

# PROJECT REPORT

On

**“SYNTHESIS AND CHARACTERIZATION OF  
BIODEGRADABLE PLASTIC FILMS FROM BANANA PEELS-  
A GREEN CHEMICAL APPROACH”**

Submitted by

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*In partial fulfillment for the award of the  
BSc Degree in Chemistry*



**POST GRADUATE AND RESEARCH  
DEPARTMENT OF CHEMISTRY**

**ST. TERESA'S COLLEGE (AUTONOMOUS)  
ERNAKULAM**

**2018-2019**

**POST GRADUATE AND RESEARCH  
DEPARTMENT OF CHEMISTRY  
ST. TERESA'S COLLEGE (AUTONOMOUS)  
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## **DECLARATION**

We hereby declare that the project work entitled **“SYNTHESIS AND CHARACTERIZATION OF BIODEGRADABLE PLASTIC FILMS FROM BANANA PEELS-A GREEN CHEMICAL APPROACH”**

submitted to Department of Chemistry, St. Teresa’s College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, is a record of an original work done by us under the guidance of Mrs. Safalya AS, Assistant Professor, Department of Chemistry, St. Teresa’s College (Autonomous), Ernakulam and this project work is submitted in the partial fulfillment of the requirements for the award of the degree of Bachelor of Science in Chemistry.

**Betsy PR  
Giya Johnson  
Lucy Grace VB**

## *Acknowledgements*

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We thank GOD Almighty for the blessings bestowed upon us that have helped us to complete the project successfully.

We thank Dr. Sajimol Augustine, our principal for providing various facilities for the training. We are sincerely grateful to Dr. Ushamani.M., our Head of Department for continuous support extended to us throughout the course of training.

We express our profound sense of gratitude to Mrs. Safalya AS, Assistant Professor, our teacher in charge and our project guide for her valuable guidance, innovative suggestions and encouragement throughout, this humble endeavor. We also thank her for the keen interest she had shown in our training.

We would also like to thank our co-guide Miss Georgina Grace, Assistant Professor, for her encouragement and moreover her timely support for the completion of our project work.

We would like to remember here, with a sense of gratefulness, the cooperation given to us by all other respected teachers.

We wish to record our gratefulness to our parents and lab assistants for their dedicating support everytime.

We also wish to acknowledge to the authors whose work we have consulted during the preparation of this project.

*Acknowledgements*

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Finally, we express our sincere thanks to our dear friends for their support and cheerful cooperation.

***Betsy PR, Giya Johnson, Lucy Grace VB***

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## **OBJECTIVES OF THE WORK**

1. Synthesis of biodegradable polymeric films from banana peels and to determine the impact of parameters such as ;
  - (a) Residence time
  - (b) Baking temperature
  - (c) pH
  - (d) Plasticizers
2. Characterization of biodegradable polymeric films using FTIR spectroscopy and Scanning Electron Microscopic (SEM) techniques.
3. Analysis of biodegradability of the plastic films by soil burial degradation test.
4. Analysis of tensile stress of the biodegradable polymeric films prepared using different sets of plasticizers.
5. Calculation of the Young's modulus and conversion percentage of the samples.
6. To determine the maximum tensile stress and Young's modulus of elasticity exhibited by the polymeric films.

# Chapter 1

## Introduction

### 1.1 PLASTICS

Plastics are derivatives of petroleum, natural gas or similar substances. Plastics offers a variety of benefits, due to its wide variety of physical and economical properties such as light weight, strong, visually aesthetic, flexible size and shape and cheaper price. Low-density polyethylene, high-density polyethylene and polyvinyl chloride are some of the most commonly available plastic polymers.

However, the increase in demand of plastics resulted in massive harmful effects. It takes longer time to degrade which is estimated about 500 years to degrade and will become toxic after decomposed [1]. Plastic pollution can unfavorably affect lands, waterways and oceans. Humans are also affected by plastic pollution, such as through the disruption of the thyroid hormone axis or hormone levels. Since plastics are so vital to our lives and so versatile in their usage; their use cannot be completely stopped. Hence alternative solution to this problem is being looked into .The most promising answers seem to be coming in the form of bioplastics.

### 1.2 BIOPLASTICS

Bioplastics can be defined as natural biopolymers made from the renewable biomass sources such as vegetable fats, oils, corn, straw, sugar cane, banana peels, food waste etc[2][3][4].They are fully or partially bio-based, biodegradable or compostable. Hence, these can be readily eliminated from our biosphere in an “eco-friendly” fashion.

Commonly used types of bioplastics are based on cellulose, starch, proteins and some aliphatic polyesters. Starch is one of the major sources in the development of bioplastic. Many previous studies have been

conducted by using starch as a natural biopolymer. Starch consists of a long chain of two glucose units joined together, namely branched polymerized amylopectin and linear chained amylose. Due to its large availability, low cost, renewability and biodegradability, starches are commonly used in the production of bioplastics. Starch can behave like a thermoplastic in the presence of plasticizer, with application of heat and mechanical treatment [5]. Cellulose bioplastics are mainly the cellulose esters, (including cellulose acetate and nitrocellulose) and their derivatives, including celluloid. Cellulose can become thermoplastic when extensively modified. For example cellulosic fibers added to starches can improve mechanical properties, permeability to gas and water resistance due to being less hydrophilic than starch [6].

Bioplastics that can be made from proteins from different sources are called protein based bioplastics. For example, wheat gluten and casein show promising properties as a raw material for different biodegradable polymers [7]. The aliphatic polyesters that can be used for the synthesis of bioplastics are polyhydroxyalkanoates (PHAs) like the poly-3-hydroxybutyrate (PHB), polyhydroxyvalerate (PHV) and polyhydroxyhexanoate (PHH).

### **1.2.1 APPLICATIONS**

Commercial applications of biodegradable plastics are packaging, bags and sacks, disposable housewares, agriculture and horticulture, medical devices, consumer electronics and electromotive. Biodegradable plastics like conventional plastics, offer a large range of packaging application. Some of the packaging options include bags for compost, agricultural foils, horticultures, nursery products, toys and textiles. Other areas include packaging for contact articles including disposable cutlery, drinking cups, salad cups, plates, overwrap, lamination film, straws, stirrers and containers. Bioplastic materials used in food packaging protect the food from the environment and maintain the food quality [8]. Biodegradable shopping bags made from bioplastics may degrade or decompose quicker than petroleum based synthetic plastics. Disposable housewares made from biodegradable plastics are now seen as marketable options, replacing traditional plastics such as poly-styrene and polyolefin. Housewares such

as kitchen tools and utensils, washable storage containers and cups, bathroom accessories, toys, hangers, and hooks are now being produced using biodegradable plastics. Horticulturalists now choose bioplastics to make agricultural mulches, seeding strips and tapes [9], [10], [11]. The tape biodegrades in the soil as the seeds germinate and take root. Mulch films are used to give the new seedlings a head start in the spring; helps to reduce evaporation and conserve moisture, increases soil temperature, and keeps control on the weeds. Compostable seed belts and active component encapsulations made out of bioplastics have also proven to be beneficial. Nontoxic biodegradable polymer sutures are being used each year by surgeons in life-saving heart operations and other procedures. Easily sterilized, the sutures remain strong and intact until the surrounding tissues have healed. The sutures dissolve and are readily metabolized in the body leaving no trace. Bioplastic products are now introduced in the fast moving consumer electronics sector such as touch screen computer casings, loud speakers, keyboard elements, mobile casings, vacuum cleaners, and a mouse for a laptop [12]. Bio-based plastics such as bio-based polyamides and bio-based polyesters are used by leading automotive brands around the world today with the aim of reducing their products environmental impact.

