“PREPARATION & PROPERTIES EVALUATION OF PACKAGING FILM USING POTATO STARCH AND LDPE BLENDS”

A

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Submitted in partial fulfilment of the requirements for the award of the degree of

MASTER OF TECHNOLOGY

IN

FOOD TECHNOLOGY

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BY

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This to certify that I have personally worked on the thesis entitled **“Preparation & properties evaluation of Packaging Film using Potato Starch and LDPE blends”.** The data mentioned in the thesis report have been generated during the work and are genuine. Data obtained from other agencies have been duly acknowledged. The result embodied in this thesis report has not been submitted to any other University/ Institute for the award of any degree or diploma.

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PREPARATION & PROPERTIES EVALUATION OF PACKAGING FILM USING POTATO STARCH AND LDPE BLENDS

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ABSTRACT

This research work was done by incorporating potato starch to LDPE to develop a biodegradable film using LDPE, glycerol, urea and talc powder. Potato starch incorporated LDPE film was made by blown film extrusion method with different proportions of potato starch T1 (5%), T2 (7.5%), and T3 (10%). Film was then tested for the mechanical and other properties for its suitability in food packaging. The thickness of the film was found in the range of 0.66mm to 1.25mm. The tensile strength was found better for T2 (13.02 MPa). Similarly dart impact resistance of the film was found to be in the range of 0.8 N to 1.2 N. Water absorption of the bio-based film increases from 0.030% to 0.066%. This result was promising so that this biodegradable film can be used for food packaging and it will not deteriorate the environment. The need for bio-plastics is now more than ever as the rate of plastic production and air pollution has increased at a rapid rate.

Keywords: Potato starch, Synthetic Polymers, Biodegradable, Blown film extruder**.**

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ASTM	American Society of Testing and Materials
BF	Breaking Force
Ca Cas	Calcium Caseinate
CIPET	Central Institute of Plastic and Engineering Technology
CH_4	Methane
Cm	Centimeter
CO_2	Carbon Dioxide
CS	Chitosan
DTA	Differential Thermal Analysis
d.f.	Degree of freedom
EAB	Elongation at Break
EDF	Extended Depth of Focus
EM	Elastic Modules
EPA	Environment protection agency
et al	and others
Etc	etcetera
FNS	Food and nutrition service
FTIR	Fourier transform spectroscopy
GAB	Guggehein Anderson deBoer

GMO	Genetically modified organisms
HDPE	High Density Polyethylene
ISO	International standards Organization
i.e.	That is
Kg	Kilogram
LLDPE	Linear Low Density Polyethylene
MC	Moisture Content
MFI	Melt Flow Index
MPa	Mega Pascal
MW	Mega Watt
Min	Minute
N/mm^2	Newton Square Millimeter
PCL	Polycaprolactone
PE	Polyethylene
PE-g-MA	Polyethylene grafted Maleic Anhydride
P^H	Potential Hydrogen
PLA	Polylactic Acid
PS	Polystyrene
PVOH	Polyvinyl Alcohol
PWS	Plasticized Wheat Starch
RH	Relative Humidity
SEM	Scanning Electron Microscopy

SS	Sum of Square
TGA	Thermogravimetric Analysis
TE	Tensile Strain
TPCS	Thermoplastic Cassava Starch
TPS	Thermoplastic Starch
TS	Tensile Strength
WPC	Whey Protein Concentrate

INTRODUCTION

As well known, synthetic polymer materials have been widely used in every field of human activity during the last decades. These artificial macromolecular substances are usually originating from petroleum and most of the conventional ones are regarded as non-biodegradable. However, the petroleum sources are limited and the blooming use of non-biodegradable polymers have caused serious environmental problems. In addition, the non-biodegradable polymers are not suitable for temporary use such as sutures. Thus, the polymer materials which are biodegradable have being paid more and more attention. Now a days most of the food items are packed. In everyday life packaging is an important area where biodegradable polymers can be used. **(Pawar *et al.* 2013).**

This research aspect has been developed to study a "material" as substitute for petrochemical materials, i.e. plastic products, and to decrease the environmental pollution resulted from the petrochemical materials. Biotechnological processes are being developed as an alternative to existing route or to get a biodegradable biopolymers. This material is called as a biodegradable **"Green Plastic"** which is derived from plants and microorganisms. Biodegradable plastics arc those that can be degraded in landfills, composters or sewage treatment plants by the action of naturally occurring micro-organisms. Truly biodegradable plastics leave no toxic, visible or distinguishable residues following degradation **(Mooney 2009).**

The market demand for biodegradable plastic is 40,000 - 50,000 and 70,000-80,000 ton/year and expands to 20 and 16% per year, respectively. This is due to the growing problem of waste, resulting in the general shortage of landfill availability and the need for environmentally responsible use of resources together with the CO_2 neutrality aspect (**Simon *et al.* 1998**). However, to solve these problems, biodegradable plastic was used instead of synthetic plastic.

Starch a renewable source, appears to be the best raw material for biodegradable polymer with low cost. Starch from different sources has been studied as a potential film-forming agent, including that from potato, barley, wheat and rice. Films developed from starch are described as odourless, colourless, non-toxic and biologically degradable **(Flores *et al* 2007)**.

Starch contains amylose and amylopectin, the former, especially in amylomaize starches containing 70% amylase gives stronger and more flexible films. The branched structure of amylopectin generally leads to film with poor mechanical properties **(Srinivasa and Biji 2004).**

Starch

Starch is a polymeric carbohydrate consisting of a large number of glucose units joined together by α-1,4 gycosidic bonds. This polysaccharide is produced by most green plants as energy reserve. It is contained in large amounts in potatoes, wheat, corn, rice, etc. Native starch is a white tasteless and odorless powder. It is insoluble in water and alcohol. It consists of two types of molecule amylase and amylopectin. Amylase is a linear structure of monomer units while amylopectin is a branched structure of monomer units.

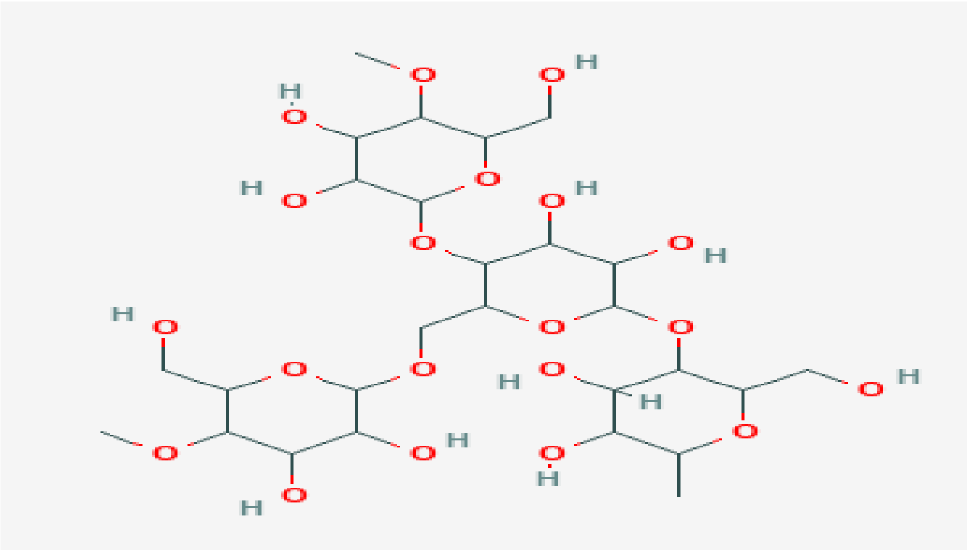

Fig 1.1 Chemical Structure of Starch

Potato is one of the world's major agricultural crops. It has been in cultivation since its introduction in the early part of 17th century **(Choudhury, 2003).** It is considered of significant importance as food crop. Potatoes are used as such or as ingredients in various foods, for starch extraction and other industrial purposes. India is the 2nd largest producer of potato with a production of about 48.7 million MT after China which produces 99.2 million MT **(FAOSTAT, 2017).**

Potato starch is starch extracted from potato. The cells of the root tubers of the potato plant contain starch grains (leucoplast). Potato starch is a very refined starch, containing minimal protein or fat. This gives the powder a clear white colour, and the cooked starch typical characteristics of neutral taste, good clarity, high binding strength, long texture and a minimal tendency to foaming or yellowing of the solution.

Starch as packaging material alone does not form good films with adequate mechanical properties, unless it is treated with plasticizers or blended with other materials. To overcome brittleness of starch-based films, common plasticizers like glycerol, sorbitol etc are used. Use of plasticizers such as glycerol, sorbitol etc in the film formulation is advantageous to impart pliability and flexibility, which will improve handling **(Tharanathan and Saroja (2001).**

Polyethylene is the plastic most well known to the consumer and is used in greater volume worldwide than any other plastic. This versatile polymer is used in a variety of areas from packaging to agriculture, building to electronics as illustrated in Table.1.1 Packaging is the major application for Polyethylene. The second most important one is wholesale. The continuous growth of polymer materials for food packaging applications in conjunction with their recalcitrance toward degradation and their visibility in the environment when discarded have stimulated further research in the field of food packaging. It has been estimated that 2% of all plastics eventually reach the environment, thus contributing considerably to a currently acute ecological problem.

Polyethylene, the first commodity plastic used on packaging, came into general use in the 1950s. Polyethylene is a family of polymers including for example the following: low-density polyethylene (LDPE), linear low density polyethylene (LLDPE) and high-density polyethylene (HDPE). These PE differ principally in their density, which is related with their degree of crystallinity. Consequently, this property will influence their melting point ranges. Some typical properties values ranges for PEs are illustrated in Table 1.1

Table 1.1 Typical Properties of Polyethylene

Type of PE	Density (g/cm^3)	Melting point C)	Mol. Wt (KDa)
LDPE	0.915-0.940	105-115	10-50
LLDPE	0.915-0.926	112-124	50-200
HDPE	0.940-0.970	120-130	Above 250

Plastics are typically composed of artificial synthetic polymers. Their structure is not naturally occurring, so plastics are not biodegradable. Bio-plastics can be defined as plastics made of biomass such as potato, corn, wheat, sugarcane etc. These substances have been increasingly highlighted as means for saving fossil fuels, reducing CO_2 emission and plastic wastes. Biodegradability of bio-plastics has been widely publicized in society and the demand for packaging is rapidly increasing among retailers and the food.

Biodegradable plastics or biodegradable polymers are plastic materials that undergo bond scission in the backbone of a polymer through chemical, biological and/or physical forces in the environment at a rate which leads to fragmentation or disintegration of these plastics (**ASTM sub-committee D20.96**).

In India, 15,000 tonnes of plastic waste is generated every day, out of which 9,000 tonnes is collected and processed, but 6,000 tonnes of plastic waste is not being collected. The rapid rate of urbanization and development has led to increase in consumption of plastic products and plastic waste generation. The burning of plastics waste under uncontrolled conditions lead to generation of different hazardous air pollutants depending upon the type of polymers and additives used (**CPCB, 2017)**

The term "biodegradable" materials is used to describe those materials which can be degraded by the enzymatic action of living organisms, such as bacteria, yeasts, fungi and the ultimate end products of the degradation process, these being CO_2, H_2O and biomass under aerobic conditions and hydrocarbons, methane and biomass under anaerobic conditions **(Kuorwel *et al*. 2011).**

Biodegradable plastic made from renewable resources will decreases dependence on petroleum and reduces the amount of waste material, while still yielding a product that provides similar benefits of traditional plastics (**Stevens 2002**). Bio-based polymers have been shown to be a viable alternative to replace these fossil sources while also having environmental advantages, such as decreasing toxic emissions. So the development of biodegradable packaging derived from renewable natural resources, has gained increasing interest. Target markets for biodegradable polymers include packaging materials like trash bags, loose-fill foam, food containers, film wrapping, laminated paper, hygiene products like diaper back sheets and cotton swabs, consumer goods like fast-food tableware and containers, egg cartons, and toys, and agricultural tools like mulch films and planters etc. **(Erksk *et al.* 2006**)

Cellulose, lignin, and starch are commonly available in nature. Cellulose is abundant in all plants, although some plants produce more than others. Lignin is typically found in wood, and starch is common in plants such as potatoes, and wheat. Plants, wood, corn, and wheat are all raw materials that are renewable, biodegradable and easily available (**Scott *et al.* 2006).** Packaging materials based on these natural materials may be a solution to help control the environmental pollution and resolve other problems posed by non-degradable synthetic polymers **(Butschli *et al.* 2005**).

The need for packaging can be linked to the progress of civilization and need to preserve perishables for longer period of time. Our whole world seems to be wrapped in plastics. Almost every product people buy, most of the food product in solid and liquid form come encased in plastic. These are usually artificial macromolecular synthetic plastics originating from petroleum and most of the conventional ones are regarded as non- biodegradable. However, the petroleum resources are limited and the blooming use of non- biodegradable polymers has caused serious environmental problems. Plastic packaging being used are non-biodegradable as they cause ecological imbalance and aesthetic deterioration of nature. Incineration may generate toxic air pollution, and satisfactory landfill sites are limited. Also, the petroleum resources are finite and are becoming limited. The environmental impact of persistent plastic wastes is evoking more global concern as alternative disposal methods are limited therefore great need to develop environment friendly biodegradable packaging materials which do not cause environmental pollution. Biodegradable packaging materials neither promote any waste disposal problems nor affect the trade and safety of the food product (**Sharma, 2000**).

