Development of a biodegradable transparent packing material

By

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Declaration

The work described in this thesis was carried out by me at the Department of Agricultural Engineering, Faculty of Agriculture, University of Peradeniya under the supervision of Dr. D. A. N. Dharmasena and Mr. M. A. J. Wansapala. A report on this has not been submitted to any other University for another degree.

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AMMA AND THATHTHA

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ABSTRACT

Increasing amounts of packaging waste are discarded into municipal waste streams each year. A considerable amount of research has been devoted to the production of biodegradable polymer films derived from natural sources to reduce synthetic polymer waste. Therefore, development of biopolymer films from protein, polysaccharides and lipid material are of high interest of the most of scientists. This research study was carried out to produce a bio-film using A*lstonia* (Hawari Nuga) tree leaves. Leaves were fermented in an inoculating water tank for few days to separate the film. Films were separated manually to remove the debris of plant tissues. Films were bleached with three different techniques. These films were pasted together by gelatin with different layers (2,3,4) to be used for the tests of mechanical properties. The effect of leaf maturity was tested on separating the film and its strength. The microorganisms responsible for the anaerobic digestion of leaf material were also investigated. Finally physical properties of the films were evaluated using standard test method (ASTM D-882-91).

Bio-films produced were flexible, transparent and had a sight yellow colour. Fully matured leaf stage was the best stage to separate the film. The isolated microorganisms were mainly decomposing fungi. With the increase in the number of layers, the ultimate tensile strength increased gradually while the transparency decreased gradually. Tensile strength and percentage elongation values of cutin films reveal that they are compatible with other bio-polymer films like corn zein protein and modified Soya protein isolate films developed by other scientists. However the leaf cutin film developed has low impact strength than those of synthetic polymer films like high-density polyethylene (HDPE) and low-density polyethylene (LDPE). Therefore, these films can be used in such applications where impact is not very important.

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CHAPTER 1: INTRODUCTION

Packaging plays a critical role in spurring economic growth, enhancing the marketability of products and reducing the cost of production and distribution. In recent years, packaging industries are experiencing increased automation of converting and packaging operations, wide use of different packaging materials and formats and pursuit of good packaging education. Environmental considerations have yet to have impact on package design and selection.

Food processing industries consume large quantity of different varieties of packaging materials. This offers excellent opportunities for the packaging industrial sector for innovations and developments. Plastic packaging contributes substantially to the high quality of living in industrial nations, and to the efficiency of distribution systems, from harvest to consumers. Therefore, plastics are increasingly consumed and consequently discarded to waste streams, particularly municipal solid waste. It's estimated that in total, plastics represents 30% of the packing materials used in USA in 2000. However, only about 2% of all plastics are recycled. The remainder is deposited off in landfills or burned, in both cases putting a serious burden to the environment. The table 1.1 summarizes the composition of waste developed at the Biyagama Free trade Zone in Sri Lanka.

Table 1.1: Composition of solid waste arising at Biyagama (Export processing zone)

Component	Weight per day	Percentage
waste rubber	1.5	3.8
polythene bags	<0.1	0.1
food waste	1.5	13.5
leather off cuts	<0.1	0.1
polyester	0.2	2.0

Source: Data by the Board of Investment Sri Lanka, 1999

Pollution of the environment was generally identified in 1960's as a major problem in the world. The existence of pollution was acknowledged with the development of new technologies. Pollution due to solid wastes greatly accompanied by polythene. They accumulate and do not degrade easily. High-income countries in the world have the highest waste generation rates, ranging from 0.5 - 1.1 kg per day. (World Bank report, 1999) Low-income countries in Asia have a low waste generation rate ranging from 0.4-0.9 kg per day. The percentage of plastics in low-income countries is rather small (2-8%) when compared with high-income countries (9-16%) (Anon, 1999).

Polythene consumption by the man has greatly increased because they are very versatile. Mostly they are used as lunch sheets, bags, film wrappers etc. Consumption makes them accumulate because of their low biodegradability. Infact polymer scientists historically have concentrated on making plastics more and more durable and reproducible. They are resistant to degradation due to many reasons.

i. Hydrophobic nature of plastic resins
ii. Microorganisms have not yet adapted to new polymers

Although polythene as a packaging material serves a number of purposes, it has increasingly become a problem to the environment, due to their low biodegradability.
Over the past century, plastic formulations have been developed to resist deterioration and provide long service. To achieve the desired durability various agents have been introduced to reduce microbial degradation and photo oxidation. Therefore, this problem becomes increasingly hazardous to the environment.

Most of the developed countries recycle plastics, polythene and other packaging materials. However, countries like Sri Lanka have not initiated such environment friendly industrialization policies due to improper planning and therefore most of the combustible waste is burnt at household level. This kind of low temperature burning of plastics release lot of hazardous chemical compounds such as Cl_2, N_2 and CO_2 to the environment.

Plastic waste is difficult to dispose off, it does not biodegrade in landfills and generates many kinds of toxic gases even during incineration at high temperatures. Biodegradable polymers, both naturally occurring such as starch, protein, cellulose, bacterial polyester, etc. and synthetic polymers such as polylactic acid, polycaprolactome, polyvinyl alcohol etc.. are particularly an attractive option for addressing the environmental concerns. Instead of looking forward, some have tried to look backward to the basic by developing natural materials from plants such as leaf, bark, fruit, husk, etc. These materials offer another attractive alternative to plastic for some applications, particularly disposable food package.

Over the last fifty years, plastics petroleum based non-biodegradable polymers, have revolutionized the way we live because of their advantages in use. Such as a wide range of available properties to fit specific applications, formability into a practically unlimited range of shapes and stability in ambient and many hostile environments, plastics now comprise of an essential component of most of the articles on which we depend in our everyday lives. This is particularly true for packaging.

All packing materials have advantages and disadvantages with regard to the total environmental impact. The plastic industry has also devoted a significant effort to increase the stability of plastic formulations to provide stable materials for a wide variety of applications. So the consumers usage and also the litter problems and environmental concerns also increase. Obviously there is a global need for biodegradable packing materials. There are production processes of co-polymers of plastics and natural polymers with biodegradable package film to reduce the environmental hazard created by polythene.

Therefore, the interest in production and utilization of edible, biodegradable and compostable films and coatings prepared from various biopolymers such as polysaccharides, proteins, lipids or combinations of these components remains grate. (Kester and fennema, 1986)

Therefore, this study was focused on developing a transparent flexible package film using naturally occurring plant tissues.

Objectives:

1. To investigate the potential of developing a transparent package film using *Alstonia* plant leaves.
2. To study the stage of leaf maturity on the strength of the film.
3. To identify the microorganism groups responsible for anaerobic digestion and separation of films from plant leaves.
4. To evaluate the physical properties of the film.

CHAPTER 2: REVIEW OF LITERATURE

2.1 Packaging

Packaging is an essential part of processing and distribution of foods. It has assumed great importance recently, whether it be for food products or any other consumer items. It is obvious that the produce in it's natural or processed form should be sold with out spoilage, in attractive form, ensuring retention of their flavor, color, texture and nutritional value. A packaging must provide:

a) Complete protection of the packed material against moisture, light, dust, air, foreign odors, pests, corrosion and microbes.
b) Strength and stability during storage, handling and marketing
c) Size, shape and appearance to promote salability of the product and convenience in use
d) A packaging material suitable for use in contact with food stuffs, and it's cost must be compatible with the value of the contents and the degree of protection needed.
(Gupta, 1993)

2.1.1 Requirements for effective food packaging

1 be non toxic
2. protect against contamination from microbes
3. act as a barrier to moisture loss or gain and oxygen ingress
4. filter out harmful UV light
5. provide resistance to physical damage
6. be transparent
7. be tamper resistance or tamper evident
8. easy to open
9. have dispensing and sealing features
10. be disposed of easily
11. meet size ,shape and weight requirements
12. have appearance, printability features

13. protect against ingress of odours or environmental toxicants
14. be low cost
15. be compatible with the food
(Potter, Hotchkiss, 1987)

2.1.2 Definition of packaging

A coordinated system of preparing goods for transport, distribution, storage, retailing and end-use. Thus packaging has also been described as a 'complex, dynamic, scientific, artistic and controversial segment of business'. At it's most fundamental packaging contains, protects, preserves and informs. Also it provides two more functions- those of selling and convenience.(Paine, Paine, 1992)

2.1.3 Types of packaging materials

1. metal: rigid metal containers, metal foils
2. glass
3. paper
4. plastic: cellophanes, polythene, vinyl derivates, polyesters, polyamides, water soluble edible films, other films

1. Metal

Two basic types of alloyed metals are used in food packaging; steel and aluminum.
Steel is used primarily to make rigid cans, where as aluminum is used to make rigid cans as well as thin aluminum foils and coatings. Until a few years ago, nearly all steel used to make cans were coated with a thin layer of tin to inhibit corrosion.
Aluminum is lightweight, resistant to atmospheric corrosion, and can be shaped and formed easily. But it has less structural strength than steel.

2. Glass

As a food packaging material, glass is chemically inert and an absolute barrier to the permeation of oxygen or water vapor. The principal limitations of glass are it's susceptibility to breakage, which may be from internal pressure, impact or thermal shock. Glass is primarily formed from oxides of metals, with the most common being silicon dioxide which is common sand.

3. Paper

The principal difference between paper, paperboard and fiberboard are thickness and use. Papers are thin, flexible and used for bags and wrappers; paperboard is thicker, more rigid and used to construct single layer cartons; fiberboard is made by combining layers of strong papers and is used to construct secondary shipping cartons. (Potter, Hotchkiss, 1997)

4. Plastics

Over the past thirty years, there has been a tremendous increase in the use of plastics replacing traditional packaging materials such as glass, metal, and paper. The raw materials for plastics are petroleum, natural gas and coal. They are formed by a polymerization method that creates linkages between many small repeating chemical units (monomers) to form large molecules or polymers. Many plastics contain very small amounts of additives such as plasticizers, antioxidants, lubricants, antistatic agents, heat stabilizers and UV stabilizers. These are added to facilitate processing of plastics or to impart some desirable properties to the plastics.

For eg. Plasticizers are added to soften plastic, thus making them more flexible and less brittle for use in cold climates or with frozen stored products.

Plastics may be made as flexible films or as semi rigid or rigid containers to meet the varied packaging and processing requirements of food. Plastic films are made with a wide range of mechanical, optical, heat-sealable and barrier properties. Further more they can be coated with another polymer or metabolized to give a laminated structure with superior properties.

Flexible films

Flexible packaging describes any type of material that is not rigid, but the term flexible film is usually reserved for non-fibrous materials which are less than 0.25mm thick.

a. Flexible Single films

Most polymer films are made by extrusion, in which pellets of the polymer are melted and extruded under pressure as a sheet or tube. Other methods are callandering and casting. The most important types of film for food packaging are described below.

Table 2.1: Properties of some flexible single films

Materials	**Structural unit**	**Important properties**
Cellulose	Glucose	Good strength, poor water and gas barrier properties, good printability, no heat sealability
Polythene	Ethylene	Good strength, flexible, extensible, high water barrier, poor gas barrier, low melting point, good heat sealability
Polyester	Ethylene glycol + terephthalic acid	Stiff, strong, inert, excellent mechanical properties, poor heat sealability, moderate water and gas barrier
Polyamide	Diamine + various acids	Stiff, strong, inert, clear excellent machinability, heat sealable, poor water barrier, high gas barrier when dry

Polypropylene	Propylene	Tough, inert, clear, low melting point, high water barrier, poor gas barrier
Polystyrene	Styrene	Stiff, strong, brittle, low water and gas barrier
Polyvinyl-chloride	Vinyl chloride	Soft, inert, clear, extensible, good water barrier and moderate gas barrier
Polyvinylidene-chloride	Vinyl alcohol + vinylidene chloride	Inert, clear, not very strong, high melting point, heat sealable at high temp. , excellent water and gas barrier
Ethylene vinyl alcohol	Vinyl acetate + ethylene	Tough, clear, inert, highly extensible, low melting point, heat sealable, intermediatewater barrier, poor gas barrier
Ethylene vinyl alcohol	Vinyl alcohol + ethylene	Strong, stiff, inert, heat sealable at low temp. , low water barrier, high gas barrier
Ionomer	Ethylene + methacrylic acid	Tough, inert, clear, heat sealable at low temp. , intermediate water barrier and high gas barrier

Adopted from Jelen (1985) and Brown (1992)

b. Flexible coated films

Films are coated with other polymers or aluminum to improve the barrier properties or to impart heat sealability. eg. Nitrocellulose is coated on one side of cellulose film to provide a moisture barrier but to retain oxygen permeability. A nitrocellulose coating on both sides of the film improves the barrier to oxygen, moisture and odors and enables the film to be heat-sealed when broad seals are used.

c. Flexible laminated films

Lamination of two or more films improves the appearance, barrier properties or mechanical strength of a package. The most versatile method is adhesive laminating (or dry bonding). An adhesive is applied to the surface of one film and dried. The two films are then pressure bonded by passing between rollers.

d. Flexible coextruded films

This is the simultaneous extrusion of two or more layers of different polymers.
They have three main advantages over other types of films,

1. They have very high barrier properties, similar to multi-layer laminates but produced at low cost.
2. They are thinner than laminates and closer to monolayer films and are therefore easier to use on forming and filling equipments
3. The layers do not separate (Fellows, 1996).

2.2 Biodegradable polymers

The burden of accumulating plastic waste has stimulated research and development of degradable polymers. Roughly 16 to 18 billion pounds (7.2 to 8.1 million kilograms) of plastics are disposed of after single use in US, and in similar amounts in Western Europe.

Today's plastics are tailored with little consideration for disposability or the impact on resources used to make them. Synthetic polymers were originally developed for their durability and resistance to all forms of degradation, including biodegradation. They were also chosen for performance characteristics achieved through molecular weight control, functionality and morphology.

Biodegradable plastics offer an alternative to these traditional non-biodegradable petroleum-based polymers. The principal driving force behind the technology is the solid waste problem with regard to the decreasing availability of landfills. In the last decade there has been renewed interest in developing materials that mimic plastics but have a significant component from agricultural commodities.

2.2.1 what makes a polymer a biopolymer?

Biopolymers are defined as biologically degradable polymers. According to the American Society for testing materials (ASTM), Biopolymers are degradable polymers in which degradation results from the action of naturally occurring microorganisms such as bacteria, fungi and algae. This means that Biopolymers can either be produced from natural raw materials like starch, sugar and cellulose as well as fossil oil.

Biodegradable polymers are divided into two classifications: Natural and synthetic.

Natural polymers are isolated pure polymers with no physical or chemical modifications. The most widespread natural polymers are the polysaccharides, proteins and polyesters such as polyhydroxyalkanoates. Synthetic polyesters made from aliphatic diols and aliphatic acids that are also biodegradable. Such polymers include polyglycolic acid, polylactic acid and polycaprolactone. Other biodegradable synthetic polymers include polyamides, polyanhydrides, polyurethenes, polyethers and other condensation polymers. While natural polymers are generally inexpensive, they are difficult to process into useful end products. eg. Products made from starch or proteins are brittle, inflexible and moisture sensitive (Resource, May 2000)

2. 2. 2 Different types of biopolymers

The most common way to divide biopolymers is on the basis of the raw material used for production. However some biopolymers can be produced from different raw materials. Therefore the types of biopolymers are the starting point of the following list.