### **1.2.2 ADVANTAGES**

Traditional plastics are the petroleum based plastics which depend on fossil fuels which is an unsustainable source. Also acquiring fossil fuels does a lot of harm to the natural environment. Bioplastics on the other hand are made from bio mass like trees, vegetables, even waste which is completely bio degradable. Traditional plastics take thousands of years to degrade, these plastics lie in the environment, most notably on the ocean floor where they do the maximum damage for years. These plastics hamper the growth and kill the natural habitats. Bioplastics require considerably less time to biodegrade. This degradation can be carried out at home for some bioplastics and even for the bioplastics which require specific conditions, time required to degrade completely is considerably less. This reduces the huge pressure on our existing landfills. Some of the plastics degrade rapidly in the oceans releasing very harmful chemicals into the sea, thus harming the animals, plants and also harming the humans

by entering the food chain. Biodegradable plastics are completely safe and do not have any chemicals or toxins. This plastic harmlessly breaks down and gets absorbed into the earth.

### **1.2.3 ENVIRONMENTAL IMPACT**

Although bioplastics save more non-renewable energy than conventional plastics and emit less greenhouse gases compared to conventional plastics, bioplastics also have negative environmental impacts such as eutrophication and acidification [13]. Bioplastics induce higher eutrophication potentials than conventional plastics [13]. Biomass production during industrial farming practices causes nitrate and phosphate to filtrate into water bodies; this causes eutrophication which is the richness of the nutrients in body water [13]. Eutrophication is a threat to water resources around the world since it kills aquatic organisms, creates dead zones and causes harmful algal blooms [14]. Bioplastics also increase acidification [13]. The high increase in eutrophication and acidification caused by bioplastics is also caused by using chemical fertilizers in the cultivation of renewable raw materials to produce bioplastics [15].

### **1.2.4 GREEN CHEMICAL APPROACH FOR THE SYNTHESIS OF BIOPLASTICS**

Green chemistry, also called sustainable chemistry, is an area in chemistry and chemical engineering, focused on the designing of products and processes that minimize the use and generation of hazardous substances. Green chemistry focused on the environmental impact of chemistry, including technological approaches to preventing pollution and reducing consumption of non-renewable resources. The goals of green chemistry namely, more resource efficient and inherently safer design of molecules, materials, products, and processes can be pursued in a wide range of contexts.

The biological synthesis of plastics is a new concept. The biological synthesis of plastics can solve the environmental challenges like fossil fuel conservation, environmental protection, electronic, automobile and agricultural area. Biological synthesis of plastics is cost effective, eco-

friendly, non-toxic and consumes only low energy in compared to the conventional method which emits hazardous by-products which has some deleterious effect on the environment. Biological synthesis utilizes naturally occurring plant parts, polysaccharides and eco-friendly chemicals which are simple and viable which is the alternative to the complex and harmful chemical processes. When we make products like plastics from fossil fuels, we are contributing to the imbalance in the environment, while depleting valuable fossil resources, thereby increasing the carbon footprint of the product. Biological synthesis of plastics, on the other hand, can replace nearly 100% of the fossil fuel content found in conventional plastics, and require considerably less energy for production[16][17]. Biosynthesis of plastics are completely safe and do not include any release of chemicals or toxins. This plastic harmlessly breaks down and gets absorbed into the earth. Such advantages of bioplastics are of extreme importance, as the toxic plastic load on the earth is growing and at this rate will cause a whole range of problems for future generations.

Plastics are inseparable part of our daily life. In recent years, there has been a remarkable increase in interest in biodegradable plastics of scientists for use in medicinal sciences, agricultural sciences, biological sciences and other various areas in whole world. The diminishing supply of petroleum along with many environmental problems created by the conventional petroleum based plastics may be one of its motive drives. Bioplastic is one of the best replacements over conventional plastic. The main advantage of bioplastics over conventional plastic is that they degrade into environment without creating any pollution. Bioplastics are used in a wide variety of fields such as in food packaging, automobiles, constructions and in the casing of modern electronic devices. Due to its large availability, low cost, renewability and biodegradability, starches are commonly used in the production of bioplastics. One of the most common waste forms of starch is the banana peels. Therefore, banana peels can be suggested as a suitable source for the manufacturing of bioplastics. By using banana peels as a raw material we can produce good quality bioplastics which has a good shelf life.

### 1.3 BANANA PLANT

#### *Musa acuminata* (Banana plant)

Kingdom	: Plantae
Division	: Angiosperm
Class	: Monocot
Order	: Zingiberales
Family	: Musaceae
Genus	: Musa
Species	: acuminata



**Fig 1: Banana plant**

Banana peels are collected from bananas, the parthenocarpic fruits of banana plants. Banana peels are extremely rich sources of starch [18]. Green banana peels possess 3% starch, which reduces to 1% - 2% in intermediate ripe banana peels [19]. However, if the peels are too ripe, the starch will be converted into glucose while the least ripened peels, film becomes too firm although high in starch molecules [20]. Therefore, moderately ripened banana peels were used in the production of bioplastic as an alternative to petroleum based plastics for insulation and cosmetic applications [21].

### 1.4 POLYMERIZATION

Polymerization is a process of reacting monomer molecules together in a chemical reaction to form polymer chains or three dimensional networks [22][23][24]. The polymers are classified as homopolymers and copolymers [24]. Homo polymers consist of repeated chains of structures of same monomer units. A copolymer is a polymer derived from more than one species of monomers [25]. Different types of copolymers are:-

- Block copolymers comprises two or more homopolymers subunits linked by covalent bonds.

- An alternating copolymer has regular alternating A and B units, and is often described by the formula  $-(A-B)_n-$
- Random copolymers consists of monomers randomly linked in a given polymer chain.
- Graft copolymers consist of a main polymer chain or backbone, covalently bonded to one or more side chains. They are branched copolymers where the components of the side chain are structurally different than that of the main chain. Graft copolymers containing a larger quantity of side chains are capable of wormlike conformation, compact molecular dimension, and notable chain end effects due to their confined and tight fit structures [26]. They can be used for the manufacture of materials that are impact resistant, and are often used as thermoplastics, elastomers, compatibilizers or emulsifiers for the preparation of stable blends or alloys [27].

