Paper No.: 12
Paper Title: FOOD PACKAGING TECHNOLOGY
Module – 31: Quality evaluation of Packaging Materials

1. INTRODUCTION:

In order to evaluate the performance of a package in the market place, it is important to test the different packaging materials used for their manufacture. Thus, quality evaluation of the packaging materials is done mainly for the following purposes:

1) Comparison with competitive packaging material, e.g. to compare offers.
2) Comparison of the current supply of material with the quality of that offered for the first time; also regular checking of uniformity in new supplies of packaging materials.
3) Quality checks during the production of packaging materials.
4) Evaluation of the suitability of a packaging material for a certain specific purpose, for instance protection against mechanical or climatic hazards.

Test procedures that are applicable for general classes of materials or packages are available and published in standardized form (Paine *et al.*, 1992; Griffin *et al.*, 1972).

ASTM - American Society of Testing and Materials Standards
TAPPI - Technical Association for the Pulp and Paper Industry (USA) Standards
BIS - Bureau of Indian Standards
ISO/R - International Standards Recommendations
BS - British Standards
FEFCO - Federation Europeanne des Fabricants de Carton Ondule Test Methods
PIFA - Packaging and Industrial Films Association Standards
ABA - American Box Board Association
BPBMA - British Paper and Board Manufacturers' Association
NFPA - National Flexible Packaging Association

2. CONDITIONING OF THE SAMPLES

It is very important that the quality evaluation of the packaging material should be carried out in standard atmospheric conditions and the samples should be allowed to reach equilibrium prior to evaluation which normally takes 24 hours. This is so because the properties of many packaging materials depend on the climatic conditions to which these materials are exposed. The physical properties of paper are affected by its moisture content and the moisture content varies in proportion to the relative humidity and temperature of the surrounding atmosphere. In special cases it may be necessary to check the moisture content of the test specimen, in order to ensure that the climate has had its effect on the specimen. Quality evaluation laboratories are constructed to maintain the standard atmospheric conditions and no test is considered official if conducted under any other conditions. In a number of countries a standard atmospheric condition has been established, i.e. 20°C and 65% R.H. in Argentina, Australia, Belgium, France, Germany, Netherlands, New Zealand, UK; 23 ° C and 50% R.H. in USA, Canada, Burma, Mexico, South Africa and 27°C and 65% R.H. in India.

3. IMPORTANT QUALITY EVALUATION METHODS

Most of the tests for evaluation of packaging materials are based mainly on evaluation of one or other type of strength of packaging material to be evaluated.

3.1 Grammage or GSM

Papers, foils and films are purchased on weight basis and any deviation from the prescribed weight will affect purchaser and the vender. Most physical properties such as bursting strength, thickness are specified in accordance with a particular basis weight or bulk.

Method of Test: The samples are cut by selecting the suitable template considering the type of the sample. For heavy paper (weighing above 100 GSM) template of the size 10 cm x 10 cm is taken and hung on one of the arms of the instrument. Reading is taken directly on the scale "A". In case the paper or paper board is light and reading remains below 100 GSM then template of size 10 cm x 20 cm is used to get more accurate results and reading is taken on scale "B". At least 5 readings are taken and results are expressed in range as g/m^2.

Suitable size of paper is cut with a template and then weighed on a balance. The weight is recorded and converted in to g/m^2 and expressed as grammage or GSM.

3.2 Thickness

Many physical and mechanical properties of paper, paperboard and flexible packaging materials are dependent upon the thickness of the material. Properties like tensile strength, sealability, and seal strength, moisture, gas and light barrier properties are directly related to thickness. In case of laminates the thickness of the constituent plies are more important as they influence the barrier properties. This test is useful for routine control.

Method of Test: Cut a piece from sample without any irregularities of size 10 cm x 10 cm. Place the specimen between two points of the micrometer, one of which has to be lifted gently to insert the paper and note the corresponding reading. The thickness can be expressed in any unit such as micron, inches, mil etc. Take at least 10 readings.

0.001" = 25 micron = 100 gauge = 1 mil.

3.3. Bursting Strength: (IS 1009-1966 Part I)

Bursting strength of paper and paperboard is determined in order to assess both strength and toughness of the material. It is essentially the ability of the sample to absorb energy.

Method of Test: The sample is fixed between clamps. The area exposed is 1.2 in^2. The sample is subjected to steadily increasing pressure hydraulically exerted on a rubber diaphragm beneath the sample until it ruptures. The maximum pressure required to rupture the sample is automatically recorded by a pressure gauge. This test is of importance in routine quality check of packaging material during manufacture. Eg. Corrugated Boxes.

3.4 Tear Strength or Tearing Resistance: (ASTM, D 689-79 Part 20)

This test is performed on papers and it gives an indication towards the strength of the paper. It is helpful in making selection of papers based on material for packaging purposes. The tear strength requirements may be high or low according to end use of the packaging material. This test measures the energy absorbed by the test sample in propagating a tear that has already been initiated by cutting a small nick in the test piece.

Method of Test: The Elmendorf tearing tester has two grips set side by side with only a small separation. One grip is stationary and is mounted on an upright on the instrument base. The second grip is movable and is mounted on a pendulum. The pendulum is mounted on a frictionless bearing and swings on a shaft. The sample of 50 x 62 mm size is clamped in the two grips and a cut is made using a sharp knife fixed on the tester. When the pendulum is released, it swings down on pre-cut sample. This indicates the residual energy lost in tearing and expressed in mN (milli Newton).

3.4 Water Penetration-Cobb Test: (IS: 4006-1966 Part I):

This test measures the amount of water absorbed by the sample during penetration from one side to another. It is useful in assessing the suitability of paper and paperboards to be used for shipping containers, which may be exposed to water spray.

<u>Method of Test:</u> A weighed sample is clamped under a metal base plate and exposed to water for one minute (paper/paper board). The area exposed is 100 cm^2. After the specific time, the sample is removed, blotted and reweighed. The difference in weight indicates the amount of water absorbed by the sample. The results are expressed as gm / m^2.

3.5 Grease Resistance: (ASTM D-722; TAPPI T - 454)

This test is important for the packaging materials used for fat rich food products like butter, ghee, oil etc.

<u>Method of Test:</u> This test is performed by putting 5 g sand on the specimen through a hollow cylinder metallic piece and then topping the sand with 1.1 ml of coloured turpentine dye. This is placed on a white paper sheet and at specified intervals, the indicator sheet is examined for the first spot and after it the experiment is discontinued. The time between the application of turpentine dye and appearance of first stain is recorded as transudation time in seconds.

3.6 Water Vapour Permeability: (IS: 1060- 1960 Part II)

One of the prime functions of the packaging materials is to act as barrier to gases and vapours. Many hygroscopic foods have to be protected from oxygenated water vapour pick up. The measurement of permeability is therefore very important.

<u>Method of Test:</u> The water vapour permeability may be measured by means of high-vacuum techniques, although there are simple gravimetric methods available which determine Water Vapour Transmission Rate (WVTR) much easily. In this method the value of water vapour permeability is determined by the increase in weight of a dish filled with desiccant (Eg. anhydrous calcium chloride), covered with the test specimen and sealed with molten wax or vacuum grease. The sealed dish is placed in a humidity cabinet maintained at 38 ± 1°C and R.H. 90 ± 2 %.

The WVTR is computed by the following formula:

$$\text{WVTR} = \frac{G \times 24 \ \text{g/m}^2/24 \ \text{hrs}}{A \times T}$$

Where;

G = weight gained in gm

T = time during which gain in weight is observed

A = area of the sample exposed in m^2

4. IDENTIFICATION OF PACKAGING MATERIALS:

Over the past decades packages have become more complex and we are presently using number of plastic films in packaging. Film users and converters may use a variety of instruments from high-powered microscopes to spectrophotometers. However, for easy and rapid identification of films, film users and converters usually go for simple non-instrumental techniques.

Method of Test: The first step in analyzing a flexible film is to measure some of its physical properties by density, hardness, stiffness etc. The laminates are to be delaminated into its separate plies so that they can be separately identified. There are several methods available to separate and isolate laminated components. They can be separated by heating on a flame or by immersing in boiling water up to 5 hours.

Tetrahydrofuran vapours are used for rapid separation of different laminates. The plastics can be identified by solubility or by the observation of burning characteristics where the colour of the flame, the way of dripping and the odour of the fumes assist to identify the type of plastic. The characteristics are shown in Table 1. Solubility of films in a particular solvent is an excellent and more dependable test for identification. In this test one-square inch of sample is put into 15 ml of the appropriate solvent and heated to boiling point with proper care to prevent solvent loss.

Table 1: Characteristics of different plastics

Film	Density range (gm/cc)	Flammability (self extinguishing)	Colour	Behaviour	Odour
Polyethylene	0.910 - 0.965	No	Top yellow bottom blue	Melts and drips	Burnt wax

			white smoke		
Polypropylene	0.900 - 0.915	No	Top yellow bottom blue white smoke	Melts and drips	Burnt wax and acrid
PVC	1.28-1.38	Yes	Yellow orange with green edge	Darkens, softens & decomposes	Chlorine
PVDC	1.68	Yes	As above with green spurts	Black, hard residue	Chlorine
PVA	1.21-1.33	Yes but slowly	Yellow with gray smoke	Swells, softens and turns brown	Pungent
Poly carbonate	1.2	Yes	Yellow orange with black smoke	No drips, decomposes	Pleasant
Polyester	1.38	No	Yellow-black smoke	No drips, softens, burns steadily	Pleasant
Polystyrene	1.04-1.09	No	Yellow orange black shoots	No drips, softens	Floral (sweet)
Nylon	1.06-1.14	Yes	Blue, yellow top	Melts, drips and froths, Rigid drips	Burnt Hair
Cellophane	1.48	No	Yellow, orange, Grey and smoke	Burns fast and complete, burnt area brittle	Burnt Paper
Cellulose acetate	1.28-1.32	No	Yellow with blue base	Melts, burns quickly and leaves beads	Burnt Vinegar
Cellulose Nitrate	1.35-1.40	No	Yellow	Burns at once and fully	Acrid

5.0 QUALITY EVALUATION OF FABRICATED PACKAGES

Once packaging materials have been fabricated into package, it is important to measure properties of these packages to ensure that they conform to the desired specifications. These tests involve measurement of critical dimensions and one or two critical properties.

5.1 Evaluation of Glass Bottles (IS: 1392 -1967)

5.1.1. Dimensional Measurements:

Height, body diameter, wall thickness and finish are measured to detect possible variations that may exceed the tolerance limits, which have been established by glass manufacturers. Adherence to these tolerance limits is an important factor in operation of high speed filling lines. For checking body dimensions, gauges are used which have been specially designed for each specific bottle. The capacity of glass container is measured by selecting a sample of 12 bottles at random and checking them for volume.

5.1.2 Pressure Test:

Bottles used for liquor, carbonated beverages and soda water etc. have to withstand certain amount of internal pressure. Devices are available which subject the bottles to internal pressure using a gas or liquid. The bottles are subjected to an internal pressure of 150 kg /cm^2 for 1 minute. The temperature at which the test is carried out is very important since, a bottle withstanding 150 kg/cm^2/at 30°C may fail to withstand the same pressure at 60°C. Bottles, which have to withstand pressure, should be carefully designed.

5.1.3 Thermal Shock Test:

This test is performed when the bottles are subjected to sudden temperature difference during actual filling and use. In food industry sterilized product/beverage is packed in bottles and in pharmaceutical industry, the bottles are sterilized by hot steam before use. In this test few bottles are immersed in a hot water bath at a temperature of 72 ±2°C for 300 ± 10 seconds and when the bottles have reached the temperature, they are taken out along with hot water inside and suddenly dipped in a cold water bath at 30 ± 2° C for 30 seconds. The difference between the hot water bath temperature and cold water bath temperature gives the thermal shock to the bottles. The time for transfer of bottles from the hot water bath should not be more than 60 seconds or less than 15 seconds.

5.1.4 Impact Test:

Bottles that are used again and again, often meets certain amount of impact in their daily use. In order to ensure that such bottles do not fail, this test is performed. In this test a steel ball of 400 gm is dropped from a height of 10 cm on the bottle held rigidly. In case of milk bottles the ball is dropped thrice on the same spot on the bottle and the bottle should not freak or crack. In the pendulum test the steel ball swings and strikes at the bottle held rigidly.

6. Conclusion

Packaging materials need specific properties in order to protect the contents. The quality evaluation of different packaging materials is important for manufacturers, raw material suppliers and as well as the users. The manufacturer is always interested in testing the basic quality and to find out what new applications can be developed based on the properties of the packaging material and also to check the quality of the material. The converter has interest in the conformity to specifications and suitability for application. The user wants to ensure that the material he is purchasing is of required quality and adequate to protect the product from spoilage. Thus, quality evaluation of packaging material is an important operation in the food packaging industry.

Reference:

Griffin C. R. and Sacharow, S. 1972. *Principles of Package Development*. The AVI Publishing Company Inc.

Paine, F.A. and Paine, H.Y. 1992. *A Hand Book of Food Packaging*. Blackie Academic & Professional.

Robertson GL. 1993. *Food Packaging Principles and Practice*. Marcel Dekker INC. New York

EVOLUTION OF PACKAGING: TREND AND GROWTH OF THE PLASTIC INDUSTRY

Ever since ancient man felt the need for storing food for the next day's consumption, technology of food packaging in its primitive form emerged. In earlier times, ancient man was using plant leaves to cover the food, which he could not consume, and perhaps that was the beginning of food packaging. As he gathered experience with vulnerability of such protection, the concept was innovated with the formation of a container made from plant leaves using tiny needles of a tree stem. With the progress of civilisation, the concept of protecting food products was developed more and more in response to increasing needs.

Traditional Packages (Bamboo Baskets, Wooden Containers and Jute Sacks) for Packaging of Fruits and Vegetables

The concept of traditional packaging itself is changing with times. Dictionary meaning of traditional is conventional, time honoured, routine or historic. If we ask a senior citizen about traditional packaging, he would cite examples of packages made from plant leaves, bamboo baskets, ceramic jar or earthen pots and other similar forms made from natural materials. For modern packaging, the senior citizen would give examples of metal containers, glass bottles, etc. However, if we ask the same question to a youngster, perhaps he may give examples of metal cans and glass bottles for traditional packaging and for modern packaging he would cite packages such as laminated cartons, blister packs, flexible bottles, PET bottles, various forms of pouches and other similar packages. With the passage of time the concept of traditional and modern packaging will further change.

In today's context, packaging is both a symbol of society's consumption habits and reflection of its progress. The user expects the packaging to have better strength, be easier handled, be lighter, more aesthetic and safer from the point of view of hygiene. In addition to its standard attributes, today's packaging is also expected to contribute to protecting the environment, besides being friendly to human health.

Emergence of Plastic Packages

For food packaging, metal container has been the choice of the food product manufacturers and for some products even glass bottle has been traditionally used for packaging.

Till recently, the quantum of steel production was considered to be an index of industrialisation of a country. Even today, the quantity of steel, cement and similar products produced in a country is taken as an indicator of its industrialisation. With development of science and technology, alternative materials like plastics have emerged either to replace natural and traditional materials with a benefit of cost reduction purpose, to cater to certain applications which were not possible with the traditional materials, as well as for enhancing utility of traditional materials.

Modern Plastic Based Packages for Confectionery

In the new millenium, use of plastics both in terms of quantity and variety is perhaps the correct index of industrialisation of a country, the status of its economy and the standard of living of its citizens.

Development of plastics received an impetus in last 30 years as natural materials such as wood was not available in adequate quantities; man-made materials such as steel, glass were found to be very expensive or could not provide economically viable solutions for packaging of products of mass consumption.

Traditional Glass Bottles for Packaging of Sauces

A few decades back, scientific tools and technology was not available to measure and predict performance of a traditional or conventional packaging. As a result, tendency of package designer/purchaser was to play safe by using over-designed packages. Stronger packages such as metal containers, glass bottles, etc. were used for quite sometime more due to the anxiety of the product marketer for preserving the product as long as possible. With the development of technology, modern gadgets became available to measure and predict protection and preservation offered by a given packaging form.

In the modern times, product marketer knows precisely the time period (shelf-life) over which the package is required to preserve the product. With the use of plastics, the marketer is in a position to select the right structure, which can give him the desired protection and preservation capabilities required for the product and market

Traditional Tinplate Containers for Packaging of Ghee

Traditional Tinplate Containers and HDPE Containers for Edible Oils needs. Added to this, consumer became more demanding and was not willing to accept inconveniences of traditional and conventional packaging forms. A typical consumer would expect an easy opening package with the features such as easy opening flip top cap or metering or similar devices for retrieving the product in the required quantity and would not like to use instruments such as scissors, knife, etc. to open the packages. Plastic packages have the versatility to provide such convenient features, which boosted development of plastic packaging.

Plastic Packages – Options

In plastics, there are a variety of materials, which are used for packaging applications. The important plastics, which find large packaging applications are HDPE, PP, PVC, Polyester, Polystyrene, etc. These are used for building the body structure of the packages, while other polymers are often used, in less thickness, as coating to improve the functional properties of basic packaging.

Examples of such speciality polymer coatings are Polyvinylidene Chloride (PVDC), Ionomer, Ethylene Vinyl Alcohol (EVOH) and others. These speciality coatings provide specific properties such as high barrier to oxygen, moisture aroma/flavour or it may impart sealability to a package even when its surface is contaminated with product. Sometimes, a speciality polymer coating may also provide a good hot tack resistance over a wider temperature range. In other situations, when rigors of handling and distribution demand higher mechanical strength, plastics can be used to enhance such properties.