1. Starch polymers

Ranging from hundred percent starches to polymers containing starch and PCL (polycaprolactone) Starch is abundantly present in many crops. It's stored in granules with in the plant. This facilitates the isolation from the plant. Starch may be modified in order to become a thermoplastic. This makes the starch polymer suitable for current processes in the plastic industry like injection moulding and extrusion. Thermoplastic starch has an affinity with moisture. The material therefore is not suitable for wet food packaging applications. Direct contact with water is only possible for a short time. By acetylation a certain resistance to water may be achieved. By adding PCL the flexibility of the boiplastic increases. Starch polymer has good Oxygen barrier properties. These are the most produced and used biopolymers at the moment.

2. Polylactic acid (PLA)

Polylactic acid is derived from lactic acid for which carbohydrates in sugar beets, potatoes, wheat, maize and milk are the source. Polylactic acid is a substance familiar to the human body, as we ourselves produce it by every muscle contraction. The body can break it down. PLA can be processed through for example injection moulding, foil blowing and deep drawing. PLA may be applied as a coating. PLA is water-resistant but cannot withstand high temperatures (> 55° C) in comparison to starch the degradation process is very slow. However, within a composting facility it can be broken down in 3 – 4 weeks.

3. Polyhydroxyalkanoates (PHA's)

Polyhydroxybutyrate (PHB), Polyhydroxybutyrate/valerate (PHB/HV)
PHA's are generally derived through fermentation of glucose, sucrose and fatty acid by microbes. PHB can be processed through injection moulding and deep drawing. It is an excellent material for the coating of paper coffee cups. The most important features of PHB are the resistant to high temperatures up to 120 °C and the resistant to water.

4. Protein polymers

Corn zein, soy protein isolates, wheat gluten, milk proteins, egg albumin have been tested on making packaging films. Few protein films are commercially used. Main limitation of protein films is limited effectiveness as a water vapor barrier.

5. Cellulose polymers

Cellulose esters, cellulose ethers, cellophane
Cellophane is one of the oldest packing materials. Cellulose pulp from trees or cotton may be used to produce cellophane. It is a relatively expensive packaging material, which can be used for a wide range of products such as cd's candy and cigarettes. The higher price is a reason why a large market share was lost to polypropylene. At the moment new applications in which the specific properties may be used, are sought for. The material is transparent and has good folding properties. The foil has a gas barrier and if a coating is applied may resist water vapor as well.

The field of biopolymers, while still in its early stages, is growing in popularity everyday. Some biopolymers can directly replace synthetic plastics in traditional applications, while others possess unique properties that may open new applications. For most biopolymers, it's too early to determine if they'll be economically feasible on a large, industrial scale.
When you run in to a spider's web, the last thing on your mind is," what a great material!" but consider that the spiders silk has a tensile strength 16 times greater than nylon! At the same time, silk maintains a very high degree of elasticity. Attempts to manufacture genetically modified silk have thus far been disappointed due to low yields.

Chitin, a polysaccharide found in the exoskeleton of insects and shellfish, possesses many desirable characteristics. Chitin's most important derivative, chitosan, is nearly a" model" biopolymer with it's useful physical and chemical properties, high strength, biodegradability and no toxicity. Infact, chitosan brings new meaning to the word "biodegradable" as the human body easily breaks it down into simple carbohydrates, carbon dioxide and water. This accounts for the research that is trying to use chitosan in drug delivery systems.

Lactic acid is produced by the microbial fermentation of sugars such as glucose or hexose. Feedstock's can include potato skin and corn. The lactic acid monomers can be used to create low or high molecular weight polylactic polymers. PLA commodity polymers are being developed for use as pulping additives in paper manufacturing and as bio packing materials. PLA's current price level of $5.00/lb (US) will have to be significantly improved upon before it gains wide acceptance. Currently, the medical community is the primary user of PLA.

Table 2.2: Commercially available biopolymers

COMPANY	PRODUCT	APPLICATION	COMMENTS
Zeneca bio products	BIOPOL resin	Films and coatings	Assessing full production
EcoChem (dupont)	PLA-PLA copolymers	Medical and packaging	100 million/lb per year
Cargill, Ins	Polylactide	Packaging	10 million/lb per year
Argonne National Labs	PLA from potato waste	Packaging	Available for license
Warner-Lambert	NOVON	Structural material	100 million/lb per year

Source: Haslego, 2001

2.2.3 Properties of Biopolymers

Properties of Biopolymers depend on the raw material they are based on, on additives used and on the (chemical) modifications during the production. Some of the most obvious properties of biopolymers are on aspects like moisture or water resistance, opacity and resistancy to high temperatures. These properties determine in what kind of application a biopolymer may be used.

Some Biopolymers are impervious to water and retain their integrity during their normal use but readily degrade in biologically rich environment. However, full biodegradability can occur only when these materials are disposed of properly in a composting site or landfill.

Today there are three major degradable polymer groups that are either entering the market or are positioned to enter the market. They are,

1. Polylactides (PLA)
2. Polyhydroxybuteratem (PHB)
3. Starch based polymers

First generation of degradable polymers, which were largely commercialized in 1980's, did not satisfy the public view of complete degradation. Second generation polymers began being introduced during the last five years. Although they are degradable, the industry has not promoted them. One reason is these new polymers are higher priced than the commodity polymers typically in used in plastic applications. However, produces are currently working towards bringing down the price of degradable polymers by increasing production capacity and improving process technology. Five years ago PLA and PHB sold for more than $25.00 USD per pound. Today PLA, depending on quantities, is between $3.00 USD /pound and PHB, in large quantities is nearly $4.00 USD /pound.

Though recent advances in production technology have helped to lower the prices of some degradable resins, prices are still higher than for petroleum-based plastics. This suggests that in the short term, companies making degradable polymers will continue to focus on niche markets. The long-term outlook for marketing these products is still uncertain.

Table 2.3: Properties of Selected biopolymers and synthetic polymer films

Film type	Thickness (mm)	TS (Mpa)	%E	Reference
Wheat gluten	0.101	2.6	276.2	Gennadios and others(1993a)
Whey protein isolate:glycerol	0.11	13.9	30.8	MacHugh and Krochta(1994b)
Dialdehyde starch-corn zein protein	-	16	2.0	Spence(1994)
Corn zein protein	0.089	0.4	<1.0	Adyt and others
Soya protein isolate	0.083	3.6	169.3	Gennadios and others
Soya protein isolate:Glycerol	0.066	5.23	90.27	Brandenburg and others(1993)
Alkali treated soya protein isolate:Glycerol	0.066	4.33	115.2	Brandenburg and others(1993)
MSPI-PEO-LDPE	0.20	2.4	100	Ghorpade and Hanna(1996)
Cellophane	0.036	114.0	20.0	Aydt and others(1991)
HDPE	0.025	17.3 to 34.6	300	Briston(1988)
LDPE	0.025	8.6 to 17.3	500	Briston(1988)

MSPI-PEO-LDPE	- modified soy protein isolate
HDPE	- High-density polyethylene
LDPE	- Low-density polyethylene

Source: Journal of food science, Vol. 65 no-4, 2000

2.2.4 Importance of Biopolymers

Waste disposal issues

According to a study by the US Environmental protection agency (EPA), plastics alone account for roughly 21%(by volume) of the nearly 200 millions tons of municipal waste generated each year in the USA. Integrated waste management practices including recycling, source reduction of packing materials, composting of degradable waste and incineration may help bring waste disposal under control. However this will not solve the importation of the petroleum products.

Instead of making plastics from conventional petroleum products, they can be made from lactic acid. Lactic acid is produced (via starch fermentation) as a co-product of corn wet milling, which can be converted to polylactides (PLA) or it can be produced using the starch from food wastes, cheese whey, fruit or grain sorghum. By using lactic-based plastics, the US could save 50-90 trillion Btu per year. This is equivalent to 9-15 million barrels of oil.

Some plastics need to be durable like parts in a car. Yet there are many plastics that are used only once or have a limited life before being thrown in to a landfill or incinerator. Microbes poorly degrade plastics unlike most organic polymers (if at all). Environmentally degradable polymers are one potential solution to replacing petroleum-based polymers. Potential uses of these polymers are plastics intended for one time or limited use, for eg. Those used as fast foods wrappers and water-soluble polymers detergents and cleaners, and for use in the printing industry. Since the main bulk of domestic waste is made up of plastic there is a great deal of interest in recycling plastic and in producing plastic materials that can be safely and easily disposed of in the environment. Current degradable polymers are design to degrade either biologically, photolytically or chemically depending on the environment that they will encounter after use. Degradation pathways will ultimately lead to the bioconversion of the polymer in to carbon dioxide (aerobic) or carbon dioxide/methane (anaerobic) and biomass. The goal is to reduce the environmental loading of polymeric wastes through biological recycling. (Biby, 1999)

2.2.5 Application areas

Due to their high prices, most current applications for degradable polymers are in niche areas with unique environmental considerations. Although these amounts are high when compared with conventional resins like PVC or styrene, they can degrade in the environment. Another important feature is that they do not typically contribute to environmental pollution during their manufacture.

In 1993, total demand for degradable polymers (including cellulose), in US, Western Europe and Japan reached (25 million pounds.)In theory conventional plastics may be substituted by biopolymers in many applications, in practice substitution is not always feasible. Besides the technical development that is needed for some applications, the application has to be economically feasible within a reasonable term. The economic feasibility depends on the investments needed for material and product development and the added value of the biopolymer in the application. The added value is of great importance because the prices of biopolymers are than these of conventional plastics.

2.2.6 Future growth

Several factors, besides cost, will be important in determining the future growth of degradable polymers. One major obstacle is lack of composting infrastructure. Large scale composting would provide the ideal disposable environment for spent degradables. Western Europe has made progress towards developing a composting infrastructure.

Many biobased resins can be processed on conventional plastic moulding equipment and, depending on the properties of the specific resin, can be converted into many types of plastic products. These include, but are not necessarily limited to single use items like: compost bags (lawn and leaf); disposable food service items (eg. curtly, plates, cups); packaging materials (eg. Loose fill, films); but they also include more durable products like; coatings (eg. Laminations, paper coatings); and other injection moulded and sheet extruded products (phone and other cards and sheet printed plastics). The desired property of the product generally determines the relative amounts of additives used in the resin.

2.3 Plant leaves as biopolymers

Plant leaves has been tested to make Eco-friendly utensils.
Gunasena and Weerasinghe in 1999 have conducted a research on using Kottamba (*Terminalia catappa*) and Kanda (*Macaranga peltata muell. Arg*) leaves to make disposable plates and dishes. They have used Beli as the binding material of the leaves. They have also tested them with different thickness of the leaves. Utensils have been dried in the sunlight. They have found that plates with two leaf layers are more appealing than others.

Banana leaf has been tested to use as a disposable food package in Thailand, and it has been very successful. (Indian food packer,1998)

2.3.1 Plant cuticle

The entire sheet system of an herbaceous plant is covered by a cuticle that slows down water loss from all of it's parts. Most of the cuticle is composed of a heterogeneous mixture of components collectively called cutin. Where as the remainder consist of overlaying waxes and of pectin polysaccharides attached to the cell wall. Cutin is a heterogeneous polymer consisting largely of various combinations of members in two groups of fatty acids, a group with 16 carbons and one with 18 carbons (kolattukudy, 1980a, 1980b;Holloway, 1980). Most of these fatty acids have two or more hydroxyl groups, similar to ricinolenic acid.
(Salisbury et al., 1992)

2.3.2 The tree "Alstonia" that has been used

Scientific name : *Alstonia macrophylla*
Sinhala name : Havari nudge
Tamil name : Velai maram
Family : Apocynaceae
Origin : Malesia
Leaves : In whorls of 3-4, cuneate base, abruptly short acuminate apex, lateral veins distant, entire margin
Trunk : smooth pale brown grey, milky latex is there
Flowers : white, in umbels
Fruits : pendulous, follicles, glabrous when ripe
(Ashton et al., 1997).

2.3.3 Fungi and litter Decomposition

The interaction within and between the fungi, bacteria and invertebrate animals leads to the decomposition of litter. Among the major groups of litter decomposing microorganisms, fungi are the most active. The colonization and successions of fungi on various leaf litters and observed a specific pattern that resembled the one proposed by Hudson,1968. Here the primary colonies were Deuteromycetes and ascomycetes.

Primary fungal colonies of Rhyzopus, Mucor, Zygorhyncus, Penicilluium and Fusarium by interacting factors of soil, climate and litter characters such as moisture, temperature, pH, depth, aeration and organic and inorganic nutrients. Moisture is one of the important factors that regulates the growth and activities of fungi on decomposing litter.

Herbaceous plants normally have a short life span. Even though most herbaceous plants die off, it decomposes quickly than woody litter. Fungal species colonize the litter and begin to sporulate with the commencement of decomposition. The old leaves are colonized first, followed by the young ones. Which normally starts with Pycomycetes followed by deuteromycetes such as Penicillium, Trichoderma, Aspergillus and Fusarium (Sharma et al., 1995)

2.4 Package testing

Many test procedures exists to measure quantitatively the protective properties of packaging materials and entire containers. These can be divided in to chemical and mechanical properties.

Examples of chemical tests are those used to identify plastics, determine if portions migrate to foods, and measure resistance to greases. Mechanical tests include such things as barrier properties, strength, heat seal ability and clarity. (Potter, Hotchkiss, 1988)
Over the last few years several standard test methods have been developed to assess biodegradability. They include enzyme assays, plate tests, biological oxygen demand (BOD), respirometry, and exposure to natural environments. Organizations that have published such tests include the American Society for Testing Materials (ASTM), the Ministry of International Trade and Industry (MITI).

CHAPTER 3: MATERIALS AND METHODS

3.0 Introduction

An observation has been made that a fairly large leaf fallen into a garden fishpond was disappeared in few days leaving a clear, thin, flexible and transparent film floating on water. This natural phenomenon of producing a flexible transparent film from a plant leaf was then investigated to study the possibility of using it for developing biodegradable transparent packaging. The specific leaf was a leaf fallen from an *Alstonia* tree in the garden and micro and macro organisms were responsible in separating the epidermis within about 10 days. This leaf structure was assumed to be very specific for the tree species as the common leaf ends up with a mesh of its ribs and veins (Dharmasena, 2000) after decomposition. Therefore the above process was investigated in detail in this research study.

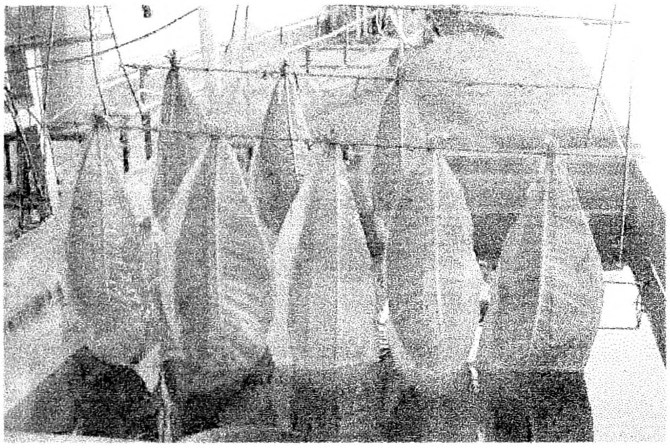
3.1 Colour measurement

The stage of the maturity of the leaf was determined by colour. Five stages of maturity of the *Alstonia macrophylla* leaves were used at the beginning of the experiment. A colour chart was prepared for the five maturity stages of leaves. (No.1- immature leaf and No. 5- fully matured leaf). Colour values of leaves were measured using a Minolta chromameter, CR-300. Six measurements were taken on upper side of each leaf and 'L', 'a*' and 'b*' values were recorded and mean values were used to prepare the color chart of the leaf.

