QUALITY CONTROL AND SIGNIFICANCE OF TESTING

Quality Control means the control of "goodness" or the "excellence" of a product. In this ever-changing pattern of producing and making things, if "quality" of a product is not maintained then it is difficult for the product to survive. Selection of raw materials to the assessment of finished product; this entire chain of functions is effectively linked through quality control. Unless a job is thoroughly and continuously checked for quality, at the strategic stages, it is never possible to always get a good quality finished product. Quality control increases output and reduces breakdown. It seeks to ensure that the finished products conform to the specified standards of performance, utility and reliability. Hence, in any field today, quality control has become an indispensable tool of modern management.

Packaging being an integral part of the product leads to the fact that all purchases, quality control and other aspects normally considered essential for the product component and the product are equally important for packaging materials and packages.

Total quality control can be divided into two components as follows:

- Product quality control
- Packaging quality control

Packaging quality control involves three main stages namely:

- Quality control on incoming raw materials
- Quality control on in-process materials and
- Quality control on finished products

The quality control is a system encompassing specifications, inspections, analysis and recommendations. It should be regarded as an inbuilt characteristic of a package and the quality implications should begin right at the procurement stage. It is important because a substandard material creates immense problems at the production stage resulting in a large amount of defects. A correct approach is that the quality aspects should be considered at the designing stage itself. While developing a package, a proper approach would be to take into account the limits within which the material is expected to perform should be defined. It is important to strike a balance between cost of product and packaging material, understand production costs and material performance so as to arrive at an optimum cost. Therefore, true quality control should be inherent in the material and its adaptability to the operations becomes only a measure of quality control, which measures it as a routine shop floor operation.

Checking and controlling of quality for any packaging material is a prelude to ensure that the performance of the packaging material is up to the mark. Therefore, before specifying any standard, it becomes essential to study the various parameters that control the performance of a packaging material at various stages of its existence.

A large number of parameters are important when one has to identify the type of tests to be selected and their significance with the end use requirements. The tests selected should be simple and quick to perform as well as have proven results in numerical values, which are easy to read and interpret. These quality control tests are carried out for comparative purposes, between similar materials or for assessment of a given material against a specific requirement or for investigational purposes with regard to performance of a material or a container.

Testing becomes necessary even in case of established materials and applications as all men and machines are fallible and liable to vary in performance and hence the need to detect the unacceptable deviations, as quality control measure is of paramount importance.

Due to increasing consumer protection legislation and higher safety standards, more testing is needed. Very often investigation testing is needed for:

- A new product – Established packaging for a new product formulation
- A new application – Over-wrap film for shrink packaging
- A new material – New plastic film for existing product

It is less costly to change/ modify material / design at the development stage by generating adequate data by testing, than at a later stage due to under or over designing.

In packaging, it is the performance required by the product to be packaged in its particular marketing environment. In order to carry this matching of properties against requirements, it is necessary to know what the various properties actually mean in practice and to have some method of quantifying them. Testing is also important for other reasons like evaluating a new product. Test results also help in making changes in new materials and processing variables specifications to be setup.

Testing is also essential for establishment of proper specifications for procurement and quality control of incoming material. The packaging specification is an effective information about all properties and special features of packaging materials and packages. The properties of packaging materials play an important role to establish optimum packaging specification.

Properties of packaging materials like plastics, paper are affected by variables like temperature and humidity. Their sensitively to temperature, light and humidity is due to their chemical nature. It is, therefore, necessary that test pieces should be subjected to standard pre-conditioning to bring them into an equilibrium state within a specified atmosphere.

The plastic packaging materials are broadly classified as flexible, which covers films, laminates, woven fabrics; semi-rigid, which cover extruded and lamitubes and rigid packages, which include moulded, blow moulded, centered and thermoformed products as well as material handling products such as crates, pallets etc.

There is a wide range of properties to be considered while selecting plastic materials for a particular purpose. The tests for plastic packages can be classified into three broad groups.

Physical Properties

This group covers basically the physical strength and performance properties of packages on converting or packaging equipment. The different properties are thickness, tensile & elongation, heat seal strength, bond strength, hot tack, shrinkage, flex resistance co-efficient of friction, pin holes, de-lamination, identification, leakage test, dart impact, seam strength, environmental stress crack resistance, closure leakage test, adhesion test, torque test.

Physico-chemical Properties

These properties include transmission properties like permeability to gases, water vapours, odours, and extractability test.

Optical Properties

These properties include haze and gloss.

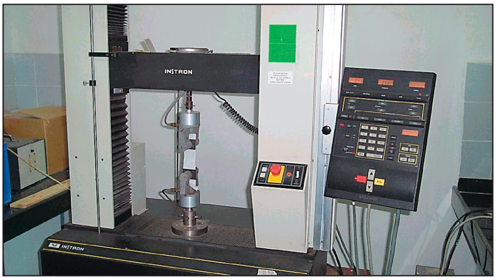
Besides the above, tests which are carried out for ascertaining the quality of the packaging material or its conformity to laid down specifications, there are some other studies which are of great significance while developing packaging systems or selecting packaging materials for food products. These are:

- Extractability / migration test
- Shelf-life Determination and Compatibility test

They are discussed later in the chapter.

Appendix Table 1 at the end of the article, gives significant tests for plastic packaging materials and packages.

Testing for Plastic Packaging



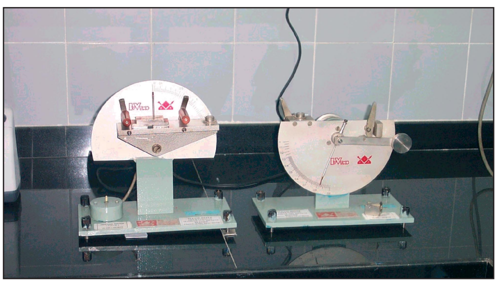
Universal Testing Machine

Differential Scanning Calorimeter

Dart Impact Tester

Haze Meter

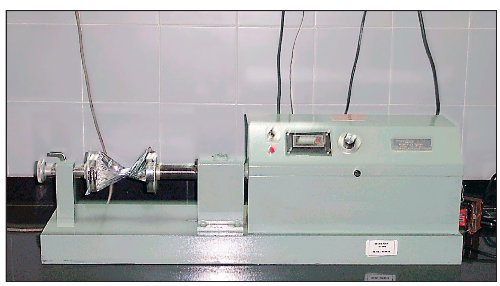
Melt Flow Index Tester



Friction Testers

Lyssy WVTR Tester

Lyssy OTR Tester



Gelbo Flex Tester

Some of the important tests and test equipment of plastic packages are discussed below:

1) Name of the test : Caliper / Thickness

Standard No : IS 2508 – 1984 / IS 1060

Equipment used : Dial gauge micrometer

Units used for results : mm / m / gauge

Significance : To check conformity of thickness to desired specification. It is an important property affecting mechanical properties, sealability, barrier properties, performance, etc.

2) Name of the test : Tensile Strength and % Elongation

Standard No : IS 2508 – 1984 / IS 1670

Equipment used : Universal testing machine (INSTRON)

Units used for results : kg / cm^2

Significance : It is a mechanical property significant in high-speed operations. By knowing the amount of deformation (strain) introduced by a given load (stress), it is possible to predict the response of the material under end-use conditions. Tensile strength is the maximum tensile stress, which a material can sustain and is taken to be the maximum load exerted in the film specimen during the test divided by the original cross section of the specimen. Elongation is usually measured at the point at which the film breaks and is expressed as the percentage of change of the original length of the specimen between the grips of the testing machine. Its importance is a measure of the film's ability to stretch. During the unwinding operation, elongation is an important property. Too low an elongation is dangerous as any sudden imbalance in the unwinding operation could lead to breaking of the film. A certain amount of tension is necessary during the unwinding operation so that films with low yield strength are in danger of being stressed beyond their yield point.

3) Name of the test : Heat Seal Strength

Standard No : ASTM F88 – 68 (1973) (Part 21)

Equipment used : Universal testing machine (INSTRON)

Units used for results : g / 15 mm width

Significance : It is the force required to pull open a seal. It is of relevance to the integrity of a package. Heat sealability of a packaging film is one of the most important properties when considering its use on wrapping or bag making equipment.

It is directly related to dwell time, temperature, pressure, seal contamination, thickness variation, MFI, type of sealant layer, type of sealing process (impulse / high frequency / ultrasonic).

4) Name of the test : Bond Strength

Standard No : ASTM D903 / F904

Equipment used : Universal testing machine (INSTRON)

	Units used for results	: g / 15 mm width
	Significance	: The performance of multi-layer structure depends upon the ability of the laminate to function as a single unit. Hence, this test is done to check the quality of lamination.

