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Food Product Shelf Life

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Why Study Shelf Life?

The modern food industry has developed and expanded because of its ability to deliver a wide variety of high quality food products to consumers on a nationwide and worldwide basis. This feat has been accomplished by building stability into the products through processing, packaging, and additives that enable foods to remain fresh and wholesome throughout the distribution process. Consumer demands for convenience have fueled new innovations in the food product development, packaging and chemical industries, and the widespread desire for products to use in the microwave oven has added further impetus to this effort.

As an increasing number of new foods vie for space on supermarket shelves, the words “speed and innovation” have become the watchwords for food companies seeking to become “first to market” with successful products. That all important market share which goes to the pioneer of each successful new product keeps that company in an excellent competitive position.

Total quality is of paramount importance to this competitive posture and needs to be built into the speed and innovation system. How the consumer perceives the product is the ultimate measure of total quality. Therefore, the quality built in during the development and production process must last through the distribution and consumption stages or all is for naught.

Shelf-life studies can provide important information to product developers enabling them to ensure that the consumer will see a high quality product for a significant period of time after production. Of course long shelf-life studies do not fit with the speed requirement and therefore accelerated studies have been developed as part of innovation.

As the mechanisms of food deterioration became known to food scientists, methods of counteracting these losses in quality have been developed. The rate at which these reactions occur, the effects of temperature, water, and the myriad of other parameters have become characterized factors contributing to the science of accelerated shelf-life studies.

Processed Food Deterioration

The principal mechanisms involved in the deterioration of processed foods are as follows:

1. Microbiological spoilage sometimes accompanied by pathogen development.

2. Chemical and enzymatic activity causing lipid breakdown, color, odor, flavor, and texture changes.

3. Moisture and/or other vapor migration producing changes in texture, water activity and flavor.

Formulation and processing variables which affect these mechanisms and which can be used to control deterioration include: (1) moisture and water activity;

(2) pH; (3) heat treatments; (4) emulsifier systems; (5) preservatives and additives; and (6) packaging.

The Importance of Moisture and Water Activity

Water has been called the universal solvent because it is a requirement for growth and metabolism of microbes, and the support of many chemical reactions which occur in food products. Water occurs in food systems in both the free and bound states. Free water is just that, free for chemical reactions, to support microbiological growth, and to act as a transporting medium for compounds. In the bound state, water is not available to participate in these reactions as it is tied up by water-soluble compounds such as sugar, salt, gums, etc., (osmotic binding) and by the surface effects of the substrate (matrix binding). These water-binding effects reduce the vapor pressure of the water over the food substrate according to Raoult's Law. By comparing this vapor pressure with that of pure water at the same temperature, one establishes a ratio which is called water activity (a_w) which by definition is 1 for pure water. A one molal solution of sugar has a a_w of 0.98, and one molal sodium chloride has a a_w of 0.9669. A saturated sodium chloride solution has a a_w of 0.755. Thus the NaC1 solution in a closed container will develop an equilibrium relative humidity (ERH) in the headspace of 75.5%.
Therefore: $a_w = ERH/100$

The ERH of a food product is defined as that relative humidity of the air surrounding the food at which the product neither gains nor loses its natural moisture; that is, it is in equilibrium with its environment. At a relative humidity above the ERH, the product will gain moisture and at a humidity below that level, it will lose moisture. A food with a a_w of 0.6 will lose moisture at a relative humidity below 60% and gain moisture above 60%.

The ERH is a valuable tool for food product and packaging development as it is an indicator of what chemical reactions may occur during distribution and of how much packaging protection should be designed for the product to give it the required shelf life. Without the protection of a package, the moisture of any food changes in direct proportion to the relative humidity. The rate at which the gain or loss of moisture occurs depends on the makeup of the product, i.e., its hygroscopicity, the temperature and the atmospheric pressure. In packaged products the changes are relatively slow, but at times a significant change can occur within a short period of time. Have you ever eaten soggy potato chips at a picnic on a hot and humid day? Have you ever received moisture test results from the lab that you were convinced were incorrect? If so, perhaps the samples were not properly packaged for transit.