3.2 Transparent film separation

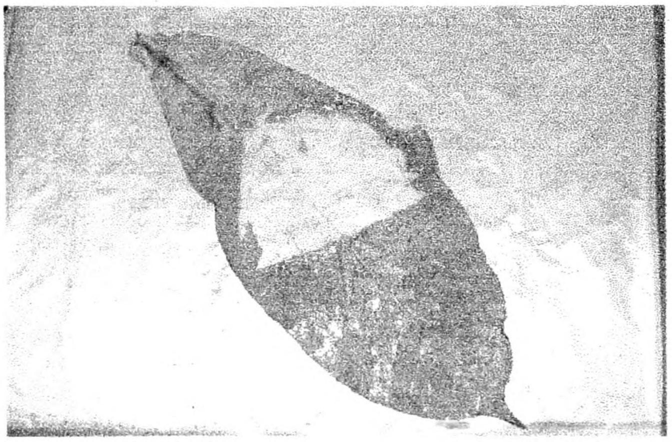
Leaves of five stages were used for separating the film. Leaves were tied to a frame and kept in an inoculation tank under submerged condition (Plate 3.1). A new tank was used for the microbial fermentation and water from the garden fishpond was used to introduce microbes.

Plate 3.1: Leaves taking out from the tank



The film separation was tested and found that transparent films were not developed after expected time period. The same observation was made with the leaves fermented in the previous fishpond. It was assumed that the film separation is a joint work of microorganisms that were in that tank. Therefore, leaves were placed on an Aluminum foil and the cellular part was removed manually. Then they were washed with clean water to remove stains and debris.

Plate 3.2: Film separation -half cleaned leaf



Film cleaning was carried out in three different techniques.

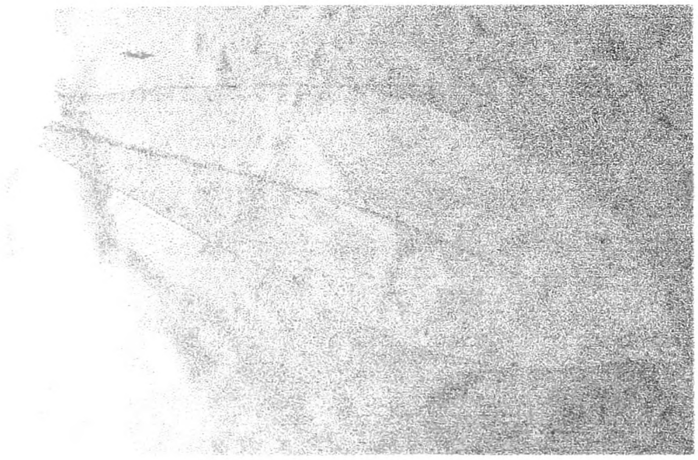
- Cleaning with bleaching (food grade chlorine)
- Cleaning with alum salt
- Cleaning with pipe water

Bleaching is a strong oxidizing agent reacting with most elements and compounds. Moist chlorine is extremely corrosive; HOCl are salts of hypochlorus acid. Ca $(Ocl)_2$ is the predominant dry form used in. 1g/*l* is the most common dosage in purifying water.

Alum is a commonly used metal coagulant in water treatments. $Al_2(SO4)_3.14.3H_2O$ is far the most widely used coagulant; the commercial product is known as alum. The dosage in water purification is in the range of 5-50g/*l*, with the higher conc. needed to clarify turbid surface waters. (Hammer and Hammer Jr., 1988)

Then the films were dried under room temperature and were collected separately.

Plate 3.3: A cleaned film from a leaf



3.2.1 Identification of decomposing microorganisms

Microorganisms were identified by inoculating a piece of plant leaf in fungal (PDA) and bacterial mediums (medium for actinomycetes). Specimens were observed under 10*25 magnification. Cultures were observed after four days from a phase contrast microscope (manual photo system, Nikon 104, E-400, Eclipse)

3.2.2 Observation of the leaf cross-section

Cross section of the *Alstonia macrophylla* leaf was observed using a phase contrast microscope (manual photo system, Nikon 104, *E-400)* to identify the location of the film.

3.3 Film preparation

Dried transparent and flexible leaf films were pasted together with gelatin (Motha,). Gum preparation was done by simply dissolving gelatin in hot water and boiling it for few minutes. Gelatin was dissolved in water until it gets to a sticky texture. Gelatin was carefully applied with a paintbrush on cutin films avoiding unnecessary damages. Finally films were allowed to dry under room temperature. Layers of 1,2, 3, 4 of films were pasted together in the same manner and they were allowed to dry under room temperature. Three sets of each layered films were made with films cleaned under three different cleaning procedures and used for the evaluation.

3.4 Measurement of transparency

Transparency of films was measured using a photometer/radio meter/quantum (model LI-185B, Li-cor., inc.) at six random positions of the film. Mean values were taken at the end.

3.5 Film thickness measurement

Film thickness was measured using a handheld Gauge meter with an accuracy of 0.01 mm. ('Tecklock', Tecklock Corporation Japan.0.001"). Five thickness measurements were taken on each test piece used in tensile testing along the length of the stripe. The mean values were used in Tensile Strength calculations.

3.6 Tensile strength and percentage elongation at break

Tensile Strength and percentage elongation at break were evaluated with a type: BZ 2.5/TN IS, ZWICK/material testing machine (August-Naglstr 11,D-89079ulm). Films were cut into 2.5*10cm stripes and tests were performed according to ASTM D-882-91method (Appendix). Initial grip separation was set at 50mm and crosshead speed was set at 50mm/min. Tensile Strength was calculated by dividing maximum load by the cross-sectional area of the sample. Percentage elongation at break was calculated as the percentage of change by dividing film elongation at the moment of rupture by initial gauge length (150mm). Tensile properties of different types of films and treatments were determined in the experiment. Each test was conducted with 4 replicates

3.7 Statistical analysis

Statistical analysis was performed using SAS software (SAS Institute Inc. 1998). Least significant difference test was used to determine significance of difference between means of the tensile properties.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Colour measurement

Mean values of L, a* and b* (Table 4.1) were used to prepare a colour chart for leaf maturity.

Table 4.1: Colour measurement values

Leaf maturity stage		1	2	3	4	5
L	Mean	40.62	37.0	38.9	37.9	65.74
	Range	39.71-41.73	36.23 - 38.06	37.5 —41.46	36.42 – 40.08	61.08-71.76
a	Mean	-18.87	-13.68	-14.38	-14.37	0.13
	Range	-19.95- -17.66	-14.7 - -12.94	-16.9 - -12.72	-16.57- -13.02	-5.87 – 3.58
b	Mean	24.09	15.83	18.0	17.32	57.87
	Range	22.0 - 26.51	14.8 - 17.53	15.12 – 22.11	14.93 – 21.32	47.98-67.23

1,2,3,4, stages of leaves which are green in colour had almost similar values for L, a* and b* than stage 5 which was yellow in colour (Plate 4.1).

Plate 4.1: Colour chart of Alstonia

1

2

3

4

5

	Leaf No.	1	2	3	4	5
L	Mean	40.62	37.0	38.9	37.9	65.74
	Range	39.71 - 41.73	36.23 – 38.06	37.5 – 41.46	36.42 – 40.08	61.08 – 71.76
a	Mean	-18.87	-13.68	-14.38	-14.37	0.13
	Range	-19.95 – -17.66	-14.7 – -12.94	-16.9 – -12.72	-16.57 – -13.02	-5.87 – 3.58
b	Mean	24.09	15.83	18.0	17.32	57.87
	Range	22.0 – 26.51	14.8 – 17.53	15.12 – 22.11	14.93 – 21.32	47.98 – 67.23

ALSTONIA-LEAF COLOUR CHART

Department of Agricultural Engineering
University of Peradeniya, 08-388923

4.2 Film isolation

Only the stage 5 in the leaf color chart was successful in separating the film. Films of other four stages could be isolated only in water but they could not be taken out of water without damaging. They were very fragile and broke out quickly and therefore rejected for film separation. Seven to ten days were taken for microbial degradation for the easy separation of the film. The dried films were slightly yellowish. The films were strong and flexible enough to be handled, and tended to curl up. Bleached films were more transparent with a white to slight yellow tint.

4.2.1 Decomposing microorganisms

Microorganisms were mainly in the category of fungi and a few bacteria were also present. This is because fungi are the major microorganism group responsible for degradation of litter. It has also been reported that old leaves normally starts decomposition by colonization of phycomycetes followed by deuteromycetes. (Sharma et al., 1995). Classes identified in the test were mainly phycomycetes and deuteromycetes belonging to the genes of Fusarium and penicillium while the bacteria were mainly actinomycetes.

4.2.2 The leaf cross-section

Major objective of looking at the cross section was to observe the transparent cutin layer and its position. Cutin layer was found at the top and the bottom of the leaf, top layer was thicker than the bottom layer and it had one to two layers of dead cells with it (Plates 4.2 and 4.3). In some places traces of chlorophyll could be seen on the cleared cutin layers.

Plate 4.2: Cross section of a leaf

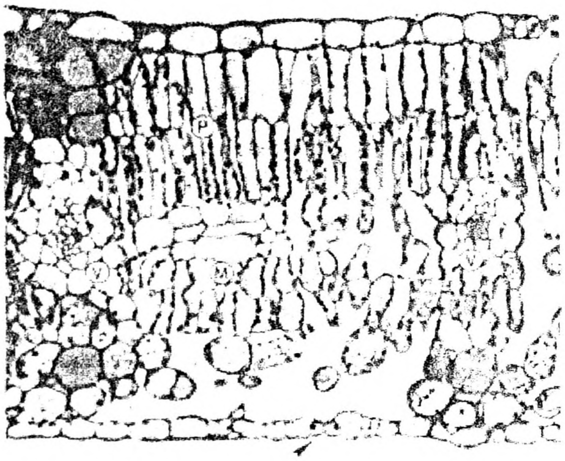


Plate 4.3: Cutin layer

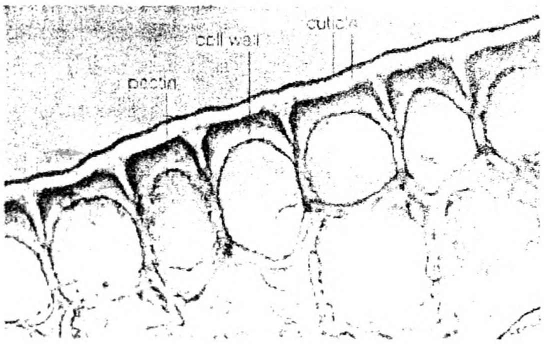


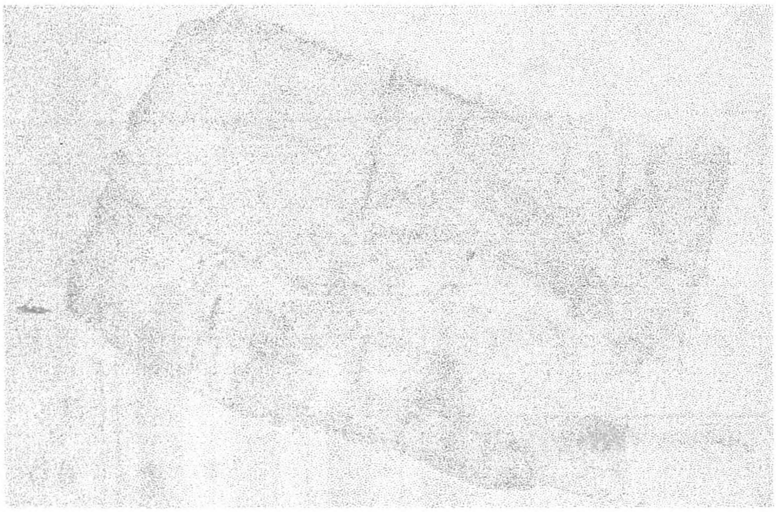
Figure 15-6 [illegible] (Courtesy P. J. Holloway.)

Source: Salisbury et al., 1992

4.3 Preparation of the biodegradable transparent film with different layers

Gelatin was used to paste films one over the other (Plate 4.4) as the adhesive is used as a food. Cornstarch and Manihot starch may also be used as adhesives although they were not tested in this method. Non-uniformity of film thickness occurred due to uneven application of gelatin and also unevenness of cutin layers of the leaves.

Plate 4.4: A film developed with number of layers



4.4 Measurement of the transparency of the film

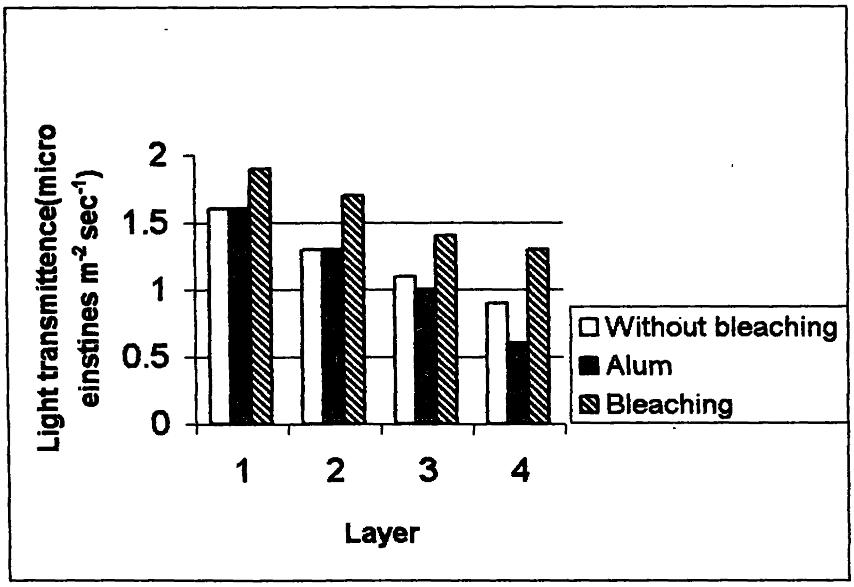
Table 4.2: Mean values of Light transmittance measurement

Number of layers	**Light transmittance (micro einsteins m^{-2} sec^{-1})**		
	Without Bleaching	***Alum***	***Bleaching powder***
1	1.6	1.6	1.9
2	1.3	1.3	1.7
3	1.1	1	1.4
4	0.9	0.6	1.3

Basic reading 2.3

Basic scale 30

Figure 4.1: Light transmittance measurement



Light transmittance values were varied from one place to another even in a single film. Films are not uniformly transparent due to many reasons.

- Cutin layers are not equal in all the places of leaves.
- Application of gelatin also makes an effect in the transparency of films

According to the figure 4.1 we can see that the bleached films were more transparent than unbleached and treated with Alum. With the increase of the number of layers transparency decreased gradually.