The test indicates:

- Reliability of continuing adhesion during packaging, sealing and storage
- The influence of oils / solvents
- The adhesive used
- The quantity of adhesive
- Affinity to moisture
- Excess solvent retention
- Climatic conditions

5)	**Name of the test**	**: Hot Tack Strength**
	Equipment used	: Universal testing machine (INSTRON)
	Units used	: g / 15 mm width
	Significance	: It is the critical factor in selecting sealant layers in applications such as vertical form / fill / seal lines where liquids and other substances may contaminate the seal area. Testing is done while the seal is still hot.
6)	**Name of the test**	**: Shrinkage**
	Standard No	: ASTM-D 1204–1978 (Part 35)
	Equipment used	: Instron Hot Air Oven
	Units used	: %
	Significance	: As a result of manufacturing process, internal stresses are locked into the film and this can be released by heating. For any given type of film the temperature at which shrinkage will begin are related to processing techniques.
7)	**Name of the test**	**: Flex Resistance**
	Equipment used	: Gelbo flex tester
	Units used	: No. of Cycles
	Significance	: This test determines the resistance of flexible packaging materials to flex-formed pin holes. The resistance to repeated flexure or creasing is important. Some films are highly resistant whereas others will fail by pinhole or total fracture after bending only a few times. In essence, the resistance to bending is measured by repeatedly folding the film backwards and forwards at a given rate. The number of cycles to failure is recorded as the flex resistance. With tough and flexible polymer films, even a large number of flexings may be worth running the test on various thicknesses since a thicker film may show failure at a relatively low number of flexings. Even if failure does not occur, certain properties of the film may be seriously impaired. Permeability

may be increased or tensile properties may be reduced. The optical properties of the film may also be affected.

8)	**Name of the test**	: **Co-efficient of Friction (Static & Dynamic)**
	Standard No	: ASTDM – D 1894
	Equipment used	: Buchel Vander – Korpt (pendulum method)
	Significance	: The co-efficient of friction is related to the slip properties of plastic film. This empirical data can be used for control of film production. It is a measure of the ease with which the surface of one material will slide over another. Thus, films which are slippery over various surfaces have a low COF. COF or slip properties of film are important in determining how that film will perform on conversion equipment and in final form such as in openability or stacking. This test determines the ability of film to slide over itself and is used to determine the effectiveness of slip additives incorporated into the resins. Both (static) and kinetic (dynamic) COF are measured.
9)	**Name of the test**	: **Identification of Plastics**
	Equipment used	: DSC / TMA
	Significance	: Differential Scanning Calorimeter (DSC) is a fast method to identify the substrates in co-extruded films from melting temperatures of individual substrates. Similarly, one can investigate the concentration of individual components in a blend. Multi-layer films can be easily checked by DSC to find out the individual and number of components alongwith their concentration.
10)	**Name of the test**	: **Leakage in Heat Sealed Packages**
	Standard No	: ASTM D 3078 (1977) Part 20
	Equipment used	: Dissector, vacuum pump
	Units used	: Pass / Fail
	Significance	: To evaluate seal performance of the pouch.
11)	**Name of the test**	: **Dart Impact Test**
	Standard No	: IS 2508 - 1984
	Equipment used	: Dart of different weights with stand
	Units used	: gf for 50% failure
	Significance	: This test is carried out to evaluate the impact resistance of flexible plastic film. The impact strength of a film is a measure of its ability to withstand shock loading.
12)	**Name of the test**	: **Tear Strength**
	Standard No	: ASTM D 1922
	Equipment used	: Elmendort Tearing strength tester
	Units used	: gf
	Significance	: Tear strength is an important property of packaging films and knowledge of both resistance to tear initiation and propagation

is necessary. In heavy duty sacks, possible rough handling may demand that tears do not run from small snaps or punctures incurred during transit. On the other hand, applications relying on a tear tend to give easy access to the contents, required ease of tear propagation in one direction.

13) Name of the test : Melt Flow Index
Standard No : ASTM D 1238 (Part 35)
Equipment used : M F I tester
Units used : g / 10 min @ 190°C for PE and 280°C for PP
Significance : Indicates the flow characteristics of the material at different processing conditions.

14) Name of the test : Seam Strength
Standard No : IS 3790
Equipment used : Universal Testing Machine (INSTRON)
Units used : Kg/cm^2
Significance : Determines the strength of the fabric used for the manufacture of sacks.

15) Name of the test : Environmental Stress Crack Resistance
Standard No : IS 6312 - 1980
Equipment used : Oven, soap solutions etc.
Units used : Pass / Fail
Significance : Indicates the stress crack resistance of plastic material.

16) Name of the test : Closure Leakage Test
Standard No : IS 6312 - 1980
Equipment used : Vibration Table
Units used : Pass / Fail
Significance : Indicates defects in closure system

17) Name of the test : Adhesion Strength of Pressure Sensitive tape
Standard No : IS 2880 - 1978
Equipment used : Universal Testing Machine (INSTRON)
Units used : kg / cm
Significance : Evaluates the quality of adhesive used.

18) Name of the test : Torque Test
Equipment used : Torque Tester
Units used : kg / lbs
Significance : Determines the torque required for proper closure.

19) **Name of the test** : **Water Vapour Transmission Rate**
Standard No : 1060 (Part I) / ASTM E-96
Equipment used : Lassy / MOCON WVTR Tester
Units used : g / m^2 / 24 hrs
Significance : Most important property for moisture sensitive products. It decides shelf-life of the products and is directly proportional to thickness. This property varies from polymer to polymer depending upon the thickness of the film.

20) **Name of the test** : **Oxygen Transmission Rate**
Standard No : ASTM F 3985
Equipment used : MOCON /Lyssy OTR Tester
Units used : cc/m^2/24 hrs at 27°C under 1 atmosphere pressure.
Significance : Most important property for gas sensitive products / vacuum packaging / gas packaging materials.

21) **Name of the test** : **Odour Pick – up Test**
Standard No : 4006 – 1972
Significance : Indicates odour transfer from packaging material to product.

22) **Name of the test** : **Haze**
Standard No : ASTM D 1003 (Part 35)
Equipment used : Hazemeter
Units used : %
Significance : In certain applications, high clarity and minimal haze or frostiness is desirable. This is the case in many packaging applications where good clarity enhances and the polymer structure diffuses light as it passes through film and cause hazy appearance. The hazemeter is setup to transmit a beam of light, which is diffused or scattered from its original path. The results are reported in terms of percentage haze. The lower haze, the better the clarity of the film.

23) **Name of the test** : **Gloss**
Standard No : ASTM D 2457 (Part 35)
Equipment used : Glossmeter
Units used : Percentage
Significance : Specular gloss correlates to the shine or sparkle of film. This trait can influence desirability of consumers to purchase the product packed in it. Gloss in film can be optimised by adjustment of extrusion parameters. Once processing conditions are perfect, changing resins to a higher melt index and higher density at constant molecular weight distribution, will yield in better gloss.

The list of different tests and their relevant IS, ISO, ASTM, BS, TAPPI Standards are given in Appendix Table 2 at the end of the article.

Extractability/ Migration Studies

Plastics are a large comprehensive family of materials with very wide range of properties to meet almost every requirement of the packaging industry. Plastics being synthetic materials can be tailor-made to cater to a specific need or combination of performance requirements. "Plastics are an essential and enduring form of packaging". It is no coincidence that around 50% of food in Europe is packed in plastics, whereas in India it is estimated to be around 15% to 25%.

Different types of plastics used in food packaging are polyethylenes (LDPE, HDPE, HM-HDPE, LLDPE), Polypropylene, Poly Vinyl Chloride, Polyester, Polyamides, Ethylene Acrylic Acid (EAA), Ionomers, Polycarbonate and their co-polymers. These materials may be used in different forms such as monofilms, co-extruded films, laminates, wrappers, pouches, injection/ thermoformed containers, blow moulded/stretch blow moulded jerry cans/ containers and bottles, drums, woven and knitted sacks, etc.

Plastics, in addition to the basic polymers derived from the petroleum industry, also contain some chemical components or additives, which are added in a small amount during manufacture and processing to impart desired properties to the polymer or to aid in their processing. These may be anti-oxidants, anti-blocking agents, anti-static agents, stabilisers, plasticizers, pigments, fillers, antislip agents, etc. The plastic packaging materials may also contain small amounts of monomers, oligomers, catalysts, polymerisation residues etc. These substances are generally low molecular weight components.

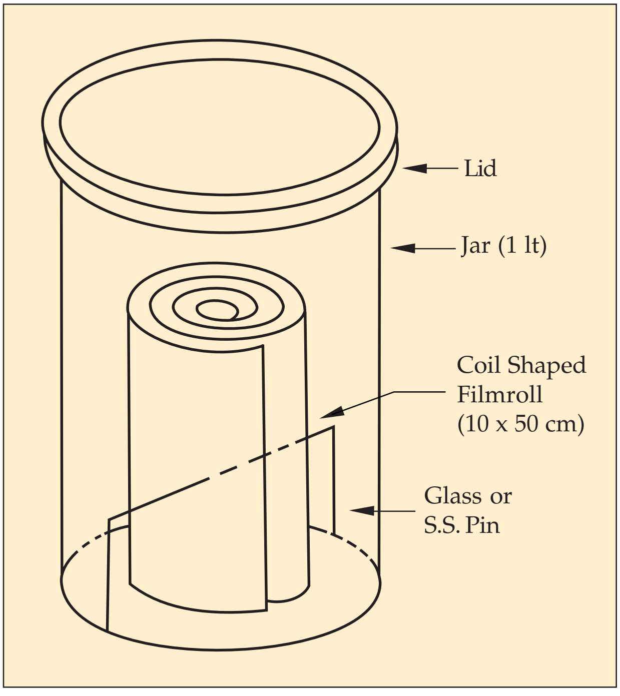
The polymers themselves, being of very high molecular weight, are inert and of limited solubility in aqueous and fatty systems and are unlikely to be transferred into food to any significant extent (Crosby, 1981). Even if fragments were accidentally swallowed, they would not react with body fluids present in the digestive system.

