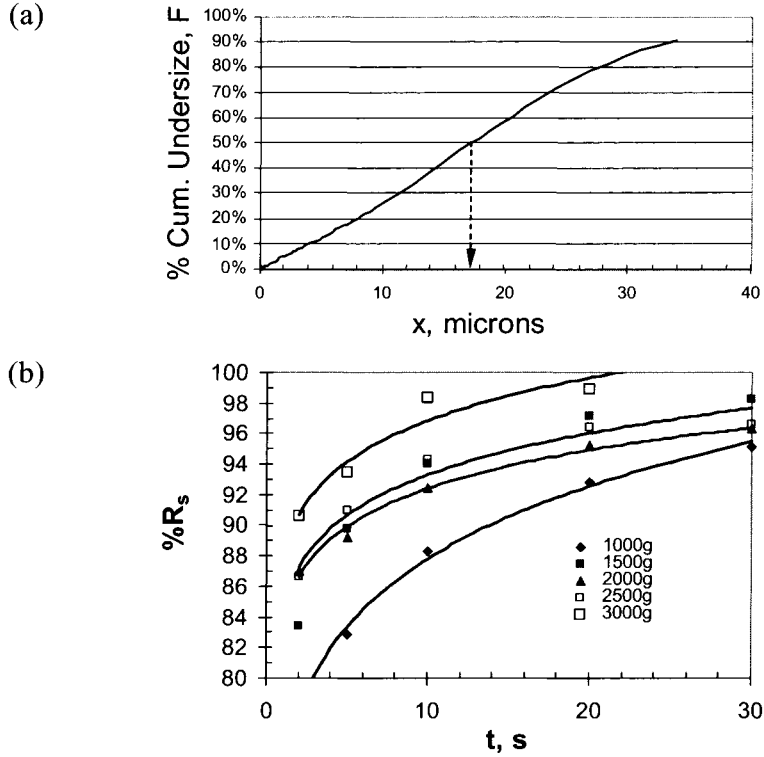

Figure 8.9 (a) PSD of feed FGD slurry. (b) Total solids recovery versus centrifugation time for different *G*'s

The tests were conducted for G = 1000, 1500, 2000, 2500, and 3000 g and t = 2, 5, 10, 20 and 30 s following similar procedures to those discussed. No chemical agent is used to enhance flocculation or coagulation. The feed solids (suspended solids only) concentration was fixed at 3.44%. The supernatant for each test condition was filtered and the suspended solids concentration was measured. Table 8.1a reports the suspended solids in the supernatant after centrifugation at a given G and t. The solids recovery is calculated from equation (19), neglecting W_e/W_s and the calculated value is reported in Table 8.1a.

Figure 8.9b plots the results of the solids recovery as a function of time with G as a parameter. As can be seen, for a given G increasing centrifugation time allows particles to settle and this increases solids recovery. Also, for a given time duration, increasing centrifugal gravity increases solids recovery. Over 95% is captured for high Gs or long time duration for which the sample is subject to centrifugation. Obviously, increasing G/g, t, or both, yields higher solids recovery.

An improvement to the analysis is to plot the solids recovery versus $(G/g)t$ instead of G or t as illustrated in Figure 8.10. Indeed, the five

Table 8.1a Spin tube test data and analysis.

$(G/g)^*$	t, s	W_e, %	$(G/g)t$, s	Le	R_s, %
1000	2	0.765	2000	8.92	77.8
1000	5	0.589	5000	5.64	82.9
1000	10	0.403	10000	3.99	88.3
1000	20	0.247	20000	2.82	92.8
1000	30	0.169	30000	2.30	95.1
1500	2	0.571	3000	7.28	83.4
1500	5	0.353	7500	4.61	89.7
1500	10	0.207	15000	3.26	94.0
1500	20	0.099	30000	2.30	97.1
1500	30	0.06	45000	1.88	98.3
2000	2	0.446	4000	6.31	87.0
2000	5	0.372	10000	3.99	89.2
2000	10	0.26	20000	2.82	92.4
2000	20	0.164	40000	1.99	95.2
2000	30	0.126	60000	1.63	96.3
2500	2	0.46	5000	5.64	86.6
2500	5	0.225	12500	3.57	91.0
2500	10	0.059	25000	2.52	94.3
2500	20	0.039	50000	1.78	96.4
2500	30	0.123	75000	1.46	96.6
3000	2	0.322	6000	5.15	90.6
3000	5	0.308	15000	3.26	93.5
3000	10	0.195	30000	2.30	98.3
3000	20	0.116	60000	1.63	98.9

*Note G is reported at the largest diameter of spin tube, R_{bowl}

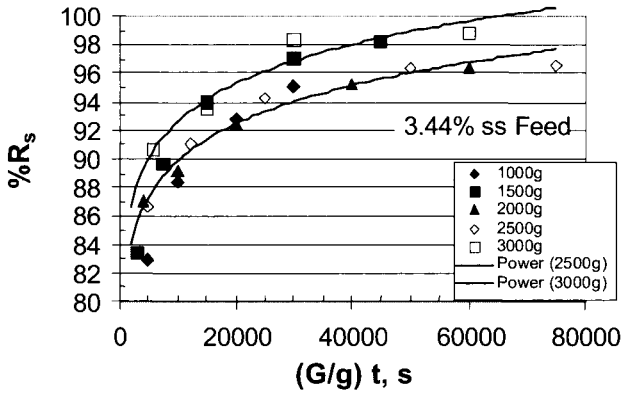

Figure 8.10 Recovery versus $(G/g)t$

sets of data from Figure 8.9b more or less collapse onto a common band of data.

More rigorously, the variable governing sedimentation in a spin tube centrifuge is the dimensionless *Le* number defined by equation (3) and the recovery data should be graphed against *Le*.

The model accounts for both constant *G* and variable *G* for which *G* varies linearly with radius (Leung, 2004a). For simplicity, the constant *G* model is used. Additional raw data on the equipment and testing are required and they are shown in Table 8.1b.

Table 8.1b

G/g	1000
t, s	2
H, cm	8.30
μ', g/(cm s)	0.025
ρ_s, g/cm^3	2.3
ρ_L, g/cm^3	1.02
$\Delta\rho$, g/cm^3	1.28
R_{bowl}/R_{mean}	1.533
Brookfield measurement:	
μ, g/(cm s)	0.035

For illustration, the first test condition with $G = 1000\ g$ and $t = 2$ s is used to calculate *Le*. From equation (3) and the data of Table 8.1b, the *Le* number for the first test condition is:

$$Le = \sqrt{\frac{2\pi\mu H}{\Delta\rho\lambda(\phi)\eta G t x_0^2}} = \sqrt{\frac{2\pi\mu' H}{\Delta\rho G t x_0^2}}$$

$$= \sqrt{\frac{2\pi(0.025)(8.30)}{(1.28)(1000/1.533)(981)(2)(1\times10^{-4})^2}} = 8.92$$

Note $G/g = 1000$ is reported at the bowl/tube radius and the geometric mean radius $R_m = (R_b(R_b - H))^{0.5}$ needs to be used (Leung, 2004a). For the spin tube geometry used in the tests, $R_b/R_m = 1.533$. The remaining test data are also analysed similarly and they are reported in Table 8.1a. The solids recovery data for different *G*'s and *t*'s are plotted in Figure 8.11 as a function of *Le*. Remarkably, the data fall into a more orderly and consistent trend regardless of the values of *G* or *t*. Also, the

effective viscosity $\mu' = 0.025$ P is used as this matches with the prediction, as will be shown later. Even if $\mu' = \mu = 0.035$ P was used, this only shifts the data (simple translation in a log scale on *Le*) and would not have affected the correlation of the test data by *Le*.

The model can predict solids recovery using the PSD of the feed suspension (Leung, 2004a). Using the feed PSD, $F_f(x)$ as delineated in Figure 8.9a and using equations (5a–d), the solids recovery R_s can be calculated from the model. The results from the prediction model (*Le*, R_s) are listed in Table 8.1c and plotted in Figure 8.11 as a solid curve to be compared with the test data. By adjusting the effective viscosity to 0.025 g cm^{-1} s^{-1} instead of the measured value of 0.035 g cm^{-1} s^{-1}, the test data compares excellently with the model prediction in Figure 8.11.

Table 8.1c Theoretical prediction of R_s using feed PSD.

Le	R_s, %	*Le*	R_s, %	*Le*	R_s, %	*Le*	R_s, %
0.00	100.0	0.63	98.8	2.96	93.3	8.84	77.1
0.18	99.6	0.76	98.5	3.55	91.9	9.68	74.4
0.21	99.5	0.91	98.2	3.89	91.1	10.60	71.4
0.25	99.5	1.09	97.7	4.67	89.2	11.61	68.0
0.28	99.4	1.19	97.5	5.12	88.0	12.72	64.3
0.30	99.4	1.57	96.6	5.60	86.6	13.93	60.2
0.33	99.3	1.72	96.2	6.14	85.1	15.26	55.8
0.37	99.2	1.88	95.8	6.72	83.4	16.71	51.1
0.44	99.1	2.25	95.1	7.37	81.6	18.30	46.2
0.53	99.0	2.70	94.0	8.06	79.5	20.05	37.5

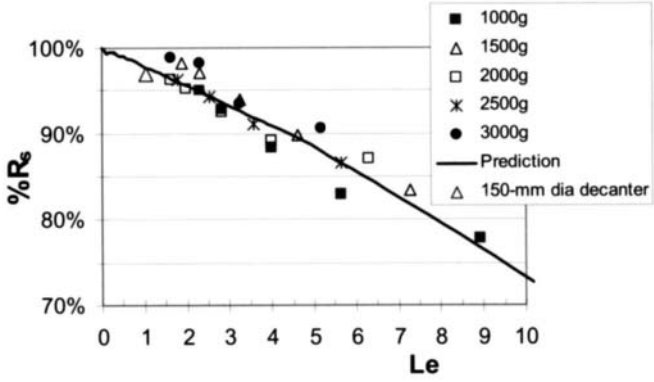

Figure 8.11 Comparing solids recovery in cake for spin-tube and decanter tests with theory on FGD slurry ($\mu' = 2.5$ cp, measured $\mu = 3.5$ cp, 3.44% suspended solids)

8.4.2.1.2 Cake Solids

It is important to determine the cake solids (or cake moisture) to be obtained from centrifugation as it dictates downstream processing and perhaps more importantly whether centrifugation is the best separation and dewatering device for the task. However, it is very difficult to determine accurately the solids concentration in the sediment in the spin tube test as the quantity is very small and is subject to large error despite some test tube bottles being tapered to facilitate such measurement.

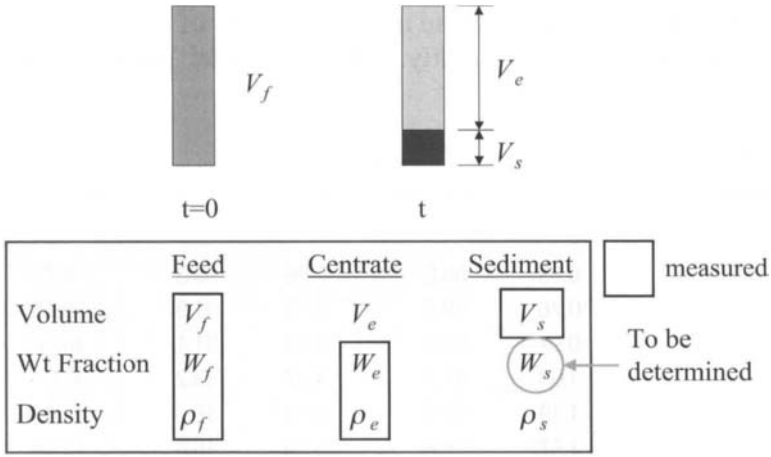

Figure 8.12 Schematic of quantity to be determined in a test tube centrifuge

With reference to Figure 8.12, the material balances of both the liquid and solids leads to the following equations for determination of the cake solids:

$$V_f\rho_f = (V_f - V_s)\rho_e + V_s\rho_s$$

$$V_f\rho_f W_f = (V_f - V_s)\rho_e W_e + V_s\rho_s W_s$$

$$W_s = \frac{\rho_f W_f - \left(1 - \frac{V_s}{V_f}\right)\rho_e W_e}{\rho_f - \left(1 - \frac{V_s}{V_f}\right)\rho_e} \qquad (20a\text{–}c)$$

Example 2 $V_f = 10$ ml, $W_f = 17.6\%$, $\rho_f = 1.145$ g cm^{-3}, $W_e \approx 0$, $\rho_e = 1$, $V_s = 1.2$ ml, the weight fraction of solids in the sediment is determined to be $W_s = 76\%$.