4.5 Film thickness measurement

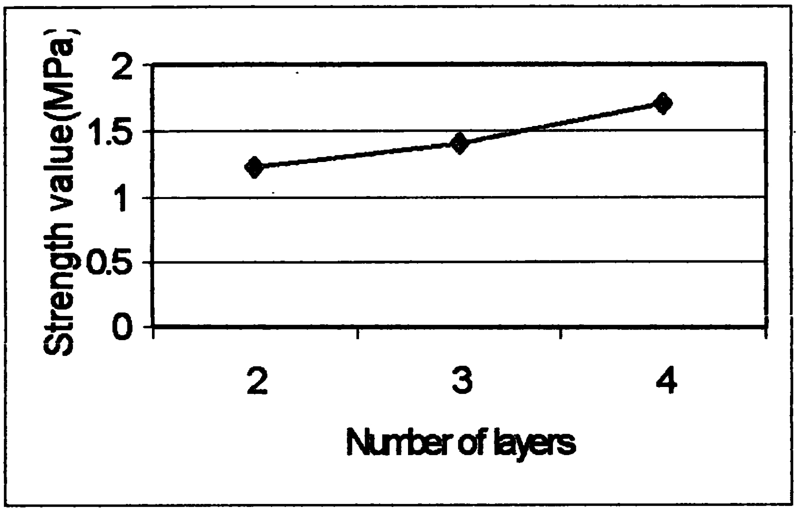
Cutin films have been isolated from naturally occurring cutin layers of plant *Alstonia macrophylla*. So it is not an even layer. Sometimes thickness values of films greatly vary due to this reason. (Appendix II, detailed results of material testing)

4.6 Tensile strength and elongation at break

Table 4.3: Average values of mechanical properties

Mechanical property	**4 layers**	**3 layers**	**2 layers**
Tensile Strength Nmm^{-2}	1.7041	1.4008	1.2291
Elongation %	1.0875	1.091	0.8958
Elastic modulus Nmm^{-2}	155.24	153.582	139.928
Thickness mm	0.1475	0.1250	0.0839

Figure 4.2: Increase of strength with the number of layers



According to the table 4.3 and figure 4.2, ultimate tensile strength and thickness values have gradually increased with the increase in number of layers. But there were no significant differences between the means of tensile strength (TS) values and the number of layers at 5% level (Appendix III). TS values of films have increased by 13% and 22% when the layer number was increased from 2 to 3 and 4 respectively. A 40% increase in the TS value could be seen from 2 layers to 4 layers.

Values of Elastic modules and elongation have almost increased with the number of layers. But the values do not show a considerable difference from 3 layers to 4 layers. This may be due to the unevenness of cutin layers of films.

Table 4.4: Change in strength values with the different treatments

No of layers	**Strength values (MPa)**		
	Without Bleaching (3)	***Alum (1)***	***Bleaching (2)***
2	0.7475	1.0075	1.9325
3	1.0725	1.2350	1.8950
4	1.5850	1.2000	1.5275

Although a statistical analysis has been carried out, there is no significant difference between the treatments and the mean values of TS of layer 3 and 4 at 5% level (Table 4.4). Only layer 2 has a significant difference from treatment 2 with 1 and 3 treatments. The TS values of the cutin films developed are compatible to the other biopolymer films like corn zein and wheat gluten developed by other scientists (Table 4.5).

Table 4.5: Properties of other biopolymers

Biopolymer	TS(MPa)	E%
Cutin film with 4 layers	1.704	1.08
Corn zein protein	0.4	<1.0
Modified Soya protein isolate film-LDPE-PEO	2.4	100

LDPE-Low density polyethylene
PEO-polyethylene glycol
Adapted from J. of food science, Vol. 65, 2000

However, it was observed that the cutin films have considerably lower impact strengths than those of synthetic polymer films such as HDPE and LDPE. Therefore, these films should be used in such applications where impact is not very important.

Chapter 5: Conclusions and recommendations

5.1 Conclusions

- A Thin, flexible and natural biopolymer film can be developed using *Alstonia* leaf.
- Only the fully matured leaf at falling stage could be used to separate the film. Microorganisms responsible in the bio-degradation process of *Alstonia* leaves and film separation were mainly fungi: Classes; phycomycetes and ascomycetes belongs to the genus penicillium and Fusarium. Actinomycetes was also responsible for the decomposition.
- Transparency of the films varies slightly with the cleaning or bleaching method of the film. Cleaning procedures also do not make much effect on mechanical properties of the films.
- The film mechanical property, Tensile Strength varied from 4.25- 0.48 MPa. Cutin films are compatible with other biopolymer films like wheat gluten and corn zein protein films.

5.2 Suggestions and recommendations

1. Other plant leaves should also be tested for separating cutin layers to make flexible transparent films.
2. Film layers should be increased beyond 4 layers (layer 5,6,7,8etc.) and should be evaluated on mechanical and barrier properties.
3. Properties other than TS and E% should be analyzed to get a better idea of the films.
4. Strength and biodegradability of the films should be studied by incorporating PGA (Propylene Glycol Alginate) and E% Values by incorporating glycerol or sorbitol. This can lead the bio-films mere towards artificial. So incorporation of natural fibre with cutin films can also be studied.
5. A detailed analysis of the composition and structure of cutin films is essential in order to form films with desired mechanical properties. So that films can be synthesized according to the requirement as a bio-film with a good appearance.

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Appendix I

ASTM D882-91: Standard Test Methods for Tensile Properties of Thin Plastic Sheeting

Designation: D 882 – 91

An American National Standard

Standard Test Methods for Tensile Properties of Thin Plastic Sheeting[1]

These test methods have been approved for use by agencies of the Department of Defense to replace Method 1013 of Federal Test Method Standard 406. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the determination of tensile properties of plastics in the form of thin sheeting, including film (less than 1.0 mm (0.04 in.) in thickness).

NOTE 1—Film has been arbitrarily defined as sheeting having nominal thickness not greater than 0.25 mm (0.010 in.).

NOTE 2—Tensile properties of plastics 1.0 mm (0.04 in.) or greater in thickness shall be determined according to Test Method D 638.

1.2 Two types of tension tests are described in these test methods, differing basically only in manner of load application. These test methods may be used to test all plastics within the thickness range described and the capacity of the machine employed.

1.2.1 *Test Method A: Static Weighing, Constant-Rate-of-Grip Separation Test*—This test method employs a constant rate of separation of the grips holding the ends of the test specimen.

1.2.2 *Test Method B: Pendulum Weighing, Constant-Rate-of-Power-Grip Motion Test*—This test method employs a constant rate of motion of one grip and a variable rate of motion of the second grip. The variable-rate grip is attached to a pendulum weighing head, and its movement is dependent on the load-deformation behavior of the material under test.

1.3 Specimen extension may be measured in these test methods by grip separation, extension indicators, or displacement of gage marks.

1.4 A procedure for determining the tensile modulus of elasticity is included, using Test Method A at one strain rate.

1.5 The values stated in SI units are to be regarded as the standard. The values in parentheses are provided for information only.

NOTE 3—This modulus determination procedure is based on the use of grip separation as a measure of extension; however, the desirability of using extension indicators accurate to ±1.0 % or better as specified in Test Method D 638 is recognized, and provision for the use of such instrumentation is incorporated in the procedure.

1.6 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* A specific precautionary statement is given in Note 19.

2. Referenced Documents

2.1 *ASTM Standards:*

D 374 Test Methods for Thickness of Solid Electrical Insulation[2]

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[3]

D 638 Test Method for Tensile Properties of Plastics[3]

D 4000 Classification System for Specifying Plastic Materials[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

3. Terminology

3.1 *Definitions*—Definitions of terms and symbols relating to tension testing of plastics appear in the Annex to Test Method D 638.

3.1.1 *line grips*—grips having faces designed to concentrate the entire gripping force along a single line perpendicular to the direction of testing stress. This is usually done by combining one standard flat face and an opposing face from which protrudes a half-round.

3.1.2 *tear failure*—a tensile failure characterized by fracture initiating at one edge of the specimen and progressing across the specimen at a rate slow enough to produce an anomalous load-deformation curve.

4. Significance and Use

4.1 Tensile properties determined by these test methods are of value for the identification and characterization of materials for control and specification purposes. Tensile properties may vary with specimen thickness, method of preparation, speed of testing, type of grips used, and manner of measuring extension. Consequently, where precise comparative results are desired, these factors must be carefully controlled. Since the actual loading rates vary between Test Methods A and B, the results obtained using these two methods cannot be directly compared. Test Method A is preferred and shall be used for referee purposes, unless otherwise indicated in particular material specifications. For many materials, there may be a specification that requires the use of this test method, but with some procedure

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[1] These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

Current edition approved Nov. 15, 1991. Published January 1992. Originally published as D 882 – 46 T. Last previous edition D 882 – 90.

[2] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vol 08.02.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Table 1 in Classification D 4000 lists the ASTM materials standards that currently exist.

4.2 Tensile properties may be utilized to provide data for research and development and engineering design as well as quality control and specification. However, data from such tests cannot be considered significant for applications differing widely from the load-time scale of the test employed.

4.3 The tensile modulus of elasticity is an index of the stiffness of thin plastic sheeting. The reproducibility of test results is good when precise control is maintained over all test conditions. When different materials are being compared for stiffness, specimens of identical dimensions must be employed.

4.4 The tensile energy to break (TEB) is the total energy absorbed per unit volume of the specimen up to the point of rupture. In some texts this property has been referred to as *toughness.* It is used to evaluate materials that may be subjected to heavy abuse or that might stall web transport equipment in the event of a machine malfunction in end-use applications. However, the rate of strain, specimen parameters, and especially flaws may cause large variations in the results. In that sense, caution is advised in utilizing TEB test results for end-use design applications.

4.5 Materials that fail by tearing give anomalous data which cannot be compared with those from normal failure.

5. Apparatus

5.1 *Grips*—A gripping system that minimizes both slippage and uneven stress distribution.

NOTE 4—Grips lined with thin rubber, crocus-cloth, or pressure-sensitive tape as well as file-faced or serrated grips have been successfully used for many materials. The choice of grip surface will depend on the material tested, thickness, etc. More recently, line grips padded on the round face with 1.0-mm (40-mil) blotting paper have been found superior. Air-actuated grips have been found advantageous, particularly in the case of materials that tend to "neck" into the grips, since pressure is maintained at all times. In cases where samples frequently fail at the edge of the grips, it may be advantageous to increase slightly the radius of curvature of the edges where the grips come in contact with the test area of the specimen.

5.2 *Thickness Gage*—A dead-weight dial micrometer as prescribed in Method C of Test Methods D 374, reading to 0.0025 mm (0.0001 in.) or less.

5.3 *Width-Measuring Devices*—Suitable test scales or other width measuring devices capable of measuring 0.25 mm (0.010 in.) or less.

5.4 *Specimen Cutter*—Razor blades, fixtures incorporating razor blades, suitable paper cutters, or other devices capable of cutting the specimens to the proper width and producing straight, clean, parallel edges with no visible imperfections, shall be used. Devices that use razor blades have proved especially suitable for materials having an elongation-at-fracture above 10 to 20 %. A device consisting of two parallel knives mounted firmly against a precision-ground base shear block (similar to a paper cutter) has also proved satisfactory. The use of striking dies is not recommended because of poor and inconsistent specimen edges which may be produced. It is imperative that the cutting edges be kept sharp and free from visible scratches or nicks.

5.5 *Extension Indicators* (if employed), shall conform requirements specified in Test Method D 638. In additi such apparatus shall be so designed as to minimize stress the specimen at the contact points of the specimen and indicator (see 8.3).

5.6 *Testing Machines:*

5.6.1 *For Test Method A*—A testing machine of t constant rate-of-jaw-separation type. The machine shall equipped with a weighing system that moves a maximu distance of 2 % of the specimen extension within the ran being measured. The machine shall be equipped with device for recording the tensile load and the amount separation of the grips; both of these measuring systems sha be accurate to ±2 %. The rate of separation of the jaws shal be uniform and capable of adjustment from approximatel 1.3 to 500 mm (0.05 to 20 in.)/min in increments necessar to produce the strain rates specified in 9.3 and 9.4. This tes method (A) shall be used for tensile modulus of elasticit measurements (Note 5).

5.6.2 *For Test Method B*—A testing machine of the pendulum type. This machine shall be equipped with a pendulum weighing head to measure the load applied to the test specimen and a device for indicating or recording the tensile load carried by the specimen with an accuracy of ±2 %. The rate of travel of the power-activated grip shall be uniform and capable of adjustment to 50.8 and 508 mm (2 and 20 in.)/min.

NOTE 5—A high response speed in the recording system is desirable, particularly when relatively high strain rates are employed for rigid materials. The speed of pen response for recorders is supplied by manufacturers of this equipment. Care must be taken to conduct tests at conditions such that response time (ability of recorder to follow actual load) will produce less than 2 % error.

6. Test Specimens

6.1 The test specimens shall consist of strips of uniform width and thickness at least 50 mm (2 in.) longer than the grip separation used.

6.2 The nominal width of the specimens shall be not less than 5.0 mm (0.20 in.) or greater than 25.4 mm (1.0 in.).

6.3 A width-thickness ratio of at least eight shall be used. Narrow specimens magnify effects of edge strains or flaws, or both.

6.4 The utmost care shall be exercised in cutting specimens to prevent nicks and tears which are likely to cause premature failures (Note 6). The edges shall be parallel to within 5 % of the width over the length of the specimen between the grips.

NOTE 6—Microscopical examination of specimens may be used to detect flaws due to sample or specimen preparation.

6.5 Wherever possible, the test specimens shall be selected so that thickness is uniform to within 10 % of the thickness over the length of the specimen between the grips in the case of materials 0.25 mm (0.010 in.) or less in thickness and to within 5 % in the case of materials greater than 0.25 mm (0.010 in.) in thickness but less than 1.00 mm (0.040 in.) in thickness.

NOTE 7—In cases where thickness variations are in excess of those recommended in 6.5, results may not be characteristic of the material under test.

6.6 If the material is suspected of being anisotropic, two

;t specimens shall be prepared having their long axes
ely parallel with and normal to the suspected
of anisotropy.

ɪr tensile modulus of elasticity determinations, a
ı gage length of 250 mm (10 in.) shall be considered
ɪrd. This length is used in order to minimize the
'grip slippage on test results. When this length is not
test sections as short as 100 mm (4 in.) may be used
)een shown that results are not appreciably affected.
', the 250-mm gage length shall be used for referee
. The speed of testing of shorter specimens must be
in order for the strain rate to be equivalent to that of
lard specimen.

ʝ—Two round robin tests[6] have shown that, for materials of
0.25-mm (10-mil) thickness, line grips padded on the round
1.0-mm (40-mil) blotting paper give the same results with a
:est section as a 250-mm test section produces with flat-face

)—Excessive jaw slippage becomes increasingly difficult to
in cases where high modulus materials are tested in thick-
ater than 0.25 mm (0.010 in.).

ltioning

'*onditioning*—Condition the test specimens at 23 ±
4 ± 3.6°F) and 50 ± 5 % relative humidity for not
ı 40 h prior to test in accordance with Procedure A of
D 618 for those tests where conditioning is required.
of disagreement, the tolerances shall be 1°C (1.8°F)
% relative humidity.

'*est Conditions*—Conduct tests in the Standard Lab-
Atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 %
humidity, unless otherwise specified in the test
s or in this specification. In cases of disagreement, the
:es shall be ±1°C (±1.8°F) and ±2 % relative hu-

ıber of Test Specimens

n the case of isotropic materials, at least five speci-
hall be tested from each sample.

n the case of anisotropic materials, at least ten
ens, five normal and five parallel with the principal
anisotropy, shall be tested from each sample.

Specimens that fail at some obvious flaw or that fail
: the gage length shall be discarded and retests made,
such flaws or conditions constitute a variable whose
s being studied. However, jaw breaks (failures at the
)ntact point) are acceptable if it has been shown that
from such tests are in essential agreement with values
ed from breaks occurring within the gage length.