The low molecular weight substances and additives possess high mobility and therefore there is a likelihood of their transfer (migration) from the packaging material into the package contents, thereby contaminating the food with a possible toxic hazard to the health of the consumer. Therefore, guidelines for proper use of plastics for food packaging applications have been realised and threshold limits have been laid down. This threshold approach has been found to be an excellent model, by which majority of plastics materials are evaluated, and on the basis of which food grade application certificates are issued.

For regulating the use of plastics in food packaging, most of the countries have formulated standards and codes for the manufacture and use of plastic materials in contact with foodstuffs, suitable to conditions and situations in the country. In our country, the Bureau of Indian Standards (BIS) has also formulated standards to regulate the positive list of constituents and their specifications for each of the plastics for its safe use in contact with foodstuffs, pharmaceuticals and drinking water.

The BIS has also published: **IS 9845:1998 – Determination of Overall Migration of Constituents of Plastics Materials and articles intended to come in contact with foodstuffs – Method of Analysis.**

This standard includes method for the determination of overall migration of single or multi-layer composites in the form of pouches or containers. The Committee, while drafting the standard, had reviewed other international test methods and had noted that the test conditions (time and temperature) and food simulant extractants stipulated by the European Union (EEC) directives and the Codes of Federal Regulations (FDA), USA represent most closely the normal and actual conditions of plastics in food contact. In view of this, the present standard is based on methods prescribed by the EU Directives, FDA, USA and Deutsche Lebensmittel Rundschau/88 Jahrg/Heft 5/1992.



Overall Migration Test

Methodology

In general, migration and extraction studies need to be conducted on actual food products under conditions, which are slightly more stringent than those encountered in normal usage. It is, however, not always possible to analyse actual food for nature and quantity of migrants from the plastics. In order to simplify such assessment, food simulants/ extractants are to be substituted for the actual foodstuff. Moreover, it is very difficult to estimate all the migrants individually, hence as a good measure, the overall migration of all the migrants put together is considered for safe use, unless they are especially toxic and their limits fixed.

The simulants specified as per IS: 9845-1998 are given in Appendix Table 3 at the end of the article.

Selection of Standard Test Conditions and Simulants for Different Foods

The choice of the simulating solvents and the test conditions (time and temperature) depends on the type of food and condition of use of food products. Food products have been classified into seven major groups. Appendix Table 4 at the end of the article gives types of foods.

Appendix Table 5 at the end of the article, highlights the simulants and test conditions for extractability studies, depending on the type of food and conditions of use.

Depending upon the different forms and shapes of containers, the standard recommends the following five methods:

Method I	For finished containers (within 2 litres capacity) or sealable single/multi-layered flexible films (one-side exposure)
Method II	For large containers made of single homogenous material above 21 litres capacity.
Method III	Both sides exposure for single homogenous film, which cannot be heat-sealed.
Method IV	For closures, sealing gaskets, liners and like materials.
Method V	Materials of articles intended to come into repeated contact with foodstuffs.

The general procedure to be followed is to keep the test specimen in contact with the simulant at test temperature for specified duration of time. After exposure, the simulant is evaporated to dryness and the extractive is weighed and calculated in mg/dm^2 and mg/1kg or mg/l or ppm of the food product with respect to the capacity of the pouch/ container to be tested. For details of the procedure, reference to the standard is suggested.

Limits

In general, the limits of overall migration are specified as 10 mg/dm^2 or/and 60 mg/litre, in food simulants for different types of materials as per IS and EU. Apart from the overall migration of plastic constituents in food simulants, there should not be any colour migration into the simulant, apparent to the naked eye. If the colour migrated is clearly visible, such materials are not suitable for food contact applications, even though the extractive value is within the limit.

Some of the plastics like PVC, Polystyrene, Polyacrylonitrile, Nylon-6 whose monomers are toxic should be tested separately for their monomer content in the plastic as well as monomers migrated into foods. The limit of different monomers in the respective polymers are 0.1 ppm, 0.2 ppm, 11 ppm and 10 ppm respectively.

Shelf-life and Compatibility Studies

The term "Shelf-life" is generally understood to be the duration of that period between packing a product and using it, for which the quality of the product remains acceptable to the product user. An attempt to predict this period from data on the product, the package, the distribution and storage conditions is appropriate, where, alternative packaging materials are available which contribute positively (but to different levels) to the extension of shelf-life of the packed food product.

Shelf-life prediction is appropriate or required when the package is permeable or semi-permeable to atmospheric agents like water-vapour or/and oxygen. In the case of impermeable containers such as tinplate containers and glass jars, it is not necessary to predict shelf-life. However, in such cases it is to be assumed that the seams and the closures of the packs are perfect. Metal containers, glass bottles, aluminium foil are used mainly for their being an absolute barrier against moisture vapour and gases. However, compatibility of metal containers with specific food items need to be ascertained and wherever necessary, suitable lacquer coatings may need to be provided to achieve product-package compatibility. The lining materials/wads of the closures/caps in glass jars also should be compatible with the packed food product.

Though polymeric packaging materials are not absolute barriers against moisture vapour and gases, they have been found to be increasingly useful due to various advantages like light weight, easy to carry, easy to transport, handle and stock. The most important function of the package is to contain the product and providde protection against changes in quality caused by adverse effects of surrounding environment. The selected packaging material has to be compatible with the product to be packed and should provide specific protection to maintain shelf-life i.e. quality preservation as well as economic considerations and competitive packaging. All these are taken into consideration in design and selection of a packaging system.

Moisture Meter

Shelf-life determinations help in:

- Selection of a package for a new product, which could be optimum i.e. provide the desired shelf-life period at the most economic cost.
- Selection of an alternative package for an already marketed product, either to extend the shelf-life or to reduce the cost by using newer materials.
- Government requirement for open dating, declaration of "Best before date" assures the consumer of wholesome, nutritious, safe food.

Food Degradation Factors

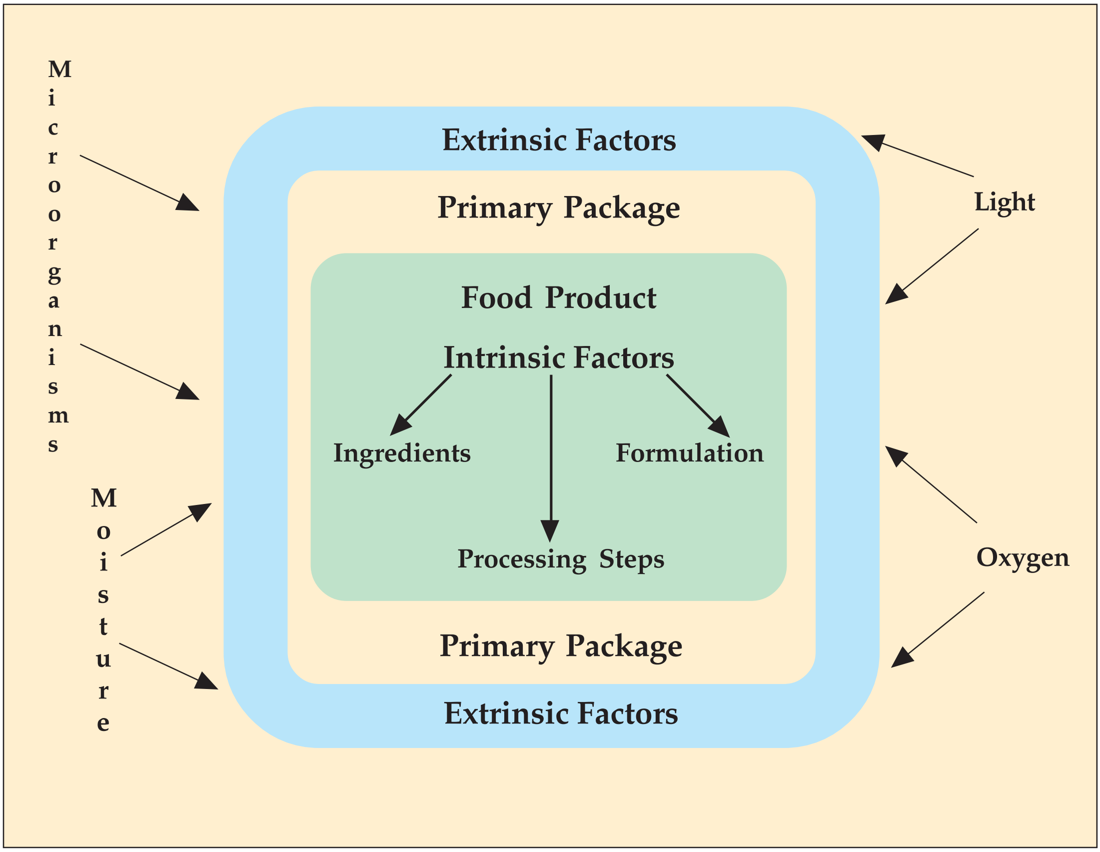
For shelf-life determination studies, it is important to understand the mechanisms of food deterioration or degradation, and the factors for the same. From the time a food product is manufactured and packed, the process of degradation commences. Protective plastic packaging slows-down some of the reactions due to the action of light, moisture, atmospheric oxygen, etc. There are many identified food degradation mechanisms, the major ones being, the gradual loss of colour, texture, flavour and nutrients. Such deteriorative changes could be due to:

- Post harvest enzymatic actions (senescene)
- Microbial spoilage
- Moisture gain or loss
- Loss of colour
- Change in texture
- Loss of nutrients

- Starch re-crystallisation (staling)
- Oxidation of fat and rancidification
- Off odours/flavours
- Entry of insects to the packaged foods
- Interactions between product and package/containers (compatibility issues)

While there are myriads of food degradation mechanisms, many are still not clearly understood. They fall into two general categories – internally driven degradation mechanism and externally driven mechanism. This can be explained by Figure 1.