8.4.2.1.3 Classification test

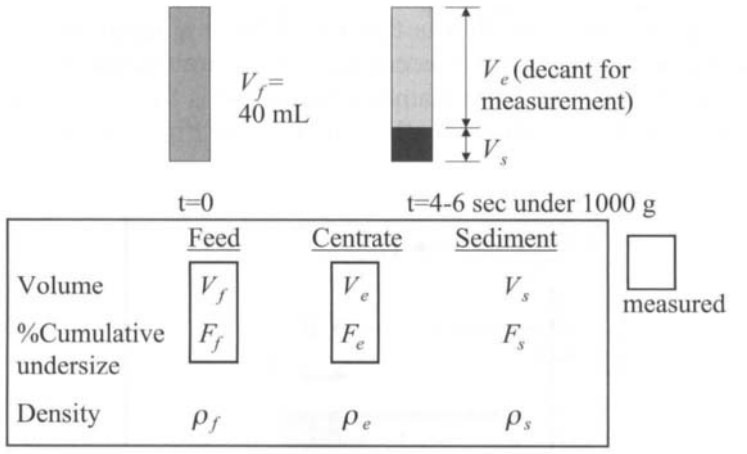

Figure 8.13 Schematic of quantities to be determined in a classification testing

In classification a finer fraction is separated to yield a finer particle size distribution (PSD) in the end product. Figure 8.13 shows the test variables. A material balance on the overall solids and liquid gives:

$$V_f \rho_f = V_e \rho_e + \left(V_f - V_s\right) \rho_s \tag{21a}$$

The product recovery in the supernatant or unsettled suspension is:

$$R_e = \frac{\rho_e V_e}{\rho_f V_f} \approx \frac{V_e}{V_f} \tag{21b}$$

The size recovery *SR* or yield of a given size requires the PSD of both the feed $F_f(x)$ and that of the supernatant $F_e(x)$:

$$SR(x) = R_e \frac{F_e(x)}{F_f(x)} \tag{21c}$$

This is similar to the standard tube test with the exception that both the PSD of the feed and resultant product need to be determined.

8.4.3 Bucket test

8.4.3.1 Deliquoring by drainage

A perforate bucket can be used for the deliquoring test to determine liquid drainage behaviour in the cake. This simulates deliquoring of a

cake in a decanter in the dry beach, or deliquoring of a cake in a basket and screen type centrifuge. A bucket resembles a spin tube but with two distinct differences. The first is that the volume is larger with a bigger diameter and a longer tube to accommodate sedimentation with a height of at least 25 mm. The large diameter round end is further equipped with a screen filter. This is shown by the schematic of Figure 8.14.

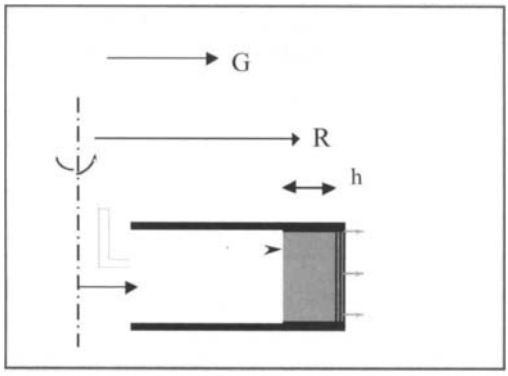

Figure 8.14 Schematic of bucket centrifuge

For a given thickened feed charged to the bucket, tests are carried out at different G, t and cake height h on the sample. The cake solids can be determined after each test. Figures 8.15a and 8.15b show respectively the cake moisture as a function of time and cake height. The cake height represents the throughput of a given centrifuge. For a given cake height, as t increases the moisture decreases as the bulk liquid drains through the coarse (large diameter) capillaries formed between solids, then the finer capillaries.

Subsequently, the liquid film on the solids starts to drain from a thick liquid layer to a thinner one. The transient behaviour of drainage can be slow resulting in a much longer time to drain the residual moisture. Cake moisture is reduced when deliquoring with a thin cake. Figure 8.15b shows schematically that as G increases lower cake moisture results. The effect of cake height is shown in Figures 8.15c and 8.15d.

The effect of G is shown in Figures 8.15e–f. It is of interest to note that as the G-force increases there is a modest reduction in moisture after which the cake seems to reach an equilibrium moisture level. A much higher G force must be imposed to ensure further reduction in cake moisture. This corresponds to removal of the pendular moisture when capillary pressure is overcome. Basket centrifuges are confined to G-forces much less than this limit.

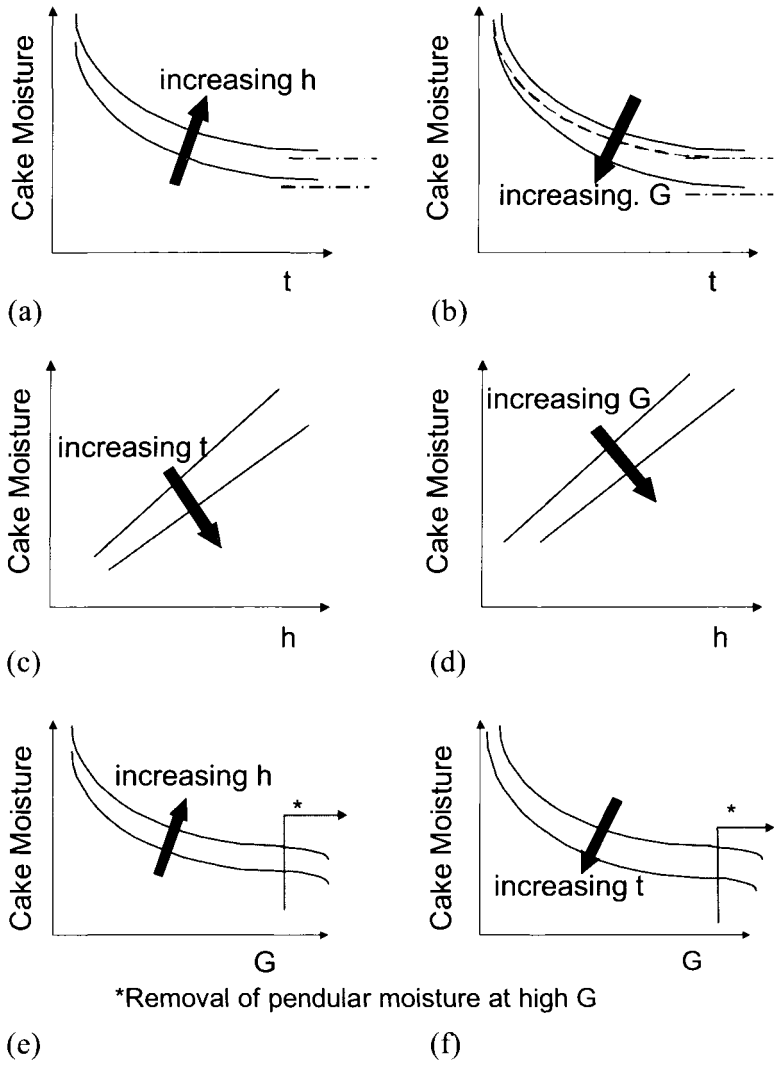

Figure 8.15 (a) Cake moisture as function of time and cake height for a given *G*. (b) Cake moisture as a function of time for different *G* and fixed cake height. (c) Cake moisture as function of time and cake height for a given *G*. (d) Cake moisture as a function of time for different *G* and fixed cake height. (e) Cake moisture as function of *G* and cake height for a given time. (f) Cake moisture as a function of *G* for different time and fixed cake height

It is appropriate to plot cake moisture versus t_d that is proportional to the group Gt/h (Leung, 1998a). This measures the final deliquoring where the liquid film coating the solid particles surfaces reduces in thickness over time. t_d is defined as:

$$t_d = \frac{\rho d_h^2 G t}{\mu h} \tag{22a}$$

where d_h is the mean hydraulic diameter of the solids making up the cake which is proportional to the square root of cake permeability K (inversely related to the specific cake resistance). Specifically,

$$d_h = \frac{2}{3}\left(\frac{\varepsilon}{1-\varepsilon}\right) d = 7.2\frac{(1-\varepsilon)K^{1/2}}{\varepsilon^{3/2}} \tag{22b}$$

where d is the mean particle diameter and ε the cake void volume fraction. Equation (22a) states that t_d is proportional to Gt/h and inversely related to the kinematic viscosity μ/ρ. A lower operating temperature often leads to higher kinematic viscosity, which can reduce the deliquoring rate.

Example 3 A suspension containing polyvinyl chloride solids with median diameter (approximated as mean diameter in this example) of 122 μm and cake void fraction as determined by bucket measurement to be 0.53. Based on equation (22b), d_h = 91.92 μm. The kinematic viscosity for the suspension is 0.01 $cm^2\ s^{-1}$. The bucket test for various G = 700 g, 1500 g, and 2500 g; t = 15, 30, 60, 120, 240, and 480 s; and h = 1.27, 2.54, 3.81 cm are tabulated in Table 8.2. t_d is further calculated in Table 8.2 using equation (22a) with d_h = 91.92 μm, μ/ρ = 0.01 $cm^2\ s^{-1}$, and the appropriate G/t, t and h values. In addition, the void fraction for each individual test is tabulated in Table 8.2, with the mean value given by 0.53.

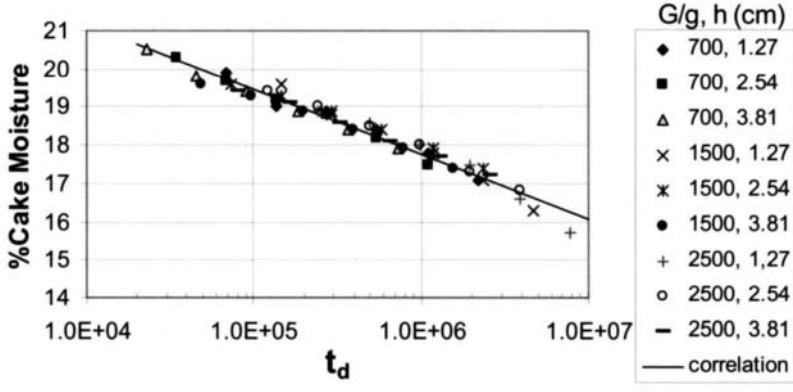

Figure 8.16a % Cake moisture as a function of t_d for bucket test results tabulated in Table 8.2