#### **1.4.1 POLYMERIC CONTENTS IN BANANA PEELS**

##### **1.4.1.1 STARCH**

Starch is a polysaccharide comprising glucose monomers joined in  $\alpha$  - 1, 4 positions by glycosidic bonds. This polysaccharide is produced by most green plants as energy storage. The basic chemical formula of the starch molecule is  $(C_6H_{10}O_5)_n$ . Starch consists of two different types of polymers of D-glycopyranose type namely amylose and amylopectin, made up of adjoined glucose molecules. Amylose is a linear polymer of glucopyranosyl and amylopectin is a branched chain polymer of glucopyranosyl.

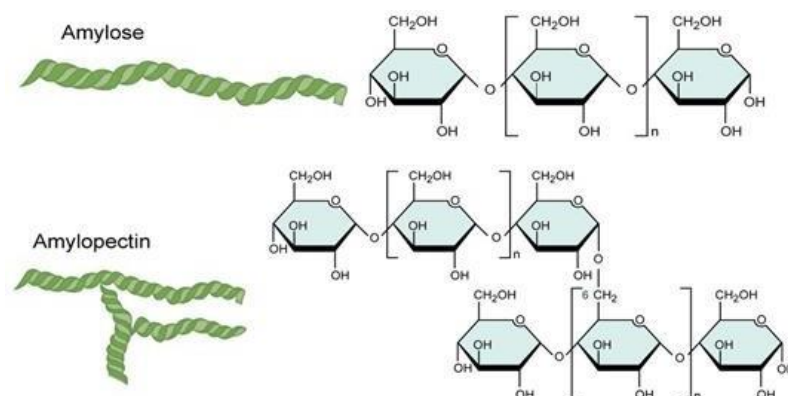
##### **1.4.1.2 AMYLOSE**

IUPAC name is (1, 4) -  $\alpha$  - D- Glucopyran. The linear polymer, amylose is the simplest unbranched form of starch. It is a polysaccharide made of roughly 500 to 20,000  $\alpha$  (1, 4) D-glucose units, bonded to each other through  $\alpha$  glycosidic bonds. It comprises approximately 20-30% of starch. Amylose is less soluble in water than the other component of starch, amylopectin.



### 1.4.1.3 AMYLOPECTIN

Amylopectin is a water-soluble polysaccharide and highly branched polymer of  $\alpha$ -glucose units found in plants. In amylopectin, glucose units are linked in a linear way with  $\alpha$  (1, 4) glycosidic bonds and branching takes place with  $\alpha$  (1, 6) bonds occurring every 24 to 30 glucose units. It comprises 80 % of starch. Amylopectin has relatively huge molecular mass but it consists of compact molecules.



*Fig 2: Amylose and amylopectin*

## 1.5 HYDROLYSIS OF STARCH (ADDITION OF HCl)

The hydrochloric acid is used in the hydrolysis of amylopectin, which is needed in order to aid the process of film formation, since the amylopectin restricts the film formation. When starch reacts with HCl, one of the polymeric components amylopectin breaks down into smaller straight-chain pieces of amylose, which results in the formation of a better polymeric film. [28] The mechanism behind the working of HCL on starch is called acid hydrolysis. The  $H^+$  of HCL attacked by lone pair of electrons present on oxygen in C-O-C of glycosidic linkage at  $\alpha$  (1,6) position of amylopectin, results in +ve charge on oxygen and makes it unstable, to get stability it pulls the electron from carbon atom attached to it and creates a carbocation. This carbocation formed is further reacts with  $H_2O$  and transfer the +ve charge to the oxygen of  $H_2O$ . This +ve oxygen takes an electron from hydrogen and release a free proton as catalyst again. This is commonly called nucleophilic substitution reaction. This

whole process breaks the glycosidic linkage at  $\alpha$  (1, 6) position of amylopectin and in turn smaller-chain pieces of amylose is formed.

### **1.5.1 HYDROLYSIS TIME**

During the initial stage of hydrolysis, due to the hydrolysis of branched chains of amylopectin, amylose content increases. However, if this hydrolysis time is increased further, there occurs a significant drop in the amylose and amylopectin content of starch. This is because once the amylopectin hydrolysed to amylose, further hydrolysis leads to the formation of glucose monomers which do not aid in polymer formation [29]. Hence an optimum residence time is maintained throughout the experiment.

### **1.6 ADDITION OF NaOH**

The sodium hydroxide is simply used to neutralize the pH of the medium, hence to stop the reaction.

### **1.7 PLASTICIZERS**

Although the straight chains of the amylose can line up together to make a good film, it is brittle. To improve the plastic properties, substances called plasticizers are used. Plasticizers or dispersants are additives that increase the plasticity or fluidity of a material. They are generally non-volatile organic liquids such as polyols like sorbitol, glycerol and polyethylene glycol (PEG). Plasticizer acts as a biocompatibilizer and an internal lubricant for the polymer. Plasticizers are added to starch to prevent crystalline areas from forming. Hence, starch can exhibit more 'plastic' properties and can improve its flexibility [30]. However, the interactions between plasticizer and starch chains are difficult to explain. It is generally accepted that plasticizers lower the number of physical cross- links between starch chains and makes a better polymeric film. Efficient plasticizers generally have low molar mass, high boiling point and exhibit low viscosities so that they can act as good copolymers and can easily blend with the starch.

As a result of graft copolymerization or blending of the plasticizers with the starch, plasticizers gets resides in the interstices within the amylose

molecules and replaces the intra and intermolecular hydrogen bonds [31]. Consequently, plasticizers alter the interface between the adjacent molecules in the polymer chains of starch leading to increase in extensibility of the polymer matrix and hence better polymeric film will be produced [32].

### **1.7.1 GLYCEROL**

Glycerol, also called glycerine is a simple polyols compound. It is a colourless, odourless, viscous liquid that is sweet tasting and non-toxic. It is widely used in the food industry as a sweetener, humectant, and as a common plasticizer. It has three hydroxyl groups that are responsible for its solubility in water and its hygroscopic nature [33]. Glycerine produces stronger hydrogen bonds with starch, increases the strength and toughness of the bioplastics. Furthermore, glycerol is a by-product generated in large amounts in the bio-fuel industry and is becoming nowadays a waste product.

### **1.7.2 POLYETHYLENE GLYCOL (PEG)**

It is a polyether compound which is also known as polyethylene oxide (PEO). The structure of PEG is commonly expressed as  $H-(O-CH_2-CH_2)_n-OH$ . It is a hydrophilic molecule. It is highly flexible and soluble in water. It is a good plasticizer and has many applications from industrial manufacturing to medicine.

### **1.7.3 POLYVINYL ALCOHOL (PVA)**

It is a water soluble synthetic polymer. It is a colourless and odourless substance. PVA is used as a polymerization aid, as protective colloid and in the manufacture of polyvinyl acetate dispersions. It is used as a plasticizer to improve plasticity of materials.