Equilibrium Moisture Conditions

When a food is exposed to a constant humidity, it will equilibrate at a final moisture content called its equilibrium moisture. By exposing the product to a wide range of relative humidities, one can develop what is called a micro moisture equilibrium curve. This test is accomplished by placing a known weight of the product in a metal dish inside of a glass jar containing a saturated salt solution. Various saturated salt solutions equilibrate at certain relative humidities. The sample dishes are weighed back at various times until they reach equilibrium. The moisture value is then calculated and the texture of the sample is noted for reference. Two examples are shown in Tables 1 and 2.

Breakfast Cereal

Initial Conditions2.5%

%RH	%H2O	Texture
0.0	1.54	Crisp
11.1	2.27	Crisp
22.9	3.18	Crisp
32.9	4.59	Soft
43.9	6.55	Soggy
53.5	8.27	Soggy
64.8	11.43	Rubbery
75.5	15.88	Moldy
86.5	23.69	Moldy

Table 1: The Effect of Relative Humidity on Moisture Content And Texture of Cereal

An equilibrium moisture curve is obtained when the product equilibrium moisture (Y-axis) is plotted against the RH (X-axis). If the RH is divided by 100 to

obtain the water activity, a moisture sorption isotherm (moisture content vs. water activity) is produced. From such a curve one can see that the breakfast cereal with a moisture content of 3.18% would have an a_w of about 0.2 and would, however, support mold growth at an a of 0.755. If this cereal were left out in an open package in Minnesota during the wintertime, the texture will remain crisp because the RH is normally below 25%. The flavor may change; insects may invade it; but the texture will remain crisp.

Flour

Initial Conditions 12.0%

%RH	%H2O
0.0	0.53
11.1	5.90
22.9	7.65
32.9	7.65
32.9	8.95
43.9	10.11
53.5	10.90
64.8	12.21
75.5	15.68
86.5	18.80

Table 2: The Effect of Relative Humidity on Moisture Content of Flour

Flour, on the other hand, will lose moisture at relative humidities below about 60%. This "drying out" accounts for many of the short weight problems that flour millers encounter with State Weight and Measure officials. Water and water activity are involved in most food product deterioration mechanisms. Microbial growth and spoilage, the action of enzymes, non-enzymatic browning, and texture changes all will be affected by moisture. The effects of water activity on relative reaction rates for these mechanisms of product deterioration are illustrated in Figure 1. The moisture content isotherm is positioned for a typical product and will vary, depending on the matrix as seen in Tables 1 and 2. Temperature will accelerate all of these reactions to some extent. This increase in reaction rate varies from 30% to 300% for each 10°C rise in the temperature. From the Figure 1 graph, at an a_w of 0.3, the product would be the most stable with respect to lipid oxidation, non-enzymatic browning, enzyme activity, and, of course, the various microbiological parameters.