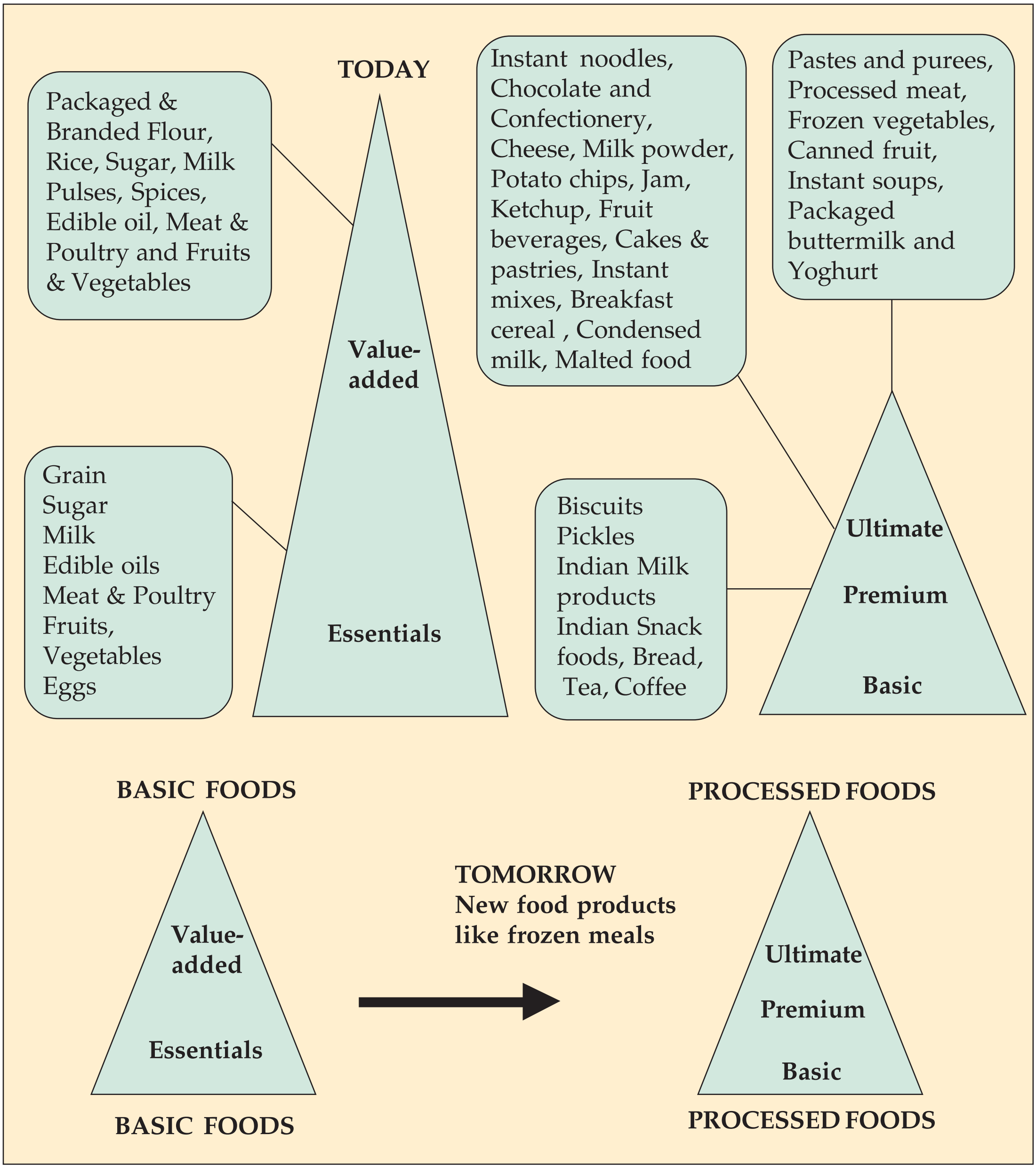
Thus, the advantage of plastics is in the range of mechanical barrier and other properties it offers and also the feasibility of tailoring a structure required to meet precisely the product protection, preservation and distribution needs.

Changing Trends in Food Processing and Packaging Technology

Yoghurt Packed in Traditional Earthern Pot and Injection Moulded Plastic Containers

The changing food consumer in India will be one of the most powerful forces for change in the food industry. Increasing incomes, increasing literacy rates, smaller family sizes, women entering the work force, urbanisation and increasing concern about health and hygiene will be motivating consumer changes in attitudes. Figure 1 depicts the markets for basic food

Figure 1: Today and Tomorrow



and processed food today and in the future, taking into account changing consumer attitudes.

In response to the changing trends, the traditional processing and packaging technologies have either been improved or a few new technologies have progressed to match new requirements.

Table 2 gives the evolution that has taken place in processing and packaging of food.

TABLE 1

Evolution of Processing and Packaging of Food

Past	Present	Future
Canning Pasteurization Freezing Dehydration	Map/Cap system Aseptic system Anti microbial Biosensors/Chemical markers Robotics, computers Infusion technologies, Irradiation, Microwave	Biotechnology High pressure processing Pulsed electric field Pulsed light field

Aseptic Packaging

These new processes either extend the shelf-life of food product or allow more sophisticated product or different packaging concepts. They also improve the nutritional and sensory properties of the food product during its entire shelf-life.

Minimal Processing Technologies for Food Preservation

Consumers increasingly demand food, which retains the natural flavour, colour and texture and contains fewer additives such as preservatives. In response to these needs, one of the most important recent developments in the food industry has been the development of minimal processing technologies designed to limit the impact of processing on nutritional and sensory quality and to preserve food without the use of synthetic additives.

Traditional thermal processing techniques can be both beneficial to food in such areas as preservation and flavour formation but detrimental in damaging other sensory and nutritional properties. Minimisation of undesirable changes can be achieved in a number of ways, whether through more effective process control, the use of High Temperature Short Time (HTST) techniques such as aseptic processing, or newer thermal technologies such as volume heating methods, infrared heating, dielectric methods such as the use of microwaves, and ohmic heating etc. The other alternates to thermal processing, range from irradiation to high pressure processing and the use of pulsed electric fields.

The safety and effectiveness of minimal processing depends on the use of the novel preservation technologies and most notably in packaging.

Numerous electromagnetic energy sources-initially developed for other purposes can now be used for food and other material processing. They are derived from generations of research aimed at much larger targets than the micro-organisms that present threat to the stability and safety of food materials. Currently, a considerable range of thermal and non-thermal processes are being assessed to improve quality and variety.

Minimal food processing technology under thermal and non-thermal methods are given in Figure 2.

Figure 2: Minimal Food Processing Technology

Minimal Processing Technology

Minimal processing of foods with thermal methods

- Thermal conduction, convection and radiation
- Heat processing in the package
- Aseptic and semi-aseptic processing
- Sous-vide processing
- Infrared heating
- Ultraviolet light
- Electric volume heating methods for foods
- Electric resistance/ohmic heating
- High frequency (HF) or radio frequency (RF) heating
- Microwave heating
- Inductive electrical heating
- Improvements in conventional thermal processing

Minimal processing of foods with non-thermal methods

- Ionising radiation
- High pressure (HP) processing
- Methods based on pulsed discharge of a high capacitor
- Pulsed white light
- Laser light
- Pulsed electric field (PEF) or High Electric Field Pulses (HEFP)
- Oscillating magnetic fields (OMF)
- Other non-thermal anti-microbial treatments
- Ultrasound
- Pulse power system
- Air ion bombardment
- Plasma sterilisation at atmospheric pressure

Current Trends in Processing and Packaging Technologies

Details of some of the trends currently prevailing in the food processing are described below:

Aseptic Filling / Processing

In this method, the food material is heated rapidly in a heat exchanger, held for sufficient time to achieve commercial sterility and then cooled before filling into a de-contaminated container within an aseptic zone. It is critical that all food contact surfaces are sterilized before production starts and that any other surfaces in proximity to the food should be sterilized too, in order to avoid re-infection. This is now a well known technology, and in the last few years, there have been interesting developments in the following areas:

- Particulate filling which allows the aseptic filling of large pieces of meat and vegetables allowing new product formulations and market opportunities. The overall quality of the product will be related to the average time and temperature treatment of the particles.
- Bulk aseptic filling which allows new markets such as foodervice applications (sauces, milk based products) and temporary storage (seasonal products). With this concept, most of the bag-in-box systems use gamma irradiated pouches.
- New methods of sterilising machine and package to replace hydrogen peroxide. Another concept would be to use superheated steam.

Aseptic processing has replaced in many applications the traditional heat processed method, as it gave to the product much better sensorial and nutritional benefits.

Within the dairy industry, the UHT treatment has gained a lot of importance to obtain shelf-stable products durable for few months. Various package forms are used from brick packs to pouches and plastic bottles.

As a consequence of application of this technology, more and more clean or ultra clean filling equipment are in use. This concept provides extra days for a refrigerated product and will not be as expensive to run as a fully aseptic process.

For flexible aseptic packages, paperboards coated or laminated with PE, BOPP, PET, EVA blends or aluminium foil or any other suitable combinations are used depending upon the product characteristics and the shelf-life required.

Ohmic / Induction Heating

As a support to aseptic filling, a system has been developed whereby liquid products containing particulate up to 25 mm can be uniformly heated through electric current. This process will prevent destruction of flavours, nutrients and keep particulate integrity.

In ohmic heating processes, food is made part of an electric circuit through which alternating current flows, causing heat to be generated within the food due to the electrical resistance of the food, thus offering a way of processing food at the rate of HTST processes, but without the limitation of conventional HTST on heat transfer to particulate.

This interesting, new continuous heating technology has a potential for commercial applications in the future.

"Sous vide" Cooking

This process is quite new and its main benefit is to retain flavours and juices in processed food. The packed food is cooked under vacuum at low temperature (close to pasteurisation) and then rapidly chilled to 2-3°C and maintained at this low temperature.

Using this method, products can be stored chilled up to 3 weeks depending on the products and the thermal process. Solid products such as roasts, ham and fish can be processed this way. Specific packaging materials have been developed for this application (barrier shrink bag). This simple process allows a centralized production of a high quality product, an optimum cooking yield and requires no skilled labour at the other end (only re-heating).

Another processing/packaging system also proposed is “DARFRESH FTF” (flex-tray-flex) for ready meals which is an extension of the “sous vide” cooking. A rigid tray (aluminium, CPET, PP) containing the food product is “sandwiched” between two flexible film webs. Both films are multi-layer films with a barrier layer. The top film moulds around the foodstuff during the vacuum skin process without crushing the food and sealing is applied between the two films with an easy-open feature built-in the bottom web. The skin package is then cooked under vacuum and then rapidly chilled. Products are sold chilled or frozen. These packages can be reheated in a microwave oven, a conventional oven or in boiling water. A very high degree of hygiene is required for these above-mentioned processing methods.

Microwave Processing

This kind of pasteurisation offers an extension of shelf-life and the short time process inherent to microwave heating will minimise product degradation but has the obvious drawback of microwave cooking, i.e. temperature is not uniform due to the different dielectric properties of food.

This method could be combined with other methods, such as hot air, in order to overcome the issue of non-uniformity of microwave cooking. The microwave processing has been made use of for drying of fruit juices, pulps, apple segments and finished drying of potato chips.

The most commonly used material today is crystallized polyester (CPET) in the form of trays. The advantages of using these trays are with respect to flexibility in the shape and design and resistance to oils and greases. These trays have another distinct advantage of dual ovenability. PP co-extruded with barrier resins such as EVOH is used when a longer shelf-life is required.

“Single-flow FSTP”

This system is designed for thermal processing of liquid food containing particulate of recognisable size. The basic principle is to have particulate being held for any desirable period of time in order to be given any required processing value. Selective holding sections can be used for different particle sizes requiring different holding times. The liquid fraction is sterilised through a conventional tubular heat exchanger.

The main advantage of this system is to reach optional cook values for the various particle sizes, without much thermal degradation of smaller particulate and liquid.

Steam / Vacuum Cooking

This concept consists of steam cooking at 130°C in an open can containing the product. In a very short time, water in the can is replaced by steam thus ensuring product sterilisation. The can is then steamed. The steam cools down, condenses on the product, keeping them moist.

A few benefits are:

- lighter can (no water)
- mineral salts and vitamins retained by the vegetables
- improved texture and flavour

However, a few drawbacks are:

- cost (additional cost of 40%)
- perception of an empty can by the consumer

Food Irradiation

This is not a new technology, gamma-rays are already used in some food applications. This technique involves exposure of food to short-wave energy and is mainly used today to reduce contamination by micro-organisms and pests, delay ripening and for sprout inhibition in vegetables, thus increasing the shelf-life of products and also reducing potential health hazards caused by micro-organisms.

This technique can be used on most fruits and vegetables, meat, poultry, seafood, spices, potatoes, grains etc. This process could be used in combination with others to develop novel food and to enhance overall product quality.

Freeze Concentration

Current efforts are focused on cost-effective dehydration processes, such as osmotic dehydration and the development of ready-to-eat, partially dehydrated intermediate moisture items. This is a means of removing water using freezing phenomenon, rather than evaporation phenomenon. Basically, ice crystals are created and filtered out, leaving behind concentrated products.

Membrane Process Technology

Membrane separation processes are basically based on the ability of semi-permeable membranes of appropriate physical and chemical nature to discriminate between molecules primarily on the basis of size, and to a lesser extent, on shape and chemical composition. Depending on the chemical/physical properties of the membrane, concentration or de-watering (reverse osmosis-RO), fractionation of components in solution (ultra filtration-UF) and classification of slurries or removal of suspended matter (micro filtration–MF) are accomplished. Membranes are commercially available. Cellulose acetate and derivatives are most widely used. A composite membrane containing polyamide and polysulfone or polyethylene is also used. Dairy industry is probably the largest user of membrane technology in food processing.

Hurdle Technology

The most important preservation technique for food is refrigeration. However, because of the difficulty in maintaining sufficiently low temperature throughout the production, distribution and storage chain, additional barriers (or HURDLES) are required to control the growth of spoilage or pathogenic micro-organisms. The concept of combining several factors to preserve food has been developed into the HURDLE effect – each factor is a hurdle that micro-organisms must overcome.

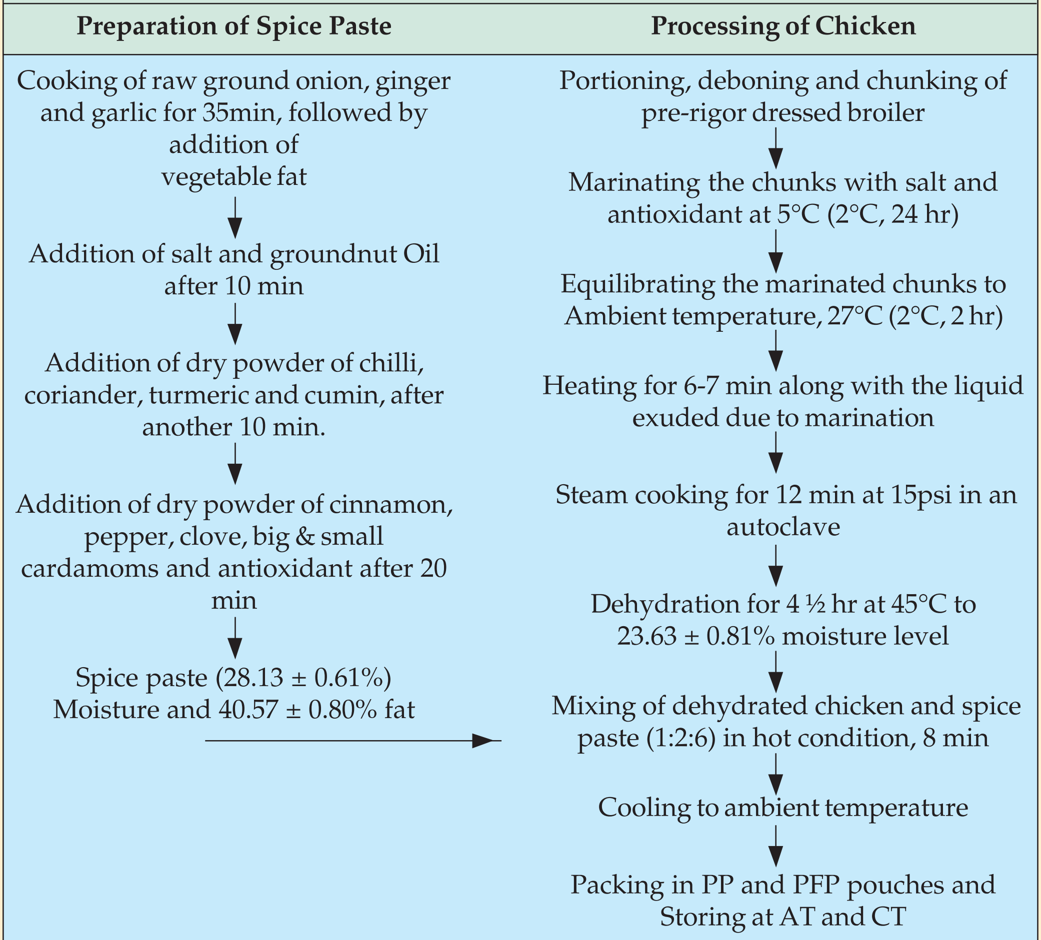
In hurdle technology the complex interactions of temperature, water activity, pH and other factors are understood and these factors are used to design a series of hurdles that will ensure microbiological safety of the food products.

Preservation of Chicken Curry by Hurdle Technology

[Studies carried out at DFRL (Defence Food Research Laboratory, Mysore)]

Intermediate moisture chicken curry was prepared from boneless chicken chunks and spices and was preserved by hurdle technology as per the flow diagram:

Flow Diagram for Hurdle Processing of Chicken Curry



Preparation of Spice Paste

Cooking of raw ground onion, ginger and garlic for 35min, followed by addition of vegetable fat

Addition of salt and groundnut Oil after 10 min

Addition of dry powder of chilli, coriander, turmeric and cumin, after another 10 min.

Addition of dry powder of cinnamon, pepper, clove, big & small cardamoms and antioxidant after 20 min

Spice paste (28.13 ± 0.61%) Moisture and 40.57 ± 0.80% fat

Processing of Chicken

Portioning, deboning and chunking of pre-rigor dressed broiler

Marinating the chunks with salt and antioxidant at 5°C (2°C, 24 hr)

Equilibrating the marinated chunks to Ambient temperature, 27°C (2°C, 2 hr)

Heating for 6-7 min along with the liquid exuded due to marination

Steam cooking for 12 min at 15psi in an autoclave

Dehydration for 4 ½ hr at 45°C to 23.63 ± 0.81% moisture level

Mixing of dehydrated chicken and spice paste (1:2:6) in hot condition, 8 min

Cooling to ambient temperature

Packing in PP and PFP pouches and Storing at AT and CT

[Source: DFRL]

The stability of the product on storage at ambient temperature of 27°C ± 4°C (AT) and chill temperature of 5°C ± 2°C (CT), in pouches of 50 micron Polypropylene (PP) film and laminate of 45 gsm paper / 37.5μ Al foil / 20μ Poly (PFP) was studied by monitoring chemical, microbiological and sensory quality parameters.

Tables 2, 3 and 4 give the effect of storage temperature and packaging on physical, physico-chemical and chemical qualities, microbiological quality and sensory quality respectively, of hurdle processed chicken curry.