## **1.8 SODIUM METABISULPHITE AS AN ANTIOXIDANT**

Sodium metabisulphite or sodium pyrosulphite is an organic compound of chemical formula  $Na_2S_2O_5$ . It is used as disinfectant, antioxidant and preservative agent. It is very soluble in ethanol and water. It is an antimicrobial agent that destroys microorganisms that lives on the surface

of biodegradable objects .It is resistant to the *Rhizopus* mould and also effective against gram positive bacteria such as *Staphylococcus* and *Clostridium* species. Also, it completely inhibits the mycelial growth of the fungus *Oxysporum* species. As the plastic film started to decay within three days of production, in order to overcome this problem, Sodium metabisulphite also known as E223 was used. It prevents the microbial growth on plastic film. It prevents the microbial attack, and hence it succeeded in improving the shelf-life of the plastic.

## **1.9 CHARACTERIZATION TECHNIQUES**

Characterization, when used in materials science, refers to the use of external techniques to probe into the internal structure and properties of a material. Characterization can take the form of actual materials testing or analysis. Analysis techniques are used to simplify or to magnify the specimen, to visualize its internal structure, and to gain knowledge as to the distribution of elements within the specimen and their interactions.

### **1.9.1 MECHANICAL CHARACTERIZATION**

This characterization technique is mainly used to reveal information about a material's mechanical properties under dynamic or static forces. It is a fundamental technique used in science and engineering fields. The specimen is subjected to a controlled tension until failure. Properties that are directly measured using this technique are ultimate tensile stress, breaking strength, maximum elongation and reduction in the surface area of the specimen. From these measurements the following properties can also be determined: Young's modulus of elasticity, Poissons ratio, yield strength and strain.

The restoring force developed per unit area of crosssection of a specimen is called stress. When the forces acting on the body produces an elongation along its length, then we call the stress as tensile stress. S.I. unit is N/m<sup>2</sup> or Pascal and its dimensional formula is [ML<sup>-1</sup>T<sup>-2</sup>]. The tensile stress produces a change in the length of the specimen, which is referred to as longitudinal strain. The tensile strength of a material is the maximum amount of stress that it can be subjected to before failure. Tensile strength measures the force required to pull something such as rope wire or a structural beam to the point where it breaks.

## **INSTRUMENTATION**

Thickness, Gauge length, Maximum force, Tensile Stress and percentage elongation all these parameters of the samples were determined using a Universal Testing Machine (UTM). The plastic films were cut according to the bounds recommended by the standard using a gelatin metal sheet cutter. The specimens were placed in the grips of a universal testing machine by aligning the long axis of the grips with an imaginary line joining the points of attachment to the machine. The tensile stress, percentage elongation at the yield and rupture points experienced by the samples were recorded by the computerized data recording system.

### **1.9.2 FTIR SPECTROSCOPY**

Fourier-transform infrared spectroscopy (FTIR) is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or a gas. FTIR spectrometer simultaneously collects high spectral resolution data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. The term Fourier transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical process) is required to convert the raw data into the actual spectrum.

FTIR spectrometer obtains infrared spectra by first collecting an interferogram of a sample using an interferometer. It then digitizes the interferogram, then performing the Fourier Transform (FT) function on it and then displays the spectrum. FTIR spectrum is used in identification of organic molecular groups and compounds, side chains and cross links involved, since all of which have vibrational frequencies in the infra-red range. It is used in the analysis of adhesives, coatings and adhesion promoters or coupling agents. Also, it is used a tool in isolating and characterizing organic contamination.

## **INSTRUMENTATION**

The instrument used for the FTIR analysis –Perkin Elmer FTIR spectrometer. It is ideally suited to everyday analysis, using it we can confidentially perform fast and accurate IR analysis. This analytical

method assures determination of the quality of our materials across a wide range of applications.

### **1.9.3 SCANNING ELECTRON MICROSCOPY (SEM)**

The scanning electron microscope (SEM) is a type of electron microscope capable of producing high-resolution images of a sample surface. SEM images have a characteristic three dimensional appearance and are useful for observing the surface structure of the sample. Scanning electron microscope consists of an electron gun and electromagnetic lens system to study the surface structure and morphology of solids. In this technique, a well-defined electron beam imposes on the specimen and leads to generation of secondary electrons ,back scattered electrons ,absorbed electrons ,characteristic X-rays etc .These electrons can be detected by suitable detectors and give information about the surface structure and morphology of the specimens. The characteristic X-rays generated are used for the identification and estimation of different elements present in the specimen by using energy dispersive spectrometer (EDS) and wavelength dispersive spectrometer (WDS).The depth of the focus is much larger than optical microscope image even at lowest magnification is one of the major advantages of SEM.

### **INSTRUMENTATION**

The instrument used for SEM analysis- High resolution Scanning Electron Microscope The high resolution three - dimensional images produced by Scanning Electron Microscopes provide topographical, morphological and compositional information makes them invaluable in a variety of science and industrial applications.

# Chapter 2

## Materials and Methods

### 2.1 MATERIALS REQUIRED

#### 2.1.1 MATERIALS USED

- Banana peels
- 500 ml beaker
- A blender
- A lab oven
- Filter paper
- Ceramic tile

#### 2.1.2 CHEMICALS REQUIRED

All the reagents used were of A.R grade.

- 0.5 N Hydrochloric acid (HCl)
- 0.5 N Sodium hydroxide solution
- 0.2 M Sodium Metabisulphite solution
- Glycerol
- Polyethylene Glycol (PEG)
- Polyvinyl Alcohol (PVA) solution

### 2.1.3 INSTRUMENTS USED

- Scanning electron microscope (SEM )
- Fourier-transform infrared spectrometer (FTIR)
- Universal testing machine (UTM)

## 2.2 BANANA PEELS SAMPLES

Banana peels were collected from bananas, the parthenocarpic fruits of *Musa acuminata* (Banana plant).

## 2.3 METHODS

### 2.3.1 PREPARATION OF BANANA PEELS PASTE

- The peels of the banana were removed using a stainless steel knife and cut into small pieces.
- Banana peels were dipped in 0.2 M sodium metabisulphite ( $\text{Na}_2\text{S}_2\text{O}_5$ ) solution for 45 minutes.
- A 500 ml beaker was filled with water and the banana peels were boiled for about 30 minutes.
- After boiling, the water was decanted from the beaker and the peels were left to dry on a filter paper for about 30 minutes.
- After the peels were dried, they were squashed using a blender until a uniform paste was formed.

### 2.3.2 SYNTHESIS OF BIODEGRADABLE PLASTIC

- 75gm of banana paste was weighed.
- 0.5 N HCl was added to this mixture according to the desired condition and stirred using a glass rod.
- 6ml Plasticizer was added and stirred.