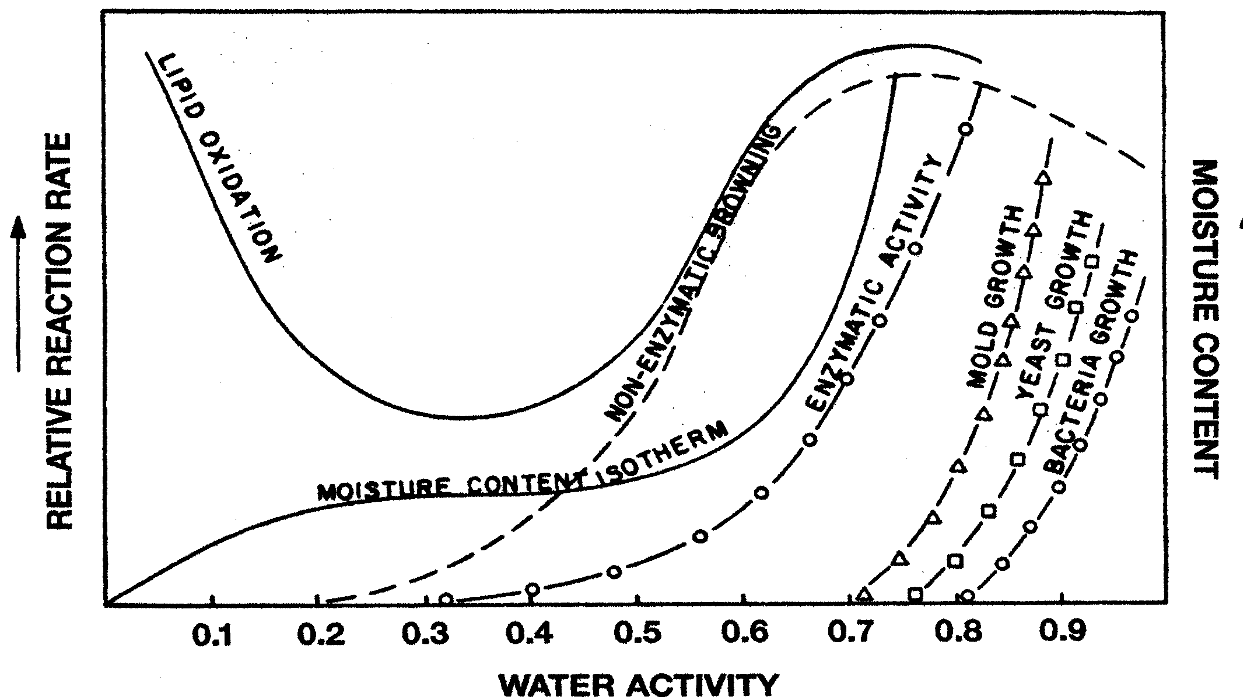

FIGURE 1: Stability map of foods as a function of water activity. Labuza, 1970

Testing Equilibrium Moisture Effects

Food products may lose quality if the moisture level changes from the equilibrium moisture in the distribution chain. The rate of moisture change can be monitored by changes in package weight which occur during storage in appropriate temperature and humidity environments. Medallion Laboratories has computerized this old technique and applied it to a wide variety of food products. For products that suffer quality losses in high humidities, the packages are stored in what is known as the "weather room". This room is kept at constant 65% RH with temperature cycling on a day/night basis between 90°F and 74°F. These conditions are used to simulate the hot, humid summers of the southeastern United States. The packages are weighed back at weekly intervals and the gain in weight, along with the original moisture determination, are then used to calculate the moisture changes. From the moisture sorption curve developed for the product and knowledge of the package material water vapor transmission rate (WVTR), a calculation can be made to determine its shelf life.

It is difficult to obtain reliable weight change data on packages at relative humidities above 75%. Most food packages contain paper or paperboard which gain and lose moisture in the same manner as food products. When stored at a relative humidity above 75%, the amount of water gained for the paper component with a small change in humidity is large and moisture condensation also becomes a problem.

The package weight method is ideal for comparing variations where only small differences are expected. This method has been able to show moisture changes due to a few pinhole leaks in impermeable pouches.

To obtain reliable results, there are some prerequisites:

1. Initial product moistures must all be the same in each set.

2. Sealed empty packages must be used as controls.

3. The heat and humidity at the storage conditions should not result in weight changes other than from moisture, i.e., no chemical changes in leavening systems.

4. The weight variation of the packages must be small with respect to net product weight. A rule of thumb is that the weight difference between the heaviest and lightest empty packages divided by the average net weight of the product must equal 0.025 or less.

If moisture gain or loss is extremely important to the packaged food product, this method may be able to help you with shelf life questions.