TABLE 2

Effect of Storage Temperature and Packaging on Physical, Chemical & Physico-chemical Qualities of the Hurdle Processed Chicken Curry

Parameters	Initial (Freshly Processed)	After 4 Months of Storage			
		AT		CT	
		PP	PFP	PP	PFP
% Moisture content (dry basis), n=6	36.13 ± 0. 54^{a}	35.73 ± 0.47^{a}	35.94 ± 0.22^{a}	35.96 ± 0.29^{a}	36.09 ± 0.35^{a}
% Fat content (dry basis), n=6	31.06 ± 0.73^{a}	30.44 ± 0.57^{a}	30.61 ± 0.33^{a}	30.98 ± 0.47^{a}	30.96 ± 0.31^{a}
% FFA (as oleic acid), n=6	1.31 ± 0.20^{a}	9.58 ± 0.33^{b}	6.86 ± 0.31^{c}	5.11 ± 0.31^{d}	3.72 ± 0.19^{e}
PV (meq peroxide O_2/kg), n=6	7.67 ± 2.07^{a}	58.25 ± 4.06^{b}	32.77 ± 3.42^{c}	19.69 ± 1.67^{d}	11.94 ± 0.93^{e}
TBA (mg malonaldehyde/kg), n=6	0.13 ± 0.06^{a}	0.58 ± 0.14^{b}	0.36 ± 0.05^{c}	0.34 ± 0.07^{c}	0.24 ± 0.03^{d}
pH (25° C), n=6	5.64 ± 0.06^{a}	5.74 ± 0.06^{b}	5.69 ± 0.04^{a}	5.67 ± 0.04^{a}	5.64 ± 0.03^{a}
W-B Shear (Kg/cm) Lengthwise (n = 10) Across	0.059 ± 0.002^{a} 0.122 ± 0.008^{a}	0.053 ± 0.003^{b} 0.107 ± 0.01^{b}	0.055 ± 0.005^{ab} 0.114 ± 0.011^{ab}	0.057 ± 0.01^{ab} 0.119 ± 0.003^{a}	0.058 ± 0.007^{ab} 0.12 ± 0.006^{a}
Reconstitution Time (min), n=3	10^{a}	10^{a}	10^{a}	10^{a}	10^{a}
Shelf-life (Day), n=24	–	116 ± 4^{a}	170 ± 6^{a}	188 ± 5^{c}	285 ± 7^{d}

FFA = Free fatty value
PV = Peroxide value
TBA = Thiobarbituric acid value
W-B shear = Warner – Brazier Shear value
AT = Ambient temperature
CT = Chill temperature
PP = Polypropylene film
PFP = Paper-Aluminium Foil-Polyethylene Laminate

Each value is Mean ± Standard Deviation, Mean values with different superscripts a,b,c etc., in a row differ significantly (P<0.01).

TABLE 3

Effect of Storage Temperature and Packaging on Microbiological Quality of Hurdle Processed Chicken Curry

Parameters	Initial (Freshly Processed)	After 4 Months of Storage			
		AT		CT	
		PP	PFP	PP	PFP
TPC (log cfu/g), n=6	3.76 ± 2.48^{a}	3.32 ± 2.60^{b}	3.44 ± 2.41^{c}	3.67 ± 2.29^{d}	3.59 ± 2.16^{e}
YM (log cfu/g), n=6	2.68 ± 1.46^{a}	2.46 ± 1.34^{b}	2.41 ± 1.25^{b}	2.17 ± 1.31^{c}	2.11 ± 1.17^{c}
SC (log cfu/g), n=6	2.26 ± 1.20^{a}	ND^{b}	ND^{b}	2.11 ± 1.25^{c}	1.98 ± 1.17^{c}
CC (log cfu/g), n=6	ND	ND	ND	ND	ND
SA (log cfu/g), n=6	1.87 ± 1.04^{a}	ND^{b}	ND^{b}	ND^{b}	ND^{b}

TPC = Total Plate Count
YM = Yeast and Mould
SC = Spores Count
CC = Coliform Count
SA = Staphylococcus Aureus
ND = Not Detected
log cfu/g = logarithum of colony forming unit per gram sample
AT = Ambient Temperature
CT = Chill Temperature
PP = Polypropylene film
PFP = Paper-Aluminium Foil-Polyethylene Laminate.

Each value is Mean ± Standard Deviation, Mean values with different superscripts a,b,c etc., in a row differ significantly ($P<0.01$).

TABLE 4

Effect of Storage Temperature and Packaging on Sensory Quality of Hurdle Processed Chicken Curry

Parameters	Initial (Freshly Processed)	After 4 Months of Storage			
		AT		CT	
		PP	PFP	PP	PFP
Colour & Appearance	7.69 ± 0.37^{a}	7.19 ± 0.22^{b}	7.33 ± 0.15^{ab}	7.56 ± 0.28^{ab}	7.60 ± 0.34^{ab}
Texture	7.60 ± 0.63^{a}	7.69 ± 0.41^{a}	7.60 ± 0.52^{a}	7.60 ± 0.37^{a}	7.60 ± 0.38^{a}
Flavour	7.57 ± 0.51^{a}	6.47 ± 0.23^{b}	6.59 ± 0.27^{b}	6.83 ± 0.44^{ab}	7.00 ± 0.59^{ab}
Rancidity	7.53 ± 0.41^{a}	6.15 ± 0.31^{b}	6.53 ± 0.46^{bc}	6.69 ± 0.29^{c}	7.04 ± 0.53^{ac}
Overall Acceptability	7.63 ± 0.23^{a}	6.36 ± 0.29^{b}	6.74 ± 0.35^{bc}	6.95 ± 0.33^{c}	7.24 ± 0.48^{ac}

Nine-Point Hedonic Scale: 9 - Like extremely, 8 - Like very much, 7 - Like moderately, 6 - Like slightly, 5 - Neither like nor dislike, 4 - Dislike slightly, 3 - Dislike moderately, 2 - Dislike very much, 1 - Dislike extremely

AT = Ambient temperature

CT = Chill temperature

PP = Polypropylene film

PFP = Paper-aluminium foil-polyethylene laminate.

Number of panelists (n) = 8. Each value is Mean ± Standard Deviation, Mean values with different superscripts a,b,c etc in a row differ significantly ($P<0.01$)

The results indicate that the chicken curry has a shelf-life of 116 ± 4 and 170 ± 6 days at ambient temperature and 188 ± 5 and 285 ± 7 days at chill temperature in polypropylene and paper/Al foil/poly pouches respectively. The product was found to have satisfactory microbiological quality, satisfactory sensory attributes, acceptable shear value and rapid reconstitution in boiling water in ten minutes. **This indicates that plastics can be used successfully for preserving hurdle processed chicken curry for a fairly long shelf-life.**

Osmotic Dehydration

Dehydrated products are wholesome, nutritious and palatable but they have not found popular acceptance because the products do not retain the flavour, colour and texture of the original fruit and vegetables after re-hydration. Recently, osmotic dehydration, which is the process of water removal by immersion of water containing cellular solids in a concentrated aqueous solution of fruits and vegetables, is gaining attention due to its important role in food processing industry. This is because the product by osmotic process is more stable during storage due to low water activity by solute gain and water loss, as at low water activity, all the chemical reactions to deteriorate the food, the growth and toxins production by micro-organisms are low. Besides that, it improves the quality of the product in terms of colour, flavour and texture and it is less energy intensive process than air or vacuum drying process (Shafiur Rahman 1992).

Studies on Osmotic Dehydration

[Studies conducted at IARI, (Indian Agricultural Research Institute) New Delhi]

The division of Fruits and Horticultural Technology, New Delhi had conducted studies on preparation of onion powder by Osmostic Dehydration.

The osmotic dehydration process as compared to the conventional air or vacuum drying process, offers better quality product in terms of colour, flavour and texture and it is a less energy intensive process, as indicated above.

A large quantity of onion is used fresh, however, some quantity is also processed into dehydrated powder mainly for exports and for armed forces personnel. The product (like any other dehydrated powder) has a tendency to absorb moisture from atmosphere resulting in lumping and loss of free flow characteristics. Physical/chemical changes can also occur during storage if the product is not suitably packed. The study was aimed at identifying suitable packaging and storage conditions.

The studies indicated that onion powder prepared from 5 mm thick osmotically dehydrated onion slices after drying in a cabinet dryer at 60°C for 10 hours, ground and sieved **can be stored up to 6 months in 400 gauge LDPE or 200 gauge HDPE pouches at 7°C with better pungency, compared to 4 months at ambient condition. Retention of colour and pungency was found to be better in the samples packed in 200 gauge LDPE pouches stored at 7°C.** The IMC (Initial Moisture Content) of the product was 3.85% and the corresponding ERH (Equilibrium Relative Humidity) was found to be 47.5% and the CMC (Critical Moisture Content) was 9.14%. The product was more stable at low temperature (7°C) as compared to high temperature (ambient conditions) in respect of colour and flavour.

Table 5 below gives the ERH of onion powder at room temperature.

TABLE 5

Relative Humidity of Onion Powder at Room Temperature (33-35°C)

Equilibrium Moisture Content, %	Equilibrium Humidity, %	Days Required to Reach Equilibrium	Remarks
1.18	10	30	Free flowing, colour normal, onion flavour
1.24	20	28	Free flowing, colour normal, onion flavour
2.18	30	26	Free flowing, colour normal, onion flavour
3.68	40	24	Free flowing, colour normal, onion flavour
5.22	50	23	Granulation, free flowing, normal colour
9.14	60	20	Colour faded, caking observed
14.47	70	18	Pasty consistency and slight darkening
21.94	80	14	Caky and mould affected after 17 days
31.23	90	9	Wet caky and mould growth appeared after 11 days
42.65	100	6	Mould growth appeared after 9 days

Tables 6 and 7 indicate the changes in chemical constitutents of onion powder stored at room temperature and at 7°C, as recorded by IARI.

TABLE 6

Changes in Chemical Constituents of Onion Powder Stored at Room Temperature (d.w.b.)

Salt Concentration, %	Storage Period, Months	Packing System	Moisture, %	Acidity as Pyruvic Acid, %	Reducing Sugars, %	Total Sugars, %	NEB O.D. at 420 nm
0	0	–	4.30	3.32	17.05	43.62	0.361
	6	200 g LDPE	6.58	3.18	18.70	44.05	1.82
		400 g LDPE	5.90	3.22	18.80	44.60	1.71
		200 g HDPE	5.75	3.28	19.22	43.62	1.58
5	0	–	2.15	3.30	18.61	42.11	0.093
	6	200 g LDPE	6.29	3.17	18.91	44.56	1.70
		400 g LDPE	5.89	3.28	18.52	43.64	1.59
		200 g HDPE	5.69	3.28	18.07	42.92	1.48
10	0	–	2.20	3.28	17.33	41.58	0.092
	6	200 g LDPE	3.58	3.23	18.61	46.57	1.76
		400 g LDPE	3.43	3.28	18.27	46.42	1.44
		200 g HDPE	3.33	3.26	18.25	46.28	1.03
15	0	–	2.23	3.24	17.10	41.47	0.09
	6	200 g LDPE	4.73	3.21	18.72	47.58	1.88
		400 g LDPE	3.54	3.22	18.58	47.28	1.34
		200 g HDPE	3.45	3.23	18.28	47.22	1.24
20	0	–	2.25	3.25	16.73	41.42	0.091
	6	200 g LDPE	3.53	3.21	16.91	46.28	1.55
		400 g LDPE	3.51	3.23	16.82	45.37	1.37
		200 g HDPE	3.41	3.23	16.81	45.28	1.20

g = Gauge, LDPE = Low density polyethylene, HDPE = High density polyethylene, NEB = Non enzymatic browning

TABLE 7

Changes in Chemical Constituents of Onion Powder Stored at Low Temperature (7°C) (d.w.b.)

Salt Concentration, %	Storage Period, Months	Packing System	Moisture, %	Acidity as Pyruvic Acid, %	Reducing Sugars, %	Total Sugars, %	NEB O.D. at 420 nm
0	0	–	4.30	3.32	17.05	43.62	0.362
	6	200 g LDPE	5.21	3.10	18.60	44.92	1.80
		400 g LDPE	4.68	3.12	18.22	44.91	1.69
		200 g HDPE	4.52	3.18	18.12	44.81	1.55
5	0	–	2.15	3.30	18.61	42.11	0.093
	6	200 g LDPE	4.65	3.21	18.59	44.93	1.78
		400 g LDPE	4.51	3.24	18.22	44.84	1.67
		200 g HDPE	4.42	3.25	18.13	44.82	1.46
10	0	–	2.20	3.28	17.33	41.58	0.092
	6	200 g LDPE	3.29	3.23	18.60	46.87	1.75
		400 g LDPE	3.17	3.25	18.52	46.07	1.44
		200 g HDPE	3.14	3.25	18.50	46.01	1.03
15	0	–	2.23	3.24	17.10	41.47	0.09
	6	200 g LDPE	3.77	3.17	18.92	47.38	1.78
		400 g LDPE	3.75	3.18	18.28	47.27	1.35
		200 g HDPE	3.08	3.18	18.23	47.25	1.03
20	0	–	2.25	3.25	16.73	41.42	0.091
	6	200 g LDPE	3.50	3.22	17.58	46.19	1.75
		400 g LDPE	3.25	3.22	17.57	46.12	1.311
		200 g HDPE	3.10	3.24	17.36	46.10	1.03

g = Gauge, LDPE = Low density polyethylene, HDPE = High density polyethylene, NEB = Non enzymatic browning

Some of the trends in packaging systems are modified atmosphere packaging / controlled atmosphere packaging/ gas flushed/ vacuum packaging and retortable pouch system.

MAP/CAP System (Modified Atmosphere Packaging / Controlled Atmosphere Packaging)

The change of the normal composition of air surrounding the product (within the pack) is one of the most effective methods of extending the shelf-life. MAP is a system where there is no control of the final atmosphere inside the package. The air inside the package is replaced partially or completely either with a single gas or mixture of gases or with vacuum, while, in CAP system, the atmosphere within the package is adjusted or controlled to specific requirement. Packaging materials with suitable barrier properties are used.

Generally, the three gases most commonly used are:

- Carbon dioxide to retard the growth of micro-organisms, taking enough care to prevent CO_2 absorption and collapsing of the pack.
- Nitrogen is largely used to displace oxygen and delay oxidation, it is also used as a filler gas to prevent vacuum to occur.
- Oxygen is reduced to as low as possible, except in some specific applications e.g. in meat where colour retention is desired.

Right gas mixture has to be established for each product taking into account the product nature and its requirements, potential gas absorption and proper use of barrier packaging material, With this technology, fresh products can have a shelf-life extended by a few days to a few weeks.

For MAP system, film with anti fog property viz. PS, PVC films are used where as for gas/ vacuum packaging very low permeability structures with good oxygen barriers such as EVOH, PVDC, Polyesters and Nylons are used.

Pre-packaged Fresh Vegetables

Fresh Strawberries Packed in Plastic Punnets

For MAP of perishables, such as fresh fruits and vegetables, which respire and meat products which need to retain the red colour, polymeric films are most suitable for use due to their permeability. Conventional materials like glass and metal are non-permeable to moisture vapour and gases and, therefore, would not be suitable for this application.

Conventional materials like glass and metal containers can be used for MAP of processed and shelf-stable food products, however, these are not cost effective, heavy in weight and fragile (in case of glass).

Retort Pack System

A retort pouch is a flexible, laminated pack having sufficient strength and heat resistance to allow it to be used in place of a metal can for the heat processing and storage of food products. The retort pouch, a flexible laminated food package, can withstand thermal processing and combines the advantages of metal can and the boil in bag and **can be used as an alternative to metal can as plastics are lighter in weight and more cost effective.** Retort pack systems are stable at ambient conditions for a long period.

It is perhaps the most significant advancement in food packaging since the development of the can. The growth of retort pouch system is rapid and dominant form of packaging for heat processed foods. The aim of retorting operation are three-folds:

- To cook the food so that it is in a form for consumption by the consumer with only a minimum further preparation.
- To destroy chemical enzymes naturally present within the food, which could lead to chemical deterioration of the food during storage.
- To destroy micro-organisms within the food to give a condition of commercial sterility, which implies the destruction of all pathogenic i.e. food poisoning, organism, together with all spoilage organisms capable of metabolism under storage conditions.

Retortable flexible plastic pouches are generally constructed from the following combinations :

- PET/Adhesive/Al foil/Primer/Adhesive/PP
- PET/PVDC/PP
- Nylon/PVDC/Nylon/Tie/Sealant/Extrudate/Sealant
- PP/Regrind/Tie/EVOH/Tie/Regrind/PP

Future Trends in Food Processing Technology

Other packing technologies envisioned for the 21st century are non-thermal processes, Pulsed Electric Field (PEF), High Pressure (HP) Preservation, Pulse Light Processing and Oscillating Magnetic Field Technology.

Pulsed Electric Field (PEF)

PEF processing is applied to liquids or pumpable products using a flow-through treatment chamber. In PEF, the flow of current is switched in very high intensity electric fields. It is a very high intensity varying electric field, through which the food passes. The temperature increase is minimal. The micro-organisms are deactivated via electro-permealization of the membrane.

Pulse Light Processing

Pulse Light Processing is an irradiation system rather than a penetration system. It is primarily for surface kill (i.e. inactivation of micro-organisms on surface, killing micro-organisms in a liquid through a very thin film).

Oscillating Magnetic Field Technology

Oscillating Magnetic Field Technology is similar to PEF but instead of using an electric field, a high intensity magnetic field is used, which inactivates micro-organisms and reduces their growth.

High Pressure (HP) Preservation

HP Preservation can be applied to either liquid or solid foods in flexible containers. The improved food preservation technologies are being used to:

- Minimize processed-induced loss of colour, flavour, texture and nutrition
- Retain the highest possible quality in stressful storage environments
- Reduce the logistics burden and cost vs. conventionally processed foods

Because non-thermal processing is relatively new compared to conventional processing, two key issues are particularly critical for PEF and HP technologies. The first major issue is safety and process assurance; the second is sensory quality and consumer acceptance over the intended shelf-life.

Industrial adoption of advanced food processing technologies will contribute to a more flexible and responsive logistics system.

Trends in Packaging Materials

High Barrier Packaging Materials

High barrier is the term, which has been associated with those plastics whose permeabilities are low enough to significantly prolong the shelf-life of food. Barrier packaging is one of the industry's most exciting developments, which has witnessed internal and external coatings, oxygen absorption and other barrier systems competing for a wide range of food and beverage applications.

Barrier packaging is designed to keep oxygen, moisture and carbon dioxide out of the packaging to preserve the flavour, colour, odour and freshness of its contents. With gas-fill techniques, barrier packaging retains CO_2 or N_2 to protect the product and extend its useful shelf-life.

Barrier resins include plastics viz. ethylene-vinyl alcohol (EVOH) copolymer, polyvinylidene chloride (PVDC), polyamide or nylon (PA), cyclic olefin (COC) copolymer, cyclodextrin (CD), MXD6 and others.

- **Ethylene Vinyl Alcohol (EVOH) Copolymer:** EVOH resins are non-toxic and can be used in direct contact with food products and also for high temperature lamination used in meat and cheese packaging. EVOH can be used in multi-layered films with other polymers. In co-extrusion technology it is used as a tie layer.

  EVOH has excellent gas barrier properties and flavour retention properties. It is effective in retaining the modified atmosphere inside the MA package. EVOH is an excellent choice for packaging of oily foods and edible oils.