- 0.5 N NaOH was added according to the desired pH, after a residence time of 15 minutes and the mixture was stirred again.
- The mixture was poured on a ceramic tile and baked at 90°C till it dry.
- The tile was allowed to cool down and the film was scraped off from the surface.

## **2.4 BIODEGRADABILITY TEST (SOIL BURIAL METHOD)**

Plastic film prepared from banana peels and polythene films were cut into 12cm x 12cm. Then, these films were buried in 10cm depth in two separate pits. At regular intervals, water was sprinkled on it so that bacterial enzymatic activities could be enriched. After 2 days, the specimens was taken from the soil and washed with distilled water, dried and the weight was taken and continued for 6 days.

## **2.5 CHARACTERIZATION TECHNIQUES**

### **2.5.1 MECHANICAL CHARACTERIZATION**

Mechanical properties were determined using Universal Testing Machine (UTM).

#### **2.5.1.1 TENSILE STRESS**

Stress is the restoring force developed per unit area of crosssection. When the forces acting on the body produces an elongation along its length, and then we call the stress as tensile stress. S.I. Unit is N/m<sup>2</sup> or Pascal (Pa) and its dimensional formula is [ML<sup>-1</sup>T<sup>-2</sup>].

$$\text{Tensile stress} = \frac{\text{Force}}{\text{Cross sectional area of the samples}}$$

#### **2.5.1.2 STRAIN**

The effect of stress on a body is called strain. Strain is measured as the ratio of the change in dimension produced to the original

dimension. The tensile stress produces a change in the length  $\delta L$ . Hence the longitudinal strain can be written as

$$\text{Longitudinal strain} = \frac{\text{Change in length}}{\text{Original length}}$$

### 2.5.1.3 YOUNG'S MODULUS OF ELASTICITY

Young's modulus of elasticity is defined as the ratio of tensile stress to the longitudinal strain.

$$\text{Young's modulus of elasticity} = \frac{\text{Tensile stress}}{\text{Longitudinal strain}}$$

### 2.5.2 FTIR ANALYSIS

The instrument used for the FTIR analysis –Perkin Elmer FTIR spectrometer. It is ideally suited to everyday analysis, using it we can confidentially perform fast and accurate IR analysis.

### 2.5.3 SEM ANALYSIS

The instrument used for SEM analysis- High resolution Scanning Electron Microscope

## 2.6 CONVERSION PERCENTAGE OF THE SAMPLES

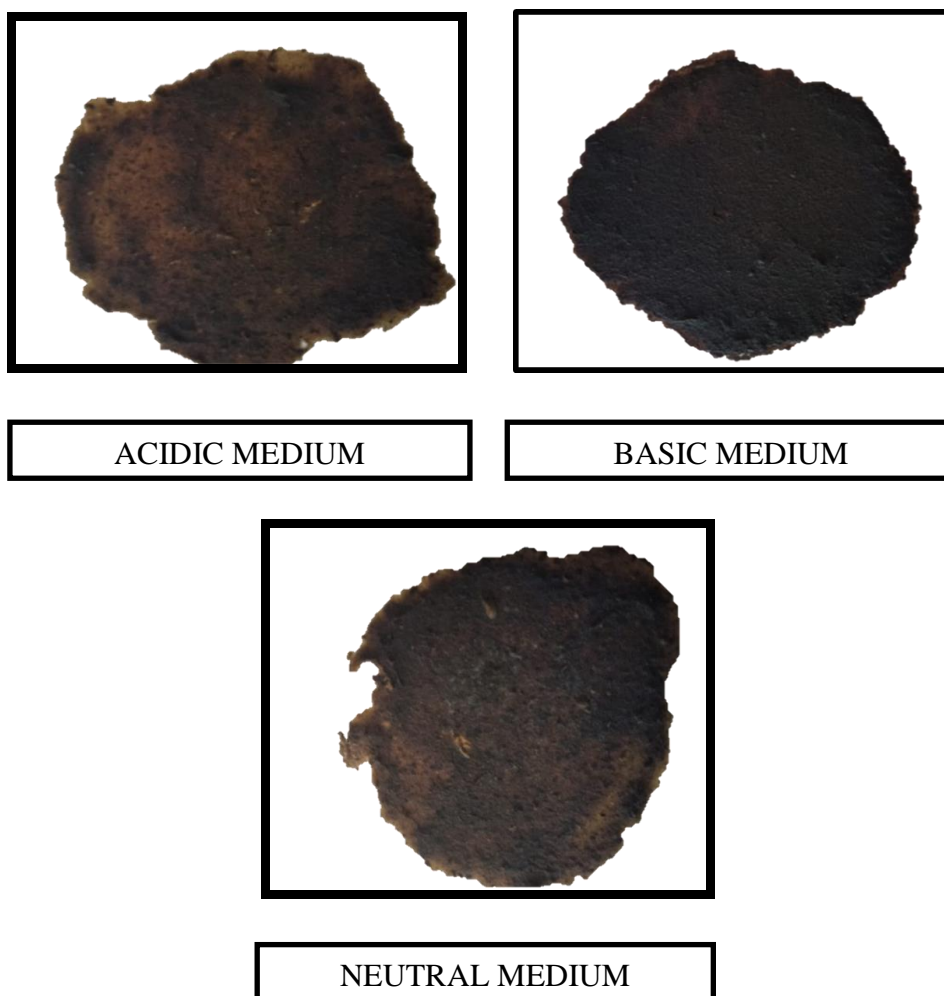
It is a relation between the net mass of polymer obtained per unit mass of the reaction mixture fed into the oven.

$$\text{Conversion \%} = \frac{\text{Weight of final paste put for baking}}{\text{weight of the film after baking}} \times 100$$

# Chapter 3

## Results and discussion

### 3.1 VISIBLE OBSERVATIONS



*Fig 3: Biodegradable polymeric films prepared at different pH conditions using glycerol as the plasticizer*

### 3.2 BIODEGRADABILITY TEST (SOIL BURIAL METHOD)

Number of days	Weight of the plastic film prepared from banana peels (grams)	Weight of polythene film (grams)
Initial day	7.5	0.9913
2 days	6.82	0.9913
4 days	5.63	0.9913
6 days	4.38	0.9913