MICROBIOLOGY AND THE SHELF LIFE OF REFRIGERATED PRODUCTS

Shelf life of food products is most familiar to consumers through the open dating used on refrigerated goods. The shelf life of these types of products is affected, for the most part, by their microbiological status. These products pose the highest food safety risk and have the shortest shelf life because they are the most susceptible to microbiological deterioration and the possibility of the growth of pathogenic organisms. Bacteria need certain conditions for growth – namely available moisture, the proper pH, the right temperature and nutrients and time. By controlling these conditions one can prevent the growth of these organisms and extend the shelf life. Once the product has been developed utilizing a combination of the proper ingredients, pH, water activity and microbiological inhibitors, its shelf life can be determined in real time at the various temperatures, which may be encountered during storage and distribution. The growth of yeasts, mold, spoilage and pathogenic bacteria, etc., can be monitored by microbiological methods. Other noticeable reactions such as gas production, syneresis (phase separation), and changes in color or viscosity can give further indications as to what might have to be changed in the formulation or packaging to increase the shelf life. Another means of ensuring the product will have an adequate shelf life is to challenge the product with inoculi of various spoilage organisms, as well as some potentially pathogenic bacteria. This step will ensure the product will be safe for the consumer as well as aesthetically pleasing at the end of its shelf life. *Listeria monocytogenes* grows under refrigerated conditions. It is imperative that it not be present in refrigerated

products, but that it be dealt with via elimination from the plant environment to assure against its presence in the product. Microbiological shelf-life determinations can usually be accomplished in real time and are, therefore, quite accurate.

CHEMICAL DETERIORATION

Chemically based deterioration of packaged food products can be classified into three different mechanisms: (1) Oxidation of lipids; (2) Enzymatic degradation; and (3) Non-enzymatic browning. All three can occur simultaneously in food systems and are accelerated to some extent or another by increasing storage temperatures.

Lipids and Shelf Life

One of the principal methods of predicting the shelf life of processed food products is to monitor the level of lipid degradation products in foods stored at elevated temperatures. Much of the science of accelerated shelf-life estimation has involved lipid degradation reaction rates.

Fat Oxidation and Hydrolysis

Fats are composed of glycerol chemically combined as the ester of fatty acids. The fatty acids attached to the glycerol molecule can be saturated, unsaturated and polyunsaturated in any combination. Each particular fat has its own unique combination of fatty acids and, therefore, its own physical and chemical properties. Saturated fatty acids are very stable because they contain no double bonds between the carbon atoms in the chain. Unsaturated fats (fats with double bonds) are subject to a variety of reactions. Monounsaturated fatty acids have one double bond while polyunsaturates have up to six double bonds and are the least stable of the fatty acids. These double bonds are the most reactive sites on the fatty acid chain and are easily attacked by oxygen, hydrogen, and enzymes. Each of these attackers produces a different result. When hydrogen reacts with the double bond, it eliminates (saturates) the bond. This process, called hydrogenation, produces a more saturated and thus more stable fat with a higher melting point. This is an industrial process that does not occur naturally during storage.

Enzymatic Degradation Mechanisms

The lipolytic enzyme lipase reacts with triglycerides to form free fatty acids in a degradation process known as hydrolytic rancidity. Lipase enzymes cleave fatty acids from triglycerides. The fatty acids have definite detectable flavors which can render the food unacceptable. Lauric acid (C12:0) has a strong soapy flavor and is readily detectable in rancid coconut and coconut oil. As little as 0.3% lauric acid will produce this effect. This hydrolysis reaction requires either high temperature, e.g., deep fat frying with water present or lipase enzyme activity. Therefore, it is not a reaction which occurs during normal food storage unless there is lipolytic enzyme activity. With the exception of lauric acid mentioned above, low levels of free fatty acids have minimal effect on the flavor of foods. Fatty acids from domestic oils (C16:0 or higher) do not affect the flavor until they reach a level of about 2% and then they impart a bitter taste. If butter is present, the short chain fatty acid released, butyric acid, have a very strong and undesirable odor and flavor at lower levels.