Table 8.2 Bucket test on a suspension of PVC particles

Test run	*G/g*	*t*, s	*h*, cm	t_d	*% moisture*	*Void fraction*
1	700	15	1.27	6.855E+04	19.91	0.55
2	700	30	1.27	1.371E+05	19	0.535
3	700	60	1.27	2.742E+05	18.9	0.528
4	700	120	1.27	5.484E+05	18.4	0.523
5	700	240	1.27	1.097E+06	17.8	0.531
6	700	480	1.27	2.194E+06	17.1	0.535
7	700	15	2.54	3.427E+04	20.3	0.54
8	700	30	2.54	6.855E+04	19.7	0.532
9	700	60	2.54	1.371E+05	19.2	0.538
10	700	120	2.54	2.742E+05	18.8	0.531
11	700	240	2.54	5.484E+05	18.2	0.533
12	700	480	2.54	1.097E+06	17.5	0.531
13	700	15	3.81	2.285E+04	20.5	0.539
14	700	30	3.81	4.570E+04	19.8	0.534
15	700	60	3.81	9.140E+04	19.4	0.531
16	700	120	3.81	1.828E+05	18.9	0.537
17	700	240	3.81	3.656E+05	18.4	0.537
18	700	480	3.81	7.312E+05	17.9	0.528
19	1500	15	1.27	1.469E+05	19.6	0.543
20	1500	30	1.27	2.938E+05	18.8	0.532
21	1500	60	1.27	5.876E+05	18.4	0.533
22	1500	120	1.27	1.175E+06	17.8	0.525
23	1500	240	1.27	2.350E+06	17.1	0.523
24	1500	480	1.27	4.701E+06	16.3	0.525
25	1500	15	2.54	7.345E+04	19.6	0.536
26	1500	30	2.54	1.469E+05	19.2	0.53
27	1500	60	2.54	2.938E+05	18.8	0.524
28	1500	120	2.54	5.876E+05	18.2	0.514
29	1500	240	2.54	1.175E+06	17.7	0.523
30	1500	480	2.54	2.350E+06	17	0.523
31	1500	15	3.81	4.896E+04	19.6	0.526
32	1500	30	3.81	9.793E+04	19.3	0.524
33	1500	60	3.81	1.959E+05	18.9	0.523
34	1500	120	3.81	3.917E+05	18.4	0.533
35	1500	240	3.81	7.834E+05	17.9	0.522
36	1500	480	3.81	1.567E+06	17.4	0.52
37	2500	15	1.27	2.448E+05	18.9	0.539
38	2500	30	1.27	4.896E+05	18.6	0.528
39	2500	60	1.27	9.793E+05	18	0.533
40	2500	120	1.27	1.959E+06	17.5	0.526
41	2500	240	1.27	3.917E+06	16.6	0.521
42	2500	480	1.27	7.834E+06	15.7	0.535
43	2500	15	2.54	1.224E+05	19.4	0.531
44	2500	30	2.54	2.448E+05	19	0.516
45	2500	60	2.54	4.896E+05	18.5	0.506
46	2500	120	2.54	9.793E+05	18	0.526
47	2500	240	2.54	1.959E+06	17.3	0.514
48	2500	480	2.54	3.917E+06	16.8	0.525
49	2500	15	3.81	8.161E+04	19.4	0.523
50	2500	30	3.81	1.632E+05	19.1	0.515
51	2500	60	3.81	3.264E+05	18.6	0.533
52	2500	120	3.81	6.529E+05	18.1	0.534
53	2500	240	3.81	1.306E+06	17.7	0.536
54	2500	480	3.81	2.611E+06	17.2	0.469

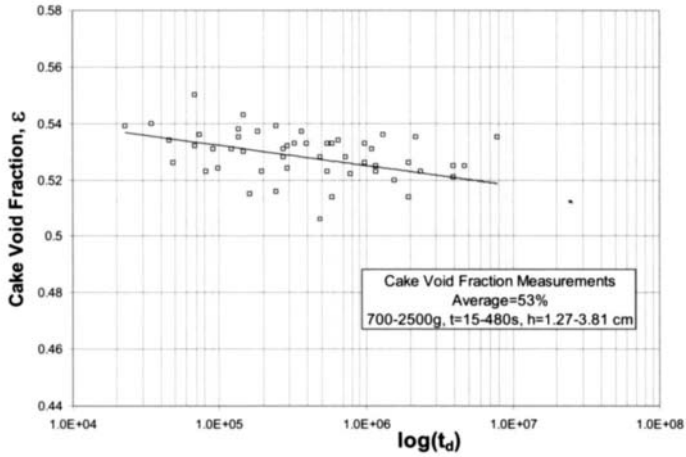

Figure 8.16b Cake void fraction as function of t_d

8.4.3.2 Deliquoring by compaction and expression

The bucket is capped to prevent drainage at the large diameter end. Thus, liquid is expressed out of the cake countercurrent to consolidation of the cake. Cake is formed and compacted for a given G and time, after which the liquid above the cake is decanted off. The cake can be removed from the bucket intact and split in to segments in which cake solid is determined from each segment. When the specific gravity of solids is known, the solids volume fraction can be determined from which the stress for a given layer is determined. The compaction pressure is determined from (Leung, 1998a):

$$\rho_s(R) = (\rho_s - \rho_L) \int_{R_c}^{R} \Omega^2 R \varepsilon_s(R) dR \tag{23a}$$

$$p_{si}(R) \approx (\rho_s - \rho_L) G_b \sum_{R=R_c}^{R=R_i} \left(1 - \frac{h_i}{R_b}\right) \varepsilon_{si} \Delta R_i \tag{23b}$$

R_c is the radius at cake surface and ε_s is the solids volume fraction of the cake. The solid stress is given by equation (23a) and can be determined approximately by the finite sum in equation (23b). After a concentrated feed is charged to the bucket with a blind wall at the large diameter end, the assembly is rotated at a given angular speed Ω and for a time duration t. After rotation has stopped, the expressed liquid is decanted from the open end of the bucket. The capped bottom is removed and a plunger is used to push out the cake intact. The cake is then dissected into small thickness ΔR_i, not necessary in equal

thickness, and each is treated as a sample for determining the total solids by weight from which the solids volume fraction ε_{si} can be inferred knowing the solid density. This is illustrated in Figure 8.17a. Note, liquid is expressed radially inward countercurrent to cake compaction. At "large" time t (i.e. equilibrium condition) all liquid in the cake should have expressed out under an imposed solid pressure p_s. While at short duration t, the cake is still subject to the slow transient associated with expression and compaction, in consequence the cake sample is wetter when compared to its equilibrium condition. The exact amount has to be determined from testing.

Example 4 Cake solids compaction As an example, the cake solids versus compaction pressure for a cake with fine particles with 2 micron median size is depicted in Figure 8.17b. The analysis of the test data is shown respectively in Table 8.3a for a thinner cake of height 2.54 cm dissected into three equal thicknesses for various time t = 60–1200 s, and Table 8.3b for a thicker cake of 4.0 cm dissected into four equal

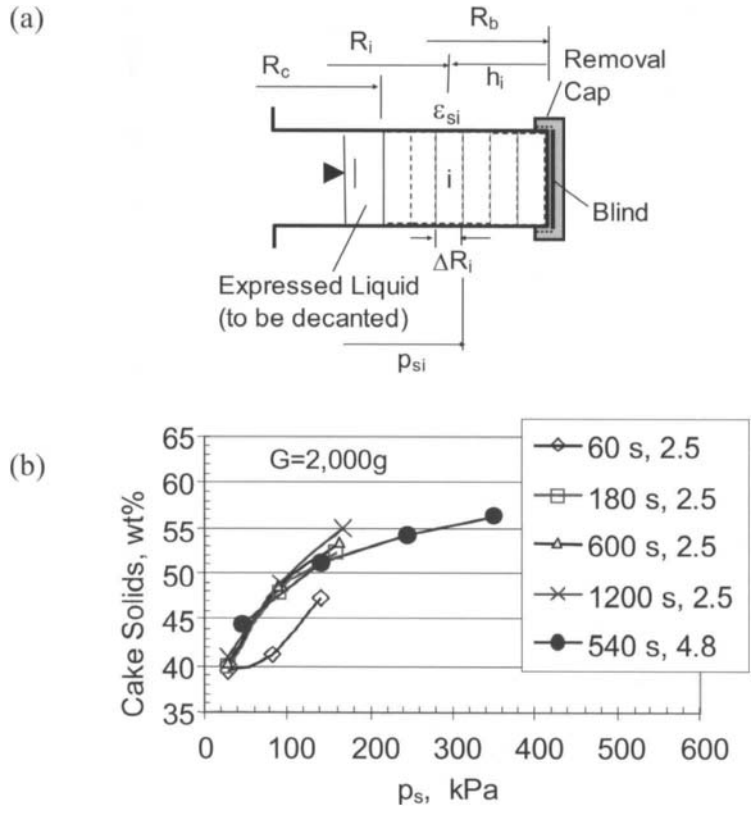

Figure 8.17 (a) Schematic of a bucket with blind bowl wall at the large diameter end. (b) Cake solids of fine particles (2 micron median size) as function of compaction pressure

Table 8.3a Calculation of cake solids and compaction pressure (cake dissected into 3 equal segments), h = 2.54 cm, G = 2000 g.

	% by Weight			*% by Volume*			Δp_{si}, *kPa*			p_{si}, *kPa*		
t, s	*bottom*	*middle*	*top*	*bottom*	*middle*	*top*	*bottom*	*middle*	*top*	*bottom*	*middle*	*top*
60	47.3	41.2	39.4	24.9	20.5	19.3	63.4	54.8	53.8	140	81	27
180	52.3	48.1	39.9	28.8	25.5	19.7	73.4	67.9	54.7	159	89	27
600	53.3	48.6	40.3	29.6	25.9	19.9	75.5	69.0	55.4	162	90	28
1200	55.0	49.0	41.0	31.1	26.2	20.4	79.2	69.8	56.7	166	92	28

Table 8.3b Calculation of cake solids and compaction pressure (cake dissected into 4 equal segments), h = 4.0 cm, G = 2000 g.

	% by Weight				*% by Volume*				Δp_{si}, *kPa*				p_{si}, *kPa*			
t, s	*bottom*	*middle 2*	*middle 1*	*top*	*bottom*	*middle 2*	*middle 1*	*top*	*bottom*	*middle 2*	*middle 1*	*top*	*bottom*	*middle 2*	*middle 1*	*top*
540	56.4	54.3	51.2	44.6	32.3	30.5	27.9	22.9	104	105	103	90	349.2	244.8	140.9	44.8

thicknesses for t = 540 s. The density of the solid is 2700 kg m^{-3} and the median particle size is 2 microns. It is clear that as $t > 180$ s, the compaction and expression reach equilibrium and further time elapse under centrifugal force has no further impact on deliquoring and compaction. Figure 8.17b shows a very interesting fact that during deliquoring of a compactible cake at low p_s the cake solids (% by weight) increases proportionally with increasing p_s, however at higher compaction pressure the resistance to compaction and expression increases dramatically such that further increases of p_s result in only diminishing to no increase in cake solids. This is atypical with a compactible cake. Note, the compaction pressure increases approximately as the product G and cake height h, and more accurately according to equations (23a) or (23b). Therefore, cake solids increase also with higher G and thicker cake. This is important in deliquoring paste-like cakes formed from fine particle slurries (Leung, 2002c) and high-solids dewatering of environmental sludges (Leung, 1992, 1998a, 2001).

8.4.3.3 Cake washing

If the bucket centrifuge is equipped with a stationary feed pipe, wash liquid can be introduced after the pool of liquid recedes from the cake surface. A test can be carried out to determine the optimal time to introduce wash liquid for a fixed charge/feed to the basket. A fixed amount of wash liquid is introduced at different elapsed times from the original spin-up. Subsequently the cake is spun dry for a fixed dewatering period. Upon completion the contaminant concentration in the cake is determined. When the wash liquid is introduced too early, the liquid would mix with the mother liquor resulting in dilution and poor washing. On the other hand, when the wash liquid is introduced too late, cake pores are drained with air filling the empty pores. Under this circumstance air blockage in the cake prevents wash liquid further penetrating into the pores. This also leads to poor washing despite the high wash ratio. The optimal time elapsed corresponds to the lowest contaminant level whereby the liquid pool barely recedes below the cake surface. As a result, there is a minimum contaminant in the cake corresponding to the optimal wash time. This is depicted in Figure 8.18a.

A different amount of wash liquid is introduced at this optimal time for the same cake thickness. A wash can be defined as the volume of wash liquid to the cake pore volume. For an ideal displacement wash, a wash ratio of unity would remove all cake impurities. (It is also known that in some applications cake washing is primarily by diffusion in which the time and wash liquid requirements both escalate.) Figure 8.18b shows the contaminant concentration as a function of wash ratio. The

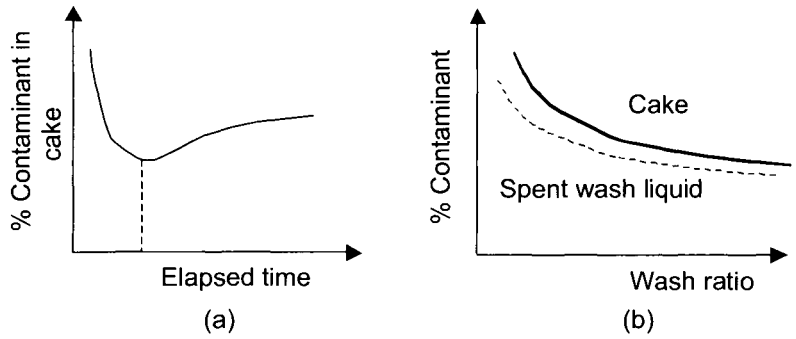

Figure 8.18 (a) Elapsed time, and (b) washing as function of wash ratio

top curve represents the contaminant level of the cake while the lower curve represents that of the spent wash liquid after passing through the cake. As the wash ratio increases contaminant decreases. A typical wash ratio between 2 and 5 is required to reduce contaminants to an acceptable level.