Figure 1: Food Degradation Mechanism



Product Quality Attributes

As a freshly prepared food product ages, it eventually reaches a stage, where it becomes unacceptable or unsaleable. The first product quality attribute that passes this critical level into a state of being unacceptable, is the primary product quality attribute, and the degradation pathways that govern the change in that attribute are the primary product degradation pathways. Similarly, a product may have secondary, tertiary and even a quaternary product quality attribute and associated degradation pathways.

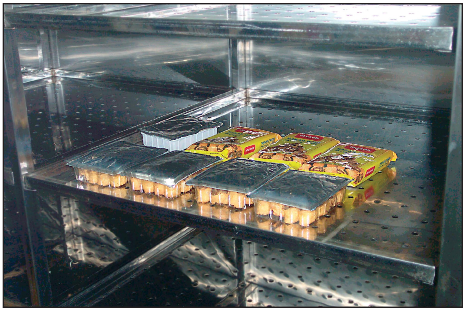
When a food product passes through the critical level of its primary product quality attribute, it does not necessarily mean that the food product is inedible or non-nutritious, it simply means that the product is beyond a certain quality standard. Thus, in practical shelf-life testing, one may not be concerned about all the degradation mechanisms for a particular food item under study, but mainly those degradation mechanisms that bring the product to the end of its commercial life.

Basics of Shelf-life Determination

Shelf-life testing in general, is the holding of a food product under a set of appropriate test conditions and monitoring the state of the product, over a time period until it fails (end of commercial life). The elapsed time to failure is the shelf-life of the product under those test conditions.

In the food industry, shelf-life (SL) testing, or storage studies are most often conducted for:

- determining the basic stability of a food product
- determining the effect of changes in ingredients
- determining the effects of change in manufacturing process
- determining the effects of different types of packaging materials, or headspace flush gases
- determining the effect of distribution abuse on the packed product
- determining the product-package compatibility



Storage Study

Accelerated shelf-life testing is the exposure of the product under a set of more severe conditions, usually higher-than-normal temperature and relative humidity, or higher-than-normal temperature and lower-than-normal relative humidity. These severe conditions accelerate the normal degradation rate, so that the product failure occurs significantly earlier. Data from a normal shelf-life study on the same product is then combined with the accelerated conditions data to establish a normal/accelerated ratio. As a thumb rule, the Indian food industry considers the ratio of 3 to establish shelf-life between normal (27°C±1°C, 65%±2% R.H.) and accelerated conditions (38°C±1°C, 90%±2% R.H.). However, practical experience, based on shelf-life studies conducted for a large number of products, indicates that this ratio does not hold good for all products and could provide misleading results. Therefore, it is recommended to conduct shelf-life testing both at accelerated as well as at standard conditions/normal conditions.

Moreover, theoretically minor changes in a product could be studied using accelerated conditions, so that shelf-life results could be obtained earlier. Unfortunately, in practice, accelerated conditions often initiate new degradation reactions that do not occur under normal conditions, thus distorting the normal/accelerated ratio, and therefore, long term storage at normal conditions must be carried out simultaneously.

Shelf-life Testing Methodology

Shelf-life studies are lengthy and expensive and they should never proceed without a detailed and specific plan, which should include the following:

- purpose of shelf-life study
- selection of packaging materials/methods
- sampling plan
- sampling instructions
- experimental factors
  - test methodologies
  - test frequency
  - control and reference materials
  - storage conditions
  - product quality attributes
  - packaging material/package attributes

  The availability of data on last three parameters is required for shelf-life determination:

  - mechanism of degradation of the product
  - agents responsible for control of rate of deterioration
  - quality of product at the time of commencement of shelf-life studies
  - the desirable size and shape of the package for marketing the product
  - the minimum acceptable quality of the product (to be saleable)
  - climatic variations likely to be encountered during storage and distribution
  - barrier properties of the packaging materials against the agents causing product degradation
  - the influence of conversion of packaging materials into packs on the barrier properties
- Data Analysis
- Conclusion
- Recommendations

Shelf-life Testing by Storage Study

Shelf-life studies can be carried out as indicated earlier by actually packing the freshly manufactured product in in different packing materials (selected for the study) and exposing adequate number of packages to different climatic conditions or to accelerated and standard conditions, which for average Indian climates are generally taken as:

Standard Condition: 27°C±1°C, 65% ±2% R.H.

Accelerated Conditions: 38°C±2°C, 90% ±2% R.H.

Walk-in Climatic Chamber

Storage studies can also be carried out at cyclic conditions. For the purpose of creating the storage conditions, humidity cabinets or environmental walk-in-chambers are used at IIP.

During the exposure period, the packages are withdrawn at fixed intervals of time to assess the quality of the product as per the product quality attributes laid down earlier. This is continued till the product becomes commercially unacceptable i.e. degradation occurs of the primary product quality attribute. To assess the product quality, organoleptic testing or sensory evaluation is also required to be carried out. The details of sensory evaluation are discussed later in the chapter.

When the packages are withdrawn, besides assessment of the product, checks are also made on the packaging material/package to observe for any softening/delamination/cracking, opening of seals, discolouration, surface stickiness etc. These checks help to establish the product-package compatibility, which is of prime importance for any packed product.

The results obtained on the assessment of the product quality and the package are tabulated and analysed for overall acceptability. Based on the analysis, shelf-life of the product in a particular package is determined. The ultimate selection of the packaging material or method would be governed by the marketing requirements and the economics of the packaging system.

Sensory Evaluation of Foods for Shelf-life Determination

A number of quality assurance procedures are used to examine and maintain the quality of a food product at different stages starting from receipt of the raw materials up to the finished product. These tests are physical, chemical, microbiological and sensory. Amongst all these methods, sensory evaluation is of paramount importance. The sensory quality has to be included in product evaluation since it is the only integrated multi-dimensional measurement. The sensory evaluation procedures have been studied in considerable details with the result that this scientific discipline has come to be recognised as fairly objective in nature (Larmond, 1987). The inherent variability of sensory evaluation by human subjects can be generally overcome through appropriate selection and training procedures, coupled with application of statistical methods so as to take full advantage of the high sensitivity of human sense organs that even today surpass the most sophisticated instrumental means for flavour, texture and colour examination (Ogden, 1993).

A successful implementation of sensory evaluation programme for shelf-life determination requires proper laboratory facilities, trained sensory panels and adoption of appropriate sensory methods. A sensory panelist works as an analytical instrument, hence should be carefully selected and rigorously trained for a particular product so as to obviate inconsistency (Pal etal 1995). Frequent re-familiarisation of investigation is indispensable.

A large number of sensory tests are available and new methods continue to develop. Broadly, these tests can be classified into three groups as follows (Stone and Sidel, 1993):

- Discriminative (Difference Testing)
- Descriptive
- Effective (Acceptance/Preference)

Verified and documented testing procedures are available from many organisations (Alan Speigel):

- AACC – American Association of Cereal Chemists
- AOAC – Association of Official Analytical Chemists
- AOCS – American Oil Chemists Society
- ASTM – American Society for Testing and Materials
- CCTI – Composite Can and Tube Institute
- PI - Packaging Institute
- TAPPI – Technical Association of the Pulp & Paper Industries
- USP - United States Pharmacopoeia

Sensory procedures are also available from the ASTM in a series of testing handbooks (refer Appendix Table 6 at the end of the article)

The storage stability/shelf-life studies strongly rely on sensory evaluation to determine the nature and extent of deteriorative changes and thereby the length of time required for the product to be commercially unacceptable. Attempts are often made to establish relationship between sensory, chemical, microbiological and instrumental data on the product quality. Various tests have been used to achieve these objectives.

For shelf-life studies, the 9 – point hedonic scale is extensively used. The scale is easily understood by consumers with minimal instructions and the product differences are reproducible with different groups of people.

The results from this scale are most informative since computations will yield means, variance measures and frequency distributions, all by order of presentation and magnitude of difference between products, by subject and by panel; and the data can be converted to ranks as well, yielding product preferences (Amerine etal., 1965).

All sensory stimuli whether simple or complex have a hedonic dimension. The term "hedonic" is derived from "hedonism" i.e. having to do with pleasure. The study of pleasure and role of sensory testing are inter-related. Though, both the parameters are inter-dependant, they cannot be qualified in measurable terms. The reason being that the same physical stimulus that arouses pleasure in one individual, may arouse displeasure in another individual. The new perspective places far greater emphasis on the essential role of pleasure and liking as a dependable variable in sensory research and on the role of consumer data relationships as a tool in understanding the importance of sensory factors to food quality. Establishing the relationship between sensory responses and pleasure associated with food is one of the most important and practical contributions that sensory science can make to the study of processed foods.

Often, certain descriptive terms are associated with product quality e.g. "excellent", "good", "fair" and "poor" and an appropriate score range corresponding to a particular description being the shelf-life cut-off. While individual sensory attributes like colour, texture, flavour, etc. may be assessed for a stored product, it is usual to have its "overall acceptance" evaluated when a hedonic or similar scale is used. However, when the evaluation is based on a score-card comprising of different attributes, often the total score is taken as the "overall acceptability" of the product.