The analytical determination of these free fatty acids by titration with a standard base solution (America Oil Chemists' Society, AOCS Ca 5a-40) provides a method for following the hydrolytic rancidity process in accelerated storage tests.

The free fatty acid (FFA) test is a simple acid base titration and will measure any acid present in the food and extracted with the organic solvent used. This will include acids that may have been added to the food or produced during storage by fermentation or microbiological spoilage, therefore the FFA of a fresh product needs to be known. In most situations, FFA is not a reliable measure of reactions other than hydrolysis. Some fatty acids are produced by lipid oxidation at lower temperatures, as will be discussed later, but the levels are too low to be accurately measured by this test. At higher temperatures, both oxidation and hydrolysis occur and since the FFA test does not differentiate between them, it is not a good measure of rancidity. Lipoxygenase and peroxidase enzymes react, for the most part, on the free fatty acids themselves as part of one of the modes of another fat degradation process called oxidative rancidity. In this mechanism, the enzymes catalyze oxidative reactions on unsaturated fatty acids at their double bonds starting a chain reaction process, which produces a wide variety of degradation products.

Since enzymes are very active in promoting food

product deterioration, it is important that they be destroyed during food processing. The best illustration of this phenomenon is the chemistry of oats and oat bran.

Oats differ considerably from other cereal grains in that the oat bran fraction contains a large amount of unsaturated fat and an extremely active lipid enzyme system. The oat kernel itself, at its normal moisture of 11-13%, is very stable as the fat is protected in several ways. The fat is stored in encapsulated globules which protect it from oxygen and enzyme activity. Secondly, the normal moisture of the oat is ideal as a minimum of oxidation takes place at that level. In addition, a high level of phenolic compounds are present in the oat and they act as anti-oxidants in a manner similar to BHA and BHT.

Once the oats are milled or ground without deactivating the enzymes, the fat no longer has the same degree of protection and fat hydrolysis will begin producing free fatty acids and mono and di-glycerides. These compounds do not significantly affect flavor but do affect the physical property of oats being converted to oat products and are substrates for other enzymes which do produce off-flavored compounds.

The mono and di-glycerides produced are emulsifiers and the long chain free fatty acids fit nicely into the starch helixes of oat flour. Through hydrogen bonding, a fatty acid/starch complex is formed. These inclusion compounds prevent the starch from forming a paste when mixed with water. In dilute solutions, they form a precipitate making the formation of a good oat cereal dough very difficult.

Oats also contain a powerful lipoxygenase that adds oxygen to the double bonds of unsaturated fat to form peroxides, as discussed above. The other enzyme present, peroxidase, reduces peroxides producing mono-, di-, and tri-hydroxy acids that are extremely bitter compounds. These compounds cause the bitter flavor of rancid wheat germ.

The activity of these enzymes is proportional to the amount of available water present and, therefore, to the water activity as seen in Figure 1. They are most readily destroyed by moist heat above 190°F for several minutes. Dry heat is much less effective at deactivating these enzymes in cereal systems.

In most biological systems, peroxidase requires much more heat to destroy than lipases, lipoxygenases or any of the other enzymes that may be present. It would follow then that if the peroxidase has been destroyed, the other enzymes have also been deactivated.

Cereals can be checked for proper enzyme deactivation by using a simple colorimetric procedure based on the peroxidase oxidation of a phenolic dye (2, 6-dichloroindophenol), which has been reduced to its leuco form by ascorbic acid. During the reoxidation, a blue color is formed which can be used to measure peroxidase activity. This test is an adaptation of an old U.S. Army Quartermaster Corps method used to ensure the proper blanching of vegetables.