Cake washing has been practised in some decanters to remove salt and contaminants in chemical applications. It is not a very effective wash as the cake is not uniform in the conical beach of a decanter. Washing can only be applied as the cake emerges from the liquid pool so that the wet cake can drain in the dry beach prior to discharge.

8.4.4 Pilot and production test

8.4.4.1 Decanter centrifuges

Separation: A test can be conducted using a small decanter to confirm feasibility of separation obtained from the bench spin tube tests (such as shown in Figure 8.11). The objectives of the tests are to confirm the separation of solids in suspension to specified solids recovery, deliquoring of the cake in the conical beach of the decanter to specified cake dryness, and demonstrate conveyance of the cake including torque measurements. The last item may present a challenge as the test run is short due to limited feed samples. In addition, the tests most likely provide useful data for scale-up of separation, deliquoring and conveyance torque, which might not be available from bench tests.

The following input parameters, where applicable, should be included and monitored in a test programme:

(a) Volumetric feed rate, Q

(b) Feed solids concentration, W_f

(c) Pool depth, h_p

(d) Centrifugal acceleration, G or rotation speed, Ω

(e) Differential speed, $\Delta\Omega$

(f) Chemical (coagulant and/or flocculant) screening and dosing especially for hard-to-separate wastewater.

In the test, each parameter should be tested over a range covering the nominal value of the parameter that is being specified. The feed rate should be recorded using a properly calibrated flow meter. Feed solids concentration may be changed with dilution or concentration. Pool depth can be changed using different size weir plates, a skimmer, or by regulating back-pressure in the case of a centripetal pump being used to remove the centrate liquid. The rotation speed and thus G should be changed where possible to confirm the effect of G-force on separation and deliquoring. It would be ideal if the test machine were equipped with variable frequency drive, otherwise the driver or driven sheave needs to be changed. All tests should be run below the recommended maximum speed of the centrifuge as stipulated by the manufacturer of the equipment. The differential speed can be varied in the tests if there is a hydraulic or electric conveyor drive, otherwise it involves changing the gear ratio in the gearbox, which is rather inconvenient. For wastewater treatment (industrial or municipal) polymer addition often is used to enhance separation. Proper screening tests should be carried out first in the laboratory and subsequently in the field prior to introducing to the centrifuge. With flocculant and polymer, it is desirable to test at different injection locations – inside the feed compartment with separate polymer introduction, several metres upstream of the feed, or tens of metres upstream of the feed. This varies the time of mixing between the feed suspension and the polymer solution prior to introducing to the separation pool of the centrifuge because some polymers require longer mixing time than others to be effective to form a stable flocculated solid (floc). Also the optimal concentration of the polymer needs to be determined as most polymers function best with proper dilution. In essence, the ideal mixing time between the polymer and the feed slurry as well as the consistency of polymer to be used are determined by trial and error.

In the test programme outlined above, the solids concentration (% suspended solids by weight) respectively of the feed W_f, centrate stream W_e, and cake W_s should all be measured for each test condition. Test data should be sampled repeatedly at a later time to get statistically meaningful data as well as confirming that steady state is being

reached. In addition to recording the input parameters, feed suspension temperature, torque (T_{NL} under no-load without feeding and total torque $T_{NL}+T_L$ for a given solids throughput), amperage, vibration, and bearing temperatures should all be recorded. For classification of suspension, the particle size distribution should be measured for the feed and the centrate product using the particle size analyser available or the standard instrument used by the specific process industry (for example, in kaolin processing a sedimenting particle size analyser is the standard). As is well known, a particle size analyser provides only relative measurement of particle size distribution.

The test data on solids concentration can be used to calculate the solids recovery R_s in the cake via equation (19). On the other hand, solids recovery in the centrate during classification can be obtained from $R_e = 1 - R_s$. For each test, the appropriate Le and r_p as defined in equations (12) and (13) should be determined based on the test condition.

For separation and clarification, the solids recovery (suspended solids) in the cake R_s can be plotted against Le with r_p as a parameter. The result can be compared with the prediction based on equation (5a).

In classification, the solids recovery in the centrate product R_e should be plotted against Le with r_p being a parameter. This can be compared with the theoretical prediction as given by equation (6). Also the % cumulative undersize for a given particle size x of interest (such as 1 micron and 2 microns for kaolin classification) in the product, denoted as $F_e(x)$, tracked by the particle size counter should be plotted against the Le of the test condition to generate a trend. This trend can be compared with the theoretical prediction, equation (7). The size recovery is related to the size distribution of the product F_e and the feed F_f as well as to the total solids recovery in the product R_e.

$$SR = R_e \frac{F_e(x)}{F_f(x)} \tag{24}$$

The test deduced SR can be plotted against Le of the test and can be compared with the theoretical prediction as given by equation (8).

Other than plotting using the scale-up variable Le, it is also prudent to plot the solids recovery (in the cake or centrate), and cake solids (or cake moisture) as a function of feed solids throughput, which is a vital metric for production. The solids throughput is given by:

$$m_{fs} = \rho_f Q W_f \tag{25a}$$

In equation (25a), ρ_f is the feed density, Q the volumetric rate of the feed, W_f the % by weight of suspended solids in the feed. Also,

$$m_{cs} = m_{fs} R_s \tag{25b}$$

and R_s is the solids recovery as determined by equation (19) based on the test data on solids concentration of all three streams in and out of the centrifuge.

The differential speed should not affect separation. However, too low a differential speed may result in inadequate transport of sediment leading to transient build-up of sediment interfering with the high velocity centrate. Likewise equal damaging results from disturbance by secondary flow adjacent to conveyor flights rotating at very high differential speed with respect to the bowl.

Example 5 Comparing decanter and spin tube tests A test was conducted using the 150 mm decanter centrifuge on the same FGD sample as tested using the spin tube presented earlier in Example 1. The vital data are compiled in Table 8.4. Based on the feed, centrate and cake solids concentration from equation (19) the solids recovery is determined to be 96.9%. The *Le* number for a continuous feed centrifuge is determined from equation (12). For the present problem, *Le* is calculated as follows:

$$L_e = \frac{\sqrt{Q/L}\sqrt{\mu/\Delta\rho}}{\Omega R_p x_0 \eta_a} = \frac{\sqrt{[(4.7\times1000/60)/20.32][0.025/(2.3-1.02)]}}{\left(5152\times\frac{2\pi}{60}\right)\times6.1214\times(1\times10^{-4})\times0.8} = 1.04$$

Table 8.4 Test data for the 150 mm diameter x 300 mm long small pilot decanter.

ρ_s, g/cm^3	2.3	Q, L/m	4.7	W_f, %	3.51
ρ_L, g/cm^3	1.04	h_p, cm	1.50	W_s, %	55.67
Δ_ρ, g/cm^3	1.26	R_p, cm	6.12	% moisture	44.33
μ', g/(cm s)	0.025	$\Delta\Omega$, rpm	51.5	W_e, %	0.114
R_b, cm	7.62	Ω, rpm	5152	R_s, %	96.95
L_{cy}, cm	20.32	G/g	2261.5	η_a	80%

The data *Le* = 1.04 and R_s = 96.9% are included in Figure 8.11 for comparison. This single data compares extremely well with both the spin-tube data as well as with the theoretical prediction (solid curve in Figure 8.11).

The spin tube data can provide a preliminary scale-up on sedimentation of a suspension. The model spells out explicitly the separation ability being related to the product of Gt, PSD of the feed, viscosity and suspension height. Despite this, it should be cautioned that there are effects that are present in spin tubes which are absent in decanter centrifuge and vice versa; also fluid flow and cake transport mechanisms are both absent and cannot be simulated using a spin tube.

Example 6 Scale-up between two similar decanters As discussed for scale-up with decanters, the Le number should be identical between the test machine and another size (presumably production sized machine). Table 8.5 shows an example of such. Other than the Le number, the pool ratio should also be comparable.

Table 8.5 Scale-up for two decanters of same design - 760 mm x 3050 mm (30 x 120) and 150 mm x 300 mm (6 x 12).

Diameter x Length	30×120	6×12
pool depth, mm	91.44	17.78
overall length, mm	3048	304.8
clarifier length, mm	2327.7	204.0
μ, 5 cp	0.05	0.05
$\Delta\rho/\rho$	0.5	0.5
Q, m^3/h	31.82	0.45
rpm	2500	5000
η_o	85%	85%
R_p, mm	289.56	58.42
Le	3.0	3.0
R_p/R_b	0.76	0.77

Deliquoring: Cake solids should be monitored as a function of volumetric feed rate (or dry solid throughput), G, differential speed, and pool depth. These effects can be graphed individually, which are schematically shown in Figures 8.19a–d.

For cake made up of granular solids deliquoring is by drainage, especially by reducing the film of liquid coated on the surface of the solid. The dimensionless parameter t_d governs the deliquoring characteristics. Figure 8.19f shows a schematic of a cake with a repose angle filling both adjacent flights of the helical channel in the dry beach section (outside the pool of liquid). The average cake height h as defined in Figure 8.19f is independent of the cake repose angle and it is related to the cake throughput m_{cs}, differential speed $\Delta\Omega$, screw pitch

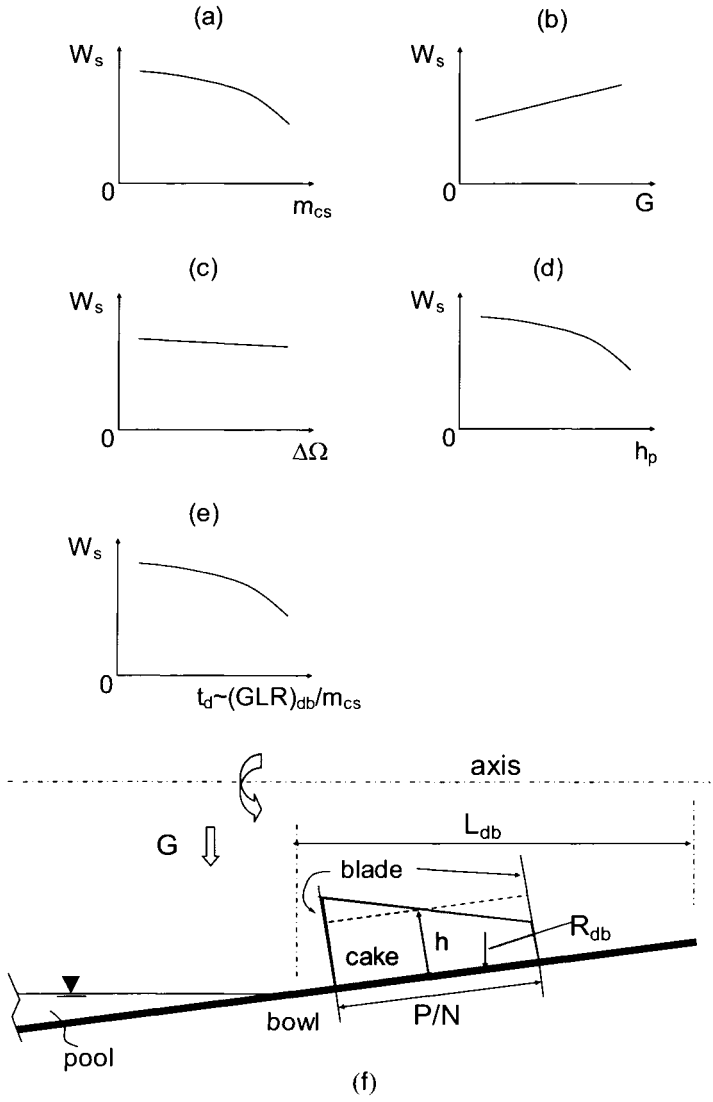

Figure 8.19 (a) Cake solids concentration plotted against cake solids throughout m_{cs} (dry basis). (b) Cake solids concentration plotted against G. (c) Cake solids concentration plotted against differential speed. (d) Cake solids concentration plotted against pool depth h_p. (e) Cake solids concentration plotted against t_d which is a combination of some of the variables. (f) Schematic of deliquoring in the dry beach of a decanter centrifuge

P, mean radius of the dry beach R_{db}, cake density ρ_c, and cake solid concentration W_s:

$$h = \frac{m_{cs}}{\Delta\Omega R_{db} P \rho_c W_s} \tag{26a}$$

Also shown in Figure 8.19e, the width of the helix channel is the pitch divided by the number of pitches N. For thin blade thickness, the number of pitches does not enter into equation (26a). (However for increasing number of pitches to, say, greater than four with small pitch P or for thick blade, N will come in play.)