To meet the growing demand of recyclable or natural packaging materials and consumer demands for safer and better quality foods, new and novel food-grade packaging materials or technologies have been and continue to be developed. Examples of these packaging materials include bio based polymers, bio plastic or edible polymer packaging products made from raw materials originating from agricultural or marine sources **(Chinnan *et al.* 2004).** It will help to reduce the packaging waste associated with processed foods and will support the preservation of fresh foods, extending their shelf life **(Gacitua *et al.* 2005).**

1.1 JUSTIFICATION

Thermoplastic packaging material has been very useful to human being in past decades but it has caused adverse health effects on the humans as well as the environment. This research studies on biodegradable packaging films that have conducted to serve consumer demands for safe, convenient and healthy food products. with extended shelf life as well as sustainability awareness as synthetic plastics last for long time and do huge damage to the environment thus bio plastics are more sustainable. Biodegradable packaging film is most efficient type of packaging material. Biopolymers present in packaging film are generally capable of being utilized by living matter (bacteria ,fungi etc), and so can be disposed in safe and ecological sound ways through disposal processes (waste management) like composting, soil application and biological wastewater treatment. Moreover these packaging films are cost effective than plastics packaging film as they are made from naturally occurring materials such as starch which are obtained from naturally occurring sources such as potato, rice, corn, barley, etc. which are readily available at market at suitable prices. Starch based-bioplastic have been shown to degrade 10-20 times quicker than conventional plastics. Producing conventional plastics consumes 65% more energy than bioplastics. Bioplastics saves 30-80% of the Green House Gas emission. Compost derived in part from bioplastics increases the soil organic content as well as water and nutrient retention, with reducing chemical inputs and suppressing plant diseases. Hence this bio-based packaging film will decrease dependency on petroleum sources and will be great answer for the problems such as saving fossils fuel, global warming and greenhouse effects .

1.2 OBJECTIVES

The present study entitled “Preparation and properties evaluation of packaging film using Potato starch and LDPE blends” was undertaken with the following objectives:

1. To develop potato starch incorporated LDPE packaging film at different composition.
2. To evaluate mechanical and biodegradable properties of the bio-based film.
3. To compare the properties of LDPE and starch incorporated LDPE and its suitability for food packaging film.

CHAPTER 2

REVIEW OF LITERATURE

The developmental of the process or product need intensive and judicious information on various aspects that can affect the efficiency of the process and the quality of the product. This chapter covers the general information about food packaging, plastics and pollution, biodegradable polyfilm, mechanical properties of bio based polyfim and its comparison with synthetic plastics.

2.1 Food Packaging Material and Pollution

Mody and Srinivasa (2000) revealed that major hurdle against increased used of plastics is their total non-biodegradability and as a result, ever increasing mounting garbage waste. With the ever growing environment consciousness among consumers and by government, the plastics packaging industry is now facing severe ecological and legislative issue for handling raw materials and eventual disposal of solid wastes and finished products. Traditional methods of handling post-consumer plastic waste include incineration, depolymerisation, recycling and land filling.

Kalia *et al.* (2000) concluded that the indiscriminate use of plastics in packaging industry and littering all around without proper disposal management has led to mounting solid wastes and causing severe environment pollution. The production and consumption of plastics have increased in geometrical progression. The annual per capita consumption of plastics in India is 2kg/person/year compared to 60kg/person/year in developed countries. In India, plastics wastes account to 3 % by weight of total of 80,000 metric tons of municipal solid waste generated daily.

Srinivasa and Biji (2004) stated that packaging is important in post-harvest preservation of fruits and vegetables and processed foods for assured shelf life extension. The raw materials of food as well as the innumerable number of processed food products made available in the market are highly perishable and need effective packaging system for their shelf life extension and availability at far off places. Protection of food products relates to the rate of quality change, including both physical (mechanical damage during transit or storage, loss of consistency or crispiness, loss of appearance, and ales appeal) and organoleptic changes (loss of taste, colour and odour).

Marsh and Bugusu (2007) studied that the primary purpose of food packaging must continue to be maintaining the safety, wholesomeness, and quality of food. The impact of packaging waste on the environment can be minimized by prudently selecting materials, following EPA guidelines, and reviewing expectation of packaging in terms of environmental impact. Knowledgeable effort by industry, government and consumers will promote continued improvement and an understanding of the functional characteristics of packaging will prevent much of the well-intentioned but ill-advised solution that do not adequately account for both pre-consumer and post-consumer packaging factors.

Gregory *et al* (2009) studied that over the past five or six decades, contamination and pollution of the world's enclosed seas, coastal waters and the wider open oceans by plastics and other synthetic, non-biodegradable materials (generally known as 'marine debris') has been an ever-increasing phenomenon. The sources of these polluting materials are both land- and marine-based, their origins may be local or distant, and the environmental consequences are many and varied. The more widely recognized problems are typically associated with entanglement, ingestion, suffocation and general debilitation, and are often related to stranding events and public perception. Among the less frequently recognized and recorded problems are global hazards to shipping, fisheries and other maritime activities. Today, there are rapidly developing research interests in the biota attracted to freely floating (i.e. pelagic) marine debris, commonly known as 'hangers-on and hitch-hikers' as well as material sinking to the sea floor despite being buoyant. Dispersal of aggressive alien and invasive species by these mechanisms leads one to reflect on the possibilities that ensuing invasions could endanger sensitive or at-risk coastal environments (both marine and terrestrial) far from their native habitats.

2.2 Development of Biodegradable Plastics for Packaging

Flieger *et al.* (2003) cited that, the field of biopolymers, while in its early stages, is growing in popularity every day. General introduction on biopolymers, a technical overview of biopolymer filed, and description of the research in Europe, Japan and

USA was summarized. Biodegradable polymers are an alternative to petroleum-based polymers. Some biodegradable polymers degrade in few weeks, while the degradation of other takes several months. In principal the properties relevant for application as well as biodegradability are determined by the molecular structure.

Tharanathan (2003) studied that the biodegradability is not only a functional requirement but also an important environmental attributes. Increased use of synthetic packaging films has led to serious ecological problems due to their total non-biodegradability. Continuous awareness by one and all towards environmental pollution by the latter and as a result the need for a safe, eco-friendly atmosphere has led to a paradigm shift on the use of biodegradable materials, especially from renewable agriculture feedstock and marine food processing industry wastes. Such an approach amounts to natural resource conservation and recyclability as well as generation of new, innovative design and use. Their total biodegradation to environmentally friendly benign products such as CO_2, water and quality compost is the turning point which needs to be capitalized and en-cashed. Polymer cross-linking and graft copolymerization of natural polymers with synthetic monomers are other alternatives of value in biodegradable packaging films. Although their complete replacement for synthetic plastics is just impossible to achieve and perhaps may be even unnecessary, at least for a few specific applications our attention and needful are required in the days to come. No doubt, eventually bio-packaging will be our future.

Torabi *et al. (2004)* had synthesized polyethylene using different formulations based on starch and urea which are biodegradable in soil and moist media. The compounds produced are injection moulded, and tested under natural conditions and their biodegradability has been studied. It had been thus possible to produce a biodegradable low-density polyethylene which is environmentally biodegradable material that is suitable for agriculture and packaging applications.

Srinivasa and Biji (2004) explained that there are two types of bio molecules viz. hydrocolloids and lipids, which are generally used in combination for the preparation of biodegradable packaging film of composites. Individually they lack structure integrity and characteristic functionality. Composite films are in fact a mixture of these and other ingredient in varying proportion, which determine their barrier and mechanical properties. Use of plasticizers such as glycerol, PEG, sorbitol, etc. in the film preparation or composites is advantageous to impart pliability and flexibility, which will improve handling.

Gacitua *et al.* (2005) reviewed current research, techniques for characterization and trends on the field of Nano-composites. Nano-composites are new materials made with fillers which have Nano size. These materials have a big potential for applications in the automotive and aerospace industry as well as in construction, electrical applications and food packing. There is a tremendous interest for using bio nanoparticles like cellulose micro fibrils or whiskers to be applied in the new era of bio- composites.

Fabunmi *et al.*, (2007) studied the diversity and ubiquity of plastic products substantially testifies to the versatility of the special class of engineering materials known as polymers. However, the non-biodegradability of these petrochemical-based materials has been a source of environmental concerns and hence, the driving force in the search for ‘green’ alternatives for which starch remains the front liner. Starch is a natural biopolymer consisting predominantly of two polymer types of glucose namely amylose and amylopectin. The advantages of starch for plastic production include its renewability, good oxygen barrier in the dry state, abundance, low cost and biodegradability. The longstanding quest of developing starch-based biodegradable plastics has witnessed the use of different starches in many forms such as native granular starch, modified starch, plasticized starch and in blends with many synthetic polymers, both biodegradable and non-biodegradable, for the purpose of achieving cost effectiveness and biodegradation respectively. In this regard, starch has been used as fillers in starch-filled polymer blends, thermoplastic starch (TPS) (produced from the combination of starch, plasticizer and thermo mechanical energy), in the production of foamed starch and biodegradable synthetic polymer like poly lactic acid (PLA) with varying results. However, most starch-based composites exhibit poor material properties such as tensile strength, yield strength, stiffness and elongation at break, and also poor moisture stability. This therefore warranted scientific inquiries towards improving the properties of these promising starch-based bio composites through starch modification, use of compatibilizers and reinforcements (both organic and inorganic), processing conditions, all in the hope of realizing renewable biodegradable substitutes for the conventional plastics.

Huneault and Li (2007) investigated the properties and interfacial modification of blends of poly lactide (PLA) and glycerol-plasticized thermoplastic starch (TPS). A twin-screw extrusion process was used to gelatinize the starch, devolatilize the water to obtain a water-free TPS and then to blend into the PLA matrix. The investigated TPS concentration ranged from 27 to 60wt%. In the absence of interfacial modification, the TPS/PLA blend morphology observed through scanning electron microscopy was very coarse with TPS particles sizes between 5 and 30μm. Interfacial modification was achieved by free-radical grafting of maleic anhydride (MA) unto the PLA and then by reacting the modified PLA with the starch macromolecules. Blends comprising MA-grafted PLA showed much finer dispersed phase size, in the 1–3μm range and exhibited a dramatic improvement in ductility. The paper discusses the effects of two interfacial modification strategies on the blend morphology and tensile properties and investigates the compatibilization efficiency for glycerol plasticizer contents between 30 and 39wt% and for starches from three different sources: wheat, pea and rice.

Lafargue *et al.*, (2007) reported that the characteristics of film based on biopolymers strongly depend on their composition and their preparation procedure. It is known that the film thickness influences its properties.

Thompson *et al.* (2009) reviewed that plastics have transformed everyday life; usage is increasing and annual production is likely to exceed 300 million tonnes by 2010. In this concluding paper to the Theme Issue on Plastics, the Environment and Human Health, we synthesize current understanding of the benefits and concerns surrounding the use of plastics and look to future priorities, challenges and opportunities. It is evident that plastics bring many societal benefits and offer future technological and medical advances. However, concerns about usage and disposal are diverse and include accumulation of waste in landfills and in natural habitats, physical problems for wildlife resulting from ingestion or entanglement in plastic, the leaching of chemicals from plastic products and the potential for plastics to transfer chemicals to wildlife and humans. However, perhaps the most important overriding concern, which is implicit throughout this volume, is that our current usage is not sustainable. Around 4 per cent of world oil production is used as a feedstock to make plastics and a similar amount is used as energy in the process. Yet over a third of current production is used to make items of packaging, which are then rapidly discarded. Given our declining reserves of fossil fuels, and finite capacity for disposal of waste to landfill, this linear use of hydrocarbons, via packaging and other short-lived applications of plastic, is simply not sustainable. There are solutions, including material reduction, design for end-of-life recyclability, increased recycling capacity, development of bio-based feed stocks, strategies to reduce littering, the application of green chemistry life-cycle analyses and revised risk assessment approaches.

Momani (2009) expounded that the mainstreams media has been devoting more attention bio-plastics as demonstrated by newspaper and magazine articles cited in the paper. Bio-plastics have many advantages over petro-plastics but they have yet to be live up to the hype. Bio-plastics production consumes fewer fossils fuel resources than petroleum based plastics because no fossils fuel feed stocks are used. They emit less carbon dioxide than petro-plastics over their lifecycle. Bio-plastics consume less energy to produce than petroleum based plastics. They have fewer health concerns associated with them. Also, bio-plastics are generally compostable.

Mohanty *et al.* (2010) studied a biodegradable blend based on low-density polyethylene (LDPE) and 36 % starch that can be used for horticultural application. The mechanical properties and the processing by moulding injection were also studied. It was found that the starch helps to increase surface area on which the microorganism can attack the polymer matrix. The increase in the carboxyl index, which indicates the concentration of carboxyl blend, with time, also indicates the progress of biodegradation. Although this type of blend is not completely biodegradable it is preferred to those completely biodegradable which are expensive and with many processing problems.

Aini (2010) studied that producing bio-degradable plastic which exhibit better mechanical property and presentable appearance for food packaging purpose. Since the use of plastics in food packaging in undeniable, the finding in this research might be considered for commercialization. This study also may contribute to the reduction of pollution to environment since the petroleum-based plastics caused serious damage to environment both during its production and disposal. Besides, the effect of other contamination from plastics waste also can be decrease and lack of landfill area might be resolve since he plastics produce is biodegradable. This study is also hoped to provide other alternative materials to substitute petroleum role in polymer industry due to depletion in petroleum source with a renewable source.

Ming *et al.*, (2011) characterized the biodegradable film from corn starch with different amylose content. They concluded that amylose content significantly affected the mechanical and thermal properties of the biodegradable starch-based film. The high amylose starch film exhibited better mechanical properties such as higher modulus and tensile strength, and very high impact strength. The reason for this include not only the easy entanglement of long linear amylose chains, but also retained granular structure in high amylose films, which may act as self-reinforcement.