Modern Plastic Based Packages for Food Products

- **Polyvinylidene Chloride (PVDC):** PVDC film is a clear film with good strength characteristics specially burst stretch. It is heat sealable at low temperatures (120°C to 150°C). It has low permeability to water vapour and gases and even a thin film can provide good barrier properties. Foods for vending and microwave re-heating can be wrapped in PVDC films because of its resistance to hot fats and oils and because it does not melt or fuse while it is in microwave cooking. These films known as Saran are used for cheese, meat, sausages and dried fruits wrapping.
- **Polyamide (PA) / Nylon:** Nylon 6 is widely used in food packaging for its dry barrier properties, toughness and heat and grease resistance. Semi-crystalline PA resins when dry, limit the transfer of gases and food components such as flavours and aromas, and are therefore used in multi-layer barrier constructions with other plastics for packaging of products such as meat, cheese, and edible oils.
- **Metaxylylene Diamine (MXDA):** MXDA is offered as a bi-axially oriented film. This crystalline polyamide differs in properties from Nylon 6, in presenting a significantly high barrier to gas and moisture transmission and greater strength and stiffness when extruded and bi-axially oriented film. The relative insensitivity of MXD6 to moisture is its most noteworthy property when compared to other polymers in use.
- **Liquid Crystals of PET (LCP):** LCP are also high gas barriers. A new option is being examined in terms of its potential for use in packaging films. LCPs are expected to be used in LCP/thermoplastic co-extruded structure for high barrier packaging.
- **Metal Oxide Coated (MOC) and Silicon Coated Films:** MOC films are environmentally friendly transparent barrier films. These coated films are available as:
    - Standard Grade: High barrier transparent film, where aluminium oxide is deposited on PET film.
    - High Barrier Grade: "Ultra" barrier transparent film, where aluminium oxide is deposited on PET film.
    - Retort Grade: Retortable high barrier transparent film, where aluminium oxide is deposited on PET film.
    - Anti-sticking Grade: Anti-sticking high barrier transparent film, where aluminium oxide is deposited on an OPA film.
    - SiOx Grade: High barrier transparent film, where silicon oxide is deposited on a PET film.

These films have wide applications in dried/dehydrated food packaging and in retortable packages.

- **Semi-rigid Thermoforming Films:** Expanded polyester is combined with EVOH to result in PE/EVOH/PE/PET matrix. Clear trays with straight or tapered walls and depths of 35-60mm can be prepared. The trays are best suited for packing fresh meat, fish, poultry and deep frozen foods.
- **Combitherm Films:** This new development is a PA/PE based film that has oxygen barrier as well as an oxygen scavenging layer, which absorbs any oxygen molecules still inside the package, even after evacuation. The scavenging layer is embedded in the film and does not come into contact with the packed product. The film remains

unimpaired during high temperature sterilisation that is used to prolong the shelf-life of a food product.

- **Multi-layer Barrier PET Bottles:** Compared to the conventional monolayer PET bottles, the barrier coinject – higher barrier bottles with PET + EVOH would have 100% improvement in the barrier properties.

  The higher temperature resistant PEN-PET bottles have been developed which have temperature resistance up to 90°C and can withstand sterilisation temperatures.

  PET bottles are developed with Amorphous Carbon Treatment for coating a layer of highly hydrogenated amorphous carbon, obtained from food safe gas in the plasma state. Oxygen barrier properties are increased 30 times and CO_2 barrier properties are increased 7 times as compared to the conventional PET bottles.

  The major applications of barrier PET bottles are for packaging of products such as beer, fruits juices, milk and tomato ketchup.

Future Developments in Plastic Material

Bio-degradable Plastics (Films)

Interest has been created to consider the use of packaging materials, particularly films which are bio-degradable to overcome the hottest issue of ecological problem becoming serious because films are light weight and bulky. Some of the bio-degradable plastics are already commercialised with improved performance and reduced costs. Research work is still going on to bring about further improvements and innovate newer materials. Some of the major bio-degradable plastics are briefly listed below:

- One of the earliest bio-degradable plastics was polyhydroxybutyrate valerate (PHBV), a bacterially grown polyester with properties similar to PP. This was discontinued later in 1998.
- Starch based plastics constitute a major volume of biodegradable plastics. By carefully using selected starch feedstock and water as a plasticizer, thermoplastics from nearly 100% starch or from blends of starch with other biodegradable components are produced. Many of these materials are water soluble in addition to being biodegradable.
- Biodegradable plastics based on lactic acid are available for packaging applications.
- The other biodegradable plastics include protein-based plastics, polysaccharides, polyoxyethylene, which is water-soluble, polyvinylalchohol etc.

Speciality Additives in Polymers

Additives such as pigments, master batches, antioxidant, antistatic agent, slip additives, UV stabilisers etc. are some of the commonly used and known additives. Some of the not so common additives with great potential are:

- Antifogging agents in transparent bottles or films for packaging of beverages and perishables.

- Nucleating agents for obtaining clarity in polymers such as PP, Adipic acid, Benzonic acid and some metal salts of these acids are used for PP. For Nylon, collodial silica is commonly used. With these additives, PP sheets can be made highly transparent, and has a large market segment potential for thermoformed disposables for food packaging.
- Other additives include slip additives for smooth operation on FFS machines, anti-microbial additives for preventing bacterial growth.
- Active and Intelligent Packaging: A lot of action in this field is going on, particularly with the growth of moisture controllers, ethylene absorbers, oxygen scavengers and theft deterrents in the ranks. Perhaps, a time has come to control internal relative humidity and oxygen, remove undesirable odours and disperse anti-microbials and desirable aromas into package interiors from a single package surface.

Plastics Industry and its Growth

Packaging is constantly evolving to meet the changing demands of the society. A major change has been our ability to produce and preserve products with packaging. Plastics with their unique combination of properties are the key in meeting these demands in a sustainable manner in both the developed and the developing world. In India, an impressive growth is recorded vis-a-vis other packaging materials as shown in Table 8.

Share of various packaging materials in the Table 10 shows not much of a deviation from the past except that the share of plastics is increasing from 4% in 1983 to 12% in 1997 and to over 32% by 2000.

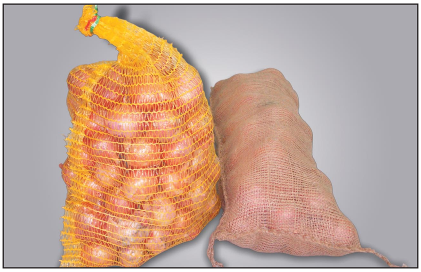
Plastics have made significant inroads at the global level as well as in the life of Indian consumers. The global consumption of plastics went up from 154 million metric tonnes in 2000 to 183.5 million metric tonnes in 2003, and is projected to touch 300 million tonnes in 2010. By 2020 the expected global demand is 540 million tonnes with the overall annual growth rate of 6% to 7% in the next few years. Table 9 gives a comparison of plastic consumption by major world areas.

TABLE 8

Growing use of Plastics in Packaging

Material (by weight)	Consumption Level (%) during Different Years		
	1983	1997	2000
Paper & Paper board	33	30	22
Glass Containers	15	22	15
Tin-Plate & Steel	16	13	9
Plastics	4	12	32
Composites	1	4	10
Aluminum	1	1	2
Jute	30	18	10
Total	100	100	100

{Source: Plastics for Environment and Sustainable Development (ICPE/CIPET, Ministry of Chemicals & Fertilisers}



Traditional Jute Bags and Knitted Plastic Bags for Onions

Modern Plastic Based Packages for Food Products

In India, however the consumption of major plastics is about 4% of the global consumption. According to the latest available data, the polymers consumption in India will touch 12.5 million metric tonnes in 2010 from 4.4 million metric tonnes in 2003, registering a compounded annual growth rate (CAGR) of 14%.

TABLE 9

Plastics Consumption by Major World Areas (Year 2000)

Region	Plastics Consumption ('000 metric tonnes)	Population (Million)	Per Capita Consumption (Kg / annum)
Europe W, C, E	40,000	450	90
Eurasia, Russia & Others	4,000	285	14
North America	45,000	310	145
Latin America	11,000	500	22
Middle East, incl. TR	4,000	200	20
Africa, North & South	2,500	190	13
Other Africa	500	610	<1
China	9,000	1,285	14
India	4,000	1,025	4
Japan	11,000	125	90
Other Asia Pacific, rest	13,000	1,120	11
Total World	1,54,000	6,100	25

[Source: Indian Rubber & Plastics Age - 7]

This growth rate is likely to sustain and by the year 2010, India is expected to be the third largest consumer in the world at 12.5 million metric tonnes as against the US consumption level of 39.9 million metric tonnes and China's consumption level of 37.5 million metric tonnes.

On the basis of the CAGR of polymers, India is leading the world at 14.1% followed by CIS-9.1%, China-8.1% and the US at 6.6%.

The per capita consumption of plastics in India has improved to 4kg/annum in 2002 from less than 1 kg a decade ago and still lagging behind the world's average of 20 kg/annum.

At the global level, the higher growth sectors or demand drivers for plastic consumption are consumer and bulk package, building construction, electrical and electronics, automotive, consumer goods, medical, telecommunications, furniture and household applications. The output value of commodity engineering and high performance polymers was US $ 115 billion, accounting for about 7% of total chemical output value globally.

The estimated growth of plastics in India in the various end use segments is shown in Table 10.

TABLE 10

Growth of Plastics in Various End Use Segments

Segments	CAGR (%)
Packaged Consumer Goods	19
Other Consumer & Industrial Packaging	14
Automobiles	12
Consumer Durable Goods	11
Fibres & Multi filaments	13
Infrastructure	9
Agriculture	12
Healthcare	15
Miscellaneous Applications	11

[Source: Financial Express, May 26, 2003.]

Table 10 depicts that "The Packaged Consumer Goods" segment is likely to achieve highest growth rate in polymer consumption. One of the major factors for this high growth rate is the increasing awareness of Indian consumers for genuine unadulterated, hygienic and safe, quality products in convenient unit packs.

Factors for Growth

There are a number of factors that are driving growth and new developments in the plastic-based packaging materials for food and other consumer products:

- Enhanced polymer manufacturing capacities in India
- Availability of wide range of materials indigenously
- Growing awareness about plastics
- Changes in lifestyles of urban and rural middle class
- Entry of multinational companies
- Shift from bulk to smaller consumer packs

The consumption of plastics in India in different sectors is given in Table 11.

TABLE 11

Consumption of Plastics in Different Sectors

Sectors	Consumption (%)
Building and construction	25
Telectronics	17
Packaging	15
Agriculture and water management	25
Transport	5
Others	13

[Source: Popular Plastics and Packaging, Feb. 2003]

At the global level plastic consumption sector-wise and resin-wise along with the corresponding growth rate from 2000 - 2010 is given in Table 12.

TABLE 12
Global Plastic Consumption, by Resin

('000 MT)

Plastics	Consumption
Commodity	**1,48,000**
PVC	26,000
LDPE	17,000
LLDPE	14,000
HDPE	23,000
PP	30,000
PS	10,500
EPS	2,600
ABS	3,800
Other styrenics	1,000
PET bottles	6,700
PET film, other	1,500
PUR	8,500
PMMA	1,000
UP	1,000
Thermosets	400
Epoxy	1,000
Engineering	**5,510**
PA	1,800
PC	1,500
POM	600
PPE	310
PBT PET	500
Alloys	800
Speciality	**213**
PTFE	70
Other FP, PVDF	30
PPS	54
PSU	27
PES	5
LCP	18
PEEK, PAEK	6
PEI	6
PI	5
All others	**1**
Total (rounded)	**1,54,000**

[Source: Indian Rubber And Plastics Age, June 2003]

Polymers, is probably the single largest family in terms of varieties and types of packaging media catering to multitude of applications. About 50% of the polymers produced/consumed domestically are estimated to be used by the packaging sector. A lion's share of this goes for food applications. Food packaging creates opportunities for more innovations as this sector is constantly and continuously influenced by the changing consumerism and market trends.

Plastics used in food packaging can be in the form of wrappers, pouches, bags, bottles, cans, trays and tubs as primary structures; seals, closures and overwraps; labels and tamper-evident devices, shrink bands, tear strips, etc as secondary packages and components; and as tertiary package such as in shrink wrap around pallet loads of boxes and cartons.

Packaging forms are broadly classified as:

- Flexibles – films and laminates, woven fabrics.
- Semi rigids – extruded and laminated tubes.
- Rigids – moulded, blow moulded, sintered and thermoformed products. Material handling products such as crates, pallets etc.

The use of plastics in the above types of packages is given in Table 13.

TABLE 13

Use of Plastics for Packaging in India

(Quantities in million tonnes)

Type of Packaging	Year 2000	Year 2010*	Growth
Rigid packaging	0.4	1.8	16%
Films	0.6	3.0	17%
Woven sacks / multi-filament	0.5	1.4	12%
Total	**1.5**	**6.2**	**16%**

* Estimated

[Source: Presentation "Polymer Industry a new Decade"
Mr. K.P. Nanavaty, RIL, Mumbai]

Flexible packaging products are gaining acceptance in the market due to their characteristics and benefits like variety and reduction in size, convenience of storage resulting in space saving, ease of opening and re-closing, light weight, disposability, cheaper than rigid packaging in most cases and effective in prevention of adulteration.

Flexibles as Economical Media

If one compares the product sale cost v/s the packaging cost for various types of packages, the most economical pack would be a flexible pouch.

It will be useful to compare traditional (conventional) packages made using modern technology such as metal can, glass bottle etc. with packages made from plastic materials.

Plastics Packages	**Conventional Packages**
Require low energy inputs for manufacturing and conversion	Require high energy inputs for manufacturing and conversion
Can be moulded to different shapes and sizes	Constraints in making odd shapes and sizes
Limitations in decoration with direct printing of multi colour graphics	Can be decorated by directly printing with multi colour graphics
Limitations for filling very hot products	Hot products can be filled
Limitations in treatment/sterilisation with steam or high temperature dry air	Can be treated/sterilised by steam and high temperature dry air
Proven storage trials required for food product compatibility	Normally compatible (with lacquer in some cases)
Mechanical properties - relatively lower	Mechanical properties - relatively better
Recycling by heating/melting is easier	Recycling by heating/melting is not easy/possible
Not perceived by consumer as a premium product packaging	Positively perceived by the consumer as a premium product packaging
Relatively low cost packages	Cost is on the higher side
Low tare weight (of empty packages)	High tare weight (of empty packages)

The materials used for laminates broadly fall into three categories – Paper, Aluminium foil and heat-sealable plastic films like Polyethylene, Polyester, Bi-axially Oriented Polypropylene (BOPP) and Cast Polypropylene.

Most of these laminates are made into pouches, bags, sachets by heat sealing and therefore the innermost ply of the laminates is heat-sealable. The other layers are selected depending upon the properties required from the pouch.

Flexible converting industry could be classified as:

- Adhesive Laminating Industry
- Co-extrusion Industry
- Extrusion Coating Industry

Amongst all the above, the adhesive laminating industry is the most rapidly growing and adhesive laminates are widely used as packaging media in the processed food industry. Some of the processed food sectors, where adhesive laminates are used are given below:

- Chocolate Bars
- Spices
- Baby Foods / Malted Foods
- Milk Powder
- Fruit Juices
- Suparies / Pan Masalas

- Gems like confectionery
- Biscuits
- Tea
- Extruded Foods / Snack Foods
- Coffee

The products that go in packages made by extrusion coating are as follows:

- Confectionery
- Biscuits
- Bread
- Tea

Co-extrusions are not generally referred to as laminates and are usually called films. It involves combining two or more layers of plastics into a multi-layer film. Up to five layers can be co-extruded in a single step. Co-extruded films are widely used in milk, vanaspati and oil pouches.

The demand estimates for adhesive laminates, extrusion coated films and co-extruded films are given in Tables 14, 15 and 16 respectively.

TABLE 14
Flexible Packaging Media in the Food Sector
Adhesive Laminates

(in tonnes)

Product	**Growth %**	**Packaging Material**	**1998-99**	**1999-2000**	**2004-05**
Chocolates Bars	15	Foil POLY/PET POLY	1,587	1,825	3,670
Gems Like Confectionery	15	BOPP/PLY/PET/POLY	396	456	917
Biscuits	15	PET or BOPP/POLY	5,951	6,843	13,765
Tea	20	PET POLY	10,800	12,960	32,248
	2	Foil/Paper	1,040	1,061	1,171
Extruded Foods/ Snack Foods	20	PET or BOPP/POLY	12,960	15,552	38,698
Coffee	10	PET/POLY Glass/POLY	1,815	1,996	3,215
Spices	15	PET/POLY	1,719	1,977	3,976
Baby Foods/Malted Foods	15	Foil/POLY	5,951	6,843	13,765
Milk Powder	15	Foil/POLY	158	182	367
Fruit Juices	10	Foil/POLY	423	465	750
Suparies / Pan Masalas	25	PET/POLY	39,062	48,828	1,49,011
Total			**81,862**	**98,988**	**261,553**

[Source: IIP – Working Group Report, MFPI]

TABLE 15

Flexible Packaging Media in the Food Sector

Extrusion Coatings/ Laminates

(in tonnes)

Product	**Growth %**	**Packaging Material**	**1998-99**	**1999-2000**	**2004-05**
Confectionery	5	Wax Paper	275	289	369
Biscuits	5	Wax Paper	2,205	2,315	2,954
	5	Glass/Poly	1,323	1,389	1,772
		Paper/Poly	4,000	4,000	4,000
Bread	5	Wax Paper	19,293	20,258	25,855
Tea	5	Paper/Poly	330	347	443
Total			**27,426**	**28,598**	**35,393**

[Source: IIP – Working Group Report, MFPI]

TABLE 16

Flexible Packaging Media in the Food Sector

Co-extruded Films

(in tonnes)

Product	**Growth %**	**Packaging Material**	**1998-99**	**1999-2000**	**2004-05**
Edible Oil	20	3 & 5 Layered Films	9,504	11,404	23,649
Vanaspati Ghee	2	LD-LLDPE and Nylon Based Films	1,591	1,623	1,757
Milk	5	LD-LDPE or LLDPE	61,354	64,421	78,305
Salt	20	LD-LLDPE	3,024	3,628	7,524
Total			**75,473**	**81,076**	**111,235**

[Source: IIP – Working Group Report, MFPI]

Semi Rigid and Rigid Plastic Containers

Plastics have played a major role in containing and protecting foods. Their contribution to the world's food protection and distribution system is immense and growing. They have been widely adopted in packaging of foods for the advantages they offer and their versatility. Foremost among the features are their formability into unlimited range of shapes and sizes to suit most end use applications.

Typical examples of rigid and semi-rigid forms include drums, bottles, containers, carboys, jars, cups, trays, crates, caps, closures, lids etc. Some common applications in food packaging are given in Table 17.

TABLE 17
Common Applications in Food Packaging

Plastic Type	Package Type	Application
LDPE	Bottles and containers	Fruit juices
HDPE	Bottles, containers, jars Carboys & drums	Edible oils, vanaspati, dairy products
PP	Bottles and jars	Edible oil, milk powder
EPS/HIPS	Cups, trays & Containers	Processed food, dairy products
PVC	Bottles, cups, trays	Mineral water, edible oil, processed foods
PET	Bottles and jars	Edible oils, hot & cold beverages, malted foods, preserves.