Oxidative Rancidity Mechanisms

Hydrolytic rancidity, for all practical purposes, does not occur during low temperature storage of foods. However, the oxidative rancidity process does occur at these temperatures because it is a chemical reaction which requires much lower activation energies to initiate. Lipid oxidation and the formation of hydroperoxides occur via three different mechanisms.

1. The Free Radical Route

2. The Lipoxygenase Route
 (which has been discussed above)

3. The Photo-Oxidation Route

The free radical route is an oxidation reaction producing free radicals that lead to peroxides that in turn are converted to alcohols, aldehydes, ketones, and free fatty acids. Many of these compounds have very objectionable flavors and odors and are what is smelled and tasted in rancid fat. This reaction, called autoxidation or peroxidation, takes place at the double bonds of unsaturated fatty acids, principally oleic C18:1, linoleic C18:2, and linolenic C18:3. The oxidation reaction occurs 64 times faster with linoleic and 100 times faster with linolenic when compared to oleic acid.

The initial reaction or initiation step for the free radical route requires the presence of a catalyst, e.g., heat, light or high-energy radiation or metal ions, e.g., copper ions. This is followed by the propagation steps that are chain reactive or autocatalytic through formation of more free radicals. Free radicals are both initiators of and products of these propagation reactions. At some point during this propagation process, free radicals themselves combine to form products that eventually can terminate the reaction.

The third mechanism involves photo-oxidation and singlet oxygen in the formation of hydroperoxides from free fatty acids. Photosensitive compounds, e.g., riboflavin, chlorophyll, myoglobin, erythrosine, and heavy metal ions are excited to a high-energy state by absorbing light. This energy reacts with the normal triplet ground state of oxygen atoms to form a very reactive singlet oxygen atom. Singlet oxygen attacks double bonds 1500 times faster than the triplet state molecule.[1]

The process of oxidative reactions during storage studies can be followed analytically by determining the peroxide value in a fat or oil or by quantitating the aldehyde, n-hexanal, in food products.

Hydroperoxides

Most peroxides are odorless and tasteless *per se*, but they are further involved in the chain of reactions which produce the objectionable compounds. Oxidation produces a number of different types of peroxides, some fairly stable, others so unstable that one cannot measure them by the normal iodometric method for peroxide value (PV) (AOAC Official Method 965.33). Such highly unstable peroxides tend to be formed from compounds that contain conjugated double bonds (R-CH=CH-CH=CH-R) such as fish oil, vitamin A, etc. In normal fats and oils containing methylene interrupted double bonds (R-CH=CH-CH2-CH=CH-R), the peroxides formed are primarily hydroperoxides which are stable for weeks or even months at room temperature. When one monitors the rate of oxidation of a refined oil using the Oil Stability Index Method (OSI) (AOCS Cd12b), or the Active Oxygen Method (AOM), (AOCS Cd12) one can demonstrate that after the induction period, the PV increases quite rapidly. This induction period is a time during which there is little change in the peroxide value measurements (often too small to measure) and can last for some time with high stability oils. As oxidation proceeds, the rate of peroxide decomposition with respect to formation increases. Eventually the peroxide values will actually begin to decrease because the formation is slower than the decomposition. Some foods contain substances such as metal ions or enzymes that enhance the decomposition of the peroxides making the PV meaningless as a method of measuring rancidity. The PV test is most properly applied as a measure of the quality of fresh oils which should have a PV of one or less. Shortening with a PV of greater than 5 should not be used in the manufacture of mix products, as it will significantly decrease the shelf life of the product.

Aldehydes

Hydroperoxides break down to form a wide variety of decomposition products that are further oxidized and degraded. Compounds called aldehydes are one of the groups produced and can be used as a means of tracking the progress of fat degradation. The aldehyde n-hexanal can be determined at very low levels in foods using a steam driven headspace gas chromatographic technique.[2] This method has provided excellent results on low fat products such as cereals and cereal grains and is the method of choice for shelf-life studies designed to study oxidative rancidity.