The retention time t in the dry beach is

$$t = \frac{2\pi L_{db}}{\Delta\Omega P} \tag{26b}$$

L_{db} is the axial length of the dry beach and is affected by the pool level. Thus, combining equations (26a) and (26b), t_d from equation (22a) becomes:

$$t_d = \frac{\rho d_h^2 G t}{\mu h} = \frac{\rho_c W_s d_h^2}{(\mu/\rho)} \frac{2\pi R_{dh} L_{dh}}{m_{cs}} G_{dh} \tag{26c}$$

This translates to a deliquoring parameter t_d that is proportional to the unwrapped peripheral area of the dry beach $2\pi(RL)_{db}$, the average G of the dry beach G_{db} and inversely proportional to the cake solid throughput m_{cs} which is proportional to the feed solid throughput m_{fs}. It can be stated that the cake solid or cake moisture is a function of t_d, such as determined by the bucket test as shown for example in Figure 8.16a. Decreasing pool depth (leading to longer dry beach L_{db}), increasing G (increasing G_{db}), decreasing feed rate (decreasing m_{cs}) all result in larger t_d and hence higher cake solid and lower solid moisture. This is illustrated in Figure 8.19e. Thus for scale-up with the fluid and solid properties all identical:

$$\left(\frac{R_{db} L_{db} G_{db}}{m_{cs}}\right)_1 = \left(\frac{R_{db} L_{db} G_{db}}{m_{cs}}\right)_2 \tag{26d}$$

Remarkably, t_d does not depend on the differential speed as it affects both cake height and retention time to the same extent and the two effects cancel out. Also, the pitch and number of pitches do not enter into the analysis.

By way of illustration, Table 8.6 shows two different deliquoring decanters with similar geometry but different sizes, operating G and solid throughput. The solids recovery in the cake is comparable, thus the ratio of the cake solid rate between the two machines $(m_{cs})_1/(m_{cs})_2$

is as the feed solid rate between the two machines $(m_{fs})_1/(m_{fs})_2$. The geometry required by the scale-up law, equation (26d), is summarized for the two machines in Table 8.6. If machine 1 (small machine of 300 mm diameter) has a feed solid throughput of 6 t h^{-1} (dry basis), the larger 600 mm diameter machine can process 35 t h^{-1} (dry basis) yielding the same cake solids as the smaller machine.

Table 8.6 Comparing two deliquoring decanters.

	300 mm diameter	*600 mm diameter*
R_b, mm	30.48	60.96
h_{sp}, mm	6.35	12.7
R_{sp}, mm	24.13	48.26
h_p, mm	3.81	5.08
R_p, mm	26.67	55.88
R_{db}, mm	25.4	52.07
L_{db}, mm	14.4	43.2
G/g	1300	1200
G_{db}/g	1083	1025
$(RLG)_{db}$	396,379	2,306,469
$(RLG)_2/(RLG)_1$	1	5.82
m_{fs}, tph	6	35

Note: Same R_s, for both machines

Conveyance Torque Analysis: The spline torque data should be plotted against the cake solids throughput m_{cs} as given by equation (25b) similar to Figure 8.6b. Equation (17) with an assumed C_f is used to match the test data after which the best matched C_f can be used to compare with other test data, to predict torque for a different sized machine, or to predict performance under different operating conditions for the same process application. An example is shown in Table 8.7, demonstrating how C_f is determined from the single measured torque data T_L = 57,500 N m after the no-load torque has been subtracted from the total conveyance torque. Also, it is best to use equation (17) to generate a comparison of predicted torque trend with a larger amount of torque data under different test conditions, from which the best fitted prediction with the matched C_f can be determined. C_f is then used for further comparison with other test data or for scale-up for similar design machine using equation (17).

Example 7

Table 8.7 Machine and process data for torque calculation.

Machine Data:				
D_b = 762 mm	L = 3048 mm	h_{sp} = 101.6 mm	R_{sp} = 279.4 mm	
R_b = 381 mm				
Operating Data:				
Ω = 2,654 rpm	$\Delta\Omega$ = 20 rpm	G/g = 3,000	h_p = 76.2 mm	R_p = 304.8 mm
Process Data:				
W_f = 16%	W_e = 1%	W_s = 50%	R_s = 95.66%	
ρ_f = 1,112 kg/m^3	ρ_c = 1,780 kg/m^3	ρ_L = 1,000 kg/m^3	$\Delta\rho/\rho_c$ = 0.438	
m_{fs} = 5.26 t/h	m_{cs} = 5.03 t/h			
T_L = 57,500 N	C_f = 3.5	$C_f(\Delta\rho/\rho_c)$ = 1.53		

Example 8 High-solids scale-up Two decanters of different diameters, 457 mm and 914 mm, each with a length-to-diameter ratio of 3.0 were tested on the same environmental sludge operating in high solids mode in a wastewater treatment plant (Leung, 1992). The torque data T_L, $\Delta\Omega$, G, and m_{cs} are measured or deduced based on other raw data as described. The "buoyant" frictional coefficient $(\Delta\rho/\rho_c)C_f$ is calculated in accordance with equation (18b). The results are as shown in Figure 8.20. As can be seen, the effective buoyant frictional coefficient is relatively constant and independent of the magnitude of cake solids achieved and also independent of the machine sizes.

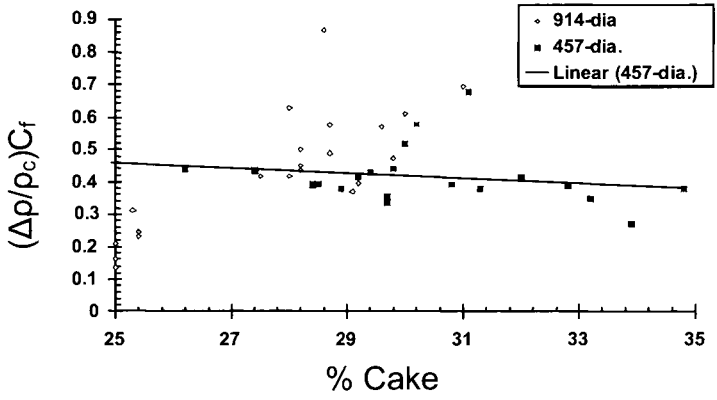

Figure 8.20 Effective frictional coefficient determined for two different size high-solids decanters

This index was found to depend on the type of sludge being dewatered, and it may increase for certain bio-solid sludges (Leung, 1998b).

8.4.4.2 Tubular centrifuges

In testing a tubular centrifuge, the important variables are feed rate, rotational speed, and possibly pool depth depending on the design. The solids concentration of the feed and centrate should be measured. The average cake is measured for the entire test as the cake is stored temporarily in the bowl not to be discharged until the centrate turns turbid and feeding stops.

The testing of the tubular centrifuge follows the same procedure as a decanter especially for separation, clarification, and classification. There is no conveyor and differential speed between conveyor and bowl.

8.4.4.3 Disk centrifuges

The test variables for a disk centrifuge are feed rate, rotational speed, discharge adjustment (changing time elapse for cake discharge or changing total nozzle area for continuous flowable cake discharge).

It is important that there should be continuous smooth solids removal for both nozzle and dropping bottom disk centrifuges, as with decanters.

For a dropping bottom centrifuge, cake solids should be discharged when the solids holding space in the centrifuge bowl fills up, this determines the time at which the bottom should drop to discharge cake solids. Thus

$$t = \frac{V_s \varepsilon_s}{Q \phi_s} \tag{27}$$

t is the time duration when the bowl bottom opens for cake discharge, V_s is the solids holding volume, ε_s is the solids volume fraction of the cake, Q the volumetric feed rate, and ϕ_s the solids volume fraction of the feed. While equation (27) provides a preliminary estimate of the discharge time, during testing or operation, a turbidity meter in-line with the centrate enables fine-tuning based on the turbidity reading to trigger the opening of the bowl sleeve or piston for cake discharge.

For nozzle discharge disk centrifuges, the total area of the nozzle should be selected to remove the incoming solids on a continuous basis, thus balancing the solids in the feed and the cake solid to be discharged. The total nozzle area is calculated to be

$$A_T = \frac{\phi_s}{\varepsilon_s} \frac{Q}{\Omega D_n} \tag{28}$$

During operation, depending on the turbidity and cake solids consistency, one can adjust the total nozzle areas (i.e. number of nozzles and or diameter of the nozzles) for cake discharge fine tuning the value as suggested by equation (28).

8.5 Integration of equipment in flow sheet – application examples and design considerations

Centrifugal separation has been commonly practised in many key separation steps, some of which include separation, clarification, classification, degritting, dewatering, and purification. Literally thousands of sedimenting and filtering centrifuges are employed around the world on important applications. In the past decade, significant advances have been made on new technologies and know-how to improve process separation and to get higher grade product(s) with improved throughput and lower energy consumption. In this section, some applications and new technologies of centrifuges are discussed.

Decanter, disk, and tubular centrifuges are used in many separation processes. The process objectives for separation, classification, degritting, concentrating, cake washing, deliquoring etc. are discussed. This applies to mineral processing including coal, potash, soda ash, tar sand, kaolin, calcium carbonate, drill mud, yellow cake, bauxite, and barium sulfate, etc; chemical processing; food and biopharmaceutical, industrial, water and wastewater treatment. A few of these applications are discussed herein for illustration purpose. Additional examples can be found elsewhere (Leung, 1998a).

8.5.1 Separation of polyvinyl chloride

Decanters have been commonly used for deliquoring (specifically dewatering) of PVC suspensions. Figure 8.21 shows a schematic of the flow sheet. Liquid vinyl chloride monomer is added with emulsifier, suspension agent, and catalyst in a polymerization reactor. After polymerization, the product is sent to the stripping tank where contaminants (unreacted monomers) are removed or stripped from the rest of the suspension containing reacted products. The purified product is directed to a holding tank and subsequently sent to decanter

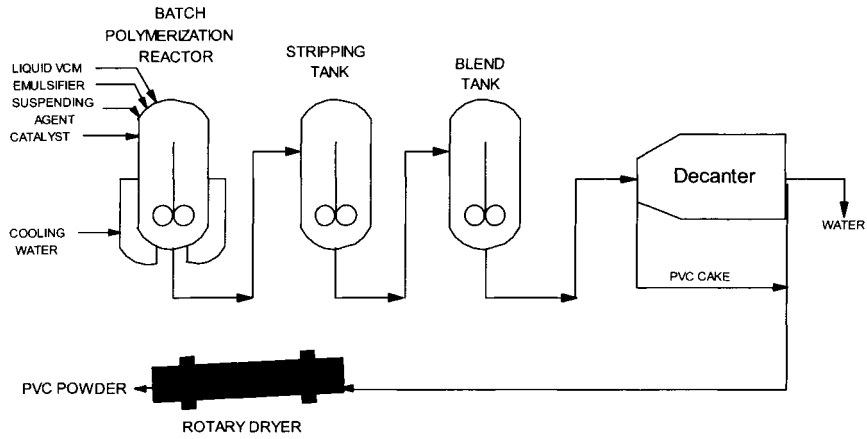

Figure 8.21 Dewatering of PVC suspension

centrifuges for dewatering. The resultant cake moisture depends on the dewatering parameters G, time, cake height in addition to the PVC intrinsic properties, i.e. resin, porosity in the solids/ polymer, and the surface morphology of the particles. The cake moisture is typically between 17 and 25% by weight for most common pipe grade resins. It is important to get the moisture within specification as the cake is fed to a dryer downstream. Higher cake moisture reduces the capacity as the dryers in most plants do not have excess capacity and more importantly wet cake can adhere to duct surfaces which cause more serious clogging problems. Different grades of pipe grade resin have their nominal operating temperature which varies between 60 and 90°C, it is recommended that the PVC slurry be dewatered at the maximum possible temperature (temperatures higher than the maximum temperature cause decolouring and fusing of the resin) to reduce the viscosity magnitude which affects dewatering through the t_d parameter.