Sharka *et al.*, *(2011)* had produced biodegradable films after blending with poly-(ε-caprolactone) (PCL) and B-starch or acetylated starch. The following mechanical characteristics of the produced films were derived from the stress-strain curves: Young modulus, yield stress, stress-at-break, and strain-at- break. Water absorption of PCL/starch (60/40) films was determined according to European standard ISO 62. The measured data were compared with those of commercial A-starch. The films containing native starch degraded in compost totally during 2 months. Acetylation of starch molecules in the composites reduced the degradation rate. Optical microscopy, in combination with the image analysis system NIS-Elements vs. 2.10 completed with an Extended Depth of Focus (EDF) module, was used to study the surface morphology of PCL/starch films after 20-day and 42-day compost incubation. Chemical changes in the compost used for the film exposition were measured.

Azahari *et al.*, (2011) prepared polyvinyl alcohol (PVOH)/Corn Starch (CS) blend films using the solution casting method. The biodegradability of the film was investigated based on enzymatic absorbency in water and an acidic solution as well as by burial in soil and compost. The tensile properties were examined using a tensile test. The structure of the film was characterized by scanning electron microscopy. Compared to the film without corn starch, the film containing starch was found to be more highly biodegradable by enzyme as well as in soil and compost. However, the result from the tensile and elongation at break test showed that as the corn starch content increased, the strength decreased. The morphology study revealed the distribution of corn starch in the PVOH.

Guerrero *et al* (2011) prepared Soya protein-based films modified with gelatin and plasticized with glycerol by compression at pH 10. The effect of different contents of lactic acid, epoxydized soybean oil and olive oil on optical, barrier, and mechanical properties was investigated and results were related to changes in the bands obtained by Fourier infrared spectroscopy and thermo-gravimetric analysis. The observed changes indicated interactions of the small molecules of lactic acid with protein and glycerol, which also caused the improvement in the hydrophobic character of the films, maintaining water vapour permeability and good values for puncture and UV barrier properties and, at the same time, decreasing the typical yellowish colour of soy protein based films.

Sabetzadeh *et al.* (2011) prepared low-density polyethylene (LDPE)/thermoplastic corn starch (TPCS) compounds containing the highest amount of starch for biodegradable application. It is intended to increase concentration of corn starch into LDPE without affecting basic required mechanical properties of LDPE plastics for disposable products. LDPE/TPCS blends containing different contents of TPCS (0–40wt %) and a constant amount of LDPE grafted maleic anhydride [PE-g-MA (3wt %)] are prepared using a single-screw extruder. The prepared blend was evaluated for their mechanical, flow, and water absorption properties. Scanning electron micrographs of the samples show improvement in dispersion of the starch particles in LDPE matrix in the presence of PE-g-MA as a compatibilizer. The Young’s modulus and impact strength properties reduce by increasing corn starch content in TPCS. The ultimate tensile strength and elongation at break of the samples show that the blends with 25wt % TPCS have the required mechanical properties to produce plastic packaging products, as evidenced by ASTM D 4976-04. The results of rheological tests indicate that as shear rate increases, the apparent viscosity of the blends decreases (shear thinning). The addition of starch to LDPE also decreases the melt flow index values of the samples. The water absorption of the samples increases with increasing starch concentrations at a constant time of water immersion.

Perez *et al.* (2013) prepared polypropylene/starch blend using different botanical starch. The influence of starch type and starch content on thermal properties and morphology of polypropylene/starch blends were studied. Corn and potato starches were used as starch source. The starch content in the blends was 7 and 15wt-%. Characterization of PP and PP/starch blends by (DSC), (TGA), and (SEM) was carried out to obtain information on miscibility among PP and starch. The starch domains exhibited characteristic voids be due to thermal degradation from the thermo degradative studies of the PP/CS and PP/PS blends, it can be concluded that the CS and PS content induces a retardant effect on the degradative process of the PP, since it increases the initial decomposition temperature of the blend, acting like an anti-oxidant agent. The presence of an amylose content as well as corn and potato starches increase in the blend. This increase of amylose content produced a greater dispersion of CS and PS in PP1 increases the miscibility. It was corroborated by DSC, TGA, and SEM. Thus conclude that PP/CS blends have lower decomposition temperatures than PP/PS because corn starch has a higher content of amylose than that potato starch.

Oromiehie *et al.* (2013) studied composites of low density polyethylene (LDPE) and thermoplastic starch (LDPE/TPS) at different ratios of TPS (40%, 60% w/w) was prepared in internal mixer. Polyethylene grafted maleic anhydride at 3wt % was used as coupling agent. Chemical reactions between functional groups of composite components were studied and confirmed by Fourier transforms infrared (FTIR) spectroscopy. The morphology of film surfaces was studied using scanning electron microscopy. The physical, mechanical, and dynamic- mechanical thermal analyses of LDPE/TPS composites were evaluated. The FTIR results showed transmission peak at 1642 cm^{-1}, which is the result of chemical reaction between the hydroxyl groups of starch and anhydride groups of coupling agent. This verifies the presence of the carboxylate group due to the formation of ester bonding. The results showed that the water absorption and density of composite films increased by increasing the starch content in LDPE/TPS composites. The tensile strength and elongation at break decreased by increasing the starch level in the composites, but the young's modulus increased. The morphological studies showed that the biodegradability of composites increased by increasing the starch content and the results was confirmed by weight loss in burring the samples in wet soil during time intervals. The dynamic mechanical thermal analyser, thermo gram showed that there are two relaxation temperature peaks. The amplitude of peaks increased by increasing the starch content from 40 to

60% probably due to increasing amorphous phase of composite. The starch was uniformly distributed throughout the LDPE polymer matrix and compatible and biodegradable composites were formed.

2.3 Quality Evaluation of bio-based film

Vinhas *et al. (*2007) evaluated the growth and the amylolytic activity of mixed and isolated cultures of ***Phanerochaete chrysosporium*** and ***Talaromyces wortmanni*** on different types of starch. The thermal and mechanical properties in polyethylene/starch blends (**proportion: 80/20 (w/w**) before and after inoculation of the mixed cultures were evaluated. The regular starch and the modified starch Fox5901 stood out in relation to the cellular growth and production of the amylase enzyme. In spite of the short time, the blends were exposed to the fungi, the microorganisms promoted physical and chemical changes in the structure of the blend, modifying its thermal and mechanical properties. The alteration of the degree of crystallinity and mechanical properties of the blends could be indications of the modification caused by the biodegradation process.

Souza *et al.* (2011) analysed glycerol content and its incorporation method on tensile and barrier properties of biodegradable films (BF) based on cassava starch. ANOVA showed that the glycerol incorporation method did not influence the results ($P > 0.05$), however the glycerol content influenced significantly the tensile and barrier properties of the films ($P < 0.05$). Films prepared with lower glycerol content presented better tensile and barrier properties than films with higher content. Films were then prepared with addition of clay nanoparticles and their tensile and barrier properties and glass transition temperature were measured. ANOVA indicated that both glycerol and clay nanoparticles influenced significantly the tensile and barrier properties ($P < 0.05$), diminishing film permeability when clay nanoparticles were present, while the glass transition temperature was not influenced ($P > 0.05$).

Manikantan *et al.* (2012) synthesized polypropylene (PP) based nanocomposite films of 15 different compositions of nano clay, compatibilizer and thickness were developed and used for packaging and storage of banana chips. The effect of nanocomposite films on the quality characteristics viz. moisture content (MC), water activity (WA), breaking force (BF) total plate count (TPC) and overall acceptability score of banana chips under ambient condition at every 15 days interval were studied for 120 days. Among all the nanocomposite films, packaging material having 5% compatibilizer, 2% nanoclay & 100µm thickness (treatment E) and 10% compatibilizer, 4% nanoclay & 120µm thickness (treatment N) showed better stability of measured quality characteristics of banana chips than any other treatment.

Reddy and Gupta (2013) studied bio-plastics are a form of plastics derived from plant sources such as sweet potatoes, soya bean oil, sugarcane, hemp oil, and corn starch. These polymers are naturally degraded by the action of microorganisms such as bacteria, fungi and algae. Bio-plastics can help alleviate the energy crisis as well as reduce the dependence on fossil fuels of our society. They have some remarkable properties which make it suitable for different applications. This paper tries to give an insight about Bio-plastics, their composition, preparation, properties, special cases, advantages, disadvantages, commercial viability, its life cycle, marketing and pricing of these products.

Ezeoha and Ezenwanne (2013) produced a biodegradable plastic film by blending cassava starch and a synthetic biodegradable polymer (PVA). The film produced was found to have a biodegradability of 41.27% compared to 10.33% and 85.99% for polythene and paper respectively. The film also has a tensile strength of 24.87N/mm^2 compared to 10.86N/mm^2 and 8.29N/mm^2 for polythene and paper. The potential of producing biodegradable packaging plastic film from cassava starch is encouraging and should be further explored especially in a country with high cassava productivity, like Nigeria. This study have provided platform for commercial production of bio-plastics with potential property.

Wagh *et al. (*2013) prepared casein and whey protein concentrate (WPC) films, plasticized with glycerol and sorbitol independently, by casting. The film thickness, water vapour and oxygen permeation and tensile and moisture sorption properties of the films were determined. The tensile strength (TS), tensile strain (TE) and elastic modulus (EM) of the films ranged from 0.71 to 4.58 MPa, 19.22 to 66.33% and 2.05 to 6.93 MPa, respectively. The film properties were influenced by the type of biopolymer (casein and whey protein concentrate), plasticizer and its concentration. Increasing the plasticizer concentration, increased the film thickness, TE and water vapour permeability (WVP), but decreased the TS and EM. As the concentration of plasticizer increased to the highest level, the film thickness increased from 0.168 to 0.305 mm for glycerol-plasticized films and from 0.251 to 0.326 mm for sorbitol-plasticized films. The film thickness increased because the amount of plasticizer in the film network increased and the amount of biopolymer remained same. Casein films showed superior tensile properties as compared to WPC films. The WVP of both casein and WPC films lied between 3.87 and 13.97g.mm/ (m2.h.kPa). The moisture sorption isotherms of both films were typical of high-protein material, and were adequately described by the GAB model. The oxygen permeability of casein films was relatively lower than that of WPC films, regardless of the plasticizer used. The sensory data revealed that the organoleptic quality of Cheddar cheese was unaffected by milk-protein film packaging.

Bonnaillie *et al.* (2014) reported that the casein film for food packaging possess good strength and low oxygen permeability, but low elasticity and high sensitivity to moisture. The mechanical properties of solvent-cast (15% solids) calcium-caseinate/glycerol films (CaCas:Gly ratio of 3:1) were characterized as a function of processing and environmental conditions, including film thickness, solution formulation and ambient humidity (from 22% to 70% relative humidity (RH) at ~20 °C). At constant RH, the elongation at break (EAB) had a strong positive dependence on the film thickness. Depending on the desired film properties and utilization conditions, pectin may be a useful addition to casein film formulations for food packaging applications.

Elizabeth *et al.* (2014) investigated the effect of zein and film formulation on mechanical and structural properties of native (FNS), and oxidized with 2.5% (FOSA) and 3.5% (FOSB) banana starch. The oxidized starch showed differences from native Starch due to the oxidation process, showing a decrease in lipids, proteins, and amylose. The increase of the sodium hypochlorite increased the content of carbonyl and carboxyl groups in the ranges 0.015–0.028% and 0.022–0.031%, respectively. The film obtained from FOSB displayed the highest tensile strength (5.05MPa) and satisfactory elongation value (27.1%). The zein addition caused a decrease in these mechanical properties, as well as a significant decrease in water vapour permeability (WVP). However, films from FOSA and FOSB showed higher permeability than that of the native starch. The addition of glycerol and the level of oxidation increased the films moisture. Micrographs showed that, during the oxidation process, impurities were largely eliminated from the starch granule, noting more homogeneous structures both in granules and films.

Seligra *et al.* (2015) observed that biodegradable and non retrogradable starch–glycerol based films were obtained using citric acid (CA) as cross-linking agent at 75 °C. This material allowed decreasing water vapour permeability (WVP) more than 35%, remained amorphous for at least 45 days as a result of the network formed by the CA that avoided starch retrogradation and maintained the degradability in compost, occurring only six days after the films without citric acid. A simulation of the gelatinization process of starch–glycerol with and without CA, using a differential thermal analysis device, showed that the system with CA completed the gelatinization 5 °C before than the other and CA first reacted with glycerol and then starch–glycerol–CA reaction occurred. The temperature at which the gelatinization process was carried out was critical to obtain the best results. An increase of gelatinization process temperature at 85 °C in system with CA, led to a worsening on WVP and its integrity after a swelling process with dimethylsulphoxide (DMSO), compared to the films processed at 75 °C.

Hendra and Ginting (2016) investigated that bio plastics can replace, the conventional plastics because bio plastics come from raw materials that is very environmentally friendly and bio plastics is degradable. The purposes of this research are to know the characteristics of starch from durian seed and to know the effect of additional chitosan as filler, sorbitol as plasticizer, and variation of heating temperature bio plastic solution on mechanical properties of bio plastics. In process, the ratio between durian-seed starch and chitosan are 7:3, 8:2 and 9:1 gram, while the concentration of sorbitol is 20%, 30%, and 40%. The heating temperature of bio plastic solution is varied at 70^oC, 80^oC and 90^oC. Result of bio plastic FTIR shows there is increation of wave number N-H from 1570.06 cm1 to 1589.34 cm-1 and O-H from 3352.28 cm-1 to 3653.18 cm-1 . The characteristic of durian seed starch has water content 12.73%, ash content 0.51%, starch content 76.65%, amylose content 22.34%, amylopectin content 54.32%, protein content 11.61%, and fat content 0.61%. Optimum mechanical properties of bio plastic from durian seed starch occurs in heating temperature 70^{o}C with composition between durian-seed starch and chitosan is 7:3 grams and sorbitol 20.0 grams.