Different sensory tests coupled with relevant statistical methodologies make sensory evaluation an important tool for shelf-life determination.

Several successful attempts have been made to develop shelf-life prediction models for various food products, each relying heavily on a sound sensory evaluation technique.

A study conducted by IIP on shelf-life of "Rabri" using 9-point Hedonic Scale method for Sensory Evaluation is summarised at the end of the chapter.

Shelf-life Prediction by Formulae

Prediction of shelf-life of moisture sensitive products is carried out by using equations/ formulae. These are based on various theories by different people. The most simple one is described here. The prediction of shelf-life based on formulae, requires the determination of the moisture absorption isotherm of the food product.

Moisture Absorption Isotherm

The water content of a foodstuff or other moisture-sensitive product, and the relative humidity (or water activity) with which it is in equilibrium, are linked by a characteristic curve for the product. If the product is placed in an atmosphere with which it is not in equilibrium, its moisture content will alter to bring it to equilibrium. The final moisture content usually differs for a given relative humidity, depending on whether the product has lost or gained moisture to reach equilibrium.

Moisture Absorption Isotherm

The experimental technique to obtain the water isotherm has been standardized following the COST 90 project of the European Cooperation in Scientific and Technical Research (Jowett, 1984). Saturated salt solutions are used in temperature-controlled enclosures to provide air of known relative humidity. Quantities of the product are exposed in these enclosures until weight equilibrium is established.

Using absorption isotherms for calculating the critical moisture content (Mc), the shelf-life 'T' of a product is the time taken for the packaged food to reach a critical moisture content, can be given by the simplified equation (Mahadeviah M. and R. V. Gowaramma, 1996):

$$T = 2.303 \times C \times \log [(W_t - W_o) / (W_e - W_o)] / P$$

where,

T = shelf-life in days

C = mass of water absorbed in g when the exposed product is in equilibrium with the storage atmosphere.

W_t = mass of the product at time 't', corresponding to Mc

W_o = mass of the product at initial moisture content M_o

W_e = mass of the product at equilibrium with the storage atmosphere

P = permeability of the pack in g/day

Shelf-life Determination by Computer Simulation

Prediction of shelf-life of food products stored under different environmental conditions depends on a large number of factors such as temperature, equilibrium relative humidity, oxygen partial pressure, light, package permeability and package configuration. The effect of these variable factors is sufficiently complex and requires numerical methods for shelf-life prediction. Mathematical models based on one or more deteriorative mechanisms can be developed, from which the shelf-life can be predicted. The greater the number of variables, more complex would be the model and more complicated would be the equation predicting the shelf-life e.g. A dry food packaged in a permeable container would be liable to spoilage by oxidation due to permeability of the oxygen, and deterioration through gain or loss of moisture due to water vapour permeability. Equations for these effects would be as follows:

(a) for oxygen deterioration

$d(VO_2/V)/dt = d(PO_2/P)/dt = T.A.KO_2/T°.V.X.(PO_2O\text{-}PO_2) – T.W.\ RATE/TO.V.1000$

where,

VO_2 = volume of oxygen (cm^3)

V = total headspace volume (cm^3)

dt = time (hr)

PO_2 = oxygen partial pressure inside package (atm)

P = total pressure (atm)

T = temperature (°K)

A = area of package film (m^2)

KO_2 = oxygen permeability ($cm^3\ O_2$STP.mil)/ m^2.hr.atm)

T° = reference temperature (273°K)

X = thickness of the film (mil)

PO_2O = outside oxygen partial pressure (atm)

W = weight of the product (g)

RATE = rate of oxidation (expressed as O_2 consumption per unit time) (ml O_2STP/g.hr)

(b) for deterioration by gain or loss of water vapour

$d(m)/dt = A.KW.PWS.(a_o\text{-}a_i)\ /X.\ w$

where,

m = moisture content of the product (g/g solids)

KW = water vapour permeability (g.mil)/m^2.hr.mmHg)

PWS = pressure of saturated water vapour

a_o = water activity outside of package

a_i = water activity inside of package

X = thickness of the film (mil)

w = weight of the product (g)

These equations are integrated over the maximum allowable extent of oxidation and maximum allowable equilibrium relative humidity of the product. Numerical techniques, when applied to the two mechanisms simultaneously aid in the prediction of shelf-life. Using equations such as above, shelf-life can be predicted for any package size and configuration as well as for different environmental conditions (Quast and Karel, 1972, Quast et al., 1972).

Deterioration by Combination of Causes

Biscuits, which contain fat and deteriorate simultaneously by loss of crispness and development of rancidity, are frequently cited as a packaging problem needing more refined treatment than monitoring deterioration of a single quality index.

Oswin comments that it can be considered as over-packing, if the deterioration by both quality indices does not reach the unacceptable limits the same storage time. This may be difficult to obtain in practice, because of the available combinations of water vapour and gas permeabilities in packaging materials. However, the concept makes economic sense.

The situation is more complex when the deterioration mechanisms interact, as in the loss of crispness and oxidative rancidity of potato crisps. A computer technique for shelf-life prediction, starting from the absorption isotherm for the product and oxygen permeation as set out by Becker, is possible (Quest and Karl, 1973). The technique is to produce three differential equations. The first is generally concerned with the change in oxygen partial pressure in the pack, the second with the progress of oxidation and the third with relative humidity changes during storage. These three differential equations are solved simultaneously. The interaction arises since alteration in humidity in the pack alters the rate of oxidation.

The computer technique allows the oxygen partial pressure and relative humidity in the pack and the state of oxidation of the potato crisps to be plotted as a function of time.

Studies Conducted at IIP on Packaging of Rabri Using 9-Point Hedonic Scale Method

Shelf-life studies were conducted on packaging of Rabri, wherein the sensory evaluation was done by using the 9-point hedonic scale method. The details are summarised as under.

Among the traditional milk based Indian sweets, 'Rabri' is one of the popular items of sweet for the Indian population. Rabri is prepared from milk with high fat content, sugar, nuts and flavouring agents, and is semi-solid in form.

'Rabri' is generally sold by sweet vendors over the counter and is not available in the packed form. Since the product is milk based, it is highly perishable in nature, due to spoilage by micro-organisms, which not only change the flavour / aroma of the product but also make it unfit for human consumption.

Packaging of this product not only reduces the handling of the product, but also makes it more hygienic. Packaging also helps to extend the shelf-life of the product. In this context, the above project was taken up to develop and recommend a suitable functional packaging system for 'Rabri'.

This product is a great delicacy. It contains about 20% fat, 17% lactose, 10% casein and 20% cane sugar.

The aim of the study was to determine the extent to which shelf-life of 'Rabri' can be prolonged by packaging and storage temperature.

The methodology adopted was to procure freshly prepared 'Rabri' from the market, pack it in different packaging materials / packaging systems and carry out storage tests at room temperature and at refrigerated conditions. Besides ordinary packaging, gas flushing with an inert gas like nitrogen was also considered.

Product Characteristics

Freshly prepared 'Rabri' was procured from one of the reputed sweets outlets for conducting the shelf-life studies. Fresh 'Rabri' when procured was creamish in colour with the characteristic aroma. The fresh product was tested for the following chemical, microbiological and sensory parameters:

i) Microbial Quality

Total Bacterial Count (Cfu/g) .... 2.85×10^1

Yeast and Mold Count (Cfu/g) .... 7.3×10^2

Presence of Lactobacilli .... absent

Presence of Coliforms .... absent

Presence of Staphylococci .... absent

ii) Chemical Parameters

Titratable Acidity (% lactic acid) 0.18
Reducing sugar (g %) 5.80
Non- reducing sugar (g%) 8.91
Total sugar (g%) 14.63
Acid Value 0.597
Peroxide Value (meq/kg) 0

iii) Organoleptic Characteristics (Sensory Evaluation)

A sensory evaluation is made by the senses of taste, smell and touch when food is eaten. The complex that results from the interaction of our senses is used to measure food quality in programmes for quality control & new product development. Product quality cannot be judged without sensory evaluation of the product. Hence, it is an important part in determining the quality of the product, which gives a clear picture of the acceptance of the product.

In the following study, sensory evaluation was done using trained panelists for the following parameters:

- Colour
- Consistency
- Aroma
- Mouth-feel
- Flavour
- Taste
- Overall acceptance

The panelists were asked to rate the product using a 9-point hedonic scale as per the score card given below. Percentages, Means and Standard deviations were calculated.

Score Card

Date: **Time:**

Name of the Panelist

Instructions: Given below are the samples of **'Rabri'** as A, B, C, D, E, F. You are requested to judge the sample on the 9 point hedonic scale for the parameters listed below:

Sample	Colour	Consis-tency	Aroma	Mouth-feel	Flavour	Taste	Overall acceptance	Comments
A.								
B.								
C.								
D.								
E.								
F.								

Key:

1.	Like extremely
2.	Like very much
3.	Like moderately
4.	Like slightly
5.	Neither like nor dislike
6.	Dislike slightly
7.	Dislike moderately
8.	Dislike very much
9.	Dislike extremely

Sensory Analysis

The results of the fresh product are given in Appendix Table 7, at the end of the article. The mean score of all the sensory parameters lies between 2.30 to 2.87, which corresponded to like extremely to like moderately. Appendix Table 8, at the end of the article, gives the frequency distribution of sensory parameters of the fresh sample.