Bucket tests can be carried out to determine the behaviour of G, t, and cake height on the moisture. However, there are additional dynamics at high feed solids rate to a decanter which leads to cake filling up the channel formed between adjacent helix blades from which moisture cannot be drained readily leading to a drastic increase in cake moisture (Leung, 1998a). Industrial centrifuges are often operated near this limit. Another limit on high solids throughput is the stick-and-slip phenomena with dewatering PVC which leads to chatter torque. Either chatter or high cake moisture can limit the throughput. The smallest resin of PVC is typically about 60 microns (i.e. relatively large cut size) and as such there is no problem for solids to settle under reasonable centrifugal gravity in a decanter. The centrate liquid leaving

the decanter contains minimal suspended solids measured at less than 100 ppm (0.1%), and in some instances below 50 ppm (0.05%).

8.5.2 Classification of kaolin slurries

Solid-bowl decanters have been commonly used to classify kaolin slurries (Leung, 1998a, 2002a, 2003; Leung *et al*, 1999). A generic flow sheet for kaolin classification using two-stage centrifuges is shown in Figure 8.22. The first and second stages can be decanters while the second stage can also be a disk stack centrifuge with higher G and more separation surface area. For feed containing a high fraction of less than 2 microns (in excess of 90%) only one stage centrifuge (decanter or disk) is used.

The solids concentration of kaolin slurry in the classification plant ranges between 30% by weight (normal feed) and 50% by weight (high

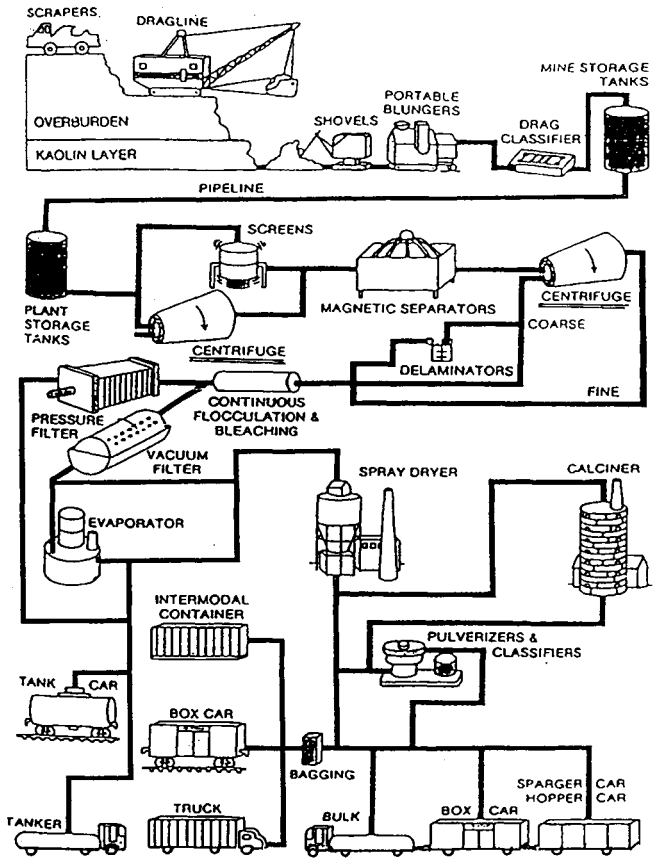

Figure 8.22 Generic two-stage centrifugal classification of kaolin processing

density feed). For example, the feed to the centrifuge may contain 70%–80% less than 2 μm and 50%–60% less than 1 μm. After classification by centrifuge, the centrate (product) may have 90–99% less than 2 μm, and 80–90% less than 1 μm. It is essential to maintain a high total solids recovery of all sizes in the overflow between 60–70+%. It is further desirable to maintain a high cumulative size recovery of less than 2 μm particles above 85%. The narrow particle size distribution together with complete removal of heavy metals in the centrifuge centrate assures the brightness of the product (for high-quality brighter paper) to be within specification. Obviously, a finer size cut can be readily achieved when the feed grade is finer as compared with a coarser feed grade.

Various decanter diameters have been used including 600 mm, 750 mm, 900 mm, and 1000 mm. Some modern decanters have an aspect ratio, length-to-diameter between 3 and 4.4. The *G*-force varies between 1000*g*–2000+*g* depending on the process requirements. Kaolin slurry is highly abrasive and components in the rotor of the centrifuge exposed to the suspension are well protected. To produce finer quality product, high *G*-force is required and it is advantageous to use improved feed accelerator design so that the feed attains the tangential speed of the pool as it is laid on the pool surface to effect classification and avoid mismatch in velocity causing disturbance at the pool (Leung and Shapiro, 1996a, b, 2001; Leung *et al*, 1999). This reduces turbulence, skidding, wear, and undesirable re-suspension of the sediment. Figure 8.23 shows that the 1 μm product obtained from the decanter with an improved feed accelerator system is much greater compared with a conventional feed accelerator (Leung and Shapiro, 1996a, b, 2001). Another way to interpret this is that the fine particles in suspension are fully accelerated with the improved feed acceleration system and

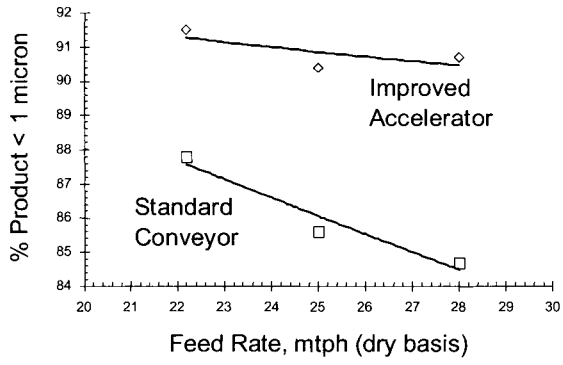

Figure 8.23 Comparing %<1 μm in product between improved feed accelerator and conventional accelerator

coarser particles (greater than 1 μm) settle out leaving only the 1 μm particles in suspension as the product. With a conventional feed accelerator, the slurry is not accelerated until after it skids in the separation pool, resulting in poor separation and a much lower percentage of the 1 μm fine particles mixed with the oversized particles. As a result, for the same capacity there is lower 1 μm fraction with the standard machine or much lower capacity is processed with the standard machine for the same separation product. As such the acceleration efficiency η_a in equation (12) needs to be borne in mind when scaling-up a given decanter for the process.

The rheology of the rejected cake is non-Newtonian and it generally exhibits shear-thinning behaviour. It may be difficult to convey fluid-like cake that flows back to the conical beach toward the pool especially under high *G*-force. A cake weir can be installed in the conical beach whereby it allows a differential hydrostatic head across the weir, with the liquid level higher in the clarifier and lower in the conical beach. This differential head facilitates cake transport in addition to the transport provided by the differential speed of the conveyor. It is critical to provide the proper driving hydrostatic head. Too large a hydrostatic head results in "wash-out" of the cake, i.e., pool spilling at the conical beach, while too little undermines the cake transport resulting in intermittent cake discharge. An adjustable cake baffle is preferred (Leung *et al*, 1999), wherein the "resistance" or gap through which the cake has to be transported across is adjustable thus compensating for the hydrostatic liquid head and cake rheology, see Figure 8.24. The solids throughput can increase by 20+% on a production-size 1000 mm decanter in kaolin processing.

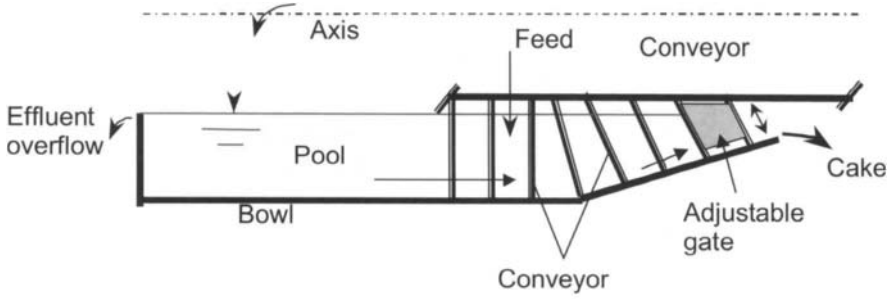

Figure 8.24 Schematic of decanter with adjustable gate/cake-baffle/cake-resistance

Nozzle decanters are employed to classify kaolin slurry (Leung, 2002a). A schematic of a nozzle decanter is shown in Figure 8.25. At

least two or more diametrically opposed nozzles (for balancing purpose) are located near the conical beach-cylinder junction in the machine for discharging the underflow instead of conveying cake up the conical beach. The feed and the centrate product both flow concurrently, and given that the centrate product overflows at the discharge diameter of the conical beach, this design takes advantage of the full length of the machine for classification of fine particles.

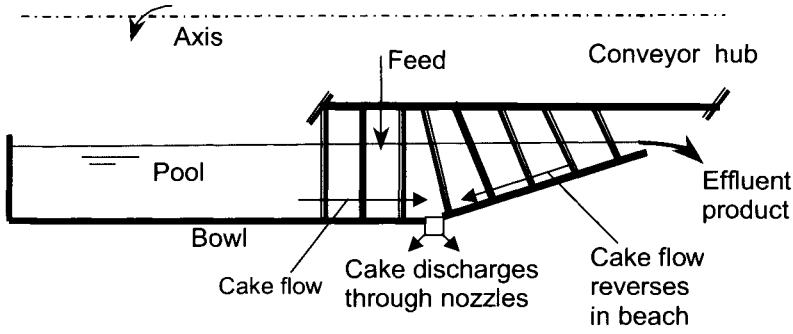

Figure 8.25 Schematic of a nozzle decanter

Figures 8.26a, b, c and d show an example of classification results from a nozzle centrifuge with 630 mm diameter. The feed particle size distribution (PSD) is given in Figure 8.26a. As shown, 73% is less than 2 microns. The size distribution of the centrate product and total solids recovery measurements in the centrate are given, respectively, by Figures 8.26b and 8.26c, are in good agreement with the theoretical prediction of the improved surface-flow model as summarized by equations (4)–(8) and (12) (Leung, 2004b). The centrifuge shown in Figure 8.26d further shows a reasonable comparison of the measurements with the theoretical "S" shaped curve for cumulative size recovery corresponding to less than 2 micron particles.

Figure 8.26b shows an important point that for a given particle size of interest, say less than 2 microns, very large *Le* (say $Le>10$, theoretically infinite *Le*) from reducing *G* or the speed and/or increasing feed rate yields no separation and the size distribution corresponds to that of the feed at 73%. On the other hand, when *Le* reduces to 1.18 or less, the product centrate contains 100% of the less than 2 micron particles. The centrifuge can be designed and scaled to operate between $1.18<Le<10$ to obtain the targeted $100\%>F_e(2\ \mu m)>73\%$. Also in this same range of *Le,* as is evident from Figure 8.26c, the total solids recovery R_e takes the range, $58\%<R_e<97\%$. In practice, *Le* should be selected to meet the centrate specification recovery R_e and the required

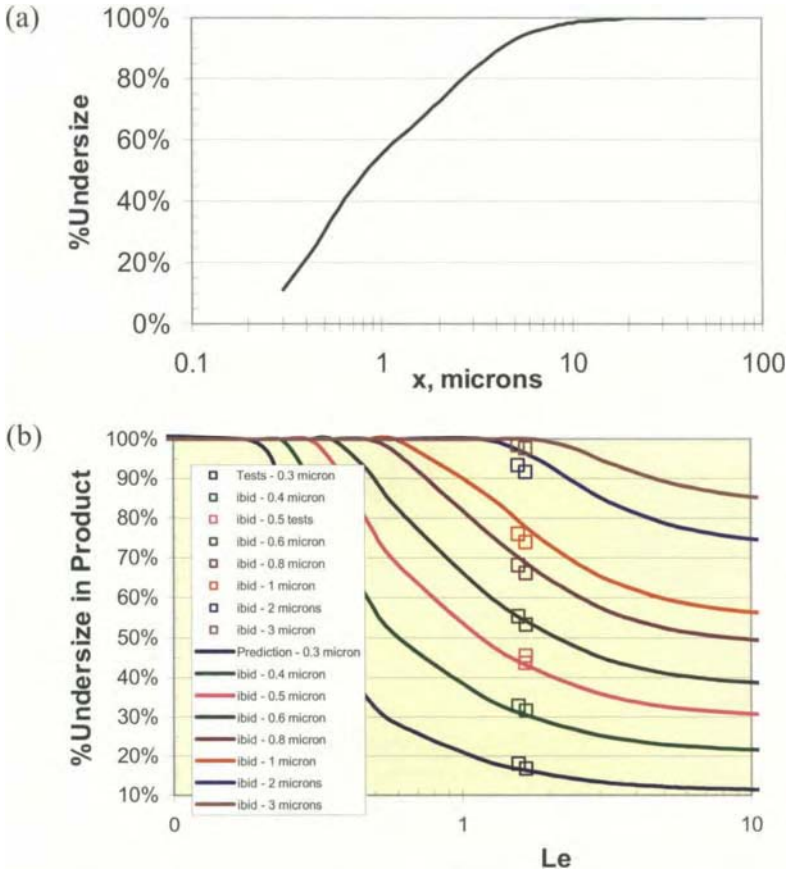

Figure 8.26 (a) Particle size distribution of feed to a centrifuge in plant A. (b) Comparing the PSD of kaolin product (□) with improved surface-slow model prediction (-) expressed as function of *Le* for a 630 mm diameter in Plant A.

percent on less than 2 micron particles, $F_e(2\ \mu m)$. The *Le* approach provides both scale-up as well as fine tuning (including performance prediction) and optimization to meet the required operating target.