The total demand for polymers in rigid and semi-rigid forms is given in Table 18.

TABLE 18
Demand in Rigid & Semi Rigid Forms

(million tonnes)

Year	LDPE + HDPE + LLDPE	PP	PVC	PS	PC	PET	Total
1994-1995	80	40	35	16	0.5	14	185.5
1995-1996	97	50	42	22	1	30	242
1996-1997	108	60	54	28	2	50	302
1997-1998	127	70	77	35	3	70	382
1998-1999	151	80	92	42	4	90	459
1999-2000	171	90	117	50	5	115	548
2000-2005 (estimated)	366	203	395	157	55	1044	2220

[Source : I I P, Working Group Report - MFPI]

As per the Packaging Consortium Report (for IIP), the packaging markets in India for different plastic based packages is given in Table 19.

TABLE 19

Packaging Market – India

Packaged Type	Variants	Market Size
Plastics		
Rigid Containers	Injection Moulded	42,000 MT
	Blow-Moulded (Growth Rate 15% pa)	120,000 MT
	PET bottles (Growth Rate 50% pa)	150,000 MT
Tubes	Total Multi-layer Tube Demand (Growth Rate 10% - 12% pa)	2,000 million nos.
	Seamless Tubes (Premium)	150 million nos.
Speciality Films		
BOPET Films	Total Indian Demand	93,000 MT
	Packaging Usage (Growth rate 3% - 4% pa)	85,000 MT
	World Packaging Usage (Growth rate 4% pa)	1,285 million MT
BOPP Films	Total Indian Demand	70,000 MT
	Packaging Usage (Growth rate 15% pa)	70,000 MT
	World Packaging Usage (Growth rate 6% - 7% pa)	2.9 million MT
Flexible Packaging		
Laminated Films	Speciality Laminates	300,000 MT
	Co-ex/ Mono Layer Films	300,000 MT
	TQPP Films (Growth Rate 15% - 20% pa)	100,000 MT
Woven Sacks	Total Market	400,000 MT/yr.
	Break-up of Market	
	HDPE	150,000 MT/yr.
	PP	250,000 MT/yr.
Short-term growth will be very high due to dilution of JPMA.		

[Source : IIP (I PC Feasibility Study – Stage 1)]

Plastics in Packaging Systems

Aseptic Packaging System

Aseptic processing and packaging system basically comprises of the following:

- Sterilisation of the products before filling
- Sterilisation of packaging materials or containers and closures before filling
- Sterilisation of aseptic installations before operation (UHT unit, lines for products, sterile air and gases, filler and relevant machine zones).
- Maintaining sterility in this total system during operation, sterilisation of all media entering the system, like air, gases, sterile water.
- Production of hermetic packages

Recent efforts of introducing aseptically packaged milk and milk derivatives in flexible pouches has tremendous scope to improve the markets for aseptically packed products for extended shelf-life at affordable prices. Similar development on aseptic flexible pack for fruit juices and fruit drinks can also result in rapid expansion of markets for such products, as there is growing awareness for consuming them for reasons such as better health and fitness.

Retortable Packaging System

Developments in plastic materials and thermal processing technologies have made it possible for the many Indian dishes to be available in ready-to-eat form. The retort pouches used in this case for packaging need to be kept in boiling water prior to serving the food. These "heat and eat" products are preferred for the ultimate convenience they offer to the consumer. They are shelf-stable, which means that they can be stored at room temperature without requiring refrigeration and, in their packed form, remain fresh for over one year. And all this without any added preservatives. The production of shelf-stable products is attained by application of heat that kills organisms, which if not destroyed, will multiply and produce enzymes, which may decompose the food and in some cases produce food poisoning toxins. The main requirement of plastic material is not only to withstand rigors of heating and cooling process, but also to maintain the overpressure correctly so that internal pressure developed during processing is balanced by the pressure of heating system. Retort pouches are flexible packages made from multi-layer plastic films with or without aluminium foil as one of the layers.

Retort trays are semi rigid containers with a high barrier plastic as one of the layers. The material commonly used in retortable container structures include polypropylene, PET, ethylene vinyl alcohol (EVOH) and polyvinyledne chloride (PVDC).

Ethnic food and delicacies like biryani and varieties of non-vegetarian and vegetarian curries can be made available in such retort pouches/trays. The continuing emphasis of Indians for ethnic taste and a growing tendency to minimise time allocation to cooking by Indian women, primarily due to busy schedule and changing lifestyles augur well for introduction of such ready-to-heat and eat products in retort pouches/trays. Popularity of such packaged food will further boost demand for suitable polymers for such applications in coming days.

Micro Ovenable Packaging

Foods packaged for microwave heating and cooking have become among the fastest growing categories of convenience foods. A microwavable package is one, which can be used for cooking or heating the product in a microwave oven. The package alters the heating pattern of the contained food, by releasing or trapping the water vapour inside the package, thereby cooking the product under controlled temperature and pressure.

The different types of materials used to pack micro–ovenable popcorn are:

- Paper / Adhesive / Met PET susceptor with OPP overwrap
- Two PET layers coated with PVDC
- Paper bag overwrapped with OPP / Tie layer / EVOH / Tie layer / Ionomer

All the above packs are shelf-stable.

MAP (Modified Atmosphere Packaging)

The most important methods to extend the shelf-life of food are Controlled Atmosphere Packaging (CAP), Modified Atmosphere Packaging (MAP) and Vacuum Packaging (VP). The principal function of such preservative packaging is to delay microbiological spoilage, but they must also delay non-microbiological deterioration of the product. CAP means that the gaseous environment in immediate contact with the food is controlled with respect to carbon dioxide, oxygen, nitrogen, water vapour and trace gases. MAP is defined as an initial alteration of the gas followed by time-based alteration stemming from product respiration, microbiological action, package gas transmission, etc. In general, all inclusive definition of a food stored in a MAP environment is one in which the product is stored in something other than air.

Vacuum packaging is removal of air from the package, with no gas replacement. Aerobic bacteria and oxidative reactions (leading to rancidity) are the two major spoilage agents of foods. Both these phenomena require oxygen. If oxygen is removed or eliminated, spoilage is inhibited and keeping quality is extended.

The effectiveness of all modified atmosphere packed food depends on the maintenance of the gaseous conditions inside the pack, for which suitable packaging materials must be used. The material should be compatible with the food and have capabilities of transmitting water vapour and gases. For MAP, plastic packaging materials are generally used and all plastics are permeable to gases, in varying degrees.

In practice, suitability of a film's gas transmission is expressed in terms of oxygen transmission rate. Transmission of carbon dioxide is usually between two and eight times that of oxygen.

In general, films with nominal oxygen permeabilities of 100 ml/m^2 /24h/atm can be used for vacuum and modified atmosphere packaging. Laminates or multi-layered structures are widely used for preservative packaging. For example, pouches may have a central gas barrier layer, like PVDC, inner food contact layer with good heat sealing properties, like PE and a damage-resistant outer layer, like nylon. Aluminium foil or metallized films may be used for additional gas barrier properties.

Active Packaging

Packaging is called active packaging when it performs an additional role, other than just exhibiting itself as an inert barrier to external influences.

The concept of active packaging has been developed to correct the deficiencies in passive packaging. For e.g., when a film is a good barrier to moisture, but not to oxygen, the film may still be used safely with the use of an oxygen scavenger to exclude oxygen from the pack. This is active, interactive or functional packaging, where the pack protects what it contains in addition to the classical functions of any packaging.

Other packaging systems, where plastics are successfully used are intelligent packaging, stretch wrapping, shrink wrapping, bag-in-box system etc.

Trends and Future Prospects

Both quantitative and qualitative trends have been identified to establish what the future holds and where the focus needs to be for technological development and upgradation of the industry. These are listed in Tables 20, 21 and 22.

TABLE 20

Indian Packaging Trends

(Growth Indication with base year as 1992)

Material	1992	1995	2000
CFB Boxes	100	123	172
Composite Containers	100	116	148
Multi-Wall Paper Sacks	100	133	214
Carton Wrappers/Bags	100	116	148
Laminates	**100**	**145**	**270**
Glass Containers	100	113	138
Jute (Sacks + Wrap)	100	111	111
Tinplate	100	112	137
Aluminium Foil	100	126	185
Aluminium Tubes	100	119	127
Plastics – Semi –Rigids	**100**	**126**	**185**
Plastics – Films	**100**	**134**	**218**

TABLE 21

World Packaging Trends

Trends

- Fastest growing **segment** is Flexible Packaging.
- Fastest growing **material** is Plastics.
- Specific high growth **areas** are:
  - Stand-up Pouches – highest growth
  - High Barrier Flexibles
  - Plastic Containers-especially High Barrier & PET
  - Labels & Shrink-Sleeves
  - Poly-Lactide Resins – Bio-degradable
  - Extreme concern on Tamper-Proofing & Brand Security
  - Demand for much better graphics

TABLE 22

Indian Packaging Trends

Package Material	Trend
Flexible packaging	Dominant
PET bottles	High growth
Paper	Moderate growth
Glass	Low growth
Metal Cans	Low growth

Conclusion

Packaging is more than a just a container with a label stuck on it. It is a vital opportunity to build new brands or reinforce and add value to a positive experience of an existing product or brand.

With development of science and technology, alternative materials like plastics have emerged as an appropriate replacement for natural/traditional materials. Apart from cost reduction, plastics also cater to certain applications, which were not possible with traditional materials. Plastics are the material of the new millennium. Now-a-days, usage of plastics gives the correct index of industrialization of the country, status of its economy and the standard of living of its citizens.

References

1. Beverage & Food World, Jan '03, Emerging Technologies in Agro Processing for Export by CT Devadas and M.Balakrishnan
2. Modern Food Packaging, IIP Publication
3. http://www.foodtechsource.com/emag/008/trend.htm
4. http://www.packaging-technology.com/informer/producteview/holoprotec/index.html
5. http://www.packaging-technology.com/informer/producteview/product46/
6. http://www.foodproductdesign.com/archive/1997/1297de.html
7. http://www.packaging-technology.com/informer/technology/tech16/index.html
8. http://www.amcor.com.au/petpackaging/news_heat_030422.htm
9. htpp://www.seabeecook.com/today/news/cook0076.html
10. Journal of Food Science and Technology, '01, Vol. 38, No. 5. Preparation of Onion Powder by Means of Osmotic Dehydration and its Packaging and Storage by V. R. Sagar, Div. of Fruits & Horticultural Technology, I A R I, New Delhi
11. Indian Food Packer, Jan–Feb '03. Preservation of chicken Curry by Hurdle Technology and its Storage Evaluation, by Himanish Das and S. Jayaraman, Defence Food Research Laboratory, Mysore
12. Packaging Technology, Apr-May '03, Application for Ceramic Coated Films with High Barrier Properties, Hiroshi Suzuki
13. Indian Rubber & Plastics Age, Apr '03 Plastics Packaging to spur EVOH>
14. www.mitsuiplastic.com
15. Indian Food Industry, Mar-Apr '01, Vol. 20 Polymer Blends in Packaging Industry, The present Status and Future Prospects, by Gotak B. Nanso IIT, Kharagpur
16. Packaging India, Dec '02-Jan '03, Vol. 35, No 5
17. Packaging India, Oct-Nov '02 Vol. 35, No 4
18. Packaging India, Dec-Jan '01 Vol. 33, No 5
19. Popular Plastic and Packaging, Feb '03, Demand for Plastic in India and Worldwide, by Anand Kumar Kulshreshlta, IPCL
20. Indian Rubber & Plastic Age, Jun '03, Plast India 2003 Propels Plastics Industry in High Growth Curve
21. IIP Report, IPC Feasibility Study – Stage 1

1988 *In:* TAPPI proceedings of the 1988 paper
preservation symposium: 1988 October 19-21;
Wahsington, DC. Atlanta, GA: TAPPI Press:
31-40

PAPER TESTING AND STRENGTH CHARACTERISTICS

D. F. Caulfield, Research Chemist
D. E. Gunderson, Research General Engineer
USDA Forest Service, Forest Products Laboratory,
One Gifford Pinchot Dr., Madison, WI 53705-2398
U.S.A.

ABSTRACT

The mechanical and strength properties of paper reflect the intrinsic chemistry, morphology, and structure of the individual fibers as well as the network structure of the paper. These properties also reflect those chemical changes that cause the paper to lose its permanence with time. Strength properties can thereby serve as indicators of the permanence of paper, even when the nature of the chemical changes responsible for the deterioration remains unknown. Careful monitoring of strength properties can also be used for evaluating the effectiveness of treatments for aging. This requires test methods and procedures that have been proven to be reliable. TAPPI test methods for evaluating the properties of tensile strength, bursting strength, tearing resistance, and folding endurance are discussed in terms of the fundamental characteristics of paper on which they depend. Also discussed is the concept of strength-property monitoring in conjunction with accelerated-aging studies. Environmental condition, primarily relative humidity, can have a great effect on measured strength properties. Testing methods are being developed that employ a greater degree of control over environmental conditions and rates of loading. The fundamental mechanical properties of paper revealed by these methods promise a more complete description of the behavior of paper in environments of changing relative humidities.

INTRODUCTION

The permanence and durability of paper were first distinguished in 1926 (1). Permanence is basically a measure of the chemical stability of paper. Durability, an the other hand, is primarily a function of the performance of paper; it is a measure of the stability of its physical and mechanical properties. A paper that is given rough treatment over a short time should be durable, but little Concern need be given to permanence. A paper that is meant to last for a century must be compounded for chemical stability. Such chemical stability can be enhanced by storing paper at a low temperature; at a constant, low relative humidity; in the dark; and in an atmosphere free of pollutants. But if the paper is also to be used--read, handled, folded, etc.--its mechanical durability is also important. Obviously, permanence and durability are not independent of each other, but rather intimately related. This relationship is so intimate, in fact, that durability or the stability of the strength and mechanical properties of paper has become the most useful indicator of its permanence or chemical stability.

Understanding the mechanical properties of paper in fundamental terms is a difficult task, and it is an area of intense study in many laboratories around the world. The ultimate aim of such studies is to provide a complete understanding of the relationships between the structure and properties of paper and papermaking procedures, so as to be able to tailor-make paper properties to meet any desired end-use requirements. Unfortunately, this goal has not yet been achieved. Instead, an alternate approach is often required; that of assessing the utility of paper to meet a desired need based on its ability to satisfy the requirements of some specified test or tests. Usually such tests are dependent upon particular test instrument designs and carefully defined procedures as to size of samples and other variables. Such tests often measure complex properties of paper that arise from a combination of several of its fundamental characteristics.

We will look at four strength properties of paper and the TAPPI test methods that are typically used to evaluate these properties. These tests evaluate the (1) tensile strength, (2) bursting strength, (3) tearing resistance, and (4) folding endurance of paper. Each of these properties, especially folding endurance, which is a fatigue strength property, has found wide acceptance as a measure of the permanence of paper. We will also discuss how environmental conditions, especially relative humidity, affect test results, and how accelerated aging coupled with strength testing provides a means of estimating permanence. Finally, we will mention recent developments of mechanical testing that incorporate a greater degree of environmental control for more accurately appraising of performance characteristics of paper under actual use conditions.

TESTS OF STRENGTH PROPERTIES

Tensile Strength

Tensile strength can be described by stress-strain graphs and measured by TAPPI tests T-404 and T-494. Stress-strain curves provide a fundamental engineering description of the mechanical behavior of paper when subjected to tensile stress. TAPPI method T-404 measures tensile breaking strength end elongation of paper and paperboard using a pendulum-type tester, and T-494 measures tensile breaking properties of paper and paperboard using constant rate of elongation appartus.

Fundamental behavior of paper in tensile loading. Figure 1 is a generalized illustration of the stress-strain tensile behavior of machine-made paper. The graph represents the behavior of two strips cut from the same sheet of paper in two directions: machine direction (MD--the direction in which the paper moves during manufacture) and cross-machine direction (CD--the direction at right agles to the machine direction). These stress-strain curves provide a fundamental engineering description of the mechanical behavior of paper strips when subjected to tensile stresses that distort or elongate the strips as they are pulled to failure by clamps attached at their ends. The x axis is a measure of the tensile strain or elongation of the sample. It is a dimensionless quantity that is defined as the change in length divided by the original Length of the strip. It is most often expressed as a percentage and called percent strain or elongation. The y axis is a measure of the tensile stress, which is defined as the tensile force applied to the sample ends divided by the cross-sectional area of the paper strip. Typical units of stress are pounds per square inch in the English system or Pascals, the preferred SI unit, which represents the force of one Newton (or 10^5 dynes) per square meter.

In the stress-strain plot (starting from the origin), as increasing stress is applied to a strip of paper, the strain or elongation of the strip increases linearly. The initial straight line section of the stress-strain curve, which is characteristic of elastic materials, has a slope that defines the fundamental property known as Young's modulus. Young's modulus is a useful concept in engineering design because it is the material characteristic which, along with the thickness of the material, determines stiffness. Further out on the stress-strain curve, the plot shows an increasing tendency of curvature towards the x axis. This nonlinear behavior of the stress-strain curve for paper indicates the inelastic or viscoelastic character of paper. It is the dual nature of paper, exhibiting both elastic and viscoelastic properties, which makes its behavior so complex.

As stress is further increased, the point of failure is reached, and the paper strip fails or ruptures. The maximum value of the stress that occurs at failure is a measure of the tensile breaking strength of the paper. The maximum value of the strain, or the elongation at rupture, is a measure of the percentage of stretch that the paper can achieve. Typically, for machine-made paper, both Young's modulus and breaking strength measured in the machine direction are 2 to 4 times the values in the cross-machine direction (2). Conversely, the machine-direction value for percentage of stretch may typically range between 0.25 and 0.5 times the percentage of stretch in the cross-machine direction. The differences in the properties of paper in these two directions arise from the tendency of the fibers to align preferentially in the machine direction and from the stretching of the paper web in this direction during manufacture.

Another fundamental measure of the tensile strength properties of paper is represented by the area under the stress-strain curve. This area represents the work expended to cause rupture of the tensile strip of paper; this work is called the tensile energy absorption (TEA) (3). Even though the breaking strength of paper in the machine direction is greater than In the cross-machine direction, it may require more work to break a tensile strip in the cross-machine direction because its greater stretch percentage makes the TEA greater in the cross-machine direction than in the machine direction. In certain instances, TEA can prove to be a significant property relating to durability. Tensile energy absorption is a measure of the ability of paper to absorb energy under variable loading conditions and can be used to gauge the durability of papers chat are subjected to repetitive straining. A stress-strain curve in which the initial slope (Young's modulus) is high, where there is little curvature, and where the value for elongation to break is low, indicates a brittle material. A tough material is one that exhibits more viscoelastic behavior, a pronounced curvature, and a high elongation to break.