As seen from Appendix Tables 7 & 8 at the end of the article, on Day '0' i.e. fresh sample, the colour of the sample is liked very much by almost three -fourth of the panelists (73.08%). Where as the consistency is liked by 50% panelists. Also flavour and aroma is 'liked very much' by 61.53% and mouth-feel by 57.69%. However aroma mouth-feel, flavour and taste was disliked slightly by small percentage of panelist. The taste is liked by 80.77% panelists and overall acceptance is liked by 69.22% panelists.

(To get consistent results, the first two keys i.e. 1 and 2 in the table have been combined).

Selection of Packaging Materials

Based on the nature of protection required by the product, the present packaging system and the commercial availability of the packaging materials, the following packaging materials / systems were selected for the study:

a) Flexible pouch of 5- layered co-extruded film of LD-TIE-NYLON-TIE-LD

b) Flexible pouch of laminate – metallised PET / LDPE

c) Thermoformed container of HIPS with peelable lid

d) Thermoformed container of PP with peelable lid

e) Thermoformed container of PET with peelable lid

The selected packaging materials were procured from the sources of supply and tested for migration to determine their suitability for food contact applications. The results of the migration tests are given in Appendix Table 9, at the end of the article.

There were no major changes observed in the flexible pouches as well as in the cups with the peelable lidding materials till the end of the studies which indicates the product – package compatibility.

Storage Studies

The shelf-life/storage studies of 'Rabri' were conducted in all the five selected packaging materials/systems. About 100 grams of the product was packed in all five types of packages. The pouches were closed by heat sealing. The thermoformed cups were closed by heat sealing with a peel-off type lid over the mouth. The cups were also provided with press-on plastic lids.

For each of the packaging systems, the product was packed in an ordinary/normal manner, and also flushed with nitrogen gas. Adequate quantity of filled packs in all the packaging systems were prepared and exposed to ambient conditions as well as to refrigerated conditions. The samples of the exposed packs were drawn at regular intervals of time and the packed 'Rabri' from these packs was assessed in the laboratory for its keeping quality for the parameters indicated earlier.

The storage study was planned to be carried out for a period of 3 days at ambient conditions and 10 days at refrigerated conditions. The samples, which showed deterioration earlier were withdrawn and studies with respect to that particular material/ system were discontinued.

The test results were obtained with respect to microbial quality, chemical parameters and organoleptic characteristics for studies conducted at ambient conditions and for refrigerated storage.

As indicated in the methodology, the packaging materials under study were also observed for any changes. The head space in the stored packs were tested periodically for oxygen content and it was noted that only negligible quantity of oxygen gas was present inside the packages. This indicates that there was no leakage of nitrogen gas from the packs. There were no major changes observed in the flexible pouches as well as in the cups with the peelable lidding materials till the end of the studies which indicates the product – package compatibility.

Sensory Analysis of Samples Stored at Room Temperature

The sensory evaluation of the product from the pouches could not be carried out after Day 1 as the product appeared to be off odour. In the case of the product from the cups, the results are given in Appendix Table 10, at the end of the article.

On comparing Day '0' sample with Day1 (room temperature), following changes in all the parameters were observed:

- Colour: The mean scores for all the different packaging materials shows a slight increase indicating slight change. This is a shift from like extremely to like moderately.

- Consistency: The consistency of product in PP cups with nitrogen & PET cups show marked increase in the mean scores compared to day '0' fresh sample, again indicating reduced liking in these two samples.
- Aroma: The aroma of product in PP cups with nitrogen & PET cups again show a similar pattern of increase in the mean score as compared to day '0', thus indicating a shift in the likeness for the product by the panelists.
- Mouth-feel: The mouth-feel of product in PP cups with nitrogen & PET cups increased in mean scores compared to day '0' score indicating that liking for mouth-feel was reduced. Thus, there is a shift from like extremely to like moderately.
- Flavour, Taste And Overall Acceptance: The flavour, taste and overall acceptance of product in PET cups show a slight increase in the mean scores compared to day '0'. Again showing a shift from like extremely to moderate liking by the panelists.

The above indicates that on Day 1 the samples are acceptable organoleptically.

Sensory Analysis of Samples Stored at Refrigerated Temperature

The sensory parameters were assessed for the product in pouches and in cups. The sensory evaluation for all parameters indicated acceptance of the product up to Day 4 in pouches.

The sensory evaluation of the product from the cups was carried out up to Day 8 and the results are given in Appendix Table 11, at the end of the article.

In the case of cups as per Table 11 the following is observed:

- Colour: The colour of all the samples on day 8 shows increase in the mean scores as compared to day '0' mean scores. But maximum increase is seen in PS cups with nitrogen (2.89) and PET cups with nitrogen (2.86) samples, indicating shift in the liking of the colour of these samples.
- Consistency: The consistency of all the samples is similar, thus indicating no major change in the consistency from day '0' value.
- Aroma: The aroma of all the samples shows very slight variation compared to day '0'.
- Mouth-feel: The mouth feel also does not show much variation amongst all the samples.
- Flavour: The flavors of all the samples were similar to that on day '0'. The mean scores ranged between (2.59 – 2.90).
- Taste: The taste also does not vary much among all the samples when compared to day '0' sample.

Overall Acceptance

The overall acceptance does not show much variation on day 8, as compared to day '0'.

Thus, on comparing mean and S.D. scores of both room temperature and refrigeration on day 8, it is seen that the samples of the product from PP cups without nitrogen gas and of PS cups without nitrogen gas are liked very much by the panelists. Also the mean scores clearly show that the first 3 samples i.e. PP, PS and PET cups without nitrogen gas have lower mean scores i.e. very close to that of day '0' indicating the retention of sensory attributes up to day 8. Only in gas flushed sample the score increased indicating reduction in the sensory qualities.

Thus, sensory analysis clearly shows that the samples of PP and PS cups are liked by the panelists as compared to other samples. PP cup is liked for its colour, aroma, flavour and taste where as PS cups for its mouth-feel and overall acceptance.

Conclusions

The overall acceptability of the product/shelf-life in different packaging materials at refrigerated conditions is given in Appendix Table 12, at the end of the article. The shelf-life is based on all the three quality parameters i.e. sensory, chemical and microbiological.

From Appendix Table 12, it can be concluded that in the pouches the product has a shelf-life of less than 4 days both when ordinarily packed and when flushed with nitrogen gas. Almost similar situation is observed for product packed in PP, PS and PET cups, which are flushed with nitrogen gas.

In PET cups, which are not nitrogen flushed, a shelf-life of 5 days is achieved. In PP and PS cups without nitrogen flushing, 8 days shelf-life can be achieved at refrigerated conditions.

When unpacked Rabri is stored at refrigerated conditions, the shelf-life is not more than 2 days and therefore by suitably packing the product the shelf-life can be increased by almost four times.

Recommendations

Based on the studies conducted and the test results obtained, it is recommended to use food grade PP (Polypropylene) or PS (Polystyrene) cups with heat-sealable, flexible, peelable lids without nitrogen gas.

The specifications of the recommended packaging materials are given in Appendix Table 13, at the end of the article.

Test Facilities and Certifications

Given below is a list of some of the organisations where the above tests can be carried out and certification obtained.

1. **Indian Institute Of Packaging**

   E-2, MIDC Area, Post Box No. 9432, Andheri (East),
   MUMBAI – 400 093.
   Phones : 2821 9803 / 2821 9469 / 2821 6751
   Cabel : Packinst – Mumbai Fax : 91-22-2837 5302 / 2825 4631
   E-mail : iip@bom4.vsnl.net.in Web: www.iip-in.com

   Regional Centres

   a. Indian Institute Of Packaging

   Plot 169, Indl. Estate, Perungudi
   CHENNAI – 600 096
   Phone : 044 – 2496 1560
   Fax : 044-2496 1077
   E-mail : iipche@giasmd01.vsnl.net.in

   b. Indian Institute Of Packaging

   Block C. P. Sector – V
   Salt Lake, Bidhan Nagar,
   KOLKATA – 700 091
   Phone : 033 – 2367 0763 / 2367 6016
   Telefax : 033 – 2367 9561
   E-mail : iipcal@cal.vsnl.net.in

   c. Indian Institute Of Packaging

   Plot No. 21, Functional Indl. Estate,
   Patparganj, Opp. Patparganj Bus Depot,
   DELHI – 110 092.
   Phone : 011 – 2216 6703 Fax : 011 – 2216 9612
   E-mail : iipdelhi@nde.vsnl.net.in

2. **Central Food Technological Research Institute (CFTRI)**
   Mysore 570 013
   Phone : 0821-2251 5910/2251 4760 Fax : 0821-2251 7233
   E-mail : prp@cscftri.ren.nic.in

3. **Shriram Institute for Industrial Research**

   (A Unit Of Shriram Scientific & Industrial Research Foundation)
   H.O. : 19, University Road, Delhi – 110 007 (INDIA)
   Tel : 011 – 2766 6008, 2766 7267, 2766 7860 Fax : 011 – 2766 7676, 2766 7207
   E-mail : sridlhi@vsnl.com

4. **Central Institute Of Plastics Engineering Technology**

   T. V. K. Industrial Estate
   Guindy, Chennai – 600 032.
   Tel: 044 – 2234 2371-4 Fax : 044 – 2234 7120
   E-mail : cipethq@vsnl.com

Conclusion

Quality control can be defined as control of product or service output quality by establishing quality level goals often involving inspection, analysis and action to make identified changes in order to maintain or achieve the required quality level. The need for quality control testing continues to grow throughout the industrial world. Manufacturers are investing more money, time and resources into their QC laboratories due to increased competition, higher quality standards and global standardisation. QC testing can be a critical driving force for the success or failure of a product line, as well as the organization. Plastics have been extensively used in food packaging where QC is of utmost importance. Many new test methods and standards have been developed for QC and testing of plastic packaging solutions in this industry and plastics have been able to meet the various requirements of the industry.