Figures 8.27a–d shows results from a much smaller nozzle decanter of 270 mm diameter operating in plant B. The test conditions are that G = 600–1800*g*, feed volumetric rate Q was 1.3–2.1 $m^3\ h^{-1}$, and solids rate (dry basis) was 0.6–0.9 $t\ h^{-1}$. The rates are obviously much less compared with the production unit in the above. All predictions are based on equations (4)–(8) and (12) using the improved model (Leung, 2004b). The feed solids are such that 70% is less than 2 μm. Figure 8.27b shows a comparison between prediction and measurement for the PSD of the fine product which is quite remarkable. Figure 8.27c shows prediction between total solids recovered in the product with test results wherein the two are in reasonable accord with each other.

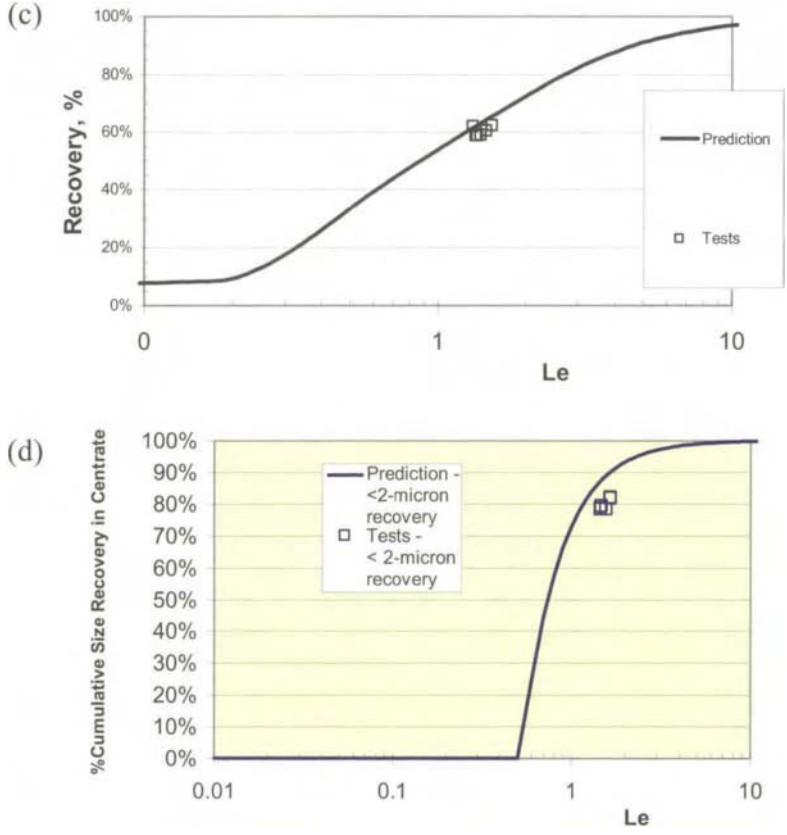

Figure 8.26 (c) Total solids recovery of kaolin as a function of *Le* for two production machines, each 630 mm diameter decanters in Plant A. (d) Cumulative size recovery of –2 μm kaolin product for two 630 mm diameter decanter in Plant A.

Finally, Figure 8.27d compares the "S-shaped" size recovery curve between theory and prediction with good results.

It can be seen from the two comparisons of the two different size centrifuges operating under different conditions, that the theoretical *Le* approach is capable of predicting performance independent of the size (production and much smaller pilot unit) and operating conditions (i.e. feed rate, *G*, feed slurry properties, etc.).

Another comparison between prediction using the improved model and production measurement was made on a conventional countercurrent design in which cake and clarified liquid travel opposite to each other (see Figure 8.4). Cake or reject is conveyed by the screw conveyor over the small diameter discharge end of the conical bowl. A 1000 mm decanter is installed in kaolin plant C. The particle size distribution

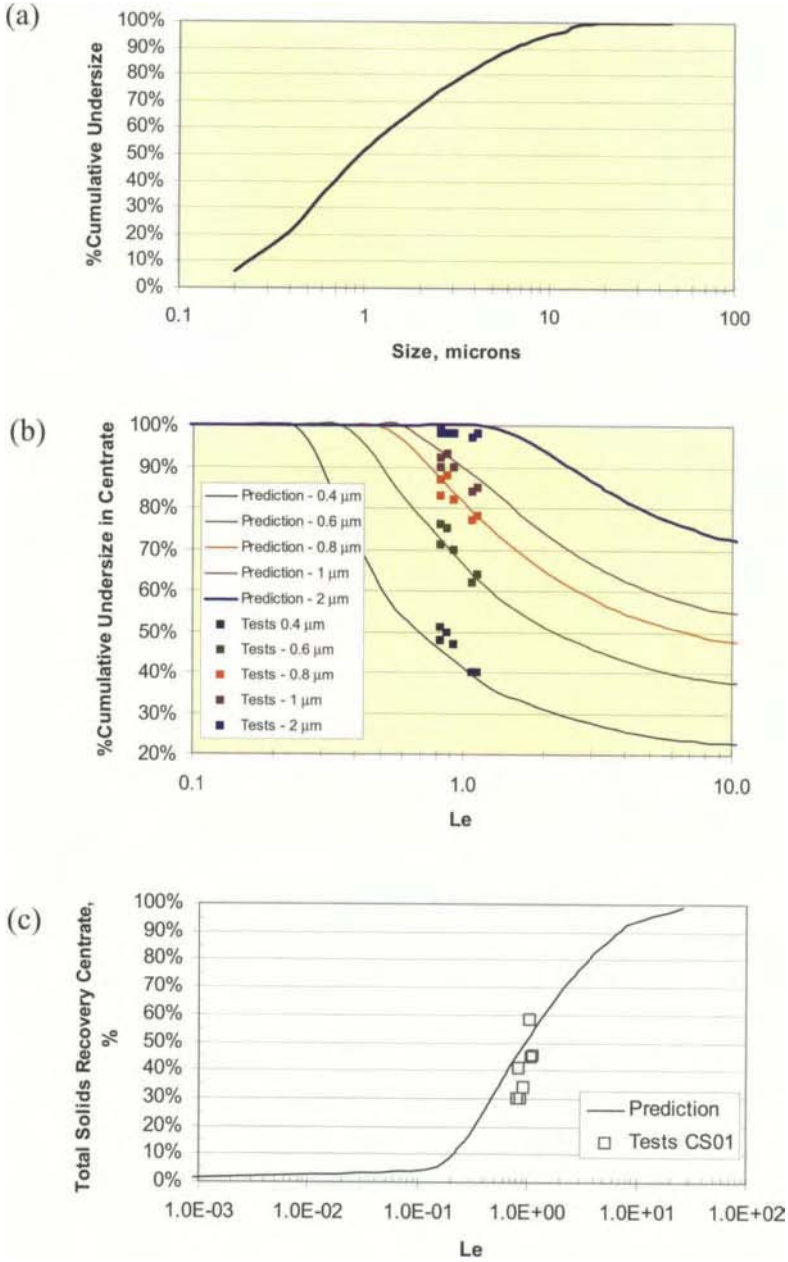

Figure 8.27 (a) PSD of feed to pilot unit in plant B. (b) PSD of centrate product. (c) Total solids recovery R_e in the centrate product.

(PSD) of the centrate product (% undersize cumulative, i.e. F_e) is plotted against Le in Figure 8.28. The agreement on a given particle size, between 0.2–10 µm, in the centrate product for a wide range of Le compares excellently with the theoretical prediction.

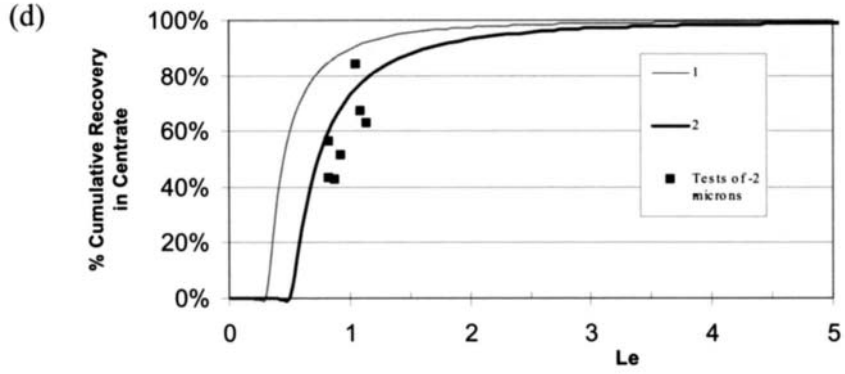

Figure 8.27 (d) Size recovery (SR) prediction of 1 and 2 μm particles and comparison with measurements for 2 μm

In some installations, nozzle disk centrifuges are used for high G (3000g+) classification. It is typically used as the second of the two-step classification process using higher G-force and more surface area to achieve the fine cut size fraction. The feed solid concentration and solid loading to the centrifuge are both somewhat limited.

8.5.3 Separation in calcium carbonate plants

There are several applications for centrifuges in processing calcium-carbonate slurries. For processing ground calcium carbonate (GCC) slurries, centrifuges are used in conjunction with the ball mill to produce finer particles with median size 5–10 μm with no oversize particles greater than 45 μm. High solids recovery of the fine particles less than 45 μm in the centrate is desirable, as the cake which may contain the finer particles is re-circulated back to the mill.

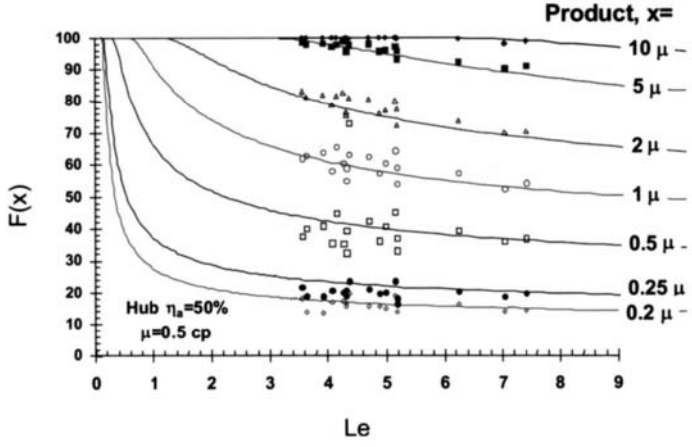

Figure 8.28 Comparing PSD of kaolin product with prediction for a 1000 mm diameter decanter centrifuge in Plant C

The second processing step is to dewater the fine-particle slurry with median size 5–8 μm to high cake dryness in excess of 70% w/w and high solids capture is essential. The cake is reslurried in dispersant prior to sending to a downstream mill to produce finer particles. At this point, the objective of the centrifuge in cooperation with the mill is to remove or degrit oversized particles in excess of 25 μm, or any foreign particles such as grinding media from the mill, from dense, high-viscosity, suspension containing 70+% solids w/w. A generic flow sheet is shown in Figure 8.29.