E 10—In the case of some materials, examination of specimens,
› and following testing, under crossed optical polarizers (polar-
ms) provides a useful means of detecting flaws which may be, or
ponsible for premature failure.

eed of Testing

The speed of testing is the rate of separation of the two
)ers (or grips) of the testing machine when running idle
r no load). This rate of separation shall be maintained within 5 % of the no-load value when running under full-capacity load.

9.2 The speed of testing shall be calculated from the required initial strain rate as specified in Table 1. The rate of grip separation may be determined for the purpose of these test methods from the initial strain rate as follows:

$$A = BC$$

where:

A = rate of grip separation, mm (or in.)/min,
B = initial distance between grips, mm (or in.), and
C = initial strain rate, mm/mm·min (or in./in.·min).

9.3 The initial strain rate shall be as in Table 1 unless otherwise indicated by the specification for the material being tested.

NOTE 11—Results obtained at different initial strain rates are not comparable; consequently, where direct comparisons between materials in various elongation classes are required, a single initial strain rate should be used. For some materials it may be advisable to select the strain rates on the basis of percent elongation at yield.

9.4 In cases where conflicting material classification, as determined by percent elongation at break values, results in a choice of strain rates, the lower rate shall be used.

9.5 If modulus values are being determined, separate specimens shall be used whenever strain rates and specimen dimensions are not the same as those employed in the test for other tensile properties.

10. Procedure

10.1 Select a load range such that specimen failure occurs within its upper two thirds. A few trial runs may be necessary to select a proper combination of load range and specimen width.

10.2 Measure the cross-sectional area of the specimen at several points along its length. Measure the width to an accuracy of 0.25 mm (0.010 in.) or better. Measure the thickness to an accuracy of 0.0025 mm (0.0001 in.) or better for films less than 0.25 mm (0.010 in.) in thickness and to an accuracy of 1 % or better for films greater than 0.25 mm (0.010 in.) but less than 1.0 mm (0.040 in.) in thickness.

10.3 The initial grip separation shall be at least 50 mm (2 in.) for materials having a total elongation at break of 100 % or more, and at least 100 mm (4 in.) for materials having a total elongation at break of less than 100 %.

NOTE 12—Since slippage is a potential problem in these tests, as great an initial distance between grips as possible should be employed

10.4 Set the rate of grip separation to give the desired strain rate based on the initial distance between the grips

TABLE 1 Speed of Testing

Test Method	Percent Elongation at Break	Initial Strain Rate, mm/mm·min (in./in.·min)
Modulus of Elasticity Determination		
A		0.1
Determinations other than Elastic Modulus		
A	Less than 20	0 1
	20 to 100	0.5
	Greater than 100	10.0
B	Less than 100	0.5
	Greater than 100	10 0

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pporting data are available from ASTM Headquarters. Request RR:
158.

(Note 13). Balance, zero, and calibrate the load weighing and recording system.

Note 13—Suggested crosshead speeds and initial grip separation to give the desired initial strain rate described in Table 1 are shown in Table 2.

10.5 In cases where it is desired to measure a test section other than the total length between the grips, mark the ends of the desired test section with a soft, fine wax crayon or with ink. Do not scratch these marks onto the surface since such scratches may act as stress raisers and cause premature specimen failure. Extensometers may be used if available; in this case, the test section will be defined by the contact points of the extensometer.

Note 14—Measurement of a specific test section is necessary with some materials having high elongation. As the specimen elongates, the accompanying reduction in area results in a loosening of material at the inside edge of the grips. This reduction and loosening moves back into the grips as further elongation and reduction in area takes place. In effect, this causes problems similar to grip slippage, that is, exaggerates measured extension.

10.6 Place the test specimen in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. Tighten the grips evenly and firmly to the degree necessary to minimize slipping of the specimen during test.

10.7 Start the machine and record load versus extension.

10.7.1 When the total length between the grips is used as the test area, record load versus grip separation.

10.7.2 When a specific test area has been marked on the specimen, follow the displacement of the edge boundary lines with respect to each other with dividers or some other suitable device. If a load-extension curve is desired, plot various extensions versus corresponding loads sustained, as measured by the load indicator.

10.7.3 When an extensometer is used, record load versus extension of the test area measured by the extensometer.

10.8 If modulus values are being determined, select a load range and chart rate to produce a load-extension curve of between 30 and 60° to the X axis. For maximum accuracy, use the most sensitive load scale for which this condition can be met. The test may be discontinued when the load-extension curve deviates from linearity.

10.9 In the case of materials being evaluated for secant modulus, the test may be discontinued when the specified extension has been reached.

10.10 If tensile energy to break is being determined, some provision must be made for integration of the stress-strain curve. This may be either an electronic integration duri the test or a subsequent determination from the area of tl finished stress-strain curve (see Annex A2).

11. Calculation

11.1 *Breaking Factor* (nominal) shall be calculated b dividing the maximum load by the original minimum widt of the specimen. The result shall be expressed in force pe unit of width, usually newtons per metre (or pounds pe inch) of width, and reported to three significant figures. Th thickness of the film shall always be stated to the neare 0.0025 mm (0.0001 in.).

Example—Breaking Factor = 1.75 kN/m (10.0 lbf/in.) o width for 0.1300-mm (0.0051-in.) thickness.

Note 15—This method of reporting is useful for very thin film (0.13 mm (0.005 in.) and less) for which breaking load may not b proportional to cross-sectional area and whose thickness may be difficul to determine with precision. Furthermore, films which are in effec laminar due to orientation, skin effects, nonuniform crystallinity, etc. have tensile properties disproportionate to cross-sectional area.

11.2 *Tensile Strength* (nominal) shall be calculated b dividing the maximum load by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures.

Note 16—When tear failure occurs, so indicate and calculate results based on load and elongation at which tear initiates, as reflected in the load-deformation curve.

11.3 *Tensile Strength at Break* (nominal) shall be calculated in the same way as the tensile strength except that the load at break shall be used in place of the maximum load (Notes 16 and 17).

Note 17—In many cases tensile strength and tensile strength at break are identical.

11.4 *Percent Elongation at Break* shall be calculated by dividing the extension at the moment of rupture of the specimen by the initial gage length of the specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation; otherwise the distance between the grips shall be used. The result shall be expressed in percent and reported to two significant figures (Note 16).

11.5 *Yield Strength*, where applicable, shall be calculated by dividing the load at the yield point by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be

TABLE 2 Crosshead Speeds and Initial Grip Separation

Test Method	Percent Elongation at Break	Initial Strain Rate, mm/mm·min (in./in.·min)	Initial Grip Separation		Rate of Grip Separation	
			mm	in.	mm/min	in./min
		Modulus of Elasticity Determination				
A		0.1	250	10	25	1.0
		Determinations other than Elastic Modulus				
A	Less than 20	0.1	125	5	12.5	0.5
	20 to 100	0.5	100	4	50	2.0
	Greater than 100	10.0	50	2	500	20.0
B	Less than 100	0.5	100	4	50	2.0
	Greater than 100	10.0	50	2	500	20.0

orted to three significant figures. Alternatively, for mate-
ls that exhibit Hookean behavior in the initial part of the
ve, an offset yield strength may be obtained as described
the Appendix of Test Method D 638. In this case the value
uld be given as "yield strength at —% offset."

1.6 *Percent Elongation at Yield*, where applicable, shall
calculated by dividing the extension at the yield point by
initial gage length of specimen and multiplying by 100.
hen gage marks or extensometers are used to define a
cific test section, only this length shall be used in the
culation. Before calculating, correct the extension for "toe
npensation" as described in Annex A1. The results shall
expressed in percent and reported to two significant
ures. When offset yield strength is used, the elongation at
offset yield strength may be calculated.

1.7 *Elastic Modulus* shall be calculated by drawing a
gent to the initial linear portion of the load-extension
ve, selecting any point on this tangent, and dividing the
sile stress by the corresponding strain. Before calculating,
rect the extension for "toe compensation" as described in
nex A1. For purposes of this determination, the tensile
ss shall be calculated by dividing the load by the average
ginal cross section of the test section. The result shall be
ressed in force per unit area, usually megapascals (or
unds-force per square inch), and reported to three signifi-
t figures.

1.8 *Secant Modulus*, at a designated strain, shall be
culated by dividing the corresponding stress (nominal) by
designated strain. Elastic modulus values are preferable
l shall be calculated whenever possible. However, for
terials where no proportionality is evident, the secant
ue shall be calculated. Draw the tangent as directed in
.3 and Fig. A1.2 of Annex A1, and mark off the
ignated strain from the yield point where the tangent line
s through zero stress. The stress to be used in the
culation is then determined by dividing the load at the
ignated strain on the load-extension curve by the original
rage cross-sectional area of the specimen.

.1.9 *Tensile Energy to Break*, where applicable, shall be
culated by integrating the energy per unit volume under
stress-strain curve or by integrating the total energy
orbed and dividing it by the volume of the original gage
ion of the specimen. As indicated in Annex A2, this may
done directly during the test by an electronic integrator, or
sequently by computation from the area of the plotted
rve. The result shall be expressed in energy per unit
lume, usually in megajoules per cubic metre (or inch-
unds-force per cubic inch). This value shall be reported to
o significant figures.

11.10 For each series of tests, the arithmetic mean of all
lues obtained shall be calculated to the proper number of
nificant figures.

11.11 The standard deviation (estimated) shall be calcu-
ed as follows and reported to two significant figures:

$$s = \sqrt{(\Sigma X^2 - n\bar{X}^2)/(n - 1)}$$

here:

= estimated standard deviation,
= value of a single observation,
= number of observations, and
$\bar{X}$ = arithmetic mean of the set of observations.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, and orientation of samples with respect to anisotropy (if any),

12.1.2 Method of preparing test specimens,

12.1.3 Thickness, width, and length of test specimens,

12.1.4 Number of specimens tested,

12.1.5 Strain rate employed,

12.1.6 Grip separation (initial),

12.1.7 Crosshead speed (rate of grip separation),

12.1.8 Gage length (if different from grip separation),

12.1.9 Type of grips used, including facing (if any),

12.1.10 Test method (A or B),

12.1.11 Conditioning procedure (test conditions, temperature, and relative humidity if nonstandard),

12.1.12 Anomalous behavior such as tear failure and failure at a grip,

12.1.13 Average breaking factor and standard deviation,

12.1.14 Average tensile strength (nominal) and standard deviation,

12.1.15 Average tensile strength at break (nominal) and standard deviation,

12.1.16 Average percent elongation at break and standard deviation,

12.1.17 Where applicable, average tensile energy to break and standard deviation,

12.1.18 In the case of materials exhibiting "yield" phenomenon: average yield strength and standard deviation; and average percent elongation at yield and standard deviation.

12.1.19 For materials which do not exhibit a yield point: average —% offset yield strength and standard deviation: and average percent elongation at —% offset yield strength and standard deviation,

12.1.20 Average modulus of elasticity and standard deviation (if secant modulus is used, so indicate and report strain at which calculated), and

12.1.21 When an extensometer is employed, so indicate.

13. Precision and Bias

13.1 Two interlaboratory tests have been run for these tensile properties. The first was run for modulus only, in 1977, in which randomly drawn samples of four thin (~0.025 mm (0.001-in.)) materials were tested with five specimens in each laboratory. Elastic (tangent) modulus measurements were made by six laboratories, and secant (1 %) modulus measurements were taken by five laboratories. The relative precision obtained in this interlaboratory study is in Table 3.

13.1.1 In deriving the estimates in Table 3, statistical outliers were not removed, in keeping with Practice E 691

13.1.2 The within-lab standard deviation of a mean value $S_{\bar{x}}$, in each case was determined from the standard deviation S_x, of the five individual specimens as follows: $S_{\bar{x}} = S_x/(5)^{1/2}$ The $S_{\bar{x}}$ values were pooled among laboratories for a given

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[7] Supporting data are available from ASTM Headquarters Request RR: D20-1084.

TABLE 3 Precision Data for Modulus

Material	Thickness, mils	Average, 10^3 psi	S_r, 10^3 psi	S_R, 10^3 psi	I_r, 10^3 psi	I_R, 10^3 psi
			Tangent Modulus			
LDPE	1.4	53.9	1.81	8.81	5.12	24.9
HDPE	1.6	191	5.47	16.2	15.5	45.9
PP	1.1	425	10.3	31.5	29.0	89.1
PET	0.9	672	13.8	55.5	39.1	157.1
			Secant Modulus			
LDPE	1.4	45.0	2.11	3.43	5.98	9.70
HDPE	1.6	150	3.29	9.58	9.30	27.1
PP	1.1	372	4.66	26.5	13.2	74.9
PET	0.9	640	10.0	27.5	28.4	77.8

material to obtain the within-lab standard deviation, S_r, of a test result (mean of five specimens). See 13.3 through 13.3.2 for definitions of terms in the tables.

13.2 An interlaboratory test was run for all the other tensile properties except modulus in 1981, in which randomly drawn samples of six materials (one of these in three thicknesses) ranging in thickness from 0.019 to 0.178 mm (0.00075 to 0.007 in.) were tested in seven laboratories. A test result was defined as the mean of five specimen determinations. However, each laboratory tested eight specimens, and the $S_{\bar{x}}$ was determined from $S_{\bar{x}} = S_x/(5)^{1/2}$ as above. This was done to improve the quality of the statistics while maintaining their applicability to a five-specimen test result. The materials and their thicknesses are identified in Tables 4 through 8, each of which contain data for one of the following properties: tensile yield stress, yield elongation, tensile strength, tensile elongation at break, and tensile energy at break (see Note 18).[8]

NOTE 18—Subsequent to filing the research report, examination of the LDPE used in this study between crossed polarizers revealed lengthwise lines representing substantial widthwise variation in molecular orientation that probably was not successfully randomized out of the between-labs component of variance.

NOTE 19—Caution: The following explanations of I_r and I_R (13.3 through 13.3.3) are only intended to present a meaningful way of considering the *Approximate* precision of this test method. The data in Table(s) 3 through 8 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 13.3 through 13.3.3 would then be valid for such data.

13.3 *Concept of I_r and I_R*—If S_r and S_R have been calculated from a *large enough body of data*, and for test results that were averages (medians/other function) from testing five specimens:

13.3.1 *Repeatability, I_r* (Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the I_r value for that material.

13.3.2 *Reproducibility, I_R* (Comparing two test results for the same material, obtained by different operators using different equipment on different days)—The two test results should be judged not equivalent if they differ by more than the I_R value for that material.

13.3.3 Any judgment made in accordance with 13.3.1 and 13.3.2 would have an approximate 95 % (0.95) probability of being correct.

13.4 *Bias*—The systematic error which contributes to the difference between a test result and a true (or reference) value. There are no recognized standards on which to base an estimate of bias for these test methods.

14. Keywords

14.1 modulus of elasticity; plastic film; plastic sheeting; tensile properties; tensile strength; toughness; yield stress

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[8] Supporting data are available from ASTM Headquarters. Request RR: D20-1101.