THE KINETICS OF SHELF-LIFE TESTING

The prediction of shelf life for food products is based on the application of the principles of temperature dependent chemical reaction kinetics. These reaction rates, as Figure 1 depicts, depend on product composition as well as environmental factors, i.e., temperature, humidity, atmosphere, etc. Basic to any predictive use of reaction kinetics is that the relationship between the measurable changing reaction parameter and time be linear. Labuza[3] has reported that quality loss follows the equation

$$dQ/dt = k(Q_A)^n$$

where dQ/dt is the change in the measurable quality factor A, with time, k is the rate constant in appropriate units, and n is the order of the chemical reaction of the quality factor. The order of reaction for most quality attributes in food products is either zero, first or second. In zero order reactions, the rate of loss of the quality factor is constant or linear

and the resulting curve will be linear on a linear plot. First order reactions are not linear but are dependent on the amount of the quality factor that remains in the sample at the time. In these types of reactions, a linear prediction curve is constructed using semilogarithmic coordinates. Typical first order reactions are: (1) rancidity, (2) microbial growth and death, (3) microbial production products, (4) vitamin losses in dried foods, and (5) loss of protein quality.

The loss of vitamin C in liquid preparations has been called a second order reaction because it is dependent on the level of both the ascorbic acid and oxygen. Reciprocal plots are linear for these reactions.

A Case Study Example

A study of vitamin losses in cereal products provides a practical example of the value of shelf life studies. A cereal product fortified with vitamins A and C was studied to find out what initial levels of these vitamins would need to be added in order to ensure that the product still would meet label claims after six months at 70°F/38%RH. The product was stored at both 70°F and 100°F and vitamin assays performed at intervals over a several week period. The vitamin A degradation reaction is first order which means the rate of change in the vitamin concentration is proportional to its concentration and the vitamin analytical values should be plotted as log values. A preferred method is to convert the analytical values to percent of original level of the vitamin or to a retention ratio of original versus remaining amounts. The log of this value is then plotted versus time. This makes data comparison and table or graph production simpler. A computer spreadsheet program is used to perform this function, figure the log value (fitting the data to the kinetic model), and run regression values. From this data one calculates the number of weeks required at the given conditions it would take to lose 25% of the original levels of the vitamins and make recommendations to the developers.

The results of this and similar tests, along with certain formula and product characteristics, were put into the computer for statistical analysis. The results showed that there were three different patterns of vitamin degradation in these cereal products. The common denominator for vitamin A degradation was certain formula characteristics while for vitamin C it was water activity. In addition, it was found that the vitamin A deteriorated roughly three times faster at 100°F than it did at 70°F which means it has a Q_{10} of 2. What is Q_{10} you ask?

The Concept of Q10

One of the most frequently asked questions regarding shelf-life studies has to be: "One week at 100°F equals how many weeks at room temperature?" The answer depends on the type of product and the mode of degradation involved.

Each of the chemical deterioration reactions requires a certain amount of energy to get started. This is called activation energy, measured in kcal/mol. Table 2 contains some typical values. The higher the activation energy is for a reaction, the greater the acceleration with increases in temperature. A simple way to express this acceleration is to use the Q_{10} concept. Q_{10} is the increase in the rate of the reaction when the temperature is increased by 10 degrees centigrade (18°F). For example, if a food has a stability of 20 weeks at 20°C and 10 weeks at 30°C, then the Q_{10} will be 20/10 or 2. The rate of reaction being followed is doubled for the 10°C temperature rise. This value can be calculated from the data of most storage tests where the product has been stored at two or more temperatures regardless of whether or not they are 10°C apart. Table 3 shows some relationships between weeks at 70°F and 100°F for various Q_{10} values. These temperatures are more than 10°C apart and were, therefore, derived from equations.