Mendes ***et al. (*****2016)** successfully prepared corn starch –chitosan blend by extrusion with a high dispersion. Incorporation of thermoplastics chitosan caused a decreased in both tensile strength and stiffness but had good thermal stability. Scanning Electron Microscopy (SEM) micrographs showed blends with homogeneous surface, and the criofractured samples displayed no agglomeration of chitosan within a completely destructurized starch matrix. These blends also had good thermal stability in which the addition of chitosan produced more thermally stable films. Moreover, addition of 5 and 10 wt.% chitosan acted as a plasticizer to TPS matrix, increasing the elongation at break (elongation at break increased by 56 to 35%, respectively) and decreasing tensile strength and elastic modulus. Therefore, the obtained blends have potential for applications in packaging, especially where a high output of processed polymer is required. Thus, this could be suitable for the sterilized food product.

Yamak (2016) successfully studied how, low density polyethylene (LDPE) was melt-blended with starch using twin screw extruder to form biodegradable polymer blends. The LDPE/starch blend films used in food packing were obtained by hot pressing of the granules produced by extrusion process. The starch content was varied from 0 to 40wt% of LDPE. To provide fine starch dispersion, glycerol and zinc stearate were used as plasticizer and compatibilizer, respectively. The effect of starch content on the properties of LDPE film was investigated. A good dispersion was achieved for low starch contents (10 and 20wt %), but agglomeration of the starch particles occurred in the presence of high amounts of starch (>20wt %). The addition of starch to LDPE reduced the tensile strength, elongation at break, crystallinity, and water resistance of LDPE. This decline in the LDPE properties was dramatic when the starch content was increased to 30% by weight in the blend. In addition, silver nanoparticles as antibacterial agent were incorporated to the biodegradable LDPE blends where LDPE/starch weight ratio of 60/40. The effects of these nanoparticles on morphological, mechanical, thermal and water resistance properties of the LDPE/starch biodegradable blends were investigated. It was observed that the incorporation of nano particles to the LDPE/starch blend was completely changed the morphology of the film, and accordingly, the mechanical, thermal, and water resistance properties were varied depending on the silver nanoparticle content in LDPE/starch blend film. Moreover, antibacterial activities of the nano-composite films against gram-negative bacteria **(*E. coli*)** and gram positive bacteria **(*S. aureus*)** were determined by measuring the inhibition zone around each film. However, the nano-composite films did not show any antibacterial activities in the presence of silver nanoparticles as an antibacterial agent.

Kormin *et al.* (2017) investigated the incorporation of different starches, such as sago starch, corn starch, potato starch, tapioca starch and wheat starch, in low-density polyethylene matrix (LDPE) to enhance mechanical properties and to obtain partially biodegradable product with the aim to reduce the plastics wastes in the environment. For comparison, virgin LDPE, LDPE with different sources of starch blends were prepared and characterized under the same conditions. The starches were mixed to the LDPE using a twin screw extruder to guarantee the homogeneity of the formulations. The compound were shaping processed by injection moulding. The characterization of those compounds was done by physical (density, MFI), mechanical (Universal tensile machine). The addition of starch to LDPE reduced the MFI values, the tensile strength, elongation at break and impact strength, whereas the elastic modulus, flexural modulus and flexural strength increased. LDPE/SS show the good mechanical behaviour compared to other formulation. The physical and mechanical properties were evident when 5 and 30 weight% were added. Water uptake increased with increased starch content and immersion time. The time taken for the composites to equilibrate was about one month even when they were immersed completely in water.

Yousofa *et al.* (2018) prepared thermoplastic starch edible films (TPS) loaded with donut-shaped starch micro-particles from two different botanical origins. The micro-particles were produced by a simple thermal aqueous-alcoholic treatment. The average size was 14.01 ± 2.65μm and 28.61 ± 6.84μm for the micro-particles from corn and pea starch, respectively. The starch films were produced using the solution casting method with glycerol as plasticizer. The starch films were found to be more thermally stable by increasing the donut-shaped micro-particles percentage as confirmed by the thermo-gravimetric analysis (TGA). The water vapour and the oxygen transmission rates through the TPS films can be modified by changing the micro-particles content. The TPS films are intended mainly for food packaging applications.

The addition of the micro-particles increased the thermal stability of the produced films. Moreover, tuning the percentage of the micro-particles in the thermoplastic films allowed to delivering the desired amount of the oxygen and water vapour to the packaged food. This is of a great importance for maintaining the packaged food fresh and healthy as long as possible.

Keziah and Priya (2018) derived bio plastics from renewable biomass sources such as vegetable fats and oils, corn starch, banana peel, and potato starch. Synthetic plastics are non-renewable, often threaten the environment, had declining impacts on marine life and increases environmental pollution. The development of bio plastics helps to solve many of these sustainability problems, offering the potential of renewability, biodegradation, and a path away from harmful additives and moreover a healthy earth. Bio plastics are produced using the mixture of glycerol, corn starch, vinegar, and food colour. The mixture was heated to give a good yield. Bio plastics or in other words green plastics are obtained in 2 days. They can be easily generated in our homes without any higher professional assistance and could be manufactured in a larger scale if the required facilities are set up. The need for bio plastics is now more than ever as the rate of plastic production and air pollution has increased at a rapid rate. Furthermore, plastics also cause many health risks because of its toxic nature. Therefore, bio plastics can be best suited as an alternative to improve healthy life and sustain a pollution-free planet.

Sreejaya and Genitha (2018). studied the development and performance evaluation of edible film by using cassava starch. The result were promising to be used as packaging material. A study was undertaken to develop edible film from cassava starch, to study its properties for its packaging property for spices mix. Edible film from cassava starch without and with addition of glycerol (T0, T1) was developed. The effects of plasticizer (edible glycerol) on the film properties were determined and its physico-chemical properties during the course of experiment. Small pouches were made from the edible films and spice mix was packed in it. This was stored at room temperature and kept for 45 days. This spice mix packed in different active films was evaluated on basis of peroxide value. Same tests were done for unpacked spice mix, packed in films and spice mix packed in low density poly ethylene (LDPE) films. The results revealed that addition of plasticizer has lowered water absorption and moisture permeability as compared to films without plasticizer. Considering all the parameters it can be said that the edible film labelled as T1 was best among all the other developed films. Edible film made from cassava can be used to pack spice mix for ready to eat (RTE) and other food products.

Shah *et al.* (2019) studied the effect of corn starch on LDPE and its suitability as food packaging film. The result were promising to be used as food packaging material.
This research work explores the possibility of incorporating corn starch to develop bio-plastics film using low density polyethylene (LDPE), glycerol, urea and talc powder. Corn starch incorporated LDPE film was made by twin screw extruder and blown film extrusion method with different proportion of corn starch T1 (5%), T2 (7.5%), and T3 (10%). The film was then tested for the physical, mechanical and other properties for its suitability in food packaging. The thickness of the film was found in the range of 0.7 mm to 1.3 mm. The tensile strength was found better for T2 (14.8 MPa). Similarly dart impact resistance of the film was found to be in the range of 0.8 N to 1.2 N. Water absorption of the bio-based film increases from 0.005% to 0.078%. This result was promising so that this biodegradable film can be used for food packaging and it will not deteriorate the environment. Bio-plastics can be easily manufactured in a larger scale if the required facilities are set up. The need for bio-plastics is now more than ever as the rate of plastic production and air pollution has increased at a rapid rate.

2.4 BIOPLASTICS APPLICATION

Kumar *et al.* (2014) reviewed packaging field is completely dominated by mineral oil derived polymers such as polyethylene (PE) and polystyrene (PS). These materials have brought so much convenience and attraction to food industry that nobody was bothered about the diminishing availability of these materials as they are coming from non-renewable sources and their safe disposal. But, due to the growing concern over environmental problems associated with these materials, renewed interest in packaging research are underway to develop and promote the use of “bio-plastic.”

Bio-plastic is a term used for packaging materials derived from renewable resources i.e. produced from agricultural sources, biological raw materials such as starch and bio-derived monomers and which are considered safe to be used in food applications. In general, compared to conventional plastics derived from petroleum, bio plastics have more diverse stereochemistry and architecture of side chains which enable research scientists a greater number of opportunities to customize the properties of the final packaging material. This review evaluates the suitability of bio-plastics for food packaging. Additionally, it identifies the challenges involved while using bio-plastics for different food products.

Jabeen *et al.* (2015) presented food packaging as a vital part of the subject of food technology is involved with protection and preservation of all types of foods. Due to economical abundance, petrochemical plastics have been largely used as packaging material due to their desirable properties of good barrier properties towards O_2, aroma compounds, tensile strength and tear strength. Meanwhile, they have many disadvantages like very low water vapour transmission rate and the major disadvantage is that they are non-biodegradable and result in environmental pollution. Keeping in view the non-renewable nature and waste disposal problem of petroleum, newer concept of use of bio plastics came into existence. Bio plastics of renewable origin are compostable or degradable by the enzymatic action of micro-organisms. Generally biodegradable polymers get hydrolysed into CO_2, CH_4, inorganic compounds or biomass. The use of bio-origin materials obtained through microbial fermentations, starch and cellulose has led to their tremendous innovative uses in food packaging in the last few years.

CHAPTER 3

MATERIALS AND METHODS

This chapter deals with the description of various materials and method used to accomplish the experimental work entitled "Preparation and Properties Evaluation of Packaging Film using Potato Starch and LDPE Blends". The work was done at Central Institute of Plastics Engineering and Technology (CIPET, LUCKNOW) during Jan -July 2019. The methodology adopted has been described. Table 3.1 shows the overall experimental plan of the study.

TABLE 3.1 EXPERIMENTAL PLAN OF THE STUDY

S. No	VARIABLES	LEVEL	DESCRIPTION
1	Product	1	Bio-based plastic film (LDPE with Potato starch)
2	Method	-	Blown Film Extrusion method
3	Constituents	5	Potato starch powder, LDPE, Talc powder, glycerol and urea
4	Properties evaluated	5	Thickness, Tensile Strength, Dart impact test, Water absorption, Biodegradability test

3.1 MATERIALS

The materials that were used for the preparation of bio-based plastics film were

Potato starch
Low density polyethylene (LDPE) granules
Talc powder
Urea and
Glycerine

3.1.1 Starch

Chemically, starch is polysaccharide composed of the elements carbon, hydrogen and oxygen in the ratio of 6:10:5, leading to the molecular formula of $(C_6H_{10}O_5)n$. A typical starch granule consists of small amounts of proteins, lipids, phosphorus and inorganic materials (**Swinkels 1985**). In addition to these materials, 97-99% of the starch molecule comprises of the polymers amylose and amylopectin. Amylose is a linearly chained molecule, which is connected by α- (1, 4) linkages, and contain between 500 and 5000 glucose units (**Swinkels 1985, Galliard and Bowler 1987**). Generally, the molecular weight of amylose ranges from 105 to 106. The linear structure of amylose is exhibited in Fig 3.1

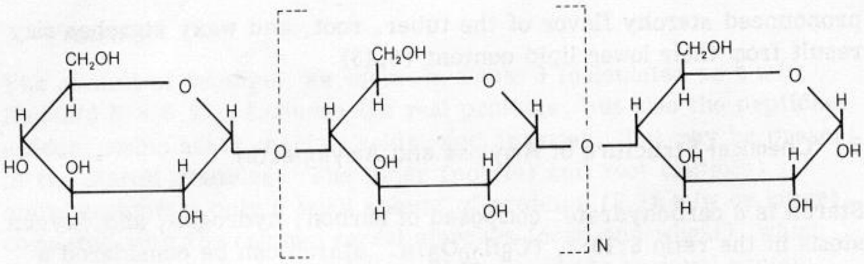

Fig. 3.1 Chemical Structure of Amylose

In contrast, amylopectin is highly branched in structure, consisting of shorter α- (1, 4) linked chains of between 10 and 60 glucose units in length (**Galliard and Bowler 1987 and Swinkels 1985**). These chains are connected to one another via α- (1, 6) linkages, as demonstrated in Fig. 3.2. The polymer molecular weight is usually in excess of 108, making it one of the largest molecules in nature. Table 3.1 shows the comparative study of amylose and amylopectin.