TABLE 4 Precision Data for Yield Stress

Material	Thickness, mils	Average, 10^3 psi	$(S_r)^A$ 10^3 psi	$(S_R)^B$ 10^3 psi	$I(r)^C$ 10^3 psi	$I(R)^D$ 10^3 psi
LDPE	1.0	1.49	0.051	0.13	0.14	0.37
HDPE	1.0	4.33	0.084	0.16	0.24	0.44
PP	0.75	6.40	0.13	0.52	0.37	1.46
PC	4.0	8.59	0.072	0.29	0.20	0.82
CTA	5.3	11.4	0.12	0.50	0.34	1.43
PET	4.0	14.3	0.12	0.23	0.34	0.66
PET	2.5	14.4	0.14	0.54	0.40	1.52
PET	7.0	14.4	0.13	0.36	0.37	1.03

[A] S_r is the within-laboratory standard deviation of the average.

[B] S_R is the between-laboratories standard deviation of the average.

[C] $I_r = 2.83\ S_r$

[D] $I_R = 2.83\ S_R$

TABLE 5 Precision Data for Yield Elongation

Material	Thickness, mils	Average, %	$(S_r)^A$, %	$(S_R)^B$, %	$I(r)^C$, %	$I(R)^D$, %
PP	0.75	3.5	0.15	0.41	0.42	1.2
PET	2.5	5.2	0.26	0.92	0.74	2.6
PET	4.0	5.3	0.25	0.60	0.71	1.7
PET	7.0	5.4	0.14	1.05	0.40	3.0
CTA	5.3	5.4	0.19	0.99	0.54	2.8
PC	4.0	6.9	0.24	0.98	0.68	2.8
HDPE	1.0	8.8	0.32	1.82	0.91	5.2
LDPE	1.0	10.0	0.55	3.41	1.56	9.6

NOTE—See Table 4 for footnote explanation.

TABLE 6 Precision Data for Tensile Strength

Material	Thickness, mils	Average, 10^3 psi	$(S_r)^A$ 10^3 psi	$(S_R)^B$ 10^3 psi	$I(r)^C$ 10^3 psi	$I(R)^D$ 10^3 psi
LDPE	1.0	3.42	0.14	0.53	0.40	1.5
HDPE	1.0	6.87	0.27	0.81	0.76	2.3
PC	4.0	12.0	0.34	0.93	0.96	2.6
CTA	5.3	14.6	0.20	1.37	0.57	3.9
PP	0.75	28.4	1.57	4.56	4.4	12.9
PET	4.0	28.9	0.65	1.27	1.8	3.6
PET	7.0	30.3	0.83	1.32	2.3	3.7
PET	2.5	30.6	1.22	2.64	3.4	7.5

NOTE—See Table 4 for footnote explanation.

TABLE 7 Precision Data for Elongation at Break

Material	Thickness, mils	Average, %	$(S_r)^A$, %	$(S_R)^B$, %	$I(r)^C$, %	$I(R)^D$, %
CTA	5.3	26.4	1.0	4.3	3	12
PP	0.75	57.8	4.4	12.7	12	36
PET	2.5	120	8.0	14.6	23	41
PET	7.0	132	5.8	10.6	16	30
PET	4.0	134	4.4	12.2	12	35
PC	4.0	155	5.4	17.1	15	48
LDPE	1.0	205	24.4	73.3	69	210
HDPE	1.0	570	26.0	91.7	74	260

NOTE—See Table 4 for footnote explanation.

TABLE 8 Precision Data for Tensile Energy to Break

Material	Thickness, mils	Average, 10^3 $\frac{\text{in.·lb}}{\text{in.}^3}$	$(S_r)^A$ 10^3 $\frac{\text{in.·lb}}{\text{in.}^3}$	$(S_R)^B$ 10^3 $\frac{\text{in.·lb}}{\text{in.}^3}$	$I(r)^C$ 10^3 $\frac{\text{in.·lb}}{\text{in.}^3}$	$I(R)^D$ 10^3 $\frac{\text{in.·lb}}{\text{in.}^3}$
CTA	5.0	3.14	0.14	0.70	0.4	2.0
LDPE	1.0	5.55	0.84	2.47	2.4	7.0
PP	0.75	11.3	1.19	3.11	3.4	8.8
PC	4.0	12.9	0.59	1.55	1.7	4.4
HDPE	1.0	26.0	1.87	5.02	5.3	14.2
PET	2.5	26.1	2.13	4.20	6.0	11.9
PET	4.0	27.1	1.42	2.75	4.0	7.8
PET	7.0	28.4	1.71	2.72	4.8	7.7

NOTE—See Table 4 for footnote explanation.

ANNEXES

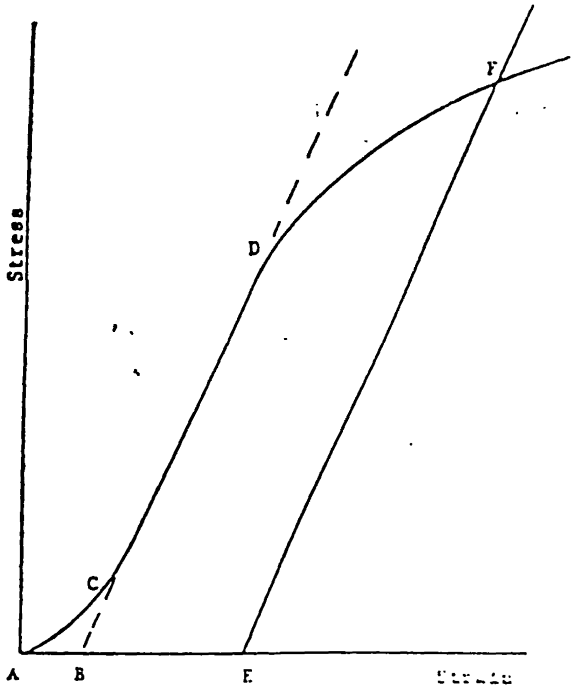
(Mandatory Information)

A1. TOE COMPENSATION

A1.1 In a typical stress-strain curve (Fig. A1.1) there is a toe region, *AC*, which does not represent a property of the material. It is an artifact caused by a takeup of slack, and alignment or seating of the specimen. In order to obtain correct values of such parameters as modulus, strain, and offset yield point, this artifact must be compensated for to give the corrected zero point on the strain or extension axis.

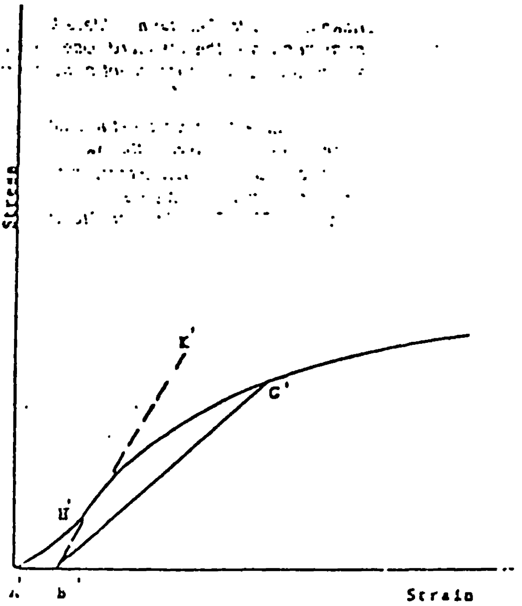
A1.2 In the case of a material exhibiting a region of Hookean (linear) behavior (Fig. A1.1), a continuation of the linear (*CD*) region of the curve is constructed through the zero-stress axis. This intersection (*B*) is the corrected zero-strain point from which all extensions or strains must be measured, including the yield offset (*BE*), if applicable. The elastic modulus can be determined by dividing the stress at any point along the line *CD* (or its extension) by the strain at the same point (measured from point *B*, defined as zero-strain).

A1.3 In the case of a material that does not exhibit any linear region (Fig. A1.2), the same kind of toe correction of the zero-strain point can be made by constructing a tangent to the maximum slope at the inflection point (*H'*). This is extended to intersect the strain axis at point *B'*, the corrected zero-strain point. Using point *B'* as zero strain, the stress at any point (*G'*) on the curve can be divided by the strain at that point to obtain a secant modulus (slope of line *B' G'*). For those materials with no linear region, any attempt to use the tangent through the inflection point as a basis for determination of an offset yield point may result in unacceptable error.



NOTE—Some chart recorders plot the mirror image of this graph.

FIG. A1.1 Material with Hookean Region



NOTE—Some chart recorders plot the mirror image of this graph.

FIG. A1.2 Material with No Hookean Region

A2. DETERMINATION OF TENSILE ENERGY TO BREAK

A2.1 Tensile energy to break (TEB) is defined by the area nder the stress-strain curve, or

$$\mathrm{TEB} = \int_0^{\epsilon_T} S \, d\epsilon$$

here S is the stress at any strain, ϵ, and ϵ_T is the strain at pture. The value is in units of energy per unit volume of e specimen's initial gage region. TEB is most conveniently nd accurately measured with a tension tester equipped with n integrator. The calculation is then:

$$\mathrm{EB} = (I/K) \frac{(\text{full scale load})\,(\text{chart speed})\,(\text{crosshead speed/chart speed})}{(\text{mean caliper})\,(\text{specimen width})\,(\text{gage length})}$$

where I is the integrator count reading and K is the maximum possible count per unit time for a constant full scale load. This whole calculation is typically done electronically. The results are best expressed in megajoules per cubic metre (or inch-pounds-force per cubic inch).

A2.2 Without an integrator, the area under the recorded stress-strain curve can be measured by planimeter, counting squares, or weighing the cut-out curve. These techniques are time-consuming and likely to be less accurate, since the load scale on some chart paper is not in round-number dimensions. Moreover, if the curve coordinates are in terms of force and extension instead of stress and strain, the calculated energy, corresponding to the measured area, must be divided by the product of gage length, specimen width, and mean caliper:

$$\mathrm{TEB} = \frac{(\text{curve area})\,(\text{force per unit chart scale})\,(\text{extension per unit chart travel})}{(\text{mean caliper})\,(\text{specimen width})\,(\text{gage length})}$$

A2.3 For example, if the area under a force-extension curve is 60 000 mm^2, the load coordinate is 2.0 N/mm of chart scale, the extension coordinate is 0.25 mm of extension per mm of chart travel, and the specimen dimensions are 0.1 mm caliper, 15 mm width and 100 mm gage length, then the calculation for tensile energy to break is:

$$\mathrm{TEB} = \frac{(60\ 000\ \text{mm}^2)\,(2.0\ \text{N/mm})\,(0.25 \times 10^{-3}\ \text{m/mm})}{(0.1 \times 10^{-3}\text{m})\,(15 \times 10^{-3}\text{m})\,(100 \times 10^{-3}\text{m})}$$

$$\mathrm{TEB} = 200\ \text{MJ/m}^3$$

Appendix II

Detailed results of material testing

A- Films cleaned with alum salt
B- Films cleaned with bleaching
O- Films cleaned with pipe water

Eg. **A 4**- Material with **four layers**, which has been cleaned with **alum** salt

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TENSILE TEST REPORT

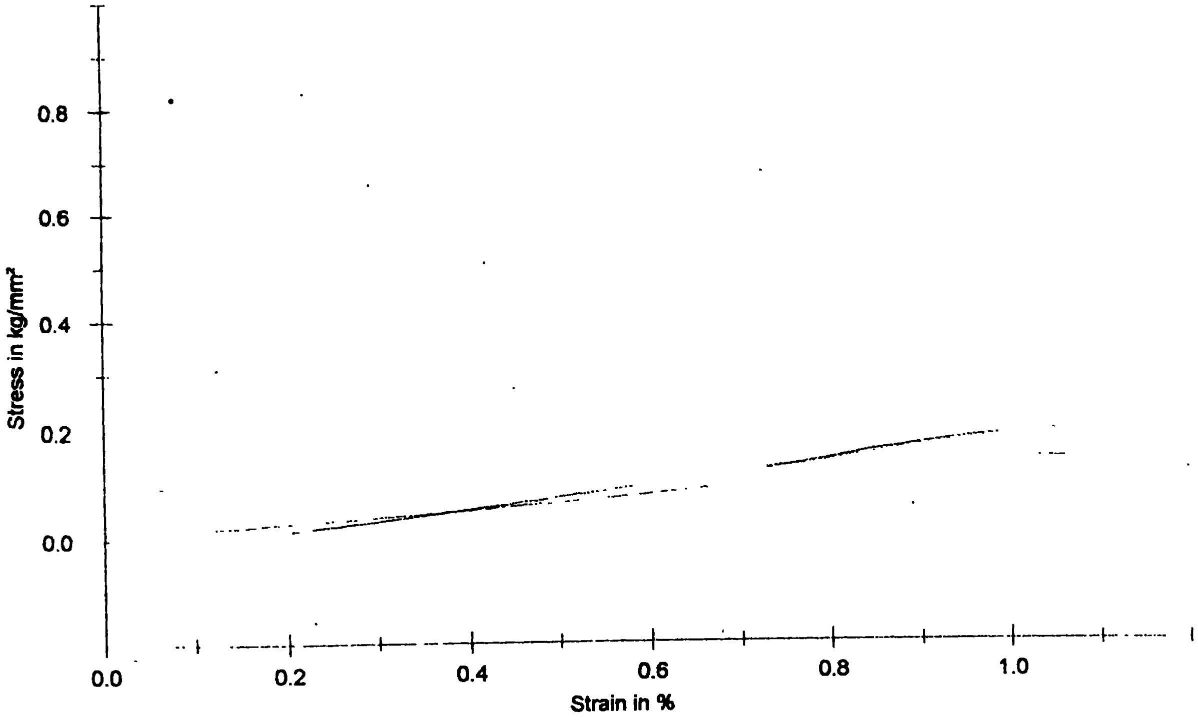
Parameter table:

Customer : Dr.Dharmasena
Tested by : T.Ex
Material/Material combination : biopolymer-(O-4)
Test speed : 25 mm/min

Results:

Legends	Nr	MD/TD	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
■	1	MD	25	0.15	116.02	0.15	1.44	1.06	0.53
■	2	MD	25	0.119	183.04	0.19	1.77	0.91	0.46
■	3	MD	25	0.113	219.35	0.19	1.90	0.80	0.40
■	4	MD	25	0.163	167.36	0.13	1.23	0.90	0.45

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x	25	0.1363	171.44	0.16	1.58	0.92	0.46
s	0.000	0.0241	42.89	0.03	0.31	0.11	0.05
v	0.00	17.69	25.02	19.67	19.29	11.82	11.82