APPENDIX TABLE 1

Significant Tests for Plastic Packaging Materials/Packages

Packaging Material/Form	Physical Observation	Important Tests			
Plastic Film	Pin holes Tearing Gauge variation Wrinkles Odour	**Conventional Film (Mono Layer)**	**Co-extruded Film**	**Shrink Film**	**Stretch Film**
		Thickness Identification of polymer Tensile strength Elongation at break Dart impact Coefficient of friction	Thickness Identification of polymer Identification of layer Tensile strength & % elongation Dart Impact test Extractibility studies (food grade)	Thickness Identification of polymer Heat shrinkage (MD, CD)	Thickness Identification of polymer Tensile strength Elongation at break
Flexible Laminates	Delamination Wrinkles Odour Easy tear Bleeding of ink	Thickness of each layer Identification of polymeric substrate Peel bond strength Heat seal strength Odour pick test Extractability studies (food grade) Flex durability Water vapour transmission rate Oxygen transmission rate			
Wrapper	Wrinkles Colour shading Smeared printing Odour Tearing	Thickness Identification Heat shrinkage (Shrink films) Elongation at break (% elongation)			
Plastic Strap	Dirt/dust deposition Cracking/ tearing	Width Identification Tensile breaking load			
Lami Tubes	Print quality Dent at nozzle Dirt / Dust	Size Capacity Heat seal strength Compatibility test Vacuum leakage test Barrier properties (WVTR & OTR)			

<table>
<tr><th>Packaging Material/Form</th><th>Physical Observation</th><th colspan="2">Important Tests</th></tr>
<tr><td>Plastic Caps /Closures</td><td>Workmanship & finish
Thread condition
Denting (Metal)
Crack (Plastics)</td><td colspan="2">Dimension
Identification of plastics
Environment Stress Crack Resistance
Compatibility of wad to product
Torque test</td></tr>
<tr><td>Pressure Sensitive Self-Adhesive Plastic Tape</td><td>Rough-cut edge
Dent at core
Dust deposition
Easy peelable</td><td colspan="2">Thickness of plastic films
Thickness of adhesive
Identification of plastic film
Adhesion strength to steel
Tensile breaking load</td></tr>
<tr><td>Flexible Intermediate Bulk Container (FIBC)</td><td></td><td colspan="2">Size and weight
Tensile strength
Top lift
Tear
Stack
Topple
Drop
Seam strength
workman ship</td></tr>
<tr><td>Sachet /Pouch</td><td>Missing colour
Improper printing
Colour shade variation
Improper sealing
Tears or holes
Dirt contamination
Delamination</td><td colspan="2">Seal strength
Hot tack strength
Peel bond strength (composite layer)
Identification of polymer substrate
Vacuum leakage test
Extractability studies (food grade)
Drop test
Resistance to product
Water Vapour Transmission Rate (WVTR)
Oxygen Transmission Rate (OTR)
Odour pick up test</td></tr>
<tr><td rowspan="2">Bag/Sack</td><td rowspan="2">Holes (Plastic bag)
Tearing
Improper Seal/seam
Dust / dirt
Odour</td><td>Plastic</td><td>Plastic Woven Sack</td></tr>
<tr><td>Thickness
Size
Tensile strength
% elongation
Heat seal strength
WVTR
OTR
Extractability studies (Food grade)
Drop test</td><td>Dimensions
Size
Seam strength
Tensile breaking load
Ends / picks</td></tr>
</table>

Packaging Material/Form	Physical Observation	Important Tests		
Thermoplastic Moulded Container	Mould marks or drawing lines Cracks or holes	**Injection Moulded Container**	**Blow Moulded Container**	**Thermoformed Container**
	Wall thickness variation Embedded foreign matter Dis-colouration	Dimensions Thickness Weight Environment Stress Crack Resistance (ESCR) Extractability studies (for food application)	Dimensions Capacity Thickness Weight To do Environment Stress Crack Resistance (ESCR) Extractability studies (for food application) Drop test Ink adhesion test Product resistance of printed container Closure leakage test	Dimensions Thickness Identification of polymer ESCR
Plastic Drum	Workmanship Dent at top / bottom and body	Dimensions Weight Environment Stress Crack Resistance Drop test Stack load test Hydraulic pressure test Leakage test		

APPENDIX TABLE 2

Tests and Relevant IS, ISO, ASTM, BS, TAPPI Standards

Name of the Test	Standards				
	IS	ISO	ASTM	BS	TAPPI
Adhesion Strength of Adhesive	2257				
Adhesion Strength of Pressure Sensitive Cellulose Tape	2280		D-2860-90		
Adhesion Strength of Pressure Sensitive Tape to self	8402		D-3330-90		
(1) Caliper (Thickness) (2) Multi-layer co-extruded film (by unitron) 2 layer film 3 layer film 5 layer film (3) PVC Sheet and Films	1060-01 2076	3034,0438	D-0645-92	4817	T-411-0M-89
Dart impact	2508				
Environmental Stress Crack Resistance (Plastics)	6312				
Extractability Studies on Plastics	9845				
Flex Crack Resistance	7016				
Flexural Properties of rigid/semi-rigid Plastics		0178	D-0790-86	6319-03	
Friction Testing (A) Dynamic Friction Test (Pendulum Method) (B) Static Friction Test (Tilting Plane) (C) Co-efficient of Friction of Plastic Film (INSTRON) (D) Co-efficient of Friction of Plastic Film (Stationary plane moving sledge) (CEAST Instrument)			 D-1894-87	2782-824A, 4618-5.6	T-815-OM-85
Gas Permeability ($O_2/N_2/CO_2$) for Film for Package / Container		2556, 0871	D-1434-92		1902-3.9
Gloss			D-2457-97		T-780-OM-90
Haze		D-1003-95			
Heat Seal Strength / Set of Condition (Fin Seal Lap Seal)			F-0088-94		

Name of the Test	Standards				
	IS	ISO	ASTM	BS	TAPPI
Identification of Adhesive (in Packaging Material)					
Leak in Heat Sealed Flexible Packages			D-3078-94		
Melt Flow Index		0489	D-1238-86	2782-720A	
Odour Pick Up (from Packaging Materials or Containers)	4006-02			PD-6459	T-483-CM-82
Porter / shots of Hessian Ends / Picks of Hessian	2818-01				
Pouch Burst Test					
Tensile Strength, % Elongation and Energy Break for Plastic Strapping / Fabric allied Materials	1670 2508	1798		2782	
Torque Test of Caps					
Water Absorption in Plastics		0062 2896	D-0570-81	3177	
Water Vapour Transmission Rate	1060-02	5633			T-448-OM-89 T-465-CM-85

APPENDIX TABLE 3
Simulants for Determination of Overall Migrants

Simulant A	Distilled water or water of equivalent quality
Simulant B	3 percent acetic acid (w/v) in aqueous solution (using the simulant A)
Simulant C[1]	10% ethanol (v/v) in aqueous solution for foodstuffs having alcohol less than 10 percent (v/v) (Using the simulant A)
Simulant C[2]	50 percent ethanol (v/v) in aqueous solution for foodstuffs, having alcohol more than 10 percent and less than 50 percent (v/v) (Using the simulant A)
Simulant D	n-heptane – shall be freshly distilled before use.
Simulant E	Rectified olive oil or mixture of synthetic triglycerides or sunflower oil.

Note : The Simulant "E" suggested by the EU for fatty foods need not be considered at present as the methodology of estimation is not yet developed.