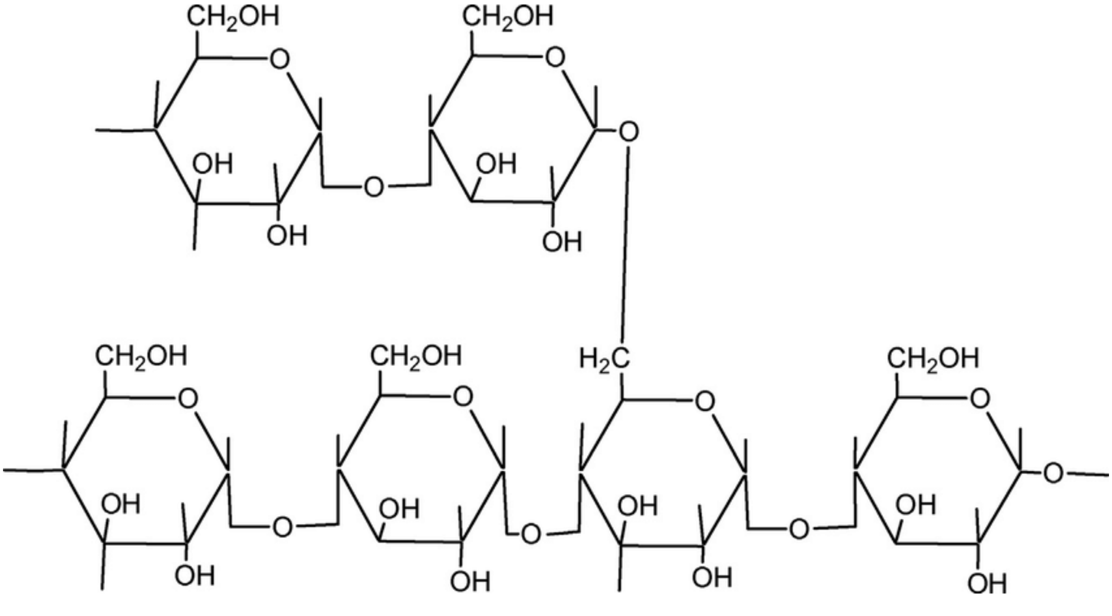

Fig. 3.2 Chemical Structure of Amylopectin

Table 3.2: Physico-chemical characteristics of amylose and amylopectin

Properties	**Amylose**	**Amylopectin**
Molecular structure	Linear(-1,4)	Branched (α-1,4; α-1,6)
Molecular weight	~106 Daltons	~108 Dalton
Degree of polymerization	1500-6000	$3*10^5$-$3*10^6$
Helical complex	Strong	Weak
Iodine colour	Strong	Weak
Dilute solution	Unstable	Stable
Retro gradation	Rapidly	Slowly
Gel property	Stiff irreversible	Stiff reversible
Film property	Strong	Weak , brittle

Source: **Jane** (2000)

Starch contributes greatly to the textural properties of many foods and is widely used in food and industrial applications as a thickener, colloidal stabilizer, gelling agent, bulking agent and water retention agent. The physicochemical properties and functional characteristics of starch systems and their uniqueness in various food products vary with starch biological origin (**Svegmark *et al.* 1993**) Native starch is a good texture stabilizer and regulator in food but limitations such as low shear resistance, thermal resistance, thermal decomposition and high tendency towards retro gradation limit its use in some industrial food applications due to which some modifications are made in native starch to change its properties.

Potato starch was obtained from the Oriental and Gums Biopolymer, Gandhi nagar Gujarat, for the study. The Potato plant is a high-capacity, factory for efficiently converting large amounts of radiant energy from the sun into stable chemical energy. This energy is stored as cellulose oil and starch in the Potato plant and in the Potato kernel. As all cereal grains, starch is the major component of Potato kernels. On a weight basis, 50–75% of the Potato grain is starch (**Rooney *et al.* 2003**) Starch is located in the endosperm both vitreous and floury.

Starch granules in Potato range from 2 to 25µm in diameter, with starch granules in Potatoeous endosperm being polygonal and smaller than those in the floury endosperm, which are more round in shape. Gelatinization temperatures of sorghum starch have been reported to vary from 62 to 72°C with starch isolated from Potatoeous endosperm having a higher gelatinization temperature than that from the floury endosperm. Potatoeous endosperm starch also has a higher intrinsic viscosity, and lower iodine-binding activity than that of the floury endosperm.

3.1.1.1 Physicochemical properties of starch

3.1.1.1.1 Gelatinization

Gelatinization is the process of granule swelling that breaks down the intermolecular bond of starch molecule in the presence of heat and heat allowing the hydrogen bonding site to engage more water followed by disruption of granule structure in which the loss of crystalline order can be observed in the disappearance of the X-ray diffraction. According to (**Jane 2004**), the temperature range at which starch granules lose their ordered structure in the presence of excess water is the gelatinization temperature reported that the gelatinization temperatures of sorghum starch vary from 71 to 80°C .Penetration of water increases randomness in the general structure and decreases the number and size of crystalline regions. Crystalline regions do not allow water entry. Heat causes such regions to be diffused, so that the chains begin to separate into an amorphous form (**Singh and Seung 2008**) The granules/grains swell to 5 times their original size. Starch gelatinization ranges of different source are shown in Table 3.2.

Table 3.3 Amylose, amylopectin content and gelatinization temperature of different starch.

Type of starch	Amylopectin (%)	Amylose (%)	Gelatinization point (^{0}C)
Sorghum	73	27	71-80
Potato	73	27	62-72
Corn	78	22	58-67
Rice	83	17	62-72
Tapioca	82	18	51-65
Wheat	76	24	58-64

Source **Jen** (1990)

3.1.1.1.2 Retro-gradation

During storage starch pastes may become cloudy and eventually deposit an insoluble white precipitate. This is caused by the re-crystallization of starch molecules; initially the amylose forms double helical chain segments followed by helix-helix aggregation. This phenomenon is termed retrogradation. Amylose is considered primarily responsible for the short-term retrogradation process due to the fact that the dissolved amylose molecules reorient in a parallel alignment. The long-term retrogradation is represented by the slow re-crystallization of the outer branches of amylopectin. The re-crystallized amylopectin in the retrograded gel can be melted at 55°C, whereas for the re-crystallized amylose the melting temperature rises to 130°C . In addition to the origin of starch, retro gradation also depends on starch concentration, storage temperature, pH, temperature procedure and the composition of the starch paste. Retro gradation is generally stimulated by a high starch concentration, low storage temperature and pH values between 5 and 7. The salts of monovalent anions and cautions can retard starch retrogradation (**Swinkels 1985**).

3.1.1.1.3 Solubility

Starch is not soluble in cold water. But its suspension starts to become viscous by heating and turns to transparent paste. It means amylopectin forms crystallized micelle and amylose arranges orderly around the gaps of the micelles in starch particle. That is why starch is not soluble in cold water. However, water molecules are getting into micelles gradually when heating the solution and resulting to loosen hydrogen bond and short molecules of amylose starts to dissolve, then amylopectin swells up.

3.1.2 Low Density Polyethylene (LDPE)

Low Density Polyethylene (LDPE) granules were obtained from Ice Eterprizes Gaziabad. The melt flow and density of the LDPE granules as per the manufacturer is 10g/10min and density 0.9g/cm^3. LDPE is made from the monomer ethylene. It was the first grade of polyethylene, produced in 1933 by Imperial Chemical Industries (ICI) using a high pressure process via free radical polymerization.

LDPE is defined by a density range of 0.917–0.930 g/cm^3. It is not reactive at room temperatures, except by strong oxidizing agents, and some solvents cause swelling. It can withstand temperatures of 80 °C continuously and 90 °C (194 °F) for a short time. Made in translucent or opaque variations, it is quite flexible and tough.

LDPE has more branching (on about 2% of the carbon atoms) than HDPE, so its intermolecular forces (instantaneous-dipole induced-dipole attraction) are weaker, its tensile strength is lower, and its resilience is higher. Also, because its molecules are less tightly packed and less crystalline due to the side branches, its density is lower.

When exposed to ambient solar radiation the plastic produces two greenhouse gases, methane and ethylene. Due to its low density properties (branching) it breaks down more easily over time, leading to higher surface areas.

LDPE is most commonly used for the production of films. About 63% of total demand stem from the production of films, with packaging films accounting for the largest market share in this sector. Bags and sacks as well as other films rank second and third. Other important applications are rigid packaging and construction products. On a global level, the segment construction product offers the largest growth potential of the next few years, thanks to new investments in infrastructure projects. In Asia, on the other hand, it is especially films, LDPE packaging films in particular, that offer the largest growth potential, since Asian countries are increasingly adapting to Western standards for packaged food.

In a global comparison, film extrusion is the most common technology for processing LDPE. Other technologies, namely extrusion coating, injection molding, and other processes like blow molding and rotomolding, account for a market share of only 37%. The various world regions show only minor differences in regard to processing technologies. For example, extrusion coating is disproportionally often used in North America, while Eastern European countries use large amounts of LDPE not only in film extrusion, but also injection molding.

LDPE is widely used for manufacturing various containers, dispensing bottles, wash bottles, tubing, plastic bags for computer components, and various molded laboratory equipment. Its most common use is in plastic bags. Other products made from it include:

- Trays and general purpose containers

- Corrosion-resistant work surfaces
- Parts that need to be weldable and machinable
- Parts that require flexibility, for which it serves very well
- Very soft and pliable parts such as snap-on lids
- Six pack rings
- Juice and milk cartons are made of liquid packaging board, a laminate of paperboard and LDPE (as the waterproof inner and outer layer), and often with of a layer of aluminium foil (thus becoming aseptic packaging)
- Packaging for computer hardware, such as hard disk drives, screen cards, and optical disc drives
- Playground slides
- Plastic wraps

3.1.3 Glycerol

Glycerine was obtained from the local market at Jhusi, Prayagraj, for the study. Glycerol is a simple polyol compound. It is a colourless, odourless, viscous liquid and has a high boiling point and freezes to form a paste. Glycerol is often used as plasticizer in the film production, because of its hydrophilic properties, making it miscible with water. A plasticizer is an additive that softens the material it is added to. Elongation of films containing glycerol in higher concentration was about 8 times greater than that of un-plasticized samples. Glycerol as shown in Fig. 3.3 also reduces the interaction between protein molecules and increases the flexibility and extensibility of the final product. The interest in using glycerol is mainly due to the factors that glycerol is acting as plasticizer, reduces the intermolecular forces and increasing the mobility of the biopolymer chains. Besides, glycerol also reduces the extent of edge-edge interactions (i.e. H-bonding interactions), hence making it possible to achieve a better dispersion.

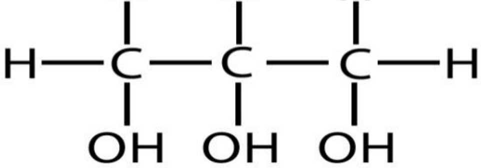

Fig. 3.3 Structure of Glycerol

3.1.4 Talc powder

Talc powder or talcum powder is a mineral that is naturally found in nature. It is the lubricant used in this work. It is used for easy flow of the mixture in the extruder. Talc powder was obtained from the local market at Jhusi, Prayagraj, for the study

3.1.5 Urea

Urea was used as crossing link agent, which is to make the starch and LDPE compatible. Urea was obtained from the local market at Jhusi, Prayagraj.

3.1.6 Glassware Used

3.1.6.1 Beaker

Borosil beaker was used for handling glycerol and were properly dried and cleaned.

3.1.6.2 Glass Rod

Glass rod was used for mixing the film forming mixture during the preparation stage.

3.1.6.3 Measuring Cylinders

Measuring cylinders of various capacities were used for measurement of liquid solvents**.**

3.2 METHODS

The flow chart for the preparation of bio-based plastic film is presented in Fig3.4

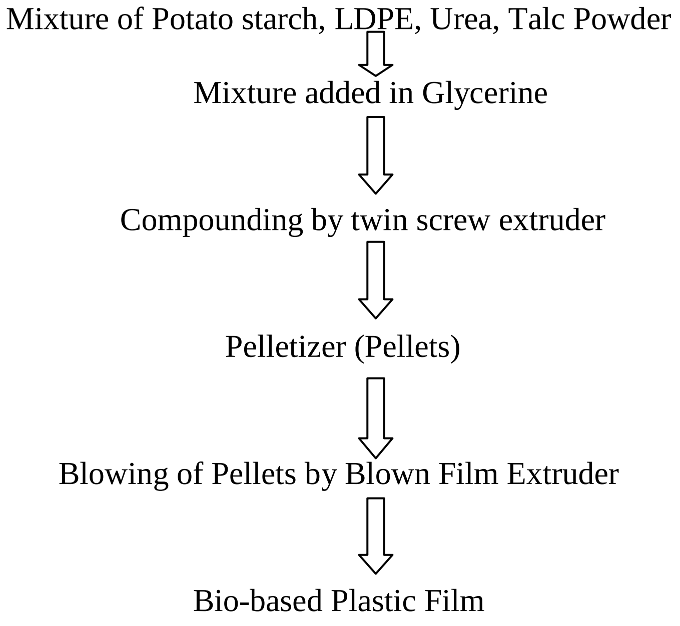

Fig. 3.4 Flow chart for film preparation

3.2.1 Film Preparation

The Potato starch was first dried in hot air oven for 1h. This was done in order to ensure that the starch contains not more than 10% moisture. Similarly LDPE was dried in hot air oven at 70^{o}C for 2h. A mixture of powdered Potato starch, talc powder, and urea was prepared, and added to the dried LDPE. The resulting mixture was added to glycerol and mixed properly. The mixture was passed through the hopper of twin-screw extruder. The biodegradable resin pellets was prepared from a twin-screw extruder and pelletizer. Thereafter, the biodegradable pellets (master batch) was then extruded with a Blown film extruder to produce a biodegradable film, which can either be used directly or modified/processed further for use. The production was carried out at Central Institute of Plastics Engineering and Technology (CIPET, LUCKNOW).

3.3 EXPERIMENTAL SET-UP

3.3.1 Hot air oven

Hot air oven as shown in Plate 3.1 **(Microtech Medicraft, Model: DAO-250 D)** is a double walled chamber of size 65×25×105 (in cm). Outer chamber is made of mild steel while the inner chamber is made of stainless steel. Hot air ovens are electrical devices used in sterilization. The oven uses dry heat to sterilize articles. Generally, they can be operated from 50 to 300 °C (122 to 572 °F). There is a thermostat controlling the temperature. These are digitally controlled to maintain the temperature. Their double walled insulation keeps the heat in and conserves energy, the inner layer being a poor conductor and outer layer being metallic. There is also an air filled space in between to aid insulation. An air circulating fan helps in uniform distribution of the heat. These are fitted with the adjustable wire mesh plated trays or aluminium trays and may have an on/off rocker switch, as well as indicators and controls for temperature and holding time. Temperature sensitive tapes or other devices like those using bacterial spores can be used to work as controls, to test for the efficiency of the device in every cycle.