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TENSILE TEST REPORT

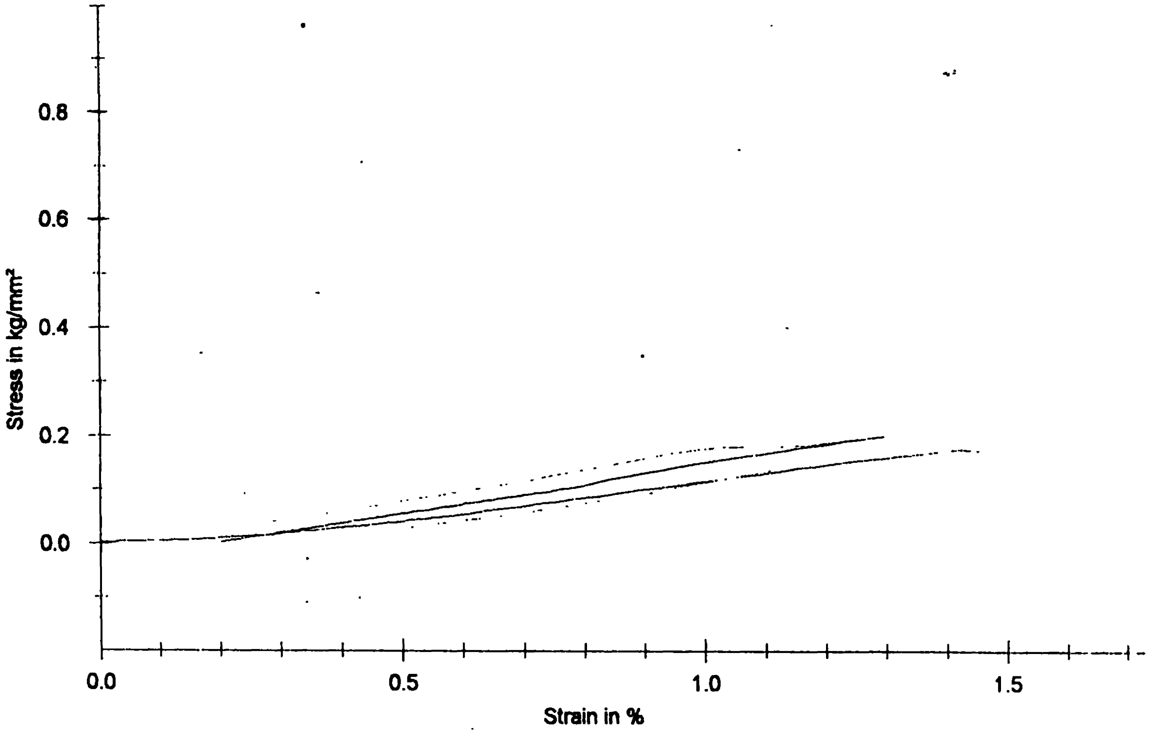
Parameter table:

Customer	:	Dr.Dharmasena
Material/Material combination	:	biopolymer~~-0.16mm~~ A4
Test speed	:	50 mm/min

Results:

Legends	Nr	MD/TD	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
■	1	MD	25	0.16	80.87	0.18	1.71	1.46	0.73
■	2	MD	25	0.143	173.56	0.20	1.93	1.17	0.58
■	3	MD	25	0.148	176.59	0.20	1.98	1.10	0.55
■	4	MD	25	0.131	147.20	0.24	2.38	1.40	0.70

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x̄	25	0.1455	144.56	0.20	2.00	1.28	0.64
s	0.000	0.01201	44.46	0.03	0.28	0.18	0.09
v	0.00	8.26	30.76	13.53	14.09	13.73	13.73

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TENSILE TEST REPORT

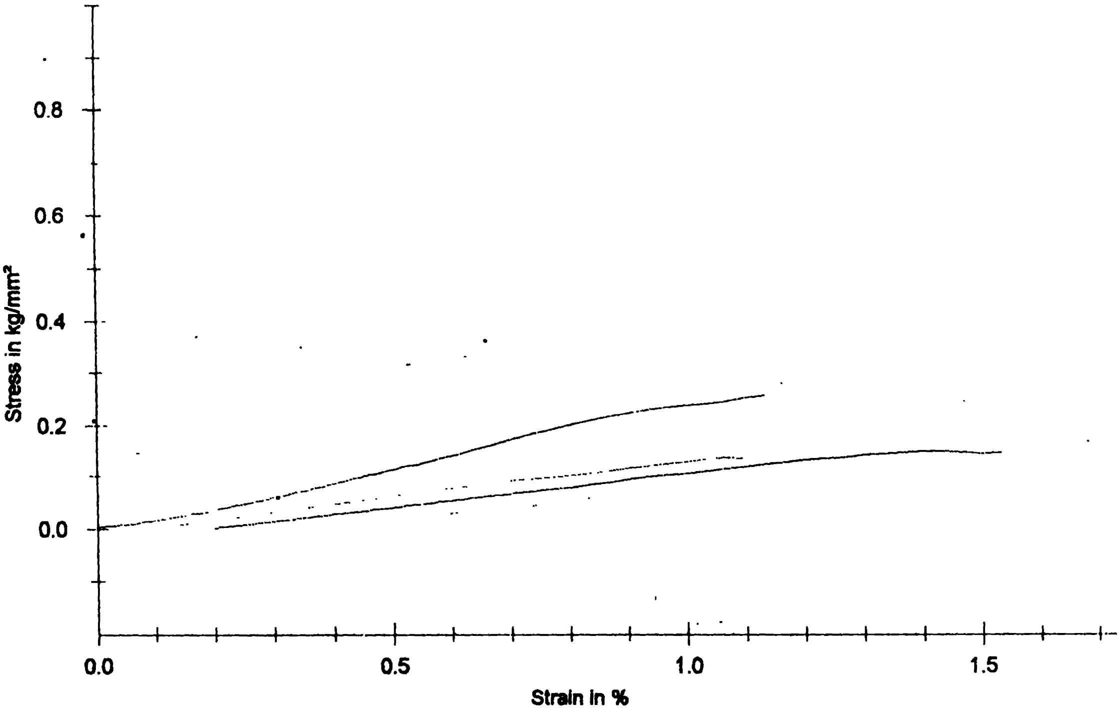
Parameter table:

Customer	:	Dr.Dharmasena
Tested by	:	T.Ex
Material/Material combination	:	biopolymer-(B4]
Test speed	:	25 mm/min

Results:

Legends	Nr	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
■	1	25	0.137	222.62	0.26	2.51	1.13	0.57
■	2	25	0.181	148.12	0.14	1.32	1.00	0.50
■	3	25	0.189	129.22	0.15	1.44	1.34	0.67
■	4	25	0.128	99.00	0.09	0.84	0.78	0.39

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x̄	25	0.1588	149.74	0.16	1.53	1.06	0.53
s	0.000	0.03071	52.63	0.07	0.70	0.23	0.12
ν	0.00	19.34	35.15	45.13	46.02	22.06	22.06

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TENSILE TEST REPORT

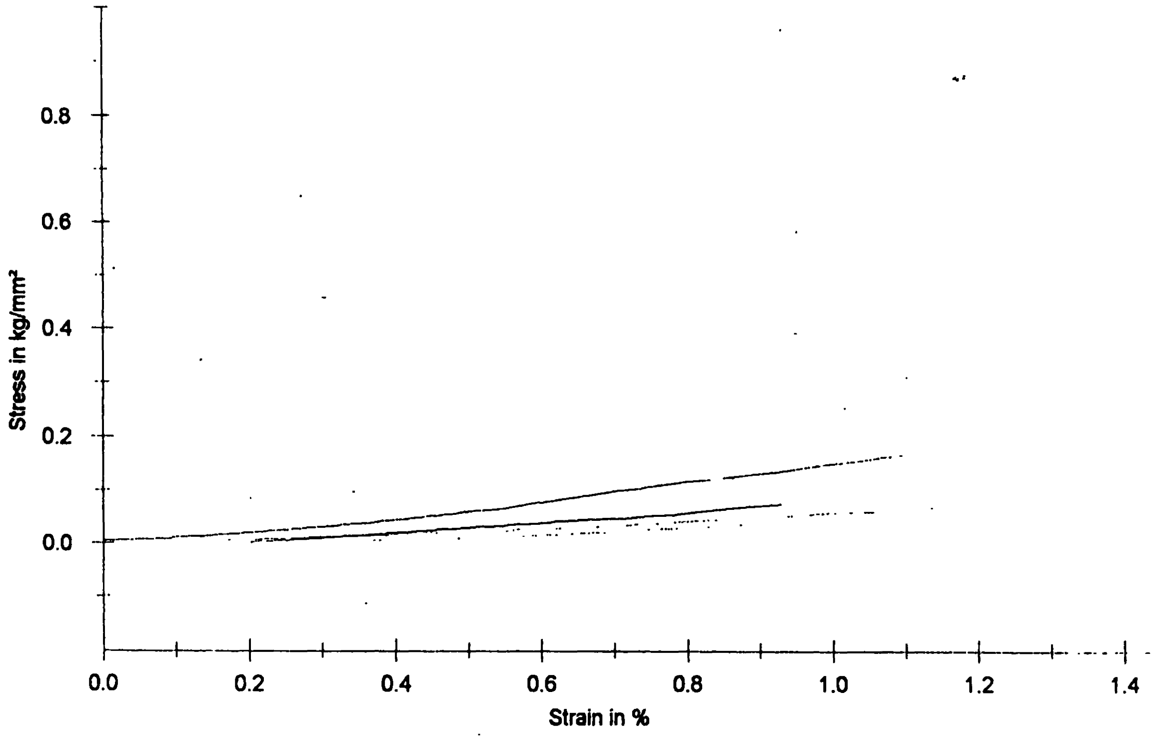
Parameter table:

Customer : Dr: Dharmasena
Material/Material combination : Bio polymer (A-3)
Test speed : 25 mm/min

Results:

Legends	Nr	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
■	1	25	0.06	117.05	0.17	1.66	1.10	0.55
■	2	25	0.1	51.80	0.08	0.83	1.20	0.60
■	3	25	0.093	93.60	0.08	0.74	0.73	0.37
■	4	25	0.101	57.36	0.07	0.64	0.93	0.47

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x̄	25	0.0885	79.95	0.10	0.97	0.99	0.50
s	0.000	0.01933	30.90	0.05	0.47	0.21	0.10
ν	0.00	21.84	38.65	48.64	48.64	20.76	20.76

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TENSILE TEST REPORT

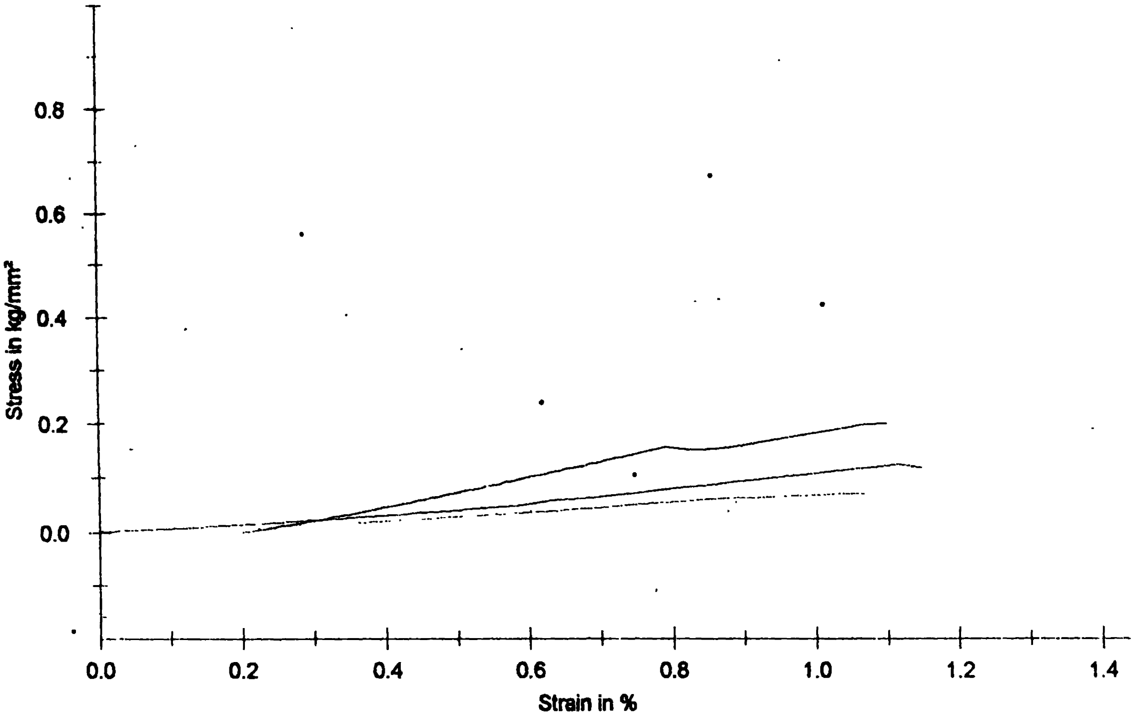
Parameter table:

Customer : Dr.Dharmasena
Material/Material combination : biopolymer-~~0.16mm~~ O_3
Test speed : 50 mm/min

Results:

Legends	Nr	MD/TD	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
■	1	MD	25	0.16	80.70	0.12	1.16	1.15	0.58
■	2	MD	25	0.173	70.40	0.07	0.70	0.97	0.49
■	3	MD	25	0.124	258.62	0.20	1.95	0.90	0.45
■	4	MD	25	0.159	64.39	0.05	0.48	0.93	0.46

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x	25	0.154	118.53	0.11	1.07	0.99	0.49
s	0.000	0.02099	93.64	0.07	0.65	0.11	0.06
v	0.00	13.63	79.00	60.22	60.71	11.34	11.34

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TENSILE TEST REPORT

Parameter table:

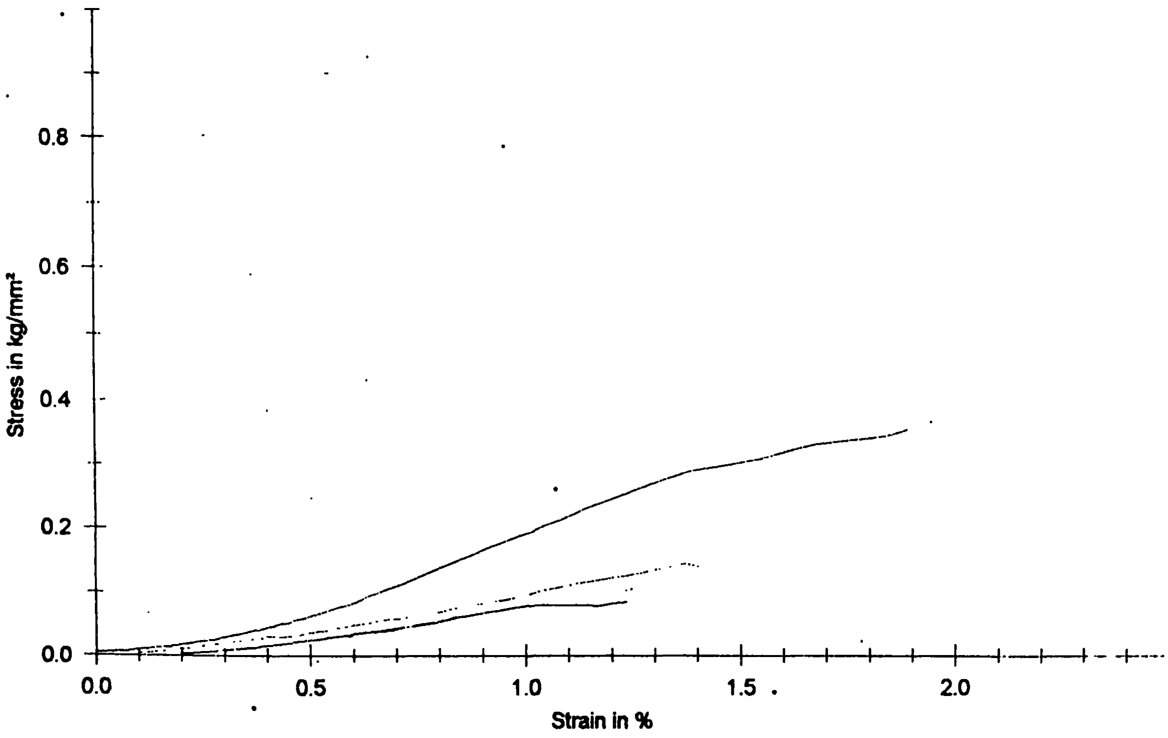
Customer : Dr.Dharmasena
Material/Material combination : biopolymer~~-0.16mm~~ B_3
Test speed : 50 mm/min