APPENDIX TABLE 4

Classification of Foods and Selection of Simulant

Type	Description	Example	Simulants
I	Aqueous, non acidic foods (pH>5) without fat	Honey, mineral water, sugar syrups, molasses, skimmed milk, *rasgulla*, infusions, *murabba*, yeast paste, etc.	'A'
II	Aqueous, acidic food (pH≤5) without fat	Fruit juices, squashes, fruit chunks or puree or paste, vinegar, jams, jellies, carbonated beverages, lemonade, processed vegetables, rennet, preparations of soups, broths, sauces, RTS beverages etc.	'B'
III	Alcoholic beverages:		
	i) Alcohol concentration less than 10 percent.	Beer and some pharmaceuticals syrups.	'C^1'
	ii) Alcohol concentration above 10 percent	Wine, brandy, whisky, arrack and other alcoholic drinks.	'C^2'
IV	Oils, fats and processed dry food with surface fat or volatile oil	Vegetable oils, ghee, vanaspati, cocoa butter, lard, biscuits, spice powder, snacks and savoury, chocolate, caramel, malted foods, egg powder, tea/coffee powder, confectionery, fried and roasted nuts, etc.	'D'
V	Non-acidic food ((*p*H>5) or high fat and having high moisture content.	Butter, bread, pastry, cakes, *shrikand,* milk based sweets, ice-cream, moist and fatty confectionery products.	'A and D'
VI	Acidic food (pH<5) or high fat and having high moisture content	Pickles, ketchup, cheese with low curd, fresh and processed meat and fish products, sauces having fat, frozen foods, mayonnaise, etc.	'B and D'
VII	Dry processed food without fat	Cereals and pulses, dehydrated vegetables and fruits, dried yeast, corn flakes, salt, sugar, milled products, barley powder, oats, vermicelli, spaghetti etc.	No end test

APPENDIX TABLE 5
Simulating Solvents for Different Types of Food and Temperature – Time Conditions

Conditions Of Use	Type of Food	Water	3 % Acetice Acid	10% Alcohol	50% Alcohol	N-Heptane
High temperature heat sterilized (Retorting)	I,II,IV, V and VI	121°C 2hours	121°C 2hours	–	–	66°C for 2 hours
Hot filled or pasteurized above 66°C, below 100°C	I,II,IV, V and VI	100°C 2hours	100°C 2hours	– –	– –	49°C for 30 minutes
Hot filled or pasteurized below 66°C	I to VI	70°C 2hours	70°C 2hours	70°C 2hours	70°C 2hours	38°C for 30 minutes
Room temperature filled and stored (no thermal treatment in container) and also in refrigerated and frozen condition	I to VI	40°C 10 days	40°C 10 days	40°C 10 days	40°C 10 days	38°C for 30 minutes

NOTES:

1. Heptane simulant are not to be used on wax lined containers.
2. Heptane extractivity results must be divided by a factor of five in arriving at the extractivity of a food product.

APPENDIX TABLE 6
Sensory Procedures Published by ASTM

Number	Details
STP 434	Manual of Sensory Testing Methods
STP 545	Sensory Evaluation of Appearance of Materials
STP 594	Co-relating Sensory/Objective Measurements
STP 913	Physical Requirements/Guidelines for Sensory Evaluation Laboratories
STP 440	Co-relation of Subjective/Objective Methods in the Study of Odour and Taste
STP 433	Basic Principles of Sensory Evaluation
STP 914	Review and Evaluation of Appearance : Methods and Techniques
STP 1035	Product Testing with Consumers for Research Guidance
STP 773	Selected Sensory Methods: Problems and Approaches to Measuring Hedonics
STP 758	Guidelines for the Selection and Training of Sensory Panel members
STP 682	Manual on Consumer Sensory Evaluation
DS 61	Atlas of Odour Character Profiles
DS 48A	Compilation of Odour and Taste Threshold Values Data
PCN 03-518088-36	ASTM Standards on Sensory Evaluation of Materials and Products
PCN 03-512087-14	ASTM Standards on Colour and Appearance Measurement

[Source: Shelf-life Testing by Alan Speigel.]

APPENDIX TABLE 7

Mean and Standard Deviation of the Samples for Sensory Parameters for Fresh Sample

Parameters	Mean	Standard Deviation
Colour	2.30	0.67
Consistency	2.87	2.46
Aroma	2.82	2.39
Mouth-feel	2.83	2.36
Flavour	2.81	2.34
Taste	2.78	2.31
Overall acceptance	2.76	2.28

APPENDIX TABLE 8

Frequency Distribution of Sensory Parameters of the Fresh Sample (% Panelists)

Sample	Parameters	Score Value % Panelists								
		1	2	3	4	5	6	7	8	9
Control (Fresh)	Colour	3.85	69.23	19.23	7.69	–	–	–	–	–
	Consistency	19.23	30.77	42.31	7.69	–	–	–	–	–
	Aroma	23.07	38.46	23.07	7.69	3.85	3.85	–	–	–
	Mouth-feel	11.54	46.15	26.90	7.69	3.85	3.85	–	–	–
	Flavour	23.07	38.46	30.77	–	3.85	3.85	–	–	–
	Taste	30.77	50.00	11.54	3.85	–	3.85	–	–	–
	Overall acceptance	23.07	46.15	23.07	7.69	–	–	–	–	–

APPENDIX TABLE 9

Extractability / Migration Test

Packaging Materials	Test Results (mg/dm^2)
Polypropylene cups	2.16
Polystyrene cups	2.20
Polyethylene terepthalate cups	8.69
Metallised Polyester/LDPE pouch	5.27
LD-TIE-NYLON-TIE-LD pouch	3.61

The test method is as per BIS: 9845-1986.

The test results obtained are within the maximum specified limit of 10 mg/dm^2 as per the following standards:

IS: 10146 -1982

IS: 10142 -1982

IS: 10910 -1984

IS: 12252 –1982

APPENDIX TABLE 10

The Mean And Standard Deviation Values of Sensory Parameters on Day 1 at Ambient Conditions

Parameters	Sample A (PP cups)		Sample B (PS cups)		Sample C (PET cups)		Sample D (PP cups with nitrogen)		Sample E (PS cups with nitrogen)		Sample F (PET cups with nitrogen)	
	M	S.D.	M	S.D.	M	S.D.	M	S.D.	M	S.D.	M	S.D.
Colour	2.58	1.25	2.67	1.24	2.67	1.26	3.05	1.42	2.87	1.39	2.86	1.37
Consistency	2.70	1.13	2.71	1.21	3.58	1.55	3.07	1.45	2.89	1.39	2.88	1.40
Aroma	2.69	1.13	2.69	1.21	3.37	1.62	3.01	1.43	2.89	1.39	2.87	1.39
Mouth-feel	2.66	1.23	2.68	1.23	3.27	1.55	2.99	1.42	2.90	1.39	3.04	3.12
Flavour	2.61	1.23	2.66	1.23	3.13	1.45	2.93	1.41	2.88	1.38	3.03	2.07
Taste	2.61	1.26	2.64	1.23	3.03	1.40	2.89	1.40	2.85	1.37	3.03	3.03
Overall Acceptance	2.67	1.24	2.63	1.23	3.08	1.43	2.89	1.40	2.87	1.38	3.04	2.99

APPENDIX TABLE 11

Mean And Standard Deviation of Day 8 Samples at Refrigerated Conditions

	Sample A (PP) Cups		Sample B (PS) Cups		Sample C (PET) Cups		Sample D (PPg) N_2 Flushed Cups		Sample E (PSg) N_2 Flushed Cups		Sample F (PETg) N_2 Flushed Cups	
	M	S.D.	M	S.D.	M	S.D.	M	S.D.	M	S.D.	M	S.D.
Colour	2.56	1.1	2.62	1.26	2.69	1.27	2.79	1.33	2.89	2.79	2.86	2.51
Consistency	2.67	1.07	2.69	1.21	2.74	1.31	2.97	3.02	2.89	2.72	2.87	2.5
Aroma	2.56	1.08	2.67	1.21	2.76	1.33	2.96	2.97	2.89	2.69	2.87	2.48
Mouth-feel	2.62	1.21	2.66	1.24	2.78	1.34	2.95	2.93	2.89	2.67	2.87	2.47
Flavour	2.59	1.21	2.65	1.23	2.77	1.32	2.89	2.82	2.88	2.61	2.9	2.57
Taste	2.56	1.24	2.63	1.23	2.76	1.31	2.93	2.85	2.87	2.58	2.91	2.55
Overall Acceptance	2.61	1.26	2.62	1.23	2.79	1.33	2.89	2.78	2.88	2.56	2.91	2.53

APPENDIX TABLE 12

Shelf-life of 'Rabri' at Refrigerated Conditions

Packaging Materials	Shelf-life (in days)
Polypropylene cups	8
Polypropylene cups Gas flushed	< 4
Polystyrene cups	8
Polystyrene cups Gas flushed	4
Polyethylene terepthalate cups	5
Polyethylene terephthalate cups Gas flushed	4
Metallised Polyester/LDPE pouch	< 4
Metallised Polyester/LDPE pouch Gas flushed	< 4
LD-TIE-NYLON-TIE-LD pouch	< 4
LD-TIE-NYLON-TIE-LD pouch Gas flushed	< 4

APPENDIX TABLE 13

Specification Details

Packaging Materials		Specification	
A. Thermoformed Polypropylene Cup			
Material of Construction	:	Food grade Polypropylene	
Thickness	:	0.15 mm	
Dimensions (Internal) (mm)			
100 grams container	:	Top diameter	- 73.00
	:	Base diameter	- 64.50
	:	Height	- 33.00
200 grams container	:	Top diameter	- 85.00
	:	Base diameter	- 71.05
	:	Height	- 44.05
B. Thermoformed Polystyrene Cup			
Material of Construction	:	Food grade HIPS (High Impact Polystyrene)	
Thickness	:	0.22 mm	
Dimensions (Internal) (mm)			
100 grams container	:	Top diameter	- 73.00
	:	Base diameter	- 64.50
	:	Height	- 35.00
200 grams container	:	Top diameter	- 85.00
	:	Base diameter	- 71.50
	:	Height	- 45.00
C. Peelable Lid for PP Cup			
Material of construction	:	Aluminium Foil coated with special heat seal lacquer.	
Thickness	:	40 µ	
D. Peelable Lid for PS Cup			
Material of construction	:	Aluminium Foil coated with special heat seal lacquer	
Thickness	:	40 µ	
E. Plastic Re-closable Lid for PP & PS Cups			
Material of construction	:	Same as the cup	
Thickness	:	Same as the cup	

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