Table 1: Analysis of biodegradability of the plastic film and polythene

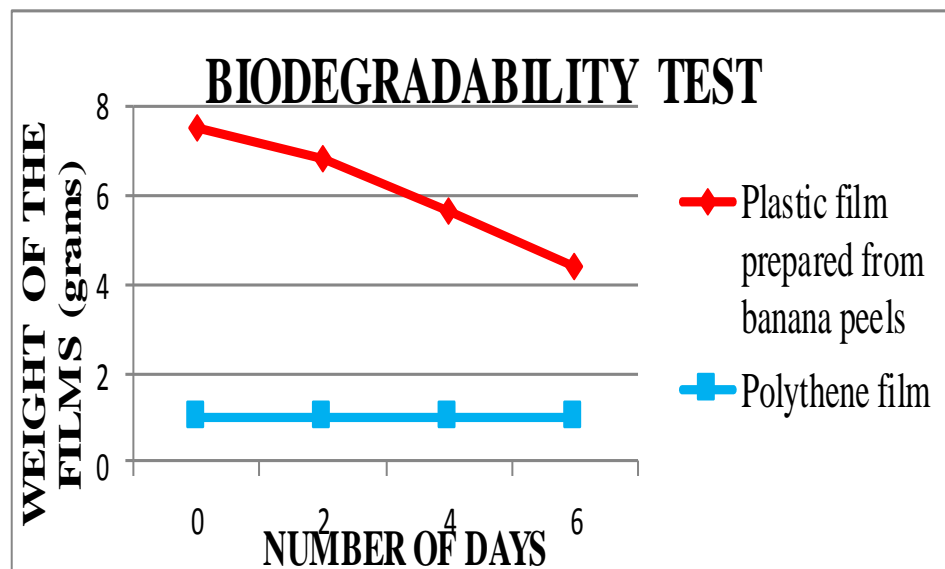


Fig 4: Biodegradability test

### 3.3 MECHANICAL CHARACTERIZATION

#### 3.3.1 AREA OF CROSS-SECTION OF THE SAMPLES

SL No	Plasticizer used	pH	Area of cross-section (mm <sup>2</sup> )		
			1	2	Mean
1	Glycerol	Acidic	0.4500	0.6029	0.5253
2	Glycerol	Neutral	0.5849	0.5850	0.5849
3	PEG	Acidic	0.4320	0.4589	0.4427
4	PEG	Neutral	0.3779	0.4049	0.3929
5	PVA	Acidic	0.5040	0.5039	0.5039
6	PVA	Neutral	0.4919	0.5072	0.4997

**Table 2: Area of cross-section of the samples**

**3.3.2 MAXIMUM FORCE OF THE SAMPLES**

Sl No	Plasticizer used	pH	Maximum Force (N)		
			1	2	Mean
1	Glycerol	Acidic	1.9469	2.5312	2.2391
2	Glycerol	Neutral	6.4312	4.2219	5.3266
3	PEG	Acidic	7.9969	5.6031	6.8000
4	PEG	Neutral	17.223	22.7656	19.9945
5	PVA	Acidic	5.9469	6.8375	6.3922
6	PVA	Neutral	6.3244	6.8562	6.5903

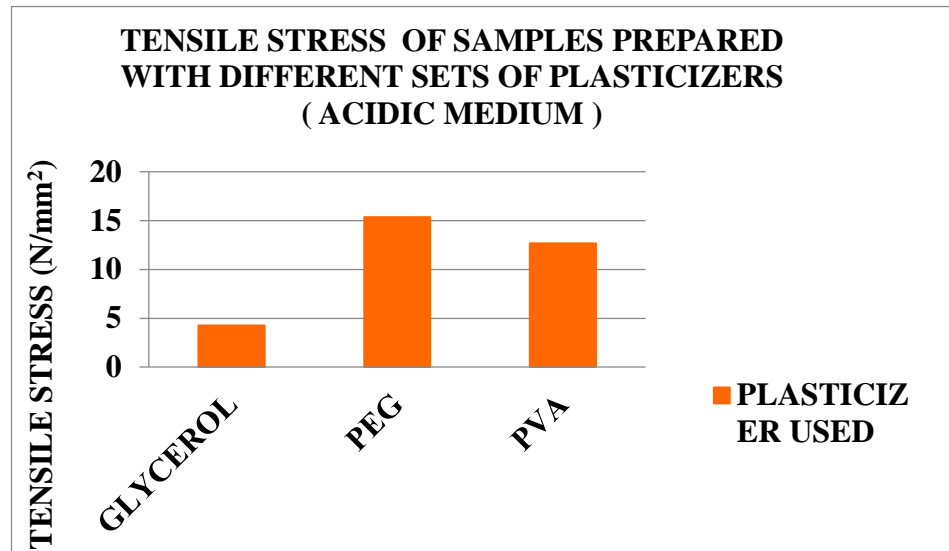
*Table 3: Maximum force of the samples*

### 3.3.3 MAXIMUM TENSILE STRESS OF THE SAMPLES

SL No	Plasticizer used	pH	Maximum Stress (N/mm <sup>2</sup> )		
			1	2	Mean
1	Glycerol	Acidic	4.3264	4.1978	4.2621
2	Glycerol	Neutral	10.9936	7.2169	9.1053
3	PEG	Acidic	18.5113	12.2072	15.3592
4	PEG	Neutral	45.5647	56.2114	50.8881
5	PVA	Acidic	11.7994	13.5665	12.683
6	PVA	Neutral	12.8569	13.5169	13.1869

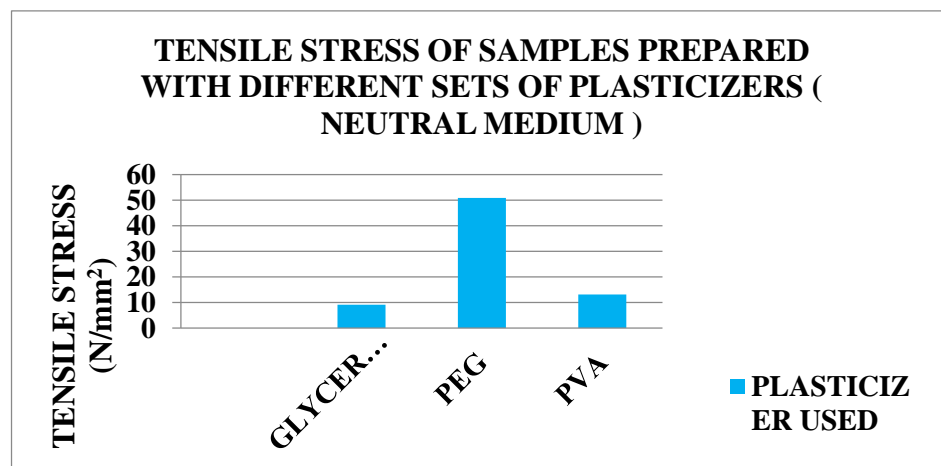
*Table 4: Maximum tensile stress of the samples*

- ANALYSIS OF ACIDIC SAMPLES



*Fig 5: Tensile stress of samples prepared with different sets of plasticizers (Acidic medium)*

- ANALYSIS OF NEUTRAL SAMPLES



*Fig 6: Tensile stress of samples prepared with different sets of plasticizers (Neutral medium)*