Results:

Legends	Nr	MD/TD	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %
■	1	MD	25	0.072	118.69	0.43	4.25	2.41
■	2	MD	25	0.11	88.74	0.14	1.37	1.31
■	3	MD	25	0.22	82.06	0.08	0.82	1.04
■	4	MD	25	0.123	109.68	0.12	1.14	1.04

Legends	Nr	ε Break mm
■	1	1.21
■	2	0.65
■	3	0.52
■	4	0.52

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x	25	0.1313	99.79	0.19	1.90	1.45	0.72
s	0.000	0.063	17.24	0.16	1.58	0.65	0.33
ν	0.00	48.00	17.27	82.87	83.54	45.20	45.20

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TENSILE TEST REPORT

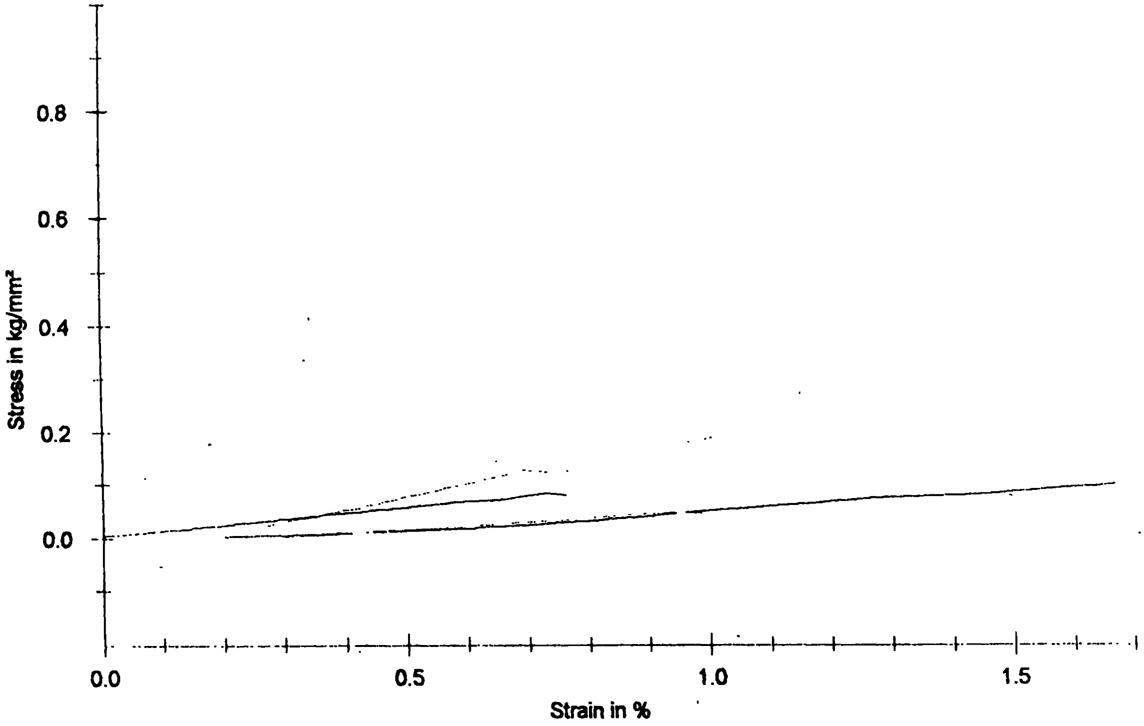
Parameter table:

Customer : Dr: Dharmasena
Material/Material combination : Bio polymer (A-2)
Test speed : 50 mm/min

Results:

Nr	MD/TD	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
1	MD	25	0.1	107.92	0.08	0.78	0.76	0.38
2	MD	25	0.05	200.61	0.19	1.84	0.91	0.45
3	MD	25	0.08	43.44	0.10	0.97	1.47	0.73
4	MD	25	0.12	70.32	0.05	0.44	0.69	0.35

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x̄	25	0.0875	105.57	0.10	1.01	0.96	0.48
s	0.000	0.02986	68.66	0.06	0.60	0.35	0.18
ν	0.00	34.13	65.03	57.80	59.17	36.83	36.83

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TENSILE TEST REPORT

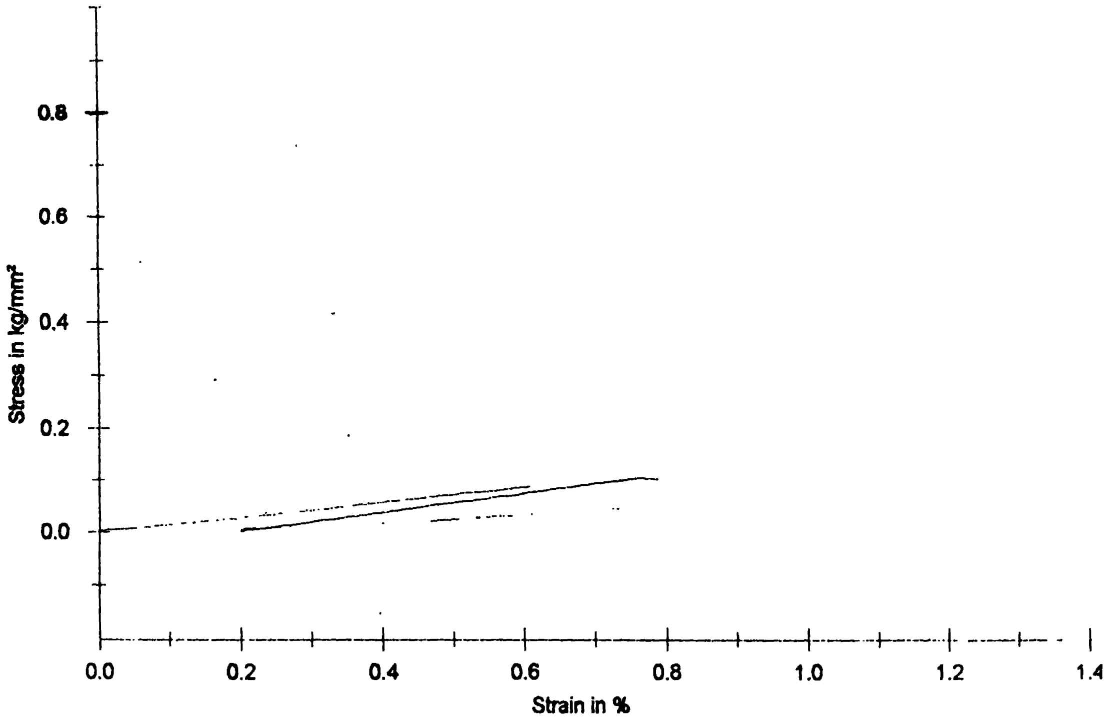
Parameter table:

Customer : Dr: Dharmasena
Material/Material combination : Bio polymer (O-2)
Test speed : 25 mm/min

Results:

Legends	Nr	MD/TD	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
■	1	MD	25	0.13	143.05	0.09	0.87	0.61	0.30
■	2	MD	25	0.08	61.76	0.11	1.05	1.31	0.65
■	3	MD	25	0.08	186.46	0.11	1.01	0.59	0.29
■	4	MD	25	0.09	136.04	0.09	0.84	0.61	0.30

Series graphics:



Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
x	25	0.095	131.83	0.10	0.94	0.78	0.39
s	0.000	0.0238	51.76	0.01	0.10	0.35	0.18
ν	0.00	25.06	39.26	11.82	10.80	45.23	45.23

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TENSILE TEST REPORT

Parameter table:

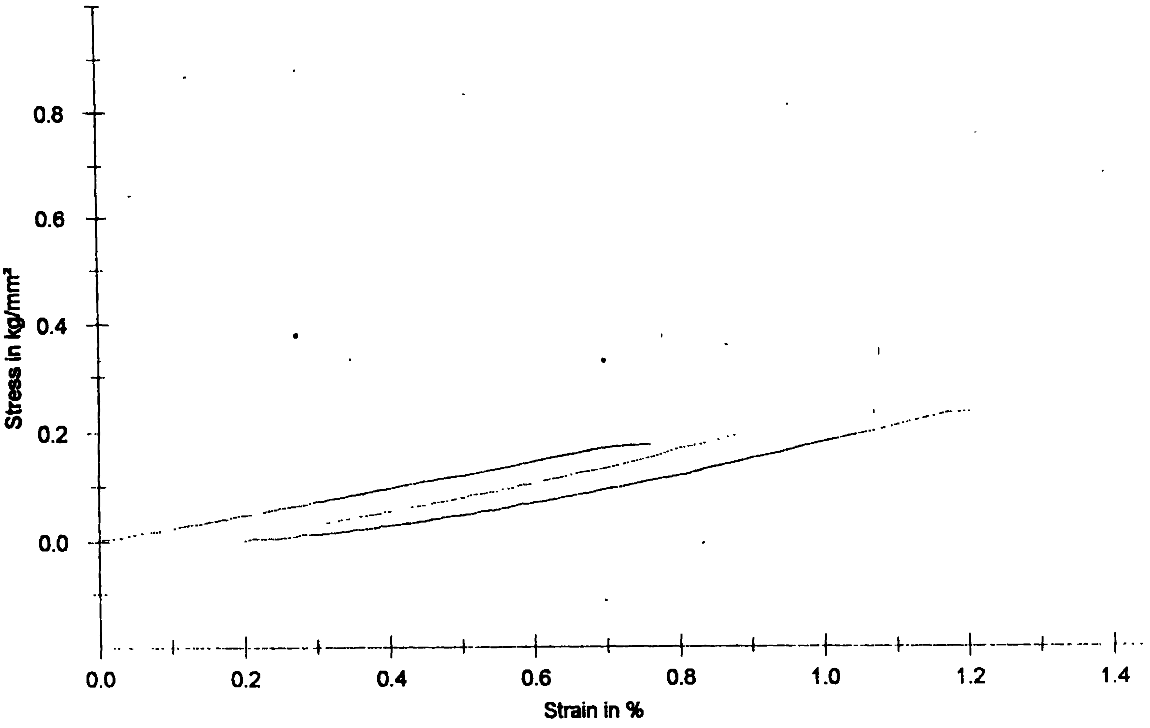
Customer : Dr: Dharmasena
Material/Material combination : Bio polymer (B-2)
Test speed : 25 mm/min

Results:

Legends	Nr	MD/TD	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %
■	1	MD	25	0.07	236.29	0.18	1.72	0.76
■	2	MD	25	0.057	206.63	0.23	2.24	0.93
■	3	MD	25	0.067	185.67	0.23	2.29	1.01
■	4	MD	25	0.083	100.96	0.15	1.48	1.10

Legends	Nr	ε Break mm
■	1	0.38
■	2	0.47
■	3	0.50
■	4	0.55

Series graphics:



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TENSILE TEST REPORT

Statistics:

Series n = 4	Width mm	Thickness mm	E-Modulus N/mm²	Maximum force kg/mm²	Force at break N/mm²	Elongation %	ε Break mm
$\bar{x}$	25	0.06925	182.39	0.20	1.93	0.95	0.48
s	0.000	0.01072	58.12	0.04	0.40	0.14	0.07
v	0.00	15.48	31.87	20.95	20.59	15.02	15.02

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Appendix III

Results of Statistical Analysis

For Materials with 2 layers - Analysis of Variance Procedure

1-Films cleaned with alum salt
2-Films cleaned with Bleaching
3-Films cleaned with pipe water

Number of observations in data set = 12

Dependent Variable: STRENG

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	2	3.10326667	1.55163333	6.52	0.0178
Error	9	2.14182500	0.23798056		
Corrected Total	11	5.24509167			

R-Square	C.V.	Root MSE	STRENG Mean
0.591652	39.68807	0.487833	1.229167

Source	DF	Anova SS	Mean Square	F Value	Pr > F
TREAT	2	3.10326667	1.55163333	6.52	0.0178

Level of TREAT	N	------------STRENG----------- Mean	SD
1	4	1.00750000	0.59673417
2	4	1.93250000	0.39676819
3	4	0.74750000	0.44768851

```
                 Analysis of Variance Procedure

               T tests (LSD) for variable: STRENG

  NOTE: This test controls the type I comparisonwise error rate not
                  the experimentwise error rate.

               Alpha= 0.05  df= 9  MSE= 0.237981
                   Critical Value of T= 2.26
               Least Significant Difference= 0.7803

       Means with the same letter are not significantly different.

T Grouping          Mean          N     TREAT

      A           1.9325          4       2

      B           1.0075          4       1
      B
      B           0.7475          4       3
```

For Materials with 3 layers - Analysis of Variance Procedure

1- Films cleaned with alum salt

2-Films cleaned with Bleaching

3-Films cleaned with pipe water

```
Number of observations in data set = 12


Dependent Variable: STRENG
                             Sum of          Mean
Source              DF      Squares         Square         F Value    Pr > F

Model                2     1.51801667     0.75900833        0.71      0.5175

Error                9     9.63007500     1.07000833

Corrected Total     11    11.14809167

            R-Square         C.V.        Root MSE       STRENG Mean

            0.136168       73.84262      1.034412        1.400833

Source             DF    Anova SS     Mean Square        F Value        Pr > F

TREAT               2   1.51801667    0.75900833          0.71          0.5175
```

```
          Analysis of Variance Procedure

        Level of     ------------STRENG-----------
        TREAT      N        Mean            SD

          1        4     1.23500000      0.52131245
          2        4     1.89500000      1.58611685
          3        4     1.07250000      0.64999359
```

For Material with 4 layers - Analysis of Variance Procedure

1- Films cleaned with alum salt
2-Films cleaned with Bleaching
3-Films cleaned with pipe water

```
                    Number of observations in data set = 12

Dependent Variable: STRENG
                              Sum of         Mean
Source              DF        Squares        Square              F Value     Pr > F

Model                2        0.53171667     0.26585833           1.19       0.3466

Error                9        2.00297500     0.22255278

Corrected Total     11        2.53469167

               R-Square      C.V.        Root MSE        STRENG Mean

               0.209776     27.68244     0.471755        1.704167

Source             DF     Anova SS       Mean Square          F Value        Pr > F

TREAT               2   0.53171667      0.26585833             1.19          0.3466


               Analysis of Variance Procedure

          Level of     -----------STRENG-----------
          TREAT     N      Mean              SD

           1        4   2.00000000       0.27916542
           2        4   1.52750000       0.70443240
           3        4   1.58500000       0.30577770
```

For materials with 2,3,4 layers - Analysis of Variance Procedure

2-Films with 2 layers
3-Films with 3 layers
4-Films with 4 layers

```
                         Number of observations in data set = 36

                              Analysis of Variance Procedure

Dependent Variable: STRENG
                                Sum of        Mean
Source              DF         Squares        Square              F Value     Pr > F

Model                2      1.38842222     0.69421111              1.21       0.3110

Error               33     18.92787500     0.57357197

Corrected Total         35  20.31629722

              R-Square         C.V.     Root MSE          STRENG Mean

              0.068340      52.42152    0.757345           1.444722

Source               DF      Anova SS     Mean Square         F Value      Pr > F

LAYER                 2    1.38842222     0.69421111             1.21      0.3110



              Level of      ------------STRENG-----------
              LAYER          N        Mean               SD

              2             12     1.22916667     0.69052626
              3             12     1.40083333     1.00670893
              4             12     1.70416667     0.48002762
```

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