Plate 3.1 Hot Air Oven

3.3.3 Twin Screw Extruder

Extruders are mechanical mixers consisting of long metallic screws used for processing solids and viscoelastic materials. Many different types of extruder can be used to produce plastics, the main two commercial types are single-screw extruders and twin-screws extruders and they differ in the transport mechanism. In most single-screw extruders the processed material rotates with the screw and is kneaded by contact with the barrel wall. The material characteristics and the channel wall affect the transport mechanism of this type of extruders. In contrast, the material cannot rotate with the screws in the twin-screws extruders as a result of the intermeshing occurred between the two screws. Therefore, twin-screws extruders are more suitable for TPS because of the water content and twin-screws extruder is less dependent on the material properties than a single-screw extruder.

Twin-screws extruder as shown in Plate 3.2-3.4 are counter rotating and they are good for the very elastic materials.

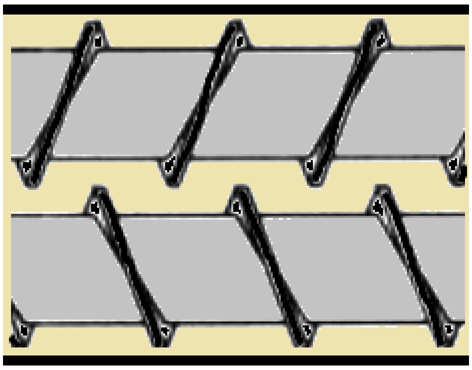

Plate 3.2 Non-intermeshing twin-screw extruder

Plate 3.4 shows a twin-screw extruder which has several temperatures zones that can be controlled. The temperature in an extruder plays a very important role in the physical and mechanical properties of the extruded material. The closest zone to feed hopper is usually cooler than the rest to start cooling the mixture after plasticisation. The gelation of starch occurs in the middle zones, where its molecular weight decreases and crystallinity changes depending on shear rate intensity, till it reaches the last zone which is completely filled, and then it comes out as TPS through a die. Thermoplastic starch rope needs to be cooled down before being cut into small chips.

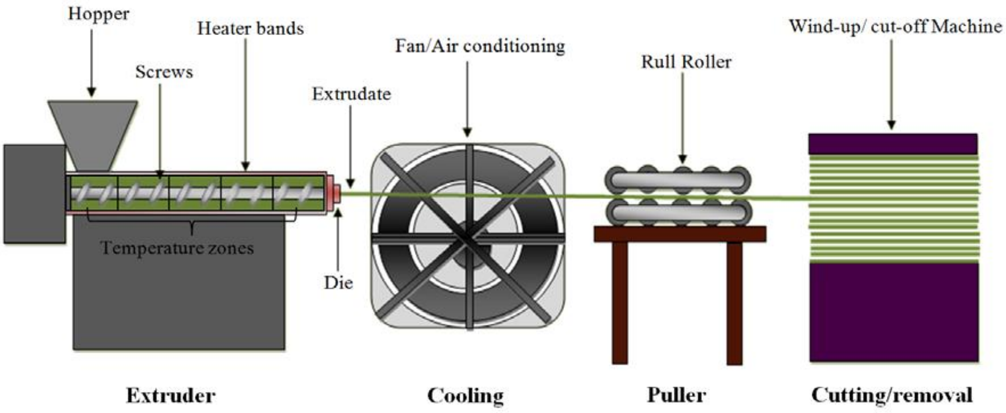

Plate 3.3 Twin Screw Extruder

In this research a five zones extruder was used to produce thermoplastic starch, the first zone, close to the feed, is to dry the mixture of starch and the appropriate plasticiser, before it starts to gel in the next zone. After starch gelation the extruder screws mix and knead the gel to be more homogeneous. The last zone is close to the die and is completely occupied with gelled starch leading to an increase in the pressure. Any gasses can escape through a venting hole. The thermoplastic starch comes out as a rope through two small dies. Plate 3.4 show the temperature zone during extrusion.

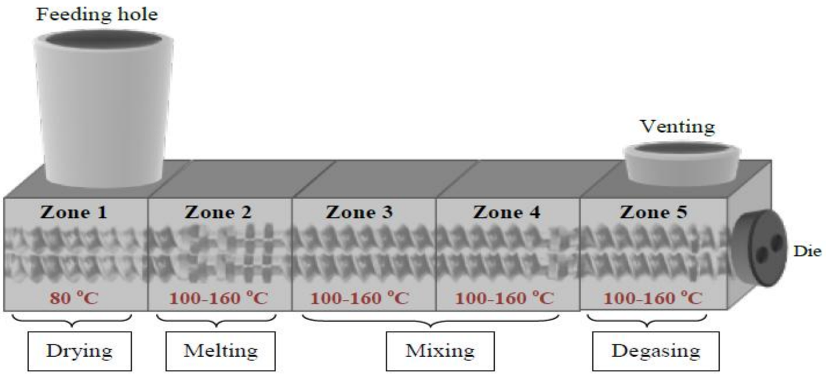

Plate 3.4 Extruder temperature zones diagram shows the process temperature during LDPE/STARCH extrusion.

3.3.4 Blown Film Extruder

Blown film extruder was used to convert the resin into an extruded film. In this, the polymer is melted by subjecting it into heat inside the barrel of an extruder and then forcing the molten polymer through a narrow slit in a die, vertically to form thin walled tube. The slit is in a circular form. Air is introduced in the centre of the die to blow up the tube like ballon, air ring blow on to the hot film to cool it (outside and inside the tube). The resulting thin film in the form of a tube, later often referred to as bubble. The tube passes through nip rolls where it is flattened. On winder the tube or film is wounded into roll. Plate 3.5 shows the complete process of blown film extruder.

3.3.4.1 Blown-Up-Ratio (BUR) and Polymer Orientation

To produce the best physical properties in an extruded film, the proper balance of film orientation in the machine and transverse direction of the film must be achieved. This relationship is achieved by adjusting the blow up ratio of the film. The blown up ratio is the ratio of the bubble diameter to the die diameter, it indicates the amount of stretching the polymer is undergoing during the shaping of the film. Plate 3.5 shows the Blown film extruder.

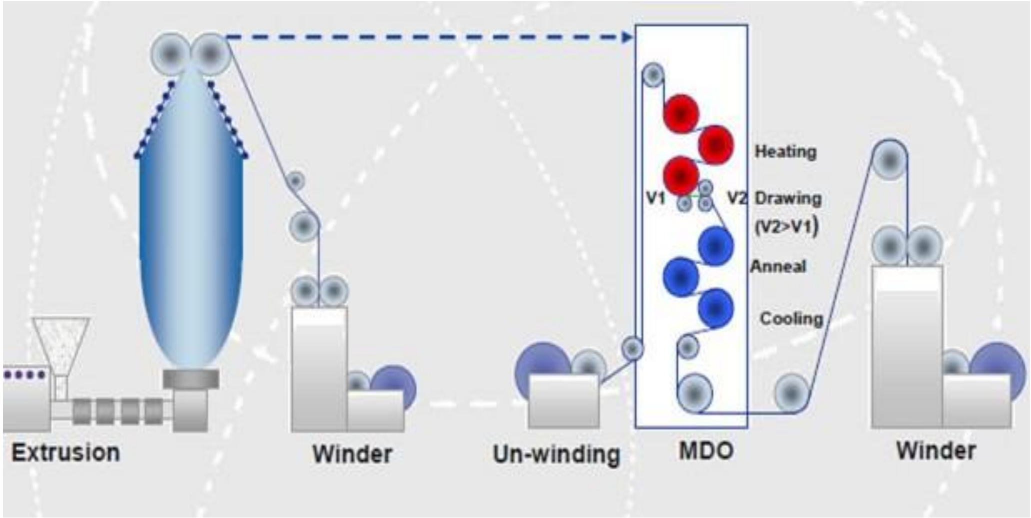

Plate 3.5 Blown film extruder

3.3.6 Micrometer

Screw gauge or micrometer as shown in plate 3.6 was used for the measurement of the film thickness. A micrometer sometimes known as a screw gauge is a device incorporating a calibrated screw widely used for precise measurement of components. The spindle is a very accurately machined screw and the object to be measured is placed between the spindle and the anvil. The spindle is moved by turning the ratchet knob or thimble until the object to be measured is lightly touched by both the spindle and the anvil. Micrometer use the principle of a screw to amplify small distances (that are too small to measure directly) into large rotations of the screw that are big enough to read from a scale. The accuracy of a micrometer derives from the accuracy of the thread-forms that are at its heart. In some cases it is a differential screw.

Plate 3.6 Micrometer

3.3.7 Dart Impact Tester

This impact resistance test simulates an object imparting a sudden force onto the film and determines the amount of energy each material can absorb. The specimen for the puncture resistance test was a disk with a diameter of 154.2 mm (6.0”). The test was performed on an Oakland Instruments Dart Drop Tester Series 8000 with a variable mass dart as seen Plate 3.7.

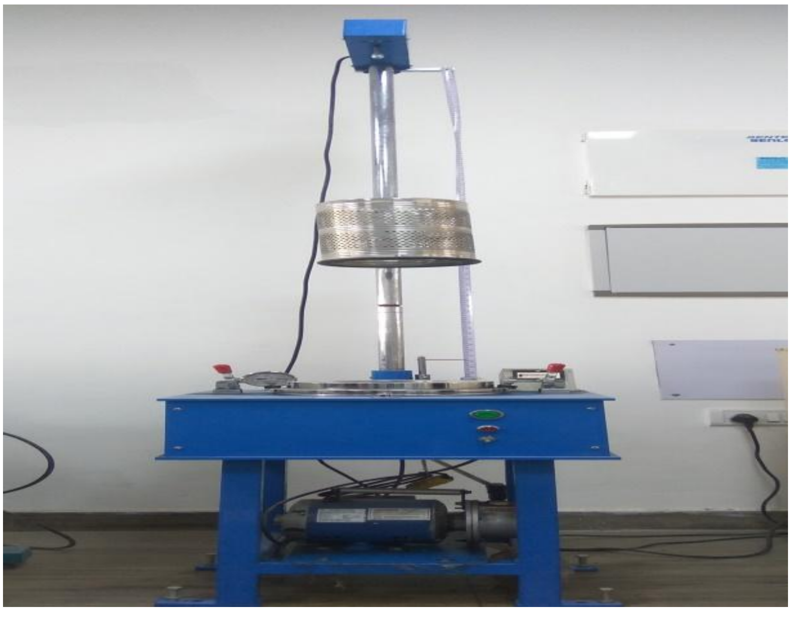

Plate 3.7 Dart Impact Tester

3.3.8 Universal Testing Machine

The texture profile analyzer (**Make: Stable Micro Systems, Model: TA TXT plus**) as shown in Plate 3.8, was used for the testing of Tensile Strength and Tear resistance. The tensile strength was performed using probe to ensure that the plastics films are appropriate for the further applications and for the food packaging also. These tests were conducted at the laboratory of CIPET Lucknow. The principle of a texture measurement system was to deform the sample in a controlled manner and measure its response. Imagine turning a laboratory balance upside down and pressing the balance pan down into the sample.

Plate 3.8 Universal testing machine

3.4 Quality Evaluation of Bio-based Film

3.4.1 Film Thickness

Pro-gauge Thickness Tester (micrometer) accurately measure the thickness of bio-based plastics film and other control sample to be compared. Each film was tested from 4 different points so that the mean thickness could found and the mean thickness was then calculated. The thickness of the film was an important parameter because the strength and other properties of a packaging film may vary with the thickness.

3.4.2 Tensile Strength

Tensile strength determines the amount of stress each material can sustain prior to failure as well as the amount of elongation at the time of failure. This test was carried out at CIPET. Each tensile test specimen follows the following guidelines:

1. The test specimen consists of strips of uniform width and thickness and at least 50 mm longer than the grip separation used.

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

Each specimen measured 100mm x 25mm at the gauge length and was cut from supplied films using hand shears. Universal testing machine (**Tensometer)** was used to test the tensile strength of the bio-based plastics film. Control sample will also be tested to compare with the bio-based plastics film.

3.4.3 Dart Impact Test

Each sample was placed in a circular grip and a dart with a hemispherical diameter of 50mm was dropped at a height of 27cm. The film was then inspected for integrity. In more detail, the condition of the film was determined using the following criteria: variable mass dart

- **Pass**: The dart did not create a hole in the film upon impact.
  Result : Increase weight of the dart by 5 g.
- **Failure**: The dart did create a hole in the film upon impact.
  Result: Decrease weight of the dart by 5 g.

Because the failure weight of the material is initially unknown, a set of pre-test drops was conducted (screening experiments) prior to testing. The screening test drops included a series of large weight step adjustments. In more detail, the highest dart weight is used (360 g of mass (3.5 N)). If the material failed, the mass of the dart was reduced by half. It is important to note that none of the materials tested withstood initial drop. This iterative approach of the testing continues in the same manner as described above with the weight of the dart changing in increments of half of the previous weight, until the incremental change is 5 g.

3.4.4 Test for Water Absorption

Water absorption was determined as per (**ASTM E96-00**) .The films were cut into a piece of 2.5×2.5 cm in size and weighed in air-dried conditions (W1). They were then immersed in distilled water (25°C) for 30 min. Wet samples were wiped with filter paper to remove excess liquid and weighted (W2). The film was kept in distilled water for 30 min, 60 min, 120 min and 240 min. The amount of absorbed water was calculated as

Water Absorption (%) = (W2-W1)/W1 ×100..............Eq.3.1

Where,

W2 and W1 represent the weights of the wet and air dried samples. The measurement was repeated three times for each type of film, and an average was taken as the result.