TAPPI tensile strength tests. TAPPI tests T-404 and T-494 are useful in evaluating the tensile properties of paper (4). TAPPI T-494 enables the simultaneous evaluation of three properties for the same test specimen: tensile breaking strength. stretch or elongation at break, and TEA (Fig. 2). Method T-404 allows only the evaluation of tensile breaking strength and elongation at break, but the test apparatus is considerably less costly. TAPPI cautions that the two methods, T-404 and T-494, are not strictly comparable because different instruments are used. These tests give similar results for tensile strength and elongation at break only when similar testing conditions apply.

For the purposes of the TAPPI tests. tensile strength is reported with different units than indicated earlier. TAPPI tests define tensile stress as the force per unit width of the test specimen. This differs from the usual engineering definition of stress, which is force per unit cross-section of the test specimen. This difference arose historically because of the difficulty of accurately measuring the thickness of test specimens of paper (5). Similarly, for the purposes of test T-494, TEA is defined in terms of energy per unit area, which differs from the usual engineering definition of energy per unit volume. Because TAPPI defines stress as force per unit width, it is possible to express tensile strength in terms of breaking length by dividing TAPPI tensile strength by grammage (or basis weight). Breaking length is the calculated upper limit of Length of a uniform paper strip that would support its own weight if it were suspended at one end. Breaking length is a convenient measure for comparing the tensile breaking strength of papers of varying grammages. Values of the tensile properties of offset, rag bond, and newsprint of typical grammages are shown in Table 1 (2).

Tensile test results reflect the intimate structure of paper and the properties of its Individual fibers. The dimensions and strength of the individual fibers, their arrangement, and the extent to which they are bonded to each other are all important factors contributing to test results. Papers made with long fibers generally have higher tensile strength properties than paper made of short fibers. However, the extent of interfiber bonding is considered the most important factor contributing to tensile strength properties (6).

Page has provided a useful theory for assessing the relative contributions of individual fiber strength and interfiber bonding to tensile strength properties (7). To separate the effects of fiber strength and bonding, Page's theory requires the measurement of zero span tensile strength. The zero span test is a tensile strength test in which the jaws of the tensile tester are placed as close as possible to each other, that is, with zero separation. The principle behind the zero span test is that the individual fibers span the gap between the jaws, and the tensile breaking strength indicates fiber strength as opposed to bond strength. The method provides can index of the ultimate strength of the longitudinal structure of individual fibers in the test specimen. The ratio between the normal tensile breaking length and the zero span breaking length provides an index of the cohesiveness of fibers in the sheet. Although TAPPI has not yet established a zero span test for papers. TAPPI method T-231, "Zero Span Breaking Length of Pulp," is available (4). Commercial instruments are also available for measuring the zero span tensile strength of paper.

Bursting Strength

Bursting strength is perhaps the most commonly measured strength property of paper. The test apparently originated from the oldtime practice of the papermaker who, in a hands-on quality control evaluation of paper strength, would attempt to push his thumb through the sheet. TAPPI method T-403 is the official test used for measuring the bursting strength of papers with thicknesses up to 0.6 mm (4). It is also commonly known as the Mullen test. Similar TAPPI tests have been adopted for measuring the bursting strength of heavier paperboards and corrugated fiberboard. TAPPI test T403 involves clamping a sheet of paper firmly between two steel plates. These plates are provided with circular openings, and a rubber diaphragm in one plate opening seals off a chamber that can be pressurized by a fluid (Fig. 3a). As the chamber is pressurized, the bulging of the rubber diaphragm is resisted by the overlying clamped paper sheet. Pressure is increased, at a specified rate, until the bulging diaphragm causes rupture of the paper sheet. A pressure gauge on the instrument provides a measure of the bursting pressure needed to rupture the paper. Bursting strength is reported as pressure in pounds per square inch (lb/in^2) or in kilopascals (kPa), the preferred SI unit.

Because of the symmetry of the test specimen, there is no possibility of assigning machine direction end cross-machine direction values. However, by observing the nature of the rupture pattern on the test specimen, it can be seen that the differences between properties in machine direction compared to cross-machine direction do influence bursting strength. The burst failure usually results in a roughly “H”-shaped tearing pattern in the specimen (Fig. 3b) (3). The orientation of the “H” with the two directions of the machine-made paper indicates that failure initiates in tension along the line at right angles to the machine direction. This occurs in spite of the fact, as shown in Table 1, that tensile strength of paper in the machine direction is greater then that of paper in the cross-machine direction. As the specimen bulges in the bursting test, it is strained equally in both directions. The specimen reaches its strength limit in the machine direction at a lower st~-ain than it does in the cross-machine direction. This indicates that stretch is probably the limiting factor (rather than tensile strength) in determining bursting strength. Tensile breaking strength and bursting strength exhibit good correlation, and those fiber properties and papermaking practices that enhance tensile breaking strength tend to also enhance bursting strength (8). For two papers of equal tensile breaking strength, however, the one with the greater stretch will consequently exhibit the higher bursting strength.

Bursting strengths of three typical papers are shown in Table 2 (2). It is more meaningful to compare bursting strength of papers of differing grammages in terms of burst index. Burst index is obtained by dividing the bursting strength in kPa by the grammage of the paper in g/m^2; burst index values are included in Table 2.

Tearing Resistance

The most commonly used tearing test, T-414, also often called the Elmendorf tear test, measures the internal tearing resistance of paper rather than the edge-tear strength of paper, which is described in T-470 (4). Internal tearing resistance is a measure of the force perpendicular to the plane of the paper necessary to tear a single sheet through a specified distance after the tear has already been started. Edge-tearing strength (T-470) is a measure of the force needed to initiate a tear. The force needed to initiate a tear may be several times the force needed to propagate the tear once it is started. This is commonly known to anyone who has experienced the difficulty of opening a cellophane bag, which, once nicked, tears open easily. Those papers and other film materials that exhibit high tensile stretch or elongation to break also exhibit high edge-tearing strength. High stretch makes it difficult to localize or concentrate stress in a sufficiently small area so that a tear can be initiated.

The Elmendorf method (T-414) measures tearing resistance of paper perpendicular to the plane of the sheet or when paper is torn in the out-of-plane mode (Fig. 4). This is the mode of tearing used for example when deliberately tearing strips of newspapers. The out-of-plane tearing mode is to be distinguished from the in-plane tearing mode. An instance of in-plane tearing is the separation of computer printout sheets along a perforation while both sheets are held on the surface of a desk. In-plane tearing is representative of the tearing failure that can occur during the transport of a paper web through a printing press. The out-of-plane tearing mode, used in the Elmendorf test, resembles the type of tearing that occurs when a nicked sheet in a book is accidentally torn by a reader while turning pages.

During actual testing, several sheets are torn simultaneously in the Elmendorf apparatus, and the work is measured. The work needed to tear a single sheet of a given length is determined by a calibration procedure. When the unit of work is divided by length, the tearing force is obtained.

The work needed to tear paper is only a fraction of the TEA or the work required for rupture in a tensile strength measurement. This is because the tearing action directs a stress concentration at the pex of an advancing tear, and the route that the tear takes will tend to be the course of least resistance. The work done in tearing is made up of two main contributions: (1) the work needed to sever individual fibers and (2) the work needed to pull out unbroken fibers from their surrounding networks. Van den Akker et al. (9) have shown that, in general, less work is needed to sever a fiber than to pull a fiber from the sides of a tear. Becuase short fibers are obviously easier to Pull out than long fibers, papers made of long fibers show much better tearing resistance than those made with short fibers. As interfiber bonding increases in a sheet of paper, a fiber lying in the path of an advancing tear becomes more likely to sever than to pull out. As bonding is improved, stress at the apex of the tear is more concentrated and is less readily shared with the rest of the structure. Consequently, tearing resistance usually shows an inverse correlation with both tensile strength and bursting strength (10). Those papermaking processes that enhance interfiber bonding, such as beating, improve tensile and bursting strength, but tend to decrease tearing resistance.

The tearing resistance of offset paper, rag bond, and newsprint are shown in Table 3 (2). The tearing resistance is reported in either grams-force or millinewtons required to tear a single sheet. Table 3 also lists the tear index, the recommended measure of tear for comparing papers of differing grammages. Tear index is obtained by dividing the tearing resistance measured in units of millinewtons (mN) by the grammage of the paper in units of grams per square meter (g/m^2).

Folding Endurance

A folding-endurmce test is used to measure the ability of a paper to maintain its strength after repeated folding. There are two TAPPI test methods (4) used for evaluating this property: T-423, which employs the Schopper tester, and T-511, which employs the MIT tester. The essential differences between these methods arise from the design and function of the test apparatus. In both methods, the test specimen is held under tension and subjected to repeated folding; the number of folds necessary to cause failure is taken as a measure of folding endurance. The Schopper instrument operates at a rate of 120 double folds per minute. A reciprocating motion folds the specimen by looping it snugly around a small cylindrical surface. During the reciprocating motion, the tension applied to the 15-mm-wide test specimen fluctuates between approximately 800 g to 1 kg (7.8 to 9.8 N) in normal operation. The MIT apparatus applies a double fold of 270° (±135°) at a rate of 175 double folds per minute by means of an oscillating jawlike clamp (Fig. 5). The applied tension to the 15-mm-wide specimen is maintained constant during the folding action by means of a spring device. In normal operation, the applied tension in the MIT test is fixed at 1 kg (9.81 N). However, this tension may be adjusted depending on the strength of the paper being tested. The advantages of the MIT tester over the Schopper tester are that the MIT allows a wider range of paper thicknesses to be used, and the calibration and adjustment of the MIT instrument is less demanding than that of the Schopper. Test results obtained with these methods are not interconvertible because the folding operation is not the same in both cases. The MIT method is viewed as more closely resembling the repeated action of opening and closing a book, and it has been found especially valuable in measuring the deterioration of paper with aging (11).

Folding endurance may be considered a modified tensile strength test because the test specimen is under a tension that eventually causes failure. But the results of the test are affected as much by the flexing ability of the paper as by its tensile strength. Test results reflect the combined elastic and viscoelastic properties of the paper. Lack of folding endurance can result from lack of sufficient fiber length, inadequate fiber bonding, or brittleness. As a rule, rag pulps produce paper high in folding endurance, whereas groundwood papers and heavily filled papers show poorer folding endurance. In the early stages of beating, folding endurance increases as does tensile strength. As beating proceeds, however, folding endurance eventually decreases as interfiber bonding increases the brittleness of the paper.

Folding endurance can be measured in both machine and cross-machine directions (for this test, machine direction signifies the line of fold perpendicular to the machine direction). As a rule, machine direction folding endurance is higher than cross-machine direction folding endurance, which reflects the higher tensile strength usually exhibited by the machine direction. For those papers for which cross-machine direction folding endurance values exceed machine direction values, flexibility or the viscoelastic characteristics of the paper play a more significant role then inherent tensile strength. Table 4 contains the MIT folding endurance values of three typical papers (12). Since, as a general rule, folding endurance increases with basis weight or grammage up to a certain optimum after which it decreases, there is no significance to a concept of "fold index" as there is to tear index and burst index.

One characteristic of folding-endurance tests is that the results vary widely even on presumably identical samples. The source of this high variability stems from the very small area of the sample that is folded. Failure is forced to occur at a specific location rather than at the naturally weakest point in the test strip, as in normal tensile strength measurement. For the folding endurance test method to meet the necessary precision requirements, the TAPPI method now recommends that folding endurance be reported as the logarithm (base 10) of the number of double folds. However, the standard does permit results also to be reported as number of double folds.

ENVIRONMENTAL EFFECTS

TAPPI test methods require strict observance of standard conditioning and environmental conditions during the strength testing of paper. These are covered in TAPPI method T-402. Some properties of paper show a dependence on temperature, and all strength and mechanical properties of paper are strongly influenced by relative humidity. The standard testing environment is 23.0 ± 1.0°C and 50.0 ± 2.0 percent relative humidity. Conditioning the sample for a time in the same environment before testing is required to ensure moisture equilibration of the specimen. Because of the phenomenon of hysteresis, in which the moisture content of a material depends somewhat on its previous history, a preconditioning step is also required. Method T-402 suggests a preconditioning environment between 22 to 40°C and a relative humidity between 10 and 35 percent. Keeping test specimens for a period of time at this lower relative humidity, before equilibrating in the standard test environment. ensures that the effects of their different histories will be minimized.

If it is impossible to maintain the standard humidity conditions, the measured strength properties can exhibit marked differences. Figure 6 demonstrates the typical trends and extents of the differences in ordinary strength properties that one would expect over a range of relative humidities (from 15 to 85 percent) as compared to standard conditions (8). As humidity and the moisture content of the paper increase, the general trends appear to reflect two interacting tendencies: (1) increasing moisture improves individual fiber strength by increasing the viscoelastic character of the fiber, increasing fiber stretch, and allowing a more uniform distribution of stresses to take place, and (2) increasing moisture content competes for bonding sites in the fiber network and interferes with the natural bonding between fibers to weaken the network structure.

Typically, tensile strength measurements tend to show a slight increase with relative humidity up to a level of about 35 percent. As relative humidity further increases (above 35 percent), tensile strength shows a continuous decrease. The initial increase is presumed to arise from a better ability of stresses to be distributed throughout the stressed paper, but above an optimal level the loss of interfiber bonds becomes the predominant effect (8). Bursting strength shows a similar trend, exhibiting a smaller maximum than tensile strength, again around 35 to 40 percent relative humidity. At higher relative humidities, the drop in bursting strength is not as great as the drop in tensile strength. This is probably because improved stretch acts to counter the loss in strength attributed to the loss in interfiber bonding. Bursting strength is a comnlex property influenced by the combination of tensile strength end elongation to break, which moisture affects in different directions. (Moisture acts to lower tensile strength but to increase elongation or stretch.) Thus, of the strength tests, bursting strength is affected least by changes in relative humidity.

Tearing resistance generally shows a continuous increase with increasing relative humidity over the range of relative humidities (15 to 85 percent). For every percent increase in relative humidity, there is an approximate 1 percent increase in tearing resistance. The increased stretch and viscoelastic character caused by the increased moisture content helps delocalize and distribute the stress at the tearing point, and promotes fiber pullout rather than fiber breakage as the dominant mode of failure (10). At very high humidities (above 85 percent) or when paper is wetted, tearing resistance also decreases because of the ultimate disruption of the interfiber network bonds caused by the water. The loss in the tearing resistance of paper on wetting, however, is not as great as the loss in tensile strength that occurs under the same conditions.

Folding endurance is the strength property of paper that is most affected by relative humidity. Folding endurance tends to show an increase of between 1 and 5 percent for every 1 percent increase in relative humidity over the range of 15 to 85 percent relative humidity. This increase reflects the increase in flexibility and vlscoelascic character caused by the increasing moisture content. At very high humidities, the tensile load that is applied to the fold specimen becomes the limiting factor, and fold-endurance measurement, too, shows a dropoff near saturation. Because folding endurance is the strength property that exhibits both the greatest variability in measurement and the greatest sensitivity to moisture content, careful humidity control and proper conditioning of specimens are essential for reliable folding-endurance testing (13).

Sorption of moisture with changes in relative humidity also causes a significant dimensional change in paper, in both in-plane and thickness directions. These changes are attributed to shrinking and swelling of the individual fibers and to release of strains built into the paper during the initial drying process. The extent of dimensional change is variable, but a gross in-plane change of 1 percent is not uncommon in the change from 25 to 90 percent relative humidity. Perhaps more significant is the fact that the dimensional change can vary significantly over the sheet, leading to sheet curl and patterns of local buckling termed cockle. New instrumentation is being developed to evaluate dimensional stability and curl (14).

ACCELERATED AGING

Two indirect indicators of the chemical changes responsible for lack of permanence are the discoloration of paper end the loss of strength properties that occur with time. The reasons for the loss in strength with aging are not completely understood, but they are presumed to be directly related to the chemical and/or structural changes that occur at the molecular level. Because of this presumption, researchers have felt justified in applying concepts of chemical kinetics in quantifying the effects of aging (11). In the chemical kinetics approach, the loss in strength property with time is used to characterize the rate of deterioration of the material. Obviously, the best way of measuring stability of strength properties is by natural aging. But realtime studies are an impractical and costly method for assessing the long-term use potential of paper. Techniques of chemical kinetics, on the other hand, provide a means of effectively accelerating the natural aging process. According to the Arrhenius kinetic theory, the rate of an ordinary chemical reaction will be accelerated by approximately a factor of two if the temperature at which the reaction is run is raised by 10°C.

Since it is usually not possible to describe a normal aging environment, TAPPI has proposed two provisional methods that simulate two different aging environments (4). Method T-453 describes a method for evaluating the effect of dry heat on the properties of paper, and T-544, a method for evaluating the effect of moist heat on the properties of paper and board. Knowledge of the environment in which the paper would likely be stored aids in selecting the most appropriate type of accelerated-aging environment. It is known that moisture greatly accelerates the aging of paper, but the rate at which this increase occurs varies greatly depending on the type of paper. Therefore, dry-heat aging of paper is less sensitive and probably does not rank papers in order of stability as accurately as moist-heat aging. However, dry-heat aging is considerably easier to accomplish. The older dry-heat method utilizes only a forced ventilation oven that is maintained at 105°C. Test specimens in the oven need only be shielded from direct radiation from the heating elements. The moist-heat method utilizes laboratory apparatus devised by Graminski for maintaining an environment of 90°C end a relative humidity of 25 percent. Because of the demanding humidity control (±1 percent), temperature must be maintained at an accuracy better than 0.5°C (preferably 0.1°C). With both methods, papers are maintained in the specified environment for varying lengths of time; 24, 48, 72 end 144 hours. Each specimen, after appropriate conditioning to the standard test environment, is tested by the TAPPI test method of interest. Test methods employed to evaluate the effects of aging typically may include brightness (T-452), pH (T-509), copper number (T-430), and sodium hydroxide volubility (T-212), in addition to any of the strength tests mentioned previously. The tested property indicates a progressive deterioration with time. Since it is impossible to describe a normal environment, TAPPI cautions against relating results from accelerated aging to actual aging. However, the results of several investigations have indicated that accelerated dry-heat aging at 105°C for 72 hours corresponds approximately to "normal" aging of 25 years (15).

A plot of the data as a function of time allows one to calculate the slope or the rate at which the property deteriorates. Figure 7 shows typical shapes of curves encountered during the aging of paper (13). Only rarely is truly straight-line behavior observed. Concave curves occur more often then convex curves. It is common practice to linearize the curve by using logarithmic plots, or by fitting the data to the best straight-line relationship. From the straight-line plots, it is possible to evaluate half-lives that are useful in comparing and ranking the relative stability of different papers end in establishing the effectiveness of chemical treatments to improve permanence.

Some organizations officially recognize the importance of heat aging in helping to determine the suitability of papers for long-term applications. The National Association of State Textbook Administrators, for example, requires that all papers shall meet strength specifications after accelerated aging of 24 hours in a dry oven at 100°C (16).