## 3.3.4. MAXIMUM STRAIN OF THE SAMPLES

SL No	Plasticizer used	pH	Maximum Strain (%)		
			1	2	Mean
1	Glycerol	Acidic	10.8633	13.53	12.1966
2	Glycerol	Neutral	15.2967	12.6483	13.9725
3	PEG	Acidic	14.3633	11.0733	12.7183
4	PEG	Neutral	17.3967	16.3067	16.8517
5	PVA	Acidic	13.7867	11.7967	12.7917
6	PVA	Neutral	13.2546	11.7845	12.5195

Table 5: Maximum strain of the samples

**3.3.5 YOUNG'S MODULUS OF ELASTICITY OF THE SAMPLES**

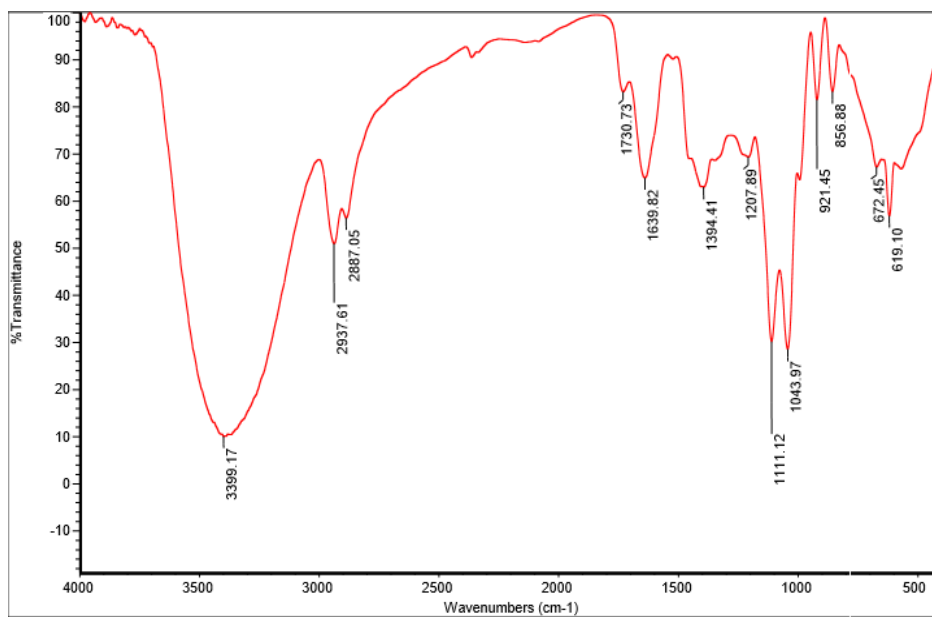
<b>Sl no.</b>	<b>Plasticizer Used</b>	<b>pH</b>	<b>Mean Stress (N/mm<sup>2</sup>)</b>	<b>Mean Strain</b>	<b>Young's modulus (N/mm<sup>2</sup>)</b>
1	Glycerol	Acidic	4.2621	12.1966	0.3494
2	Glycerol	Neutral	9.1053	13.9725	0.6516
3	PEG	Acidic	15.3592	12.7183	1.2076
4	PEG	Neutral	50.8881	16.8517	3.0198
5	PVA	Acidic	12.683	12.7917	0.9915
6	PVA	Neutral	13.1869	12.5195	1.0533

*Table 6: Young's modulus of elasticity of the samples*

### 3.4 FTIR ANALYSIS

#### 3.4.1 FTIR SPECTRAL STUDIES OF BIODEGRADABLE

#### POLYMERIC FILMS AT DIFFERENT PH CONDITIONS



*Fig 7: Acidic medium*

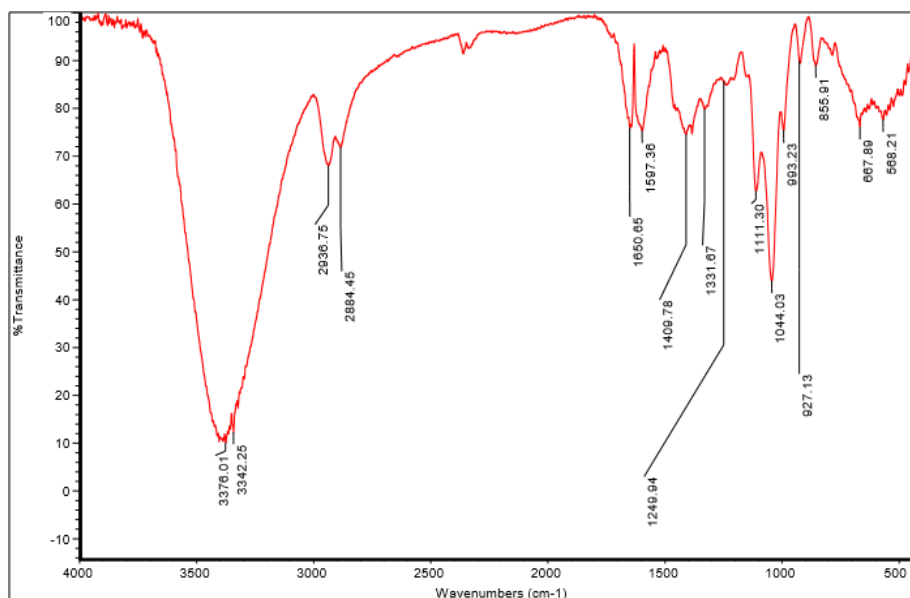


Fig 8 :Basic medium

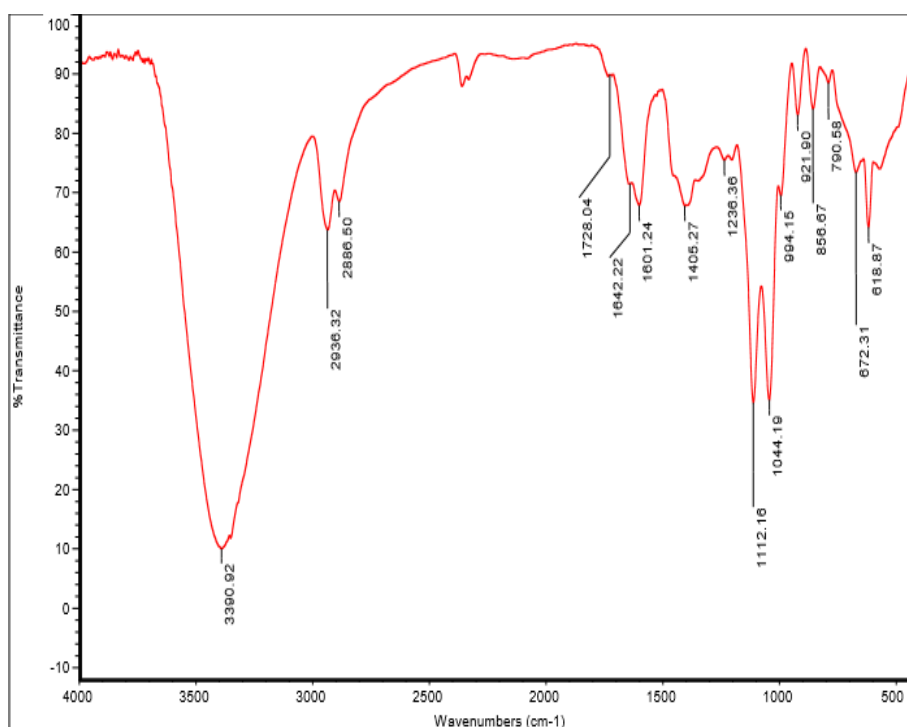
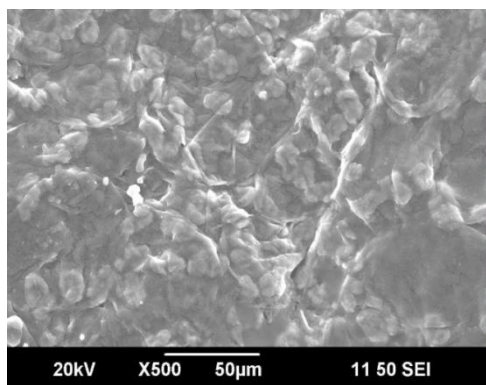