3.4.5 Test for Biodegradability

The test for Biodegradability was very important test because the problem of non-biodegradability of the conventional petroleum based plastics films was a major serious cause for the environmental pollution. So it was very crucial to obtain a fully biodegradable plastics film which can also be used for food packaging. For this purpose, the films of each samples were buried in the composted soil, the test was performed with some modifications as per the following cited paper **(Azahari *et al.* 2011).** Different small identical pots were filled with the composted soil and the samples of each film were cut into 2 cm × 2 cm pieces and buried in the soil at the depth of 5 cm. The pots were placed in laboratory for some days to study the time for biodegradation. The moisture of the soil was maintained by sprinkling water into it at regular intervals of time. The excess of water was drained out through a hole at the bottom of pots. The degradation of the samples were determined at regular intervals i.e. 5 days by carefully removing the samples from the pots and washing them to remove the soil from it and the samples were dried until the constant weight was obtained. The weight loss of the sample over the time was used to indicate the degradation rate of soil burial test. And the weight loss % was calculated according to the Eq. 3.2 to show the amount of biodegradation done.

Weight loss = (initial weight−final weight)/initial weight×100**…** Eq. 3.2

CHAPTER 4

RESULTS AND DISCUSSION

This chapter deals with the results of all tests performed on the work entitled "PREPARATION AND PROPERTIES EVALUATION OF PACKAGING FILM USING POTATO STARCH AND LDPE BLENDS". The various objectives of the study were to:

1. To develop potato starch incorporated LDPE packaging material at different composition.
2. To evaluate mechanical and biodegradable properties of the film
3. To compare the properties of LDPE and starch incorporated LDPE and its suitability for food packaging.

4.1 Development of Biodegradable packaging Film

The biodegradable film sample was prepared using LDPE different composition of potato starch and additives such as glycerol, urea and talc powder. The sample of ldpe is labelled as T0, sample with 5% potato starch labelled as T1, sample with 7.5% potato starch labelled as T2, sample with 10% potato starch labelled as T3. Table 4.1 shows the different composition of LDPE, potato starch and additives. The biodegradable film was produced as discussed in section 3.2. The film developed were shown in Fig 4.1

Table 4.1 Different composition of biodegradable film

S. No	TREATMENT	POTATO STARCH (%)	LDPE (%)
1	T_0	0	100
2	T1	5	95
3	T2	7.5	92.5
4	T3	10	90

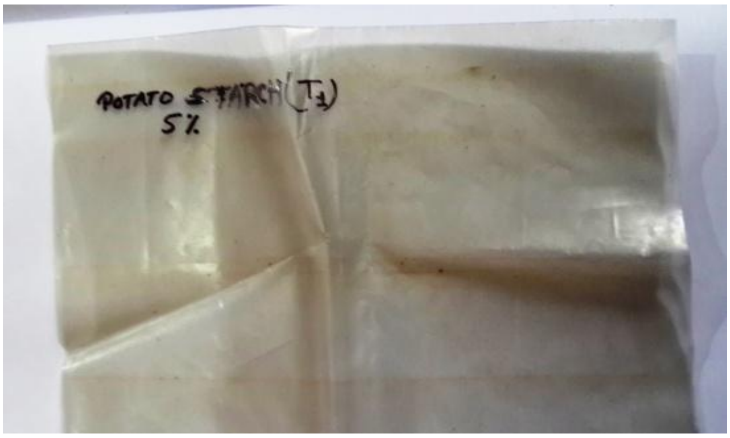

Fig 4.1 Biodegradable Film with 5% Potato Starch(T1)

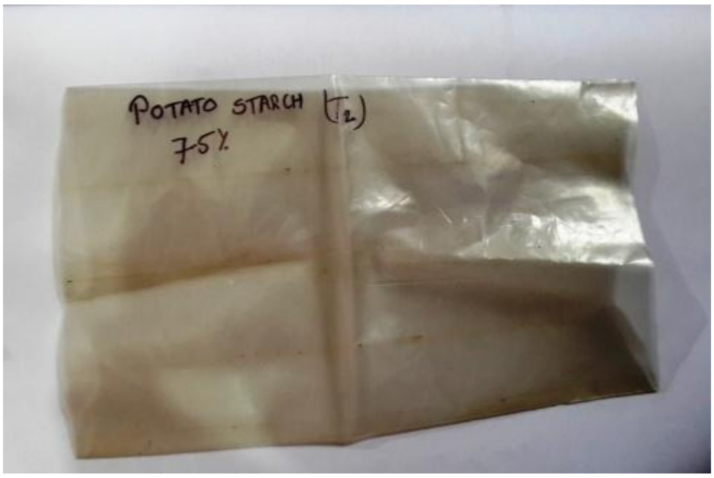

Fig 4.2 Biodegradable Film with 7.5% Potato Starch (T2)

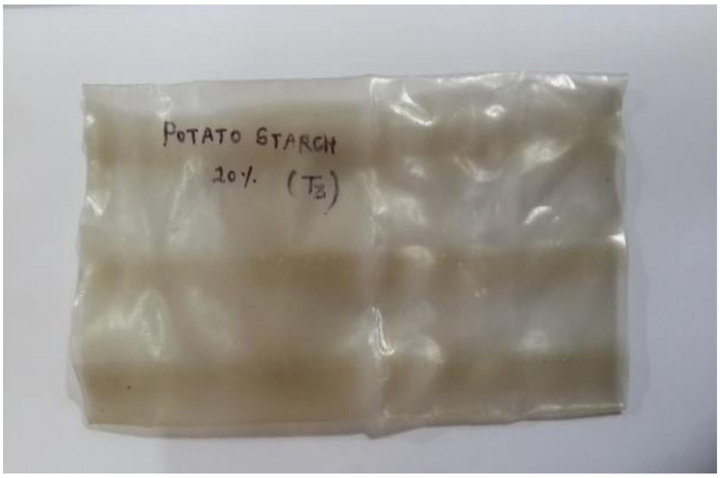

Fig 4.3 Biodegradable Film with 10% Potato Starch (T3)

4.2 Mechanical Properties of Bio-based Film

4.2.1 Film Thickness

Thickness is the physical property which is usually defined as the distance between the top and bottom. Thickness of packaging material should be same throughout as it affects the other properties of packaging materials. The film thickness was kept approximately constant and the thickness was measured at four different points of each sample. The thickness was then calculated using screw gauge and it was observed that the mean thickness was in the range of 0.66 mm to 0.95 mm. All the readings are shown in the Table 4.2 and are graphically plotted in Fig. 4.1. Approximate results were obtained in the studies conducted by **Oromiehie *et al.* (2013),** whose work was on physical and thermal mechanical properties of potato starch/LDPE composites.

From the ANOVA Table A.1, it is evident that the calculated value of F due to treatments is smaller than the tabulated value at 5% probability level. Therefore it can be concluded that significant effect of treatments on thickness of samples were observed.

Table 4.2 Thickness of bio-based film

	Thickness of film (mm)			
Treatment	**R_1**	**R_2**	**R_3**	**Mean**
T_0	0.95	0.95	0.95	0.95
T_1	0.65	0.68	0.65	0.66
T_2	0.95	0.98	0.95	0.96
T_3	1.25	1.25	1.1	1.2
S. Ed ±	**Result of F test**	**Critical Difference at 5%**		
0.049	**Treatments found Significant at 5% level of significance.**	0.099		

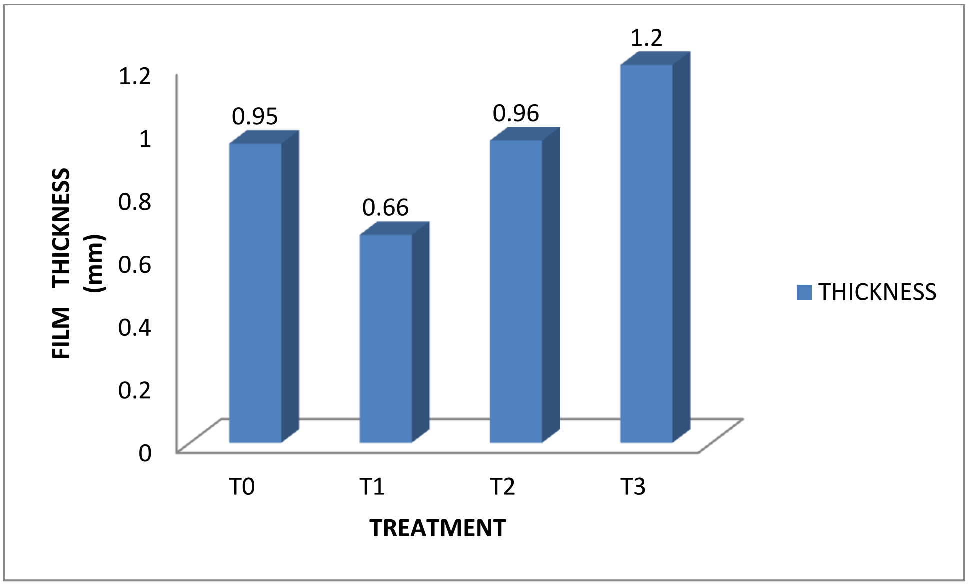

Fig 4.4 Thickness of film at different treatment

4.2.2 Tensile Strength

Tensile strength is the ability of a material to withstand a pulling (tensile) force. It is customarily measured in units of force per cross-sectional area. This is an important concept in food packaging. A higher tensile strength is generally preferred for food packets used for shipping and it helps to ensure better seal. To be plasticized with some plasticizers i.e. Glycerol was used in this study. The plasticized films were prepared and tested for tensile strength using texture profile analyser in which the exact tensile strength can be measured according to ASTM D882.

The tensile strength of films T1, T2 and T3 are depicted in Table 4.3. The results showed that the sample T2 was found to have the maximum strength i.e. 13.02 MPa force and the minimum strength was showed by the sample T3 i.e. 7.21 MPa force. These differences on the mechanical behaviour of the formulated films could be explained by the plasticizers (present in biodegradable potato starch film) interacting with film constituents causing changes in the properties of the continuous phases. Approximate results were obtained in the studies conducted by **Lu *et al.* (2009),** whose work was on starch-based completely biodegradable polymer materials, similarly **Wang *et al*. (2007)** who worked on film-forming potential and properties of protein and polysaccharide-based biopolymer films and **Ezeoha and Ezenwanne (2013)** whose work was based on cassava starch and PVA.

From the ANOVA Table 4.B, it is evident that the calculated value of F due to treatments is smaller than the tabulated value at 5% probability level. Therefore it can be concluded that significant effect of treatments on thickness of samples were observed.

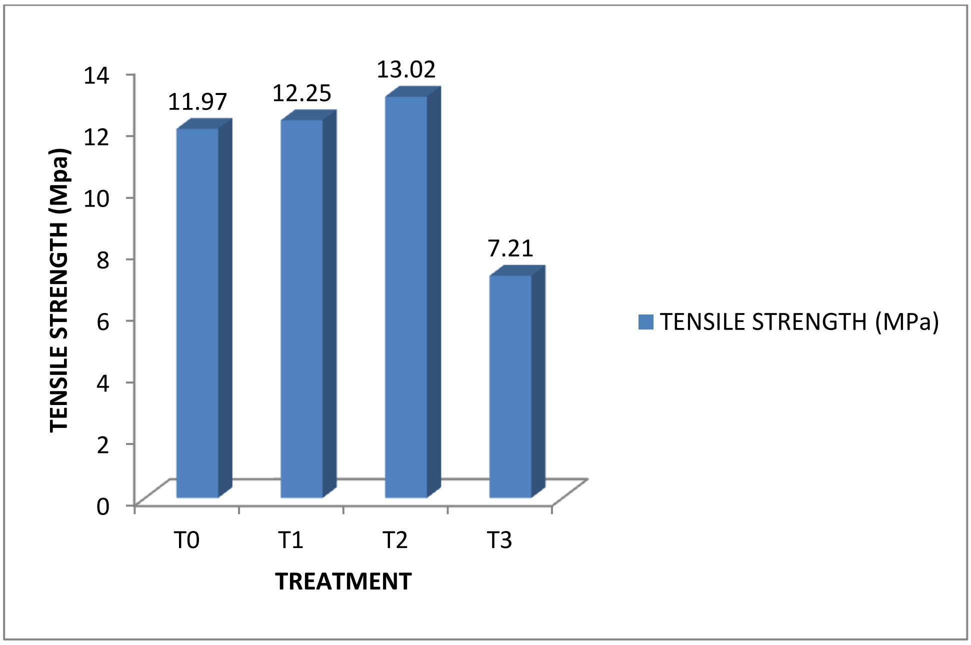

Fig 4.5 Tensile strength of film at different treatment

4.2.3 Elongation at break

The elongation at break of LDPE/Potato starch composite films is inversely proportional with starch content due to its embrittlement properties. The elongation at break decreases from 333.49% down to 176.34% with increase in starch content from 5 to 10%. This is due to (i) physical incorporation of starch in the matrix of LDPE that weakens the London forces between LDPE layers and (ii) the fact that starch, a low molecular weight polymer, has lower elongation compared to LDPE. Incorporation of starch causes discontinuity in the film matrix, leading to lower elongation due to lack of chemical interaction between starch and LDPE. Approximate results were obtained in the studies conducted by **Oromiehie *et al.* (2013),** whose work was on physical and thermal mechanical properties of Corn starch/LDPE composites. An indication of the elongation at break results achieved is depicted in Table 4.3.