Folding endurance is the most sensitive test indicator of the deterioration of paper on aging. Changes in folding endurance of paper show up long before there is a change in the tensile strength, bursting strength, or tearing resistance. Both TAPPI method for heat aging of paper suggest that folding endurance is the preferred test method. Until recently, the Library of Congress relied solely on the folding-endurance test after heat aging for demonstrating the improved life of treated paper (17). In addition to folding endurance, the Library now also recognizes the zero span tensile strength test for evaluating the effects of aging. It has been found that this test is especially useful for evaluating degraded book papers, which would not survive even a single fold, but which might still have useful life if restorative treatments could be effective (17). Zero span breaking length is considerably more sensitive as a measure of the effects of aging than is normal tensile breaking length. This suggests that fiber weakening or the creation of weak points along the fiber plays a significant role in heat aging of paper (11).

FUTURE NEEDS

We have reached the point where the performance of paper at standard conditions is fairly well understood, We have also been able to utilize artificial aging environments that simulate some of the changes in physical characteristics of paper that occur naturally with time. But until now we have not been able to measure mechanical and strength properties that are relevant in the world of changing environments, including cyclic humidity. It has long been known that the mechanical properties of paper are different at different humidities, but dramatic changes can also occur under fluctuating or nonsteady conditions. If paper is stressed slowly over a number of cyclic humidity changes, for example, the strain to failure will exceed the strain to failure that would occur under high, but constant, relative humidity conditions. This dramatic acceleration of the distortion of paper induced by long-term stresses in cyclic relative humidities is a prime example of how nonsteady environments influence the mechanical behavior of paper (11). This mechanical effect overlays the chemical changes that are believed to be accelerated by storage in changing humidity environments. With behavior like this, it seems likely that standard, constant-condition tests do not provide an adequate picture of behavior in the world of changing environments. Current research at Forest Products Laboratory seeks to observe and explain the mechanical behavior of paper in transient end cyclic humidity environments (18). For this purpose, instrumentation has been designed and constructed (Fig. 8) that allows for the characterization of the fundamental mechanical properties of paper under conditions of constant or cyclic relative humidity end constant or cyclic stress. Stresses may be applied either in tension or in compression, and strain as well as Young’s modulus may be measured continuously or intermittently as a function of time during changes in environment or stress application. Because the dimensions of paper also change with humidity along with mechanical properties, corrections must be made to obtain true strain measurements.

The combined effects of stresses and changing environmental conditions are especially revelant to the performance of paper in packaging materials, where relatively short-term failures are noticeable in cyclic humidity conditions. But these effects may also play a role in the long-term performance of library materials. Paper continually undergoes stresses because of its anisotropic properties and because of changes in temperature and humidity. These induced stresses. although small, may play a role in accelerating the loss of mechanical properties when paper is stored for decades under conditions of cyclic humidity and temperature changes. The instrumentation and methods under development should help quantify these effects and provide a fuller explanation of the connections between the mechanical durability and permanence of paper.

LITERATURE CITED

1. G. Hall, Paper Trade J. 84(14) 185 (1926).
2. Anonymous, Introduction to Paper Properties, Tappi Home Study Course #3, Tappi, Atlanta, GA (1978).
3. J. A. Johnson, K. A. Bennett, and H. M. Montrey, "Failure Phenomena", in Handbook of Physical and Mechanical Testing of Paper and Paperboard, ed. R. E. Mark, Marcel Dekker, Inc., New York (1983).
4. Anon. TAPPI Test Methods 1988, Tappi, Atlanta, GA (1987).
5. V. C. Setterholm, A new concept in paper thickness measurement, Tappi 57(3): 164 (1974).
6. J. P. Casey, Pulp and Paper, Interscience Publishers, New York (1966).
7. D. H. Page, A theory for the tensile strength of paper, Tappi 52(4): 674 (1969).
8. B. W. Scribner and F. T. Carson, "Paper Testing" Chapter 5 in Manufacture and Testing of Paper and Board, ed. J.Newell Stephenson, McGraw-Hill, New York, (1953).
9. J. A. Van den Akker, A. L. Lathrop, M. H. Voelker and L. R. Dearth, Importance of fiber strength to sheet strength, Tappi 41(8): 416 (1958).
10. R. S. Seth and D. H. Page, Fiber properties and tearing resistance, Tappi 71(2): 103 (1988).
11. P. Luner, Paper permanence, Tappi 52(5): 796 (1969).
12. W. K. Wilson and R. L. Hebert, Evaluation of the stability of record papers, Tappi 52(8): 1523 (1969).
13. R. Cardwell, L. Lyon and P. Luner, Evaluation of fold test procedure for paper aging studies, Tappi 55(2: 228 (1972).
14. L. E. Eriksson, S. Calvin, and C. Fellers, Curl and twist of paperboard- theory and measurement, Nordic Pulp and Paper Research Journal 2(2) (1987).
15. W. K. Wilson and R. L. Hebert, Effect of heating on folding endurance, Tappi 52(8): 1565 (1969).
16. Anonymous, Manufacturing Standards and Specifications for Textbooks, National Association of Textbook Administrators, Stamford, CT (1988).
17. U.S. Congress, Office of Technology Assessment, Book Preservation Technologies, OTA-0-375 U.S. Government Printing Office, Washington, D.C., (1988).
18. J. M. Considine and D. E. Gunderson, A method for mechanical property testing of paperboard during compressive creep in a cyclic humidity environment, Experimental Techniques 11(9): 18 (1987).
19. V. C. Setterholm and D. E. Gunderson, "Observations on Load-Deformation Testing", in Handbook of Physical and Mechanical Testing of Paper and Paperboard, ed. R. E. Mark, Marcel Dekker, Inc., New York (1983).

Table 1. Tensile properties of paper.

Type of paper	Tensile strength (kN/m)		Breaking length (km)		Stretch (percent)		TEA[1] (kJ/m^2)	
	MD[2]	CD[3]	MD	CD	MD	CD	MD	CD
Offset ($107\ g/m^2$)	5.55	3.21	5.30	3.06	2.5	4.1	14.9	15.8
Rag bond ($75\ g/m^2$)	3.60	2.55	4.90	3.47	1.8	4.7	6.29	13.2
Newsprint ($50\ g/m^2$)	1.79	0.90	3.65	1.84	1.1	1.4	1.78	1.29

[1] Tensile energy absorption.

[2] Machine direction

[3] Cross-machine direction.

Table 2. Bursting strength of paper.

Type of paper	Bursting strength (lb/in^2)	Bursting strength (kPa)	Burst index ($kPa \cdot m^2/g$)
Offset ($107\ g/m^2$)	33	288	2.13
Rag bond ($75\ g/m^2$)	25	172	2.29
Newsprint ($50\ g/m^2$)	4.5	31	0.62

Table 3. Tearing resistance of paper.

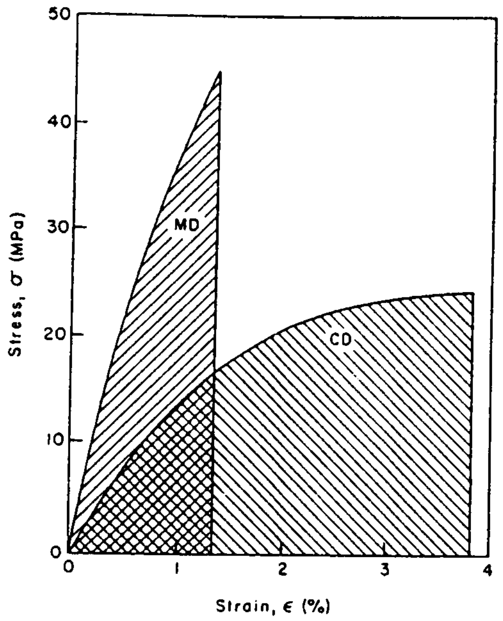
Type of paper	Tearing resistance				Tear index	
	(g)		(mN)		(mN · m^2/g)	
	MD[1]	CD[2]	MD	CD	MD	CD
Offset (107 g/m^2)	68	85	667	833	6.23	7.79
Rag bond (75 g/m^2)	65	65	637	637	8.50	8.50
Newsprint (50 g/m^2)	12	23	118	226	2.35	4.70

[1] Machine direction.

[2] Cross-machine direction.

Table 4. MIT (0.5 kg) Folding endurance of paper.

Type of paper	Folding endurance			
	Number of double folds		Log_{10} of double folds	
	MD	CD	MD	CD
Offset Paper	660	495	2.8	2.7
Rag bond	4,280	2,860	3.6	3.5
Newsprint	74	7	1.9	0.9



ML88 5588

Figure 1. Stress-strain behavior of paper in two directions: MD (machine direction) and CD (cross-machine direction). The area under the curve is a measure of the work needed to rupture and is called the tensile energy absorption (TEA).

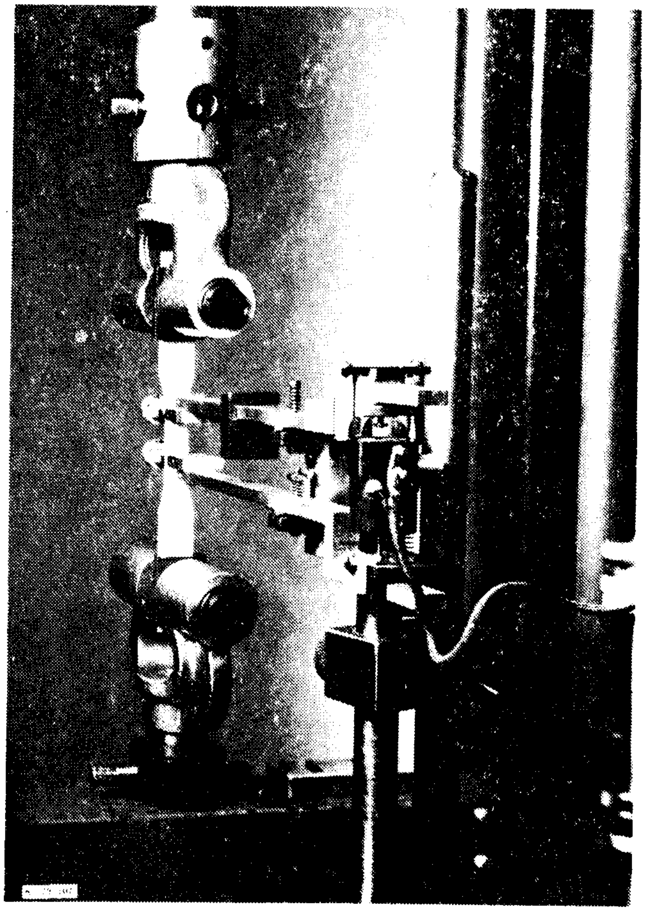
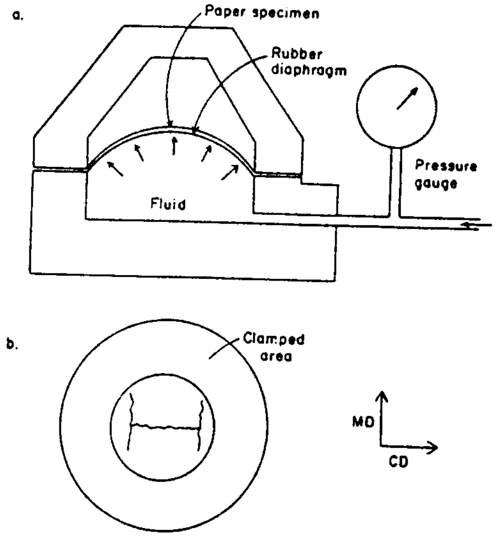
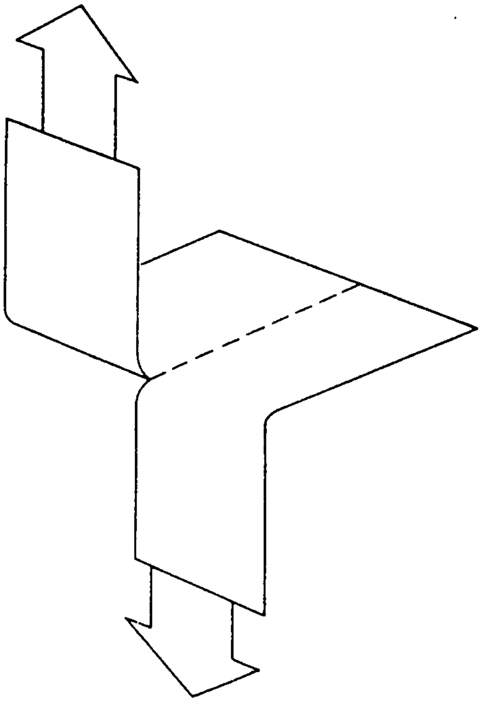


Figure 2. Test strip mounted in tensile testing apparatus, equipped with load cell and direct reading strain gage (ref. 19). (M 129107)



ML88 5589

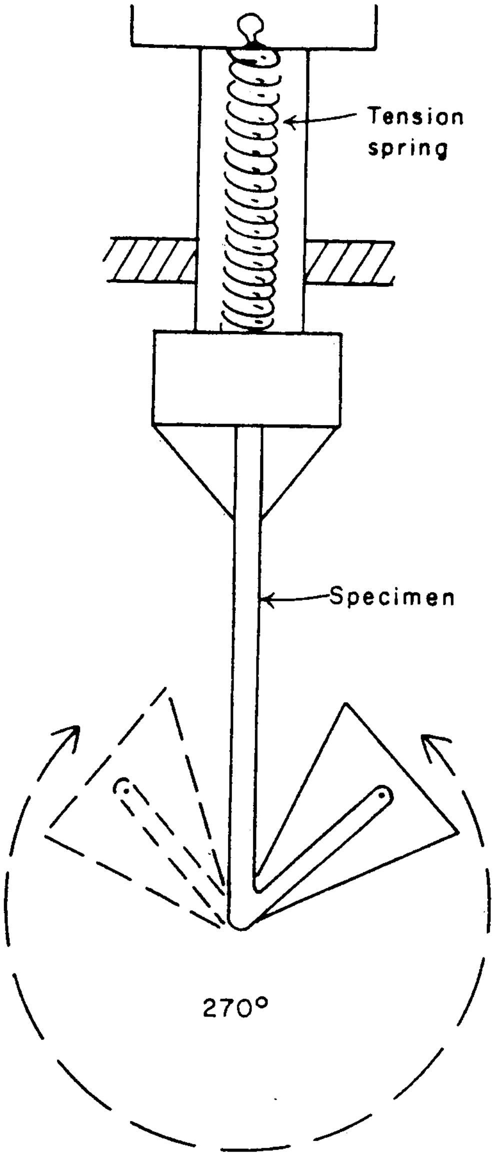
Figure 3. (a) Schematic of bursting test apparatus. (b) Appearance of a typical bursting test specimen after failure; failure initiates along the rip at right angles to the machine direction. MD, machine direction, CD, cross-machine direction.



Out-of-plane (Elmendorf) tearing mode

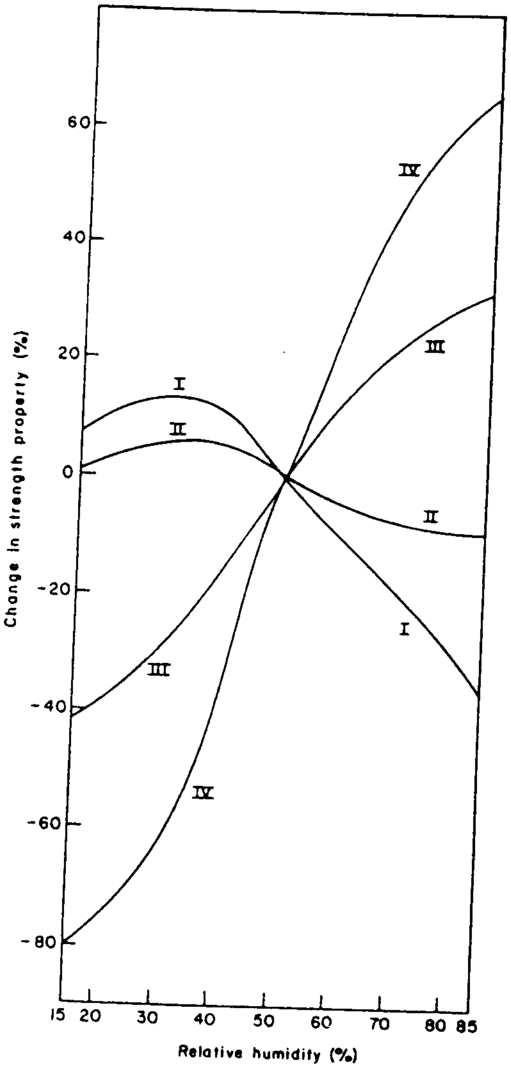
ML88 5590

Figure 4. Out-of-plane tearing mode used in the Elmendorf tearing resistance test.



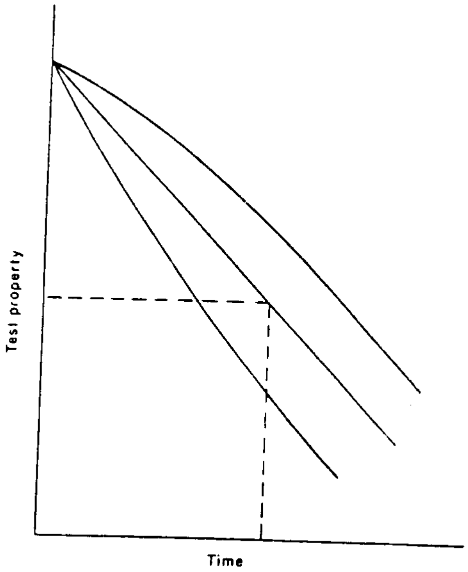
ML88 5591

Figure 5. Schematic of the MIT folding endurance apparatus.



ML88 5592

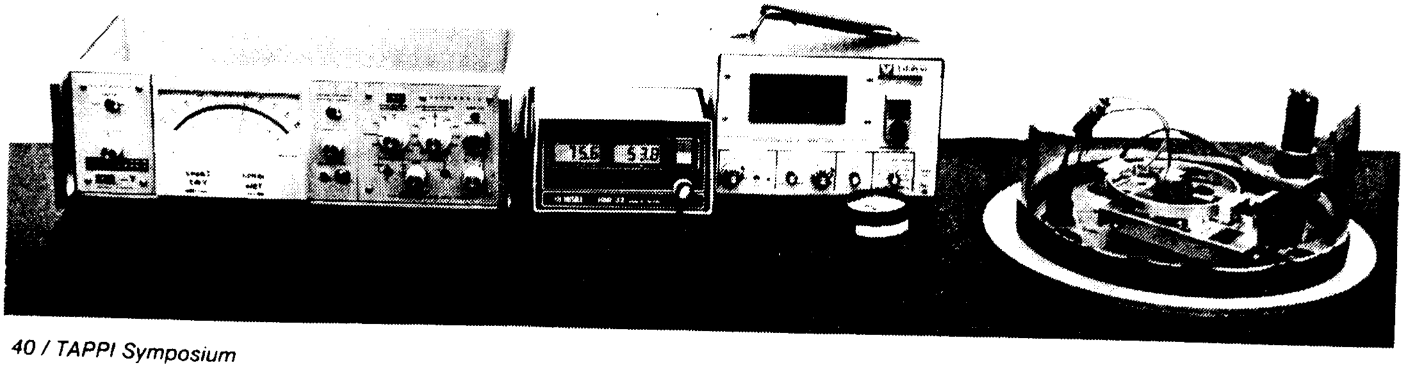
Figure 6. Generalized effect of relative humidity on strength properties of paper; I is tensile strength, II bursting strength, III tearing resistance, and IV folding endurance.