Reaction	Activation Energy (Kcal/mol)
Oxidative rancidity	10-25
Enzyme reactions	10-30
Vitamin losses	20-30
Non-enzymatic browning	25-50

TABLE 3: Food Deterioration Activation Energies

Cereals, which experience enzymatic fat oxidation,

have Q_{10}'s of about 2, while low fat cereals with no enzyme activity have Q_{10}'s of about 3. Tomato powder experiences non-enzymatic browning that has a high activation energy and, therefore, has a Q_{10} of 4 or more. The word "about" as used above is important as these values can vary somewhat for the same product produced on different days or in different plants. As can be seen from Table 3, a small difference in the value of Q_{10} can make a rather large difference in the shelf-life projection. For instance, a product which lasts 10 weeks at 100°F and has a Q_{10} of 1.5 would last 20 weeks at 70°F. A difference in the Q_{10} of 0.5 would shift the shelf life from 20 weeks at 70°F to 32 weeks at that temperature, a 60% difference.

Q_{10}	1 Week @100º F= This Number of Weeks@ 70º F
1.5	2.0
2.0	3.2
2.5	4.6
3.0	6.2
4.0	10.1
5.0	14.6

TABLE 3: Storage week equivalents for various Q_{10} values

TYPICAL SHELF-LIFE STUDY DESIGN

The first step in setting up a shelf-life study is to select one of the degradation reactions which are expected to occur in the product at typical storage temperatures, can be measured, and can be used as an index of quality loss. As discussed, these could include lipid oxidation, vitamin loss, gain or loss of moisture, etc. means the more accurate the analysis, the more precise the shelf-life prediction.

Here are a few guidelines for storage studies involving fat degradation.

1. ***Products with less than 2.5% fat***:

Do not run peroxide values or free fatty acids tests as the fat extraction process necessary to obtain enough fat is too rigorous and may end up producing artificially high results. The best test for these types of products would be hexanal.

2. ***Samples with 2.5% - 10% fat***:

Do not use PV or FFA. Select a fat acidity test which utilizes a Soxhlet type of extraction followed by the base titration.

3. ***Samples with more than 10% fat***:

FFA, PV, and hexanal may yield good results if the method measures the proper deterioration product. For the PV test to be effective on food products, the sample history needs to be known.

Peroxide value and free fatty acid tests are analytical methods typically used for fats and oils. If the fat must first be extracted from food products in order to run these tests, the following statements must be true to ensure valid results.

1. The fat extracted must be representative of the fat in the food.

2. No non-fat compounds, which would interfere with the test, should be extracted with the fat.

3. No active fat compounds can be either produced or destroyed during the extraction process.

4. The solvents used must be free of any active substances.

Next, select the package that you want to protect the product in the distribution channels. This will enable you to generate data more pertinent to the product's actual shelf life. Storage temperature conditions should then be chosen which fit the product and give reliable results in a reasonable amount of time. Common temperatures used would be 20, 30, 40, and 55°C (68, 86, 104, and 131°F). At least two temperatures are required with three or four preferred for more accurate predictions. A control, stored at 0°F, can also be used.

The frequency of the analytical testing is the next important decision. The higher the storage temperature, the more frequent should be the testing. Weekly tests are common for most products unless a Q_{10} is known.

Labuza[4] has developed the following equation for testing frequency:

$$F2 = f1 \times Q_{10}^{\Delta/10}$$

where f1 is the time between tests at the higher temperature, f2 at the lower temperature, and “delta” is the difference in degrees centigrade between the two. For a product with a Q_{10} of 2, tested each week at 30°C, the frequency at 20°C would be:

$f2 = 1 \times 2^{10/10}$ or $f2 = 2$ weeks

Each testing temperature should have at least six data points over time to make the study results statistically reliable.

The results should be plotted as they are developed in order to ensure the experimental design is working properly and changes can be made in the testing frequency, if necessary.
Thoroughly document all of the data so that results can be pooled with the results of a whole product line. Lessons learned can mean the next project is much simpler to design and carry out.

Further Reading

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