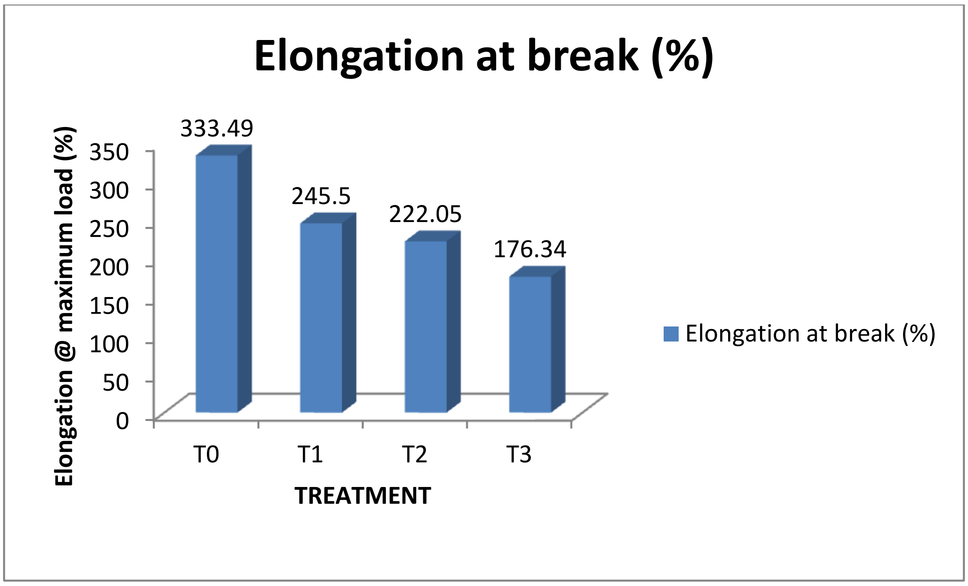

Fig. 4.6 Elongation at Break of Potato Starch incorporated LDPE Film

4.2.4 Dart Impact Test

The impact failure load obtained from a drop height of 66 cm for sample T0, T1, T2 and T3 was depicted in table 4.3. The dart impact resistance of T2 was found to be 1.02 N which is the best among the all treatment. The dart impact of biodegradable film depends upon the thickness of the film. Table 4.3 showed the dart impact resistance of different treatment of bio plastics. Similar results were reported by **Shah *et al.* (2019)**

Table 4.3 Mechanical Properties of various Composition of Biodegradable Film.

Treatment	Tensile strength (MPa)	Elongation at break (%)	Dart impact (N)
T0	11.97	333.49	1.2
T1	12.25	245.86	0.8
T2	13.02	222.5	1.02
T3	7.21	178.71	0.92

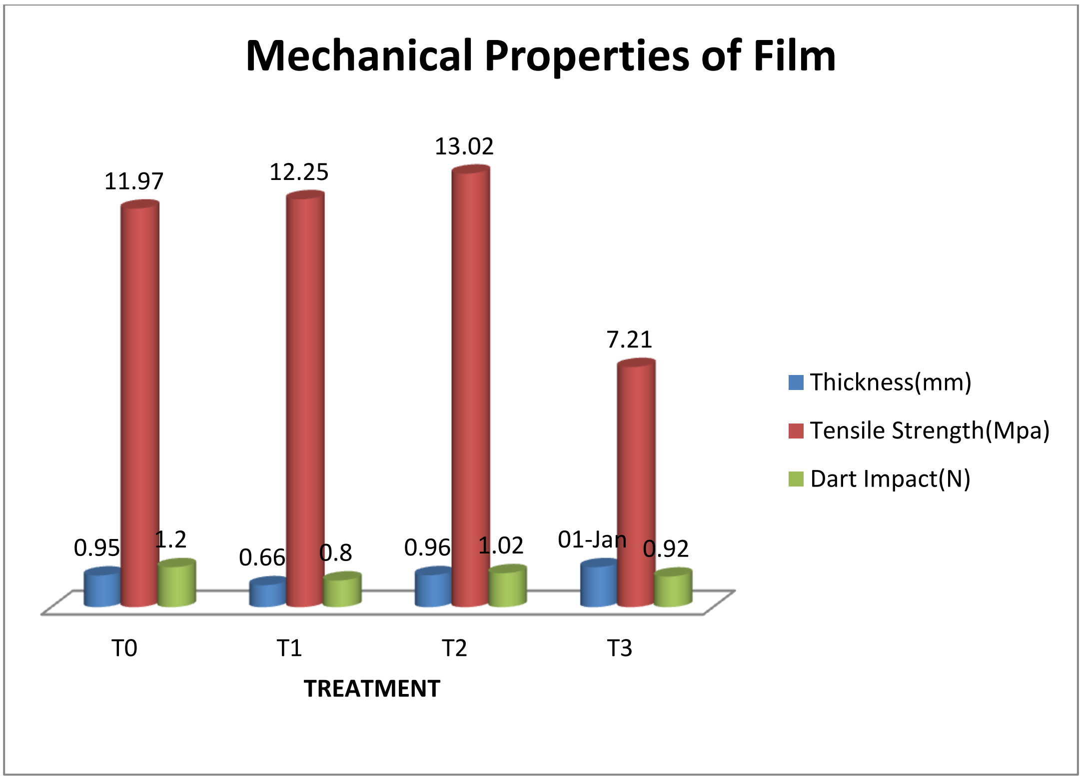

Fig. 4.7 Effect of different Treatment on Tensile Strength, Thickness and Dart Impact Test

4.2.5 Water Absorption

The water absorption of composite films is directly proportional to starch amount incorporated into polymer matrix. In other word, the water absorption would increase by increasing the starch content in LDPE. The starch is responsible for water absorption due to hydrophilic nature of starch and ionic character of hydroxyl groups of starch. Composites with more water absorption have lower mechanical properties. Regarding the starch content, the water absorption of pure PE, composites and pure starch is varied from 0.005 up to 0.081%. Table 4.4 showed reasonable water absorption in comparison with **Oromiehie *et al* (2013),** work.

From the ANOVA Table 4.C, it is evident that the calculated value of F due to treatments is smaller than the tabulated value at 5% probability level. Therefore it can be concluded that significant effect of treatments on thickness of samples were observed. Similar results were reported by **Shah *et al.* (2019)**

Table 4.4 Water Absorption of the Film

	Water Absorption of Film (%)			
Treatment	**R_1**	**R_2**	**R_3**	**Mean**
T_0	0.005	0.005	0.005	0.005
T_1	0.027	0.032	0.036	0.031
T_2	0.041	0.045	0.050	0.045
T_3	0.055	0.064	0.078	0.065
S. Ed ±	**Result of F test**		**Critical Difference at 5%**	
0.014	**Treatments found Significant at 5% level of significance.**		**0.056**	

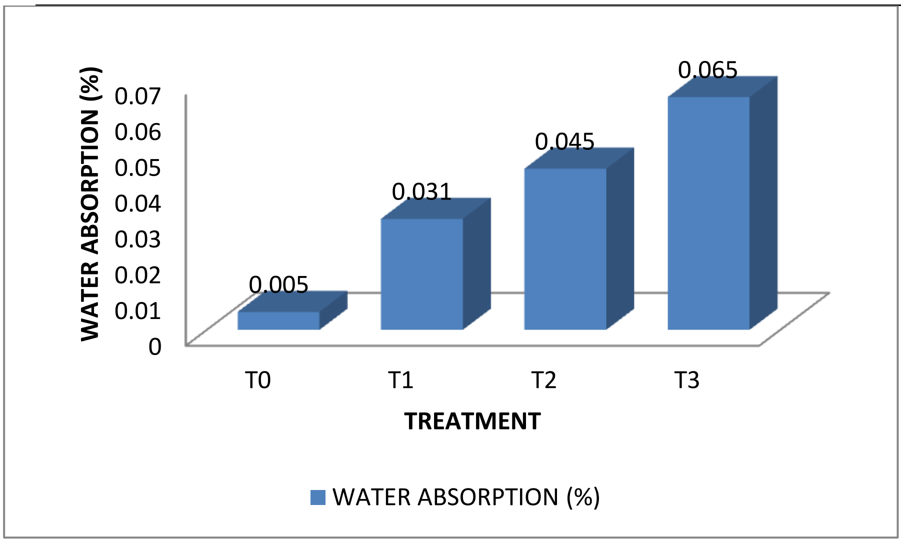

Fig 4.8 Effect of different treatment on Water Absorption of the Film

4.2.6 Soil burial biodegradation

Soil burial experiments represent a more significant approach to natural environments. In addition, using a natural complex media, with broad mixed microbial communities it should be expected a higher mineralization of polymeric materials. So samples was submitted to soil burial tests, in order to assess the biodegradability of LDPE/POTATO STARCH based blends films in an approximately natural environment. Forest soil used in biodegradation experiment was taken from agricultural field SHUATS Prayagraj has a total nitrogen content of 1.34g/kg and SO content of 2.95wt %. Before performing each characterization on LDPE/POTATO STARCH based blends films that had been incubated in soil, all samples were washed with distilled water until all visible residues were removed. Samples were then dried in a desiccator containing silica gel for at least 72 hours before analysis.

Biodegradability is property of material which ensures that it is completely compostable and does not cause any harm to the environment. Different sets of samples were kept in compost for 30 days. It was observed that all the biodegradable film with and without additives degraded within 10 days. There was no loss in weight of plastics kept in compost. 2g of each sample was taken and the weight loss percentage was studied. All the relevant readings of the biodegradation are shown in the Table 4.5. A study conducted by **Xiong *et al.* (2008)** on the structure and properties of a starch-based biodegradable film and **Azahari *et al*. (2011)** on biodegradation studies of polyvinyl alcohol/corn starch blend films in solid and solution media showed the similar results**.**

Table 4.5 Biodegradability of Film

Treatment	**10 days (%)**	**30 days (%)**	**60 days (%)**
T0	**0**	**0**	**0**
T1	**0**	**0**	**0**
T2	**0**	**2.5**	**7.5**
T3	**0**	**3.5**	**12.5**

CHAPTER 5

SUMMARY AND CONCLUSION

Synthetic packaging material has been very useful to us in the past decades but it has caused adverse health effects on the humans as well as the environment. Research shows that biodegradable packaging films have been conducted to serve consumer demands for safe, convenient and/or healthy food products with prolonged shelf life as well as sustainability awareness. For instance, biodegradable films have been improved to efficiently protect food products via their selective barrier properties. Thus search for a cheap and biodegradable alternative to the plastics as packaging material for food material has been necessitated. Varied biodegradable film had been identified to be synthesized from polysaccharides, proteins and lipids and sometimes combination of two or three components. Potato starch incorporated with LDPE and additives like glycerol, urea and talc powder were used for making film.

1. Potato starch/LDPE film was made with used of plasticizer glycerol, talc powder, and urea by blown film extruder.
2. All the prepared film were evaluated for thickness, tensile strength, dart impact test, elongation at break, water absorption and biodegradability of the film.
3. Thicknesses of films were in between 0.070 to 0.10 mm. Film labelled T3 was thickest with 0.10 mm of thickness.
4. Tensile strength of films were in between 14.08 to 7.76. Film labelled T1 has highest tensile strength and even better than LDPE. Elongation at break decreases with increasing starch content due to its embrittlement properties.
5. Water absorption of the biodegradable film increased with increasing the starch content.
6. Performance evaluation of biodegradable films was done by soil burial method for 30 days.

CONCLUSION

1. Potato starch incorporated LDPE bio-plastics film was prepared in three proportion T1, T2 and T3 using blown film extrusion method.

2. Mechanical properties of T2 sample was found reasonably better as compared with other sample.

3. Potato starch incorporated LDPE film can be used for food packaging application.

It could be concluded that the starch has direct effect on biodegradation because micro-organisms would attack to starch leading to biodegradability of the composite films

From this result it can be concluded that Potato starch can be a good polymer with LDPE and can be used as a packaging materials. It can be regarded as a boon for our society in controlling pollution.

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APPENDIX A

Statistical Analysis

Statistical analysis was conducted as per the data obtained from four treatments of and was analyzed statistically by Analysis of Variance technique- ANOVA one way classification. This technique was developed by Dr. R.A. Fisher in 1923. It gives an appropriate method capable of analyzing the variation of population variance. The significant effect of treatment was judged with the help of F'(variance ratio). Calculated F value was compared with the table value of F at 5% level of significance. If calculated value exceeded the table value the affect was considered to the significant. The significance of the study was tested at 5% level.

READINGS FOR ANALYSIS OF VARIANCE (ANOVA)

TABLE 4.A ANOVA FOR THICKNESS OF THE FILM

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	4	3.8	0.95	0.06
Column 2	4	3.86	0.965	0.0543
Column 3	4	3.65	0.9125	0.035625

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*	*Result*	*S.Ed*	*C.D.*
Between Groups	0.00585	2	0.002925	0.058529	0.943507	4.256495	S	0.049	0.099
Within Groups	0.449775	9	0.049975						
Total	0.455625	11							

TABLE 4.B ANOVA FOR TENSILE STRENGTH OF THE FILM

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	4	44.45	11.1125	6.965758
Column 2	4	44.45	11.1125	6.965758
Column 3	4	44.45	11.1125	6.965758

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*	*Result*	*S.Ed*	*C.D*
Between Groups	0	2	0	0	1	4.256495	S	6.96	13.93
Within Groups	62.69183	9	6.965758						
Total	62.69183	11							

TABLE 4.C ANOVA FOR WATER ABSORPTION OF THE FILM

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	4	0.129	0.03225	0.00048
Column 2	4	0.145	0.03625	0.000627
Column 3	4	0.167	0.04175	0.000931
				0.002038

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*	*RESULT*	*S.Ed*	*C.D.*
Between Groups	0.000182	2	9.1E-05	0.133949	0.876346	4.256495	S	0.00067	0.0013
Within Groups	0.006114	9	0.000679						
Total	0.006296	11							

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