ML88 5593

Figure 7. Types of curves found for strength property behavior as a function of aging time.

Figure 8. Instrument for mechanical stress-strain testing of paper in which the environment may be cycled between high and low relative humidities. (M87 0318-7)



Paper No.: 12
Paper Title: FOOD PACKAGING TECHNOLOGY
Module – 12: Permeability of multilayer materials and means to measure permeability

1. INTRODUCTION:

The transport behaviour of substances in polymeric materials has become progressively more important in recent years with the widespread use of plastic films and rigid plastics for food packaging. The selection of plastic materials for food packaging applications with strict design specifications relating to their transport behaviour requires knowledge and appreciation of the many features which affect those phenomena.

There are many examples of foods packaged with an obvious lack of proper consideration of the effects of the environment on properties, or of limitations forced on performance due to unfavourable transport characteristics. The plasticization of polymers by sorption of ambient vapours or liquids causing subsequent decrease in mechanical properties and the loss of beverage components are few of many examples which may be cited. An objective of research in this field is to establish mechanisms and expressions relating solubility and transport with the molecular properties and characteristics of the components.

2. PERMEATION

Permeation through a film is a three-part process:

1. Solution/absorption of penetrant into the polymer surface
2. Migration/diffusion of penetrant through polymer(s)
3. Emergence/desorption of penetrant from opposite surface of polymer.

Absorption and desorption depend on the solubility of the permeant, and solubility is greatest when penetrant and material have similar properties.

Other relevant theory comprises:

Graham’s Law (1833) states that the velocity of diffusion of a gas is inversely proportional to the square root of the density of material.

Fick (1855) stated that the quantity of diffusing gas is proportional to concentration and time and inversely proportional to the thickness of the material through which it is diffusing.

Henry’s Law (1803) states that amount of gas absorbed by a particular volume of a liquid at a specified temperature is directly proportionate to the partial pressure of the gas.

In practice, the film of packaging material may comprise more than a single polymer, and there may be discontinuities in coatings, pinholes in films, variations in molecular structure and degree of crystallinity. The penetrant molecular size, shape and degree of polarity and ambient conditions are relevant. These are all factors which affect diffusion and solubility, which in turn have a direct impact on permeability.

The permeability of plastic films to water vapour and common gases like oxygen, carbon dioxide and nitrogen has been measured by standardised test methods. Oxygen, can cause oxidative rancidity in oil or fat containing food products. Water vapour permeation into a product may cause a loss of texture, and the outflow of water vapour, from a product through the packaging may cause dehydration, textural changes and loss of weight. An example of the latter would be a plastic film-wrapped cakes or bread which would lose moisture in storage prior to sale, where a negotiation has to be made in balancing weight loss in storage with initial weight and the water vapour barrier protection provided by the plastic film. In this example, in addition to flavour retention and texture, the actual weight at the point of sale would also have to meet appropriate regulations.

The results of permeability tests give direction with respect to the choice of material(s) for the packaging of specific food products. Some other possible penetrants and the effect of the presence of polymer additives can lead to unexpected results. It is still necessary to carry out shelf life tests to establish performance of packaging material in practice with the food under consideration.

Under steady state conditions, a gas or vapour will diffuse through a polymer at a constant rate if a constant pressure difference is maintained across the film. The diffusive flux, J, of a permeant in a polymer can be defined as the amount passing through a plane surface of unit area normal to the direction of flow during unit time, i.e:

$$J = Q / A \times t$$

Where, Q is the total amount of permeant which has passed through area A during time t.

2.1 Desired Properties of a Good Barrier Material

To be a good all-round barrier material, the polymer must possess the following properties:

1. Some degree of polarity as found in nitrile, chloride, fluoride, acrylic or ester groups;
2. High chain stiffness;
3. Inertness to the permeant. Polymers containing polar groups, can absorb moisture from the atmosphere. This has the effect of swelling or plasticizing the polymer and reducing the barrier properties.
4. Close chain-to-chain packing ability got by molecular regularity, crystallinity or orientation. Linear polymers with a simple molecular structure lead to good chain packing and lower permeability than polymers in which the backbone has a bulky side groups leading to poor packing ability. The higher the degree of crystallinity, the lower the permeability, because crystalline regions are less permeable compared with the amorphous regions. Orientation of amorphous regions decreases permeation by about 10 to 15%, while in crystalline polymers reductions of over 50% can be observed.
5. Some bonding between chains. Cross-linking of polymers restricts their mobility and thus decreases permeability, due to the decrease in the diffusion coefficient. For example, in the case of polyethylene, one cross-link about every thirty monomer units leads to a one half the reduction of the diffusion coefficient. The effect of cross-linking is more prominent for large molecular sized permeants.

3. PERMEABILITY OF MULTI-LAYER MATERIALS

Multi-layer materials can be considered as a number of membranes in series. Consider the case of three layers in series. The total thickness XT = X1 + X2 + X3. Assuming steady state flux, the rate of permeation through each layer must be constant, i.e.

$$QT = Q1 = Q2 = Q3$$

Likewise, the areas will also be constant so that

$$AT = A1 = A2 = A3$$

Therefore, by substituting in equation:

$$\frac{Q}{t} = \frac{P}{X} A \ (\Delta p)$$

then

$$\frac{Q_T}{t} = \frac{P_1}{X_1} A_1 (p_1 - p_2) = \frac{P_2}{X_2} A_2 (p_2 - p_3) = \frac{P_3}{X_3} A_3 (p_3 - p_4)$$

By rearranging above equation and writing it for the case of permeation through the multi-layer material:

$$\frac{Q_T \; X_T}{t \; A_T \; P_T} = (p_1 - p_4) = \Delta p_i$$

Now since,

$$(p1 - p4) = (p1 - p2) + (p2 - p3) + (p3 - p4)$$

Therefore

$$\frac{Q_T \; X_T}{t \; A_T \; P_T} = \frac{Q_T}{t \; A_T} \left[\frac{X_1}{P_1} + \frac{X_2}{P_2} + \frac{X_3}{P_3} \right]$$

and

$$\frac{X_T}{P_T} = \frac{X_1}{P_1} + \frac{X_2}{P_2} + \frac{X_3}{P_3}$$

or

$$P_T = \frac{X_T}{(X_1/P_1) + (X_2/P_2) + (X_3/P_3)}$$

Thus if the thicknesses and permeability coefficients are known for each layer, and provided that the permeability coefficients are independent of pressure, then above can be used to calculate the permeability coefficient for any multi-layer material. If they are not, then differing permeability coefficients will be obtained depending on the positioning of the layers.

The standard methods for gas permeability measurements through plastic materials specify dry gas. But, in practice the films are almost used in humid conditions, and for materials such as ethylene-vinyl alcohol copolymers, the oxygen permeability is dependent on the humidity. In such a case the above equation cannot be used directly since P2 (the permeability coefficient of the centre layer) will depend on the average partial pressure at the centre.

An equation for forecasting the average partial pressure at the centre of a multi-layer material containing a water sensitive centre layer can be derived as follows. Consider again the case of three layers in series, but this time assume that the oxygen permeability of the centre layer is moisture dependent and that the direction of water vapour flux is from the outside to inside.

Since the partial pressure of water vapour will not be constant across the multi-layer, the equation must be modified to include a term for the partial pressure difference and the thickness:

$$\mathrm{WVTR} = \frac{Q}{A \times t} \Delta p$$

Now since the area A and time t will be the same for all three layers, the equilibrium WVTR between the outside and centre layers can be calculated as:

$$\frac{Q_1}{X_1}(p_1 - p_2) = \frac{Q_2}{X_2}(p_2 - p_3)$$

Similarly, the equilibrium WVTR between the centre and the inside layer is

$$\frac{Q_2}{X_2}(p_2 - p_3) = \frac{Q_3}{X_3}(p_3 - p_4)$$

The average partial pressure of the centre layer (p_c) will be:

$$p_c = \frac{p_2 + p_3}{2}$$

Simultaneous linear solution of equations for p_2 and p_3 and substitution in above equation:

$$p_c = \frac{p_1\left[\frac{X_2}{Q_2} + 2\frac{X_3}{Q_3}\right] + p_4\left[\frac{X_2}{Q_2} + 2\frac{X_1}{Q_1}\right]}{2\left[\frac{X_3}{Q_3} + \frac{X_2}{Q_2} + \frac{X_1}{Q_1}\right]}$$

= average partial pressure of the center layer.

By the data of pc, the permeability coefficient Pc of the centre layer can be determined at this partial pressure and equation for P_T can be used to calculate the overall permeability of the multi-layer material.

4 MEASUREMENT OF PERMEABILITY

4.1 Gas Permeability

There are many methods for measuring permeability; the some important methods will be considered here. For a complete understanding of the principles behind permeability measurements it is important that the meaning of two terms which are constantly used - total and partial pressure of gases in a mixture is clearly appreciated.

In constant volume, total pressure exerted by gases present is the sum of the partial pressures of each of the gases, a discovery made by John Dalton and known as Dalton's law. The partial pressure of any one of the constituent gases is the pressure which would result if that particular gas occupied the same volume by itself. That is, each gas in a gas mixture behaves independently of the other gases.

The permeation rate of a gas through a polymeric material is function of the partial pressure difference of that gas across the material and not of the total pressure difference between the two sides.

4.1.1. Pressure Increase Method

The ASTM standard method for measuring gas transmission rates and permeability of flat films is labelled as D 1434. It includes the manometric method which utilizes the Dow gas transmission cell. The film is backed with a filter paper and sealed with an O-ring. The pressure in the receiving compartment is measured with an open-ended mercury manometer. Detailed descriptions of the calibration and testing procedures are given in the ASTM method. The corresponding British Standard is BS 2782 Part 8 Method 821A.

On condition that the pressure on the high-pressure side remains much larger than on low-pressure side, the pressure difference remains constant and the permeability coefficient can be calculated as follows. The slope of the straight line portion of the plot of the pressure (in mm Hg) on the low-pressure side versus time (ΔpL / Δt) is determined and substituted into the following equation:

$$P = \frac{\Delta p_L}{\Delta t} \times \frac{V_L}{760} \times \frac{273}{T} \times \frac{X}{A}$$

Where V_L is the volume of the low-pressure side, X the thickness of the film, T the absolute temperature and A the film area.

4.1.2 Concentration Increase Method / Isostatic method

In this method a partial pressure difference through the film with respect to the test gas is created without change in total pressure, thus avoiding the need for rigid support of the film. A partial pressure difference is maintained by sweeping one side continuously with the test gas and keeping an inert gas on the other side into which the test gas diffuses. The concentration of the diffusing gas can be measured by chemical analysis, gas chromatography, thermal conductivity or special electrodes. As the method of measuring the concentration of the test gas can be specific to that gas, equipment can be developed in which the relative humidity of both the test and inert gases can be controlled. This is of main importance when measurements are carried out on films whose gas barrier properties are related to humidity or moisture.

Instrument in extensive commercial use for the estimation of oxygen permeability by the isostatic method is MoCon Ox-Tran (Modern Controls, Inc., Minneapolis, Minn., U.S.A.). An advantage of this instrument over the permeability cells is that the permeability of not only flat film but also containers, bottles, pouches, tubes, etc. can be measured, thus allowing the analysis of possible adverse effects of machine processing, printing and distribution. The use of this instrument is included in ASTM D 3985.

The Ox-Tran has a two-chamber measuring cell between which the test film is placed. Gas stream of known oxygen partial pressure is passed through one of the chambers; oxygen-free carrier gas is passed through the other chamber to a coulometric detector. A separate part of the instrument is fitted with two openings for the carrier gas over which containers can be fixed and sealed. A glass dome is located over this arrangement into which oxygen flows by means of a filling tube.

Modern Controls, Inc. has designed an instrument for the measurement of carbon dioxide permeability, it is known as the Permatran C, it is similar in construction to the Ox-Tran, but it uses a pressure-modulated infrared detector.

4.1.3. Volume Increase Method

In this method, the change in volume at constant pressure, because of permeation of gas through the film is measured. Variable volume permeation cells are often used for rapid estimation of relatively high steady-state permeation rates. Though, the volume increase method is simpler to implement but less sensitive than the pressure increase method, it is rarely used for high-pressure time lag measurements. Volumetric methods are used relatively infrequently compared with the use of the pressure increase or concentration increase methods.

4.1.4. Detector Film Method

A method for measuring permeability of films which requires little equipment and is both rapid and accurate, has been devised. The principle of the method is a plastic detector film saturated with a reagent which is sensitive to the measured gas. The film, having an absorption spectrum that changes with the gas or vapour when absorbed, is thus suitable for spectrophotometric measurements. The detector film is sealed between two pieces of test film in a simple cell so that the permeation rate of the penetrant gas or vapour can be readily measured. The detector film can measure much less than the minimum detectable quantity of oxygen determined by most other methods, and therefore allows the use of either smaller film samples or more rapid permeability determinations.

The oxygen detector consist a cast film of ethyl cellulose containing dimethylanthracene (DMA) and erythrosine. On absorbing blue light, the erythrosine can trigger oxygen dissolved in the ethyl cellulose to form singlet oxygen, a reactive form of oxygen. Singlet oxygen diffuses to a neighbouring DMA molecule and reacts with it. Thus, the disappearance of DMA is monitored in the UV, which is a measure of the oxygen consumed. As the ethyl cellulose detector is highly permeable to oxygen, it is capable of measuring very low rates of oxygen permeation.

4.2 Water Vapour Permeability

The standard method to determine water vapour transmission rates is to place a quantity of desiccant in an aluminium dish which is covered with a sheet of the material being tested and sealed in the same position with wax. The dish is then placed in a closely controlled atmosphere (Either 25±0.5°C & 75±2% RH for temperate conditions, or 38±0.5°C & 90±2% RH for tropical conditions) and the increase in weight noted as a function of time. If the points are plotted out they should fall more or less on a straight line since Dp is constant throughout the test. To convert WVTR into permeance (P/X), it should be divided by the driving force D_p.

This method has several disadvantages, including the length of time needed to make a determination of 2 and 14 days and the lower limit of the useful range of about 1g / m^2 /day for a typical packaging film. Another disadvantage is that, depending on the desiccant, D_p may not remain constant during the test period. When using anhydrous calcium chloride, the partial pressure of water vapour in the dish remains below 2% of the vapour pressure of water at the test temperature, while in case of silica gel the partial pressure of water adsorbed on it increases with coverage.

Newer type of film detector to measure rate of transmission of water vapour has also been developed. It comprises of transparent cellulose film which becomes bright blue when soaked in cobalt chloride solution and dried over calcium chloride but rapidly turns pink on exposure to high humidity. A humidity cabinet is used to provide the partial pressure gradient through the test film, which is sealed in the same way and in a cell of similar design to that used to measure oxygen permeability. The change in absorbance of the detector film is measured at 690 nm, and from this the quantity of water absorbed by the detector film, and hence WVTR of the test film, can be calculated. Good results have been obtained using this method.

4.3 Odour Permeability

The permeability of packaging materials to organic vapours is of substantial interest, predominantly where the contents of the package has to be protected against foreign odours or where there is a prerequisite that volatile flavouring materials are not lost from the package. The major off-flavours found in some food products may result from the packaging material itself, or may permeate via the packaging material from the outside environment. In other situation, foods may contain highly desirable but volatile flavour compounds whose loss from the packaged food will reduce its quality. In both situations, suitable tests must be undertaken to select materials which have the desired odour barrier properties.

There are no standard methods for the measurement of odour permeability. A number of methods have been described for vapour permeability measurements, although many of them are only suitable for use with saturated vapours only. A sophisticated instrument for studying the transport of aromas in polymer films has been described, which utilizes a mass spectrophotometer to detect the permeant. Temperatures up to 150°C and relative humidity from 0 to 100% can be used, making it possible to obtain data on the likely aroma, flavour and odour permeation of polymeric materials used in retortable pouches. A method for the quantitative evaluation of the aroma barrier of packaging materials has been developed which uses a permeation cell similar to that described for the concentration increase method. Nitrogen gas is bubbled through the liquid permeant and then passed with the permeant vapours through the cell. The concentrations of the permeating vapours and related humidity are monitored by gas chromatography.

A common, odour penetration test involves packaging various odoriferous substances in pouches made from the test materials. The pouches are then placed in clean glass bottles and sealed by crimping with aluminium foil. After storage for a fixed time, the bottles are sampled, either by gas chromatography and mass spectroscopy, or by sniffing using a sensory evaluation panel. By these results it is possible to rank a range of packaging materials according to their odour barrier properties.

5. Conclusion

There are various methods for determination of permeability of multilayer packaging materials. But, particular methods have often been lost and then rediscovered, their origins are forgotten. For example, the isostatic method has been reinvented many number of times, most recently in 1973, since Mitchell used it in 1831. Although the permeability of the permanent gases and of water vapour through many packaging materials are well known, there is a lack of data for the permeation of organic vapours. Much of the published work has involved the use of saturated solvent vapours, and although this data is useful in estimating how well a packaging material will withstand accidental high-level contamination, it is not generally valid to use such data to estimate permeation rates at the very much lower levels of vapour encountered in typical retailing situations. So there is a need of work to be done on measurement of permeability of organic vapours or odorous compounds in the actual retail situations.

Reference:

Ashley, R.J. 1985. Permeability and plastics packaging. In *Polymer Permeability*, J. Comyn (Ed.), Elsevier Applied Science Publishers Ltd., Essex, England

Brown, R.P. (Ed.). 1981. Permeability. In *Handbook of Plastics Test Methods*, 2nd Edn., George Godwin Ltd., Essex, England

Hernandez, R.J., Giacin, J.R. and Baner, A.L. 1989. The evaluation of the aroma barrier properties of polymer films. In *Plastic Film Technology*, K.M. Finlayson (Ed.), Technomic Publishing Co., Inc., Lancaster, Pennsylvania, USA

Paine, F.A. and Paine, H.Y. 1992. *A Hand Book of Food Packaging*. Blackie Academic & Professional, London, England

Pauly, S. 1989. Permeability and Diffusion Data. In *Polymer Handbook*, 3rd Edn., J. Brandrup and E.H. Immergut (Eds.), Wiley-Interscience, New York, USA

Robertson, G.L. 1993. *Food Packaging Principles and Practice*. Marcel Dekker INC. New York, USA