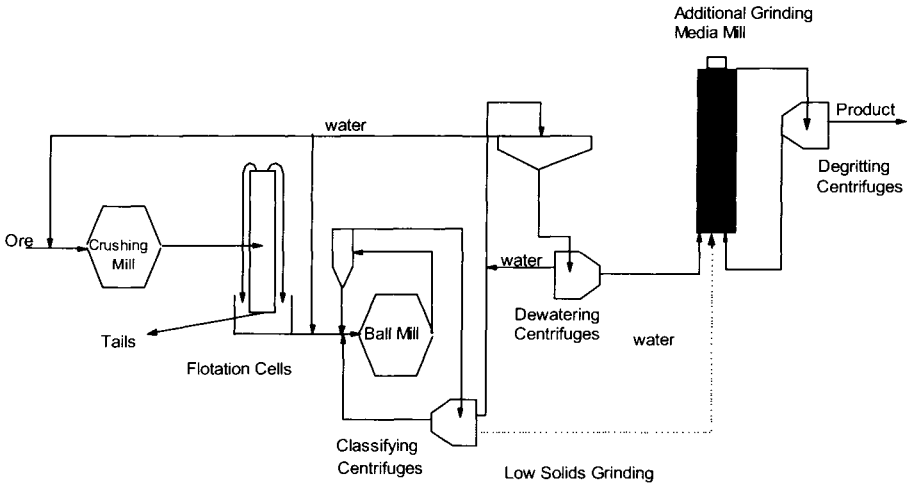

Figure 8.29 Generic flow sheet for ground calcium carbonate processing

For the precipitated calcium carbonate (PCC) process, after the slurry leaves the reactor it is sent to the centrifuge for dewatering. Particles in suspension are typical approximately mono-dispersed with mean size between 1–3 μm. Increasing feed rate beyond a critical level might result in a large drop in the solids recovery due to the feed PSD (Leung, 1998a). The cake is mixed with dispersant and sent to a second-stage centrifuge for high-density degritting similar to that of GCC.

Some common size centrifuges deployed for use in the calcium carbonate separation processes are 450 mm, 750 mm, 1100 mm and 1100 mm diameter. Dewatering of the slurry is usually limited by the solids recovery and at other times the cake dryness may be the limiting factor. In some applications, backdrive is used to provide good control on differential speed for process and machine cleaning as discussed.

The *G*-force ranges between 100–1200*g* for different classification steps to 1000–3000*g* for dewatering depending on the process

(viscosity, PSD) and the size of the machine. The feed rate also depends on the same factors.

A new compound-beach design has been developed to dewater fine-particle slurry such as PCC and GCC where cake solids are 5%–10% above and beyond that can be obtained from conventional design (Leung and Shapiro, 2002). This new high G design, as illustrated in Figure 8.30, has a compound beach profile with a steep conical beach followed by a zero-degree cylindrical beach. An adjustable gate is located near the exit of the machine to meter the cake to be discharged. Across the entire cake layer only the driest cake layer adjacent to the bowl wall which is subject to the maximum compaction solid pressure p_s (see Figure 8.17b) is skimmed for discharge while the wet cake near the cake surface is recycled back upstream. This increases the solids retention time in the machine. With the compound beach geometry, increasing solids capacity does not compromise cake dryness and vice versa as with a conventional decanter.

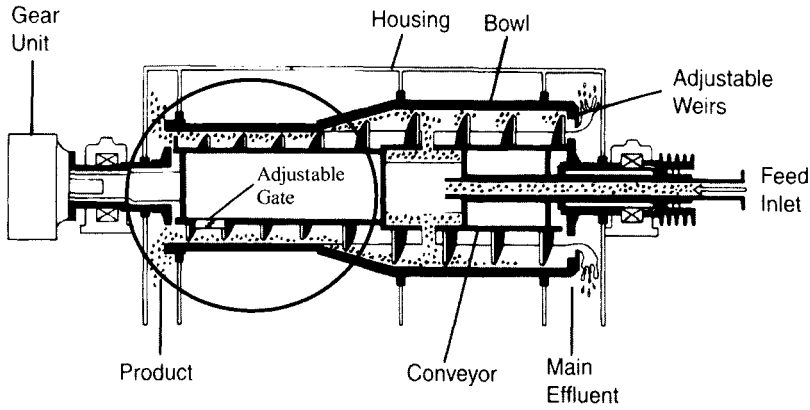

Figure 8.30 Compound-beach decanter and adjustable cake baffle for fine-particle dewatering

8.5.4 Separation of yeast cells after fermentation

A disk stack centrifuge is in operation in a bioprocessing plant. It is intended to remove the yeast cells downstream of the fermenter. The yeast cells have sizes ranging from 1–5 microns with a small density difference between solid and liquid of 0.1 g cm^{-3}. The viscosity of the suspension is 5 cp. The disk stack is equipped with a variable frequency drive such that it can tune the rotation speed to operate either at 6000g or 12000g. Based on the bowl diameter of 400 mm, the equivalent Ω for the two G's are 5139 rpm and 7268 rpm, respectively. The disk inner and outer diameters are 177 and 300 mm, respectively

and there are 100 conical disks in the centrifuge. The angle between the conical disk and the vertical is 60°. Using equations (9a–c) and the cut size as given by equation (4), the performance is scaled up in accordance to the separated cells and the results are shown in Figure 8.31. Assuming the task is to remove 1-micron biological cells, at 6000*g* the feed rate to the centrifuge is 8.3 l min^{-1} whereas at 12000*g* the feed rate doubled to 16.67 l min^{-1}. If the removal on the minimum cell size is relaxed to 2.5 microns, the rate for both speeds would have increased drastically to 83.3 l min^{-1} at 12000*g* and 42 l min^{-1} at 6000*g*. The stringent requirement of removing fine particles when there is a small density difference between solid and liquid phase is evident.

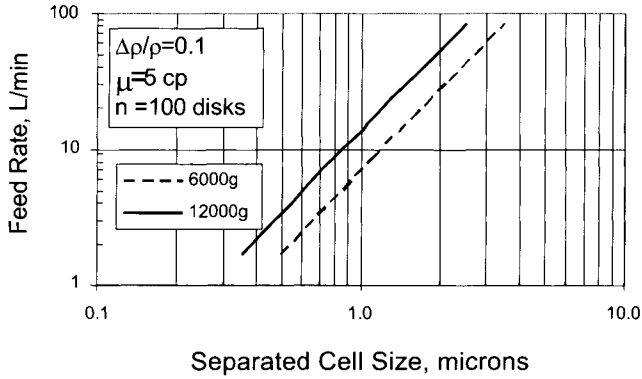

Figure 8.31 Feed rate to a 400 mm diameter disk stack centrifuge in Plant D

Table 8.8 summarizes the separated size (i.e. cut size), which is directly related to the centrifuge parameter *Le*, and the various common applications (Leung, 2002b).

Table 8.8 Separated size in various applications (Leung, 2001)

x, μm	*Le*	*Typical Applications*
0.1 0.5	0.06 0.3	High-G classification of valued ultra fines, finer biosolids, and colloids
1 2 5 10	0.6 1.2 3 6	Classification in biosolids, coatings, pigments (e.g. kaolin, calcium carbonate, silica, mica, etc.) and drill mud with high-speed centrifuges
25	15	Classification/degritting of oversize particles above 25 μm
45	27	Classification/degritting of oversize particles above 45 μm

8.6 Additional considerations

8.6.1 Decanters

When a decanter is scaled-up for production based on pilot test data, even when separation and deliquoring scale-up as well as torque scale-up are all done properly, there are other factors which can further limit throughput that might not have been considered. Some of these may be known while others may not be known until production. This applies to chemical and industrial application and more so to mineral processing as well as to wastewater treatment. These factors are mostly related to feed properties such as feed solids concentration, feed particle size distribution especially the fine fraction that can cause a headache in both separation (including thickening, classification and degritting) as well as deliquoring. For separation and classification in mineral processing, the feed size for an upstream centrifuge classifier can change depending on the seam/pile/lot which is being processed at a given time. The particle size distribution can drift from say $x_{50\%} = 60$ microns to $x_{50\%} = 10$ microns. As discussed, the particle size distribution affects separation and a centrifuge being scaled-up for the latter would have to reduce throughput significantly when processing a much finer throughput. On the other hand, if the specification is such that the feed $x_{50\%}$ is given the range of 10–60 microns upfront and when scale-up is done based on the worst case of $x_{50\%} = 10$ microns the throughput to each centrifuge is significantly reduced, resulting in many large centrifuges processing low throughputs. This expensive design might be rejected due to cost, in favour of other cheaper technical alternatives. In mineral processing some feed may contain an oily coating on the particles which render them more difficult to separate which is not foreseen in equipment testing and scale-up. When processing a very coarse fraction, high torque can reduce throughput to a lower level than anticipated. For dewatering in wastewater treatment plant, the organic content in the feed to a dewatering centrifuge can fluctuate widely daily, if not hourly, changing the cake consistency and thus the throughput of the centrifuge above and beyond the level of scale-up. There is lesser variation in chemical application involving upstream reactor processes; however change in feed stock, chemical additives, and fluctuation in reactor temperature which affect viscosity can affect the performance of the downstream deliquoring centrifuge.

Some cakes that have density difference close to the liquid may be extremely sensitive to the pool level adjustment (Leung, 1998a). If the pool height is too deep, the cake may be too wet while if the pool height is too shallow, the cake may not convey causing intermittent discharge

and periodic "torque spikes". Feed rate and G may have to be reduced to below what the scale-up level is to ensure smoother operation.

Also high vibration of the centrifuge due to (a) imbalance in cake distribution at high throughput (especially for dewatering wastewater sludge), or (b) other mechanical components not working properly may lead to operating at lower (as compared to scale-up values) throughput and/or G.

8.6.2 Disk and tubular

Some of the problems discussed for the decanter in the previous section are also found in the disk stack and tubular centrifuges. Underflow/sediment/cake discharge for the disk stack using the dropping bottom or nozzles can also affect scale-up throughput. Optimisation is needed for the production machine, as the throughput obtained from scale-up may need to be curtailed. One concern for a disk centrifuge is that the feed distribution to the disk stack is non-uniform; often this non-uniformity boils down to reasonably determining the efficiency factor in equation (9a).

Tubular centrifuges operate in a batch or discontinuous mode, the downtime for solids discharge (manual or with automatic knife) and cleaning (including clean-in-place and steam-in-place for bio-processing) should be factored into the overall cycle as it affects the equivalent overall throughput.

8.7 Conclusions

There has been a significant advancement in the understanding in scale-up of sedimenting centrifuges through research in the past years. The subject is difficult due to the complicated dynamics, especially of fluid flow (single and two-phase flow) in a rotating system which is often dictated by non-intuitive phenomena. Also there has been a continuous build-up of practical experiences in various applications which complement the scientific approach to scale-up of the equipment.

Nomenclature

C_f	frictional coefficient
D	bowl diameter, m

d	diameter, m
F	cumulative undersize distribution, %
f	frequency distribution, %
G	centrifugal acceleration, m s^{-2}
g	acceleration due to earth gravity (9.81 m s^{-2})
H	suspension height, m
h	height, m
I	integral in equation (5c), m^2
K	cake permeability, m^2
L	length, m
Le	Leung number in centrifugal separation
m	dry solid throughput, kg s^{-1}
N	number of pitches in a screw conveyor
P	pitch, m
SR	size recovery of a give particle size in centrate, %
R	radius, m
r	gear ratio
r_p	pool radius to bowl radius ratio
T	torque, N m
t	time, s
V	volume, m^3
W	weight fraction, %
x	particle size, micron
β	beach angle, deg or rad
η	efficiency, %
μ	viscosity of liquid, kg m^{-1} s^{-1} or as otherwise defined
μ'	effective viscosity, kg m^{-1} s^{-1} or as otherwise defined
ϕ_s	solid volume fraction in feed
ε	cake void fraction
ε_s	cake solid volume fraction

λ	hindered settling factor
ρ	density, kg m^{-3}
$\Delta\rho$	density difference, kg m^{-3}
θ	angle between disk and vertical, deg or rad
$\Delta\Omega$	differential speed, rev/min (rpm)
Ω	rotational speed, rev/m (rpm)

Subscripts:

a	accelerator
b, bowl	bowl
c	cake
cs	cake solid
d	dimensionless
e	centrate
f	feed
fs	feed solid
h	hydraulic
i	index
k	index
L	load
NL	no–load
n	nozzle
0	reference
p	pool, pinion
s	sediment, cake, solid
sp	spillover location of decanter (deepest pool)
1	inner radius of disk stack
2	outer radius of disk stack

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