Fig 9: Neutral medium

pH	O-H group stretching vibrations ( $\text{cm}^{-1}$ )	C-O-H group stretching vibrations ( $\text{cm}^{-1}$ )	C-H bond stretching vibrations of $\text{CH}_2$ groups ( $\text{cm}^{-1}$ )	OH group bending vibrations ( $\text{cm}^{-1}$ )	$\text{CH}_2$ group bending vibrations ( $\text{cm}^{-1}$ )	C-O group stretching vibrations ( $\text{cm}^{-1}$ )
Acidic medium	3399.17	1043.97	2937.61	1639.82	1394.41	1207.89
Basic medium	3376.01	1044.03	2936.75	1650.65	1409.78	1249.94
Neutral medium	3390.92	1044.19	2936.32	1642.22	1405.27	1236.36

**Table 7: FTIR analysis of biodegradable polymeric films at different pH conditions**

### 3.5 SEM ANALYSIS



**Fig 10: SEM image of biodegradable polymeric film prepared from banana peels**

### 3.6 CONVERSION % OF THE SAMPLES

- Parameters fixed
- Residence time : 15 minute
- Baking temperature : 90<sup>0</sup> C for 6 hours

#### TRIAL: 1 PLASTICIZER USED: GLYCEROL

Sample	pH	Weight of the final paste (grams)	Weight of the film (grams)	Conversion %
A	Acidic	102.45	5.75	5.6125
B	Basic	104.76	5.82	5.5555
C	Neutral	96.18	5.69	5.9160

*Table 8: Conversion % of the samples prepared using glycerol as the plasticizer*

**TRIAL 2: PLASTICIZER USED: PEG**

<b>Sample</b>	<b>pH</b>	<b>Weight of the final paste (grams)</b>	<b>Weight of the film (grams)</b>	<b>Conversion %</b>
D	Acidic	97.33	5.97	6.1338
E	Basic	101.904	5.98	5.8683
F	Neutral	95.677	5.76	6.0202

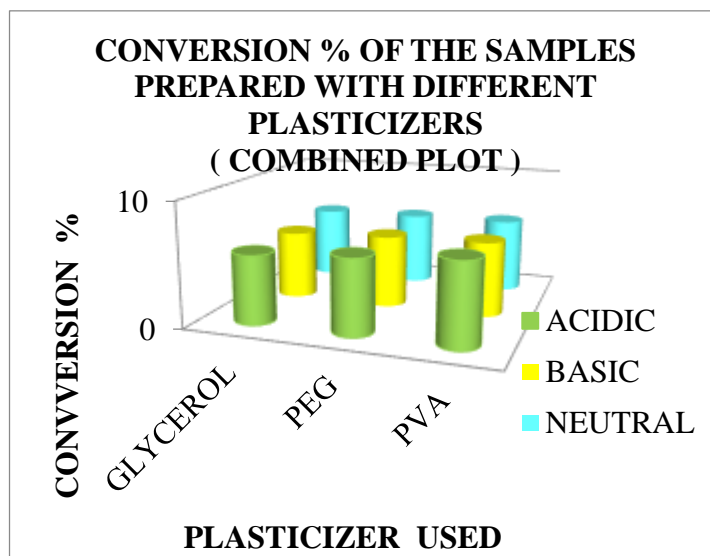
*Table 9: Conversion % of the samples prepared using PEG as the plasticizer*

**TRIAL 3: PLASTICIZER USED: PVA**

<b>Sample</b>	<b>pH</b>	<b>Weight</b>	<b>Weight</b>	<b>Conversion%</b>
G	Acidic	103.52	7.13	6.7114
H	Basic	102.83	7.24	6.0683
I	Neutral	100.674	7.73	6.0890

**Table 10: Conversion % of the samples prepared using PVA as the plasticizer**

• **ANALYSIS OF CONVERSION % OF SAMPLES**



**Fig 11: Conversion % of the samples prepared with different plasticizers (Combined plot)**

### 3.7 DISCUSSIONS

The soil burial test provided a realistic environment where soil humidity, temperature, types and the amount of microorganisms were less in control and changed with seasons. Both of the tested films have same shape and size in order to avoid the effects of film's shape on its biodegradability. The loss of weight of the film is monitored by means of sample collected from the soil at regular time interval. The films were buried in the soil and the sample was removed for evaluation at 2 days interval. It was observed that the degradation rate of the banana peel starch films increased continuously with the increase in the number of the days. In the soil burial degradation test, the intensity of degradation was tested for the two types



of films and the film prepared from banana peels degraded at a rapid rate compared to synthetic plastic (polythene film), which did not degrade at all.

Banana peel starch fibers have relatively high mechanical properties when compared to other natural fibres. The polymer obtained from banana peel show high tensile stress and Young's modulus of elasticity when gets copolymerized with the plasticizers beside of low cost and easy availability of banana peels[34]. From the graphs it was found that the maximum tensile stress was obtained for the neutral samples. Based on the paper "The effect of sodium hydroxide treatment and fibre length on the tensile property of coir fibre" by Karthikeyan et al (2013), the experimental results showed that increasing the amount of NaOH leads to a decrease in fibre diameter in a linear fashion [35]. This reduction in diameter naturally ends up with reduced tensile strength. Hence, the tensile stress of basic samples was always lower than the neutral samples. The decrease in the tensile stress of acidic samples may be because of the excessive hydrolysis occurring in the samples. The study revealed that the neutral sample prepared with PEG as the plasticizer exhibits maximum tensile stress followed by PVA and glycerol [36]. The sample which was prepared in neutral medium using PEG as the plasticizer exhibits maximum tensile stress of 50.8881 N/mm<sup>2</sup> and Young's modulus of elasticity of 3.0198 N/mm<sup>2</sup>.

The FTIR spectra of the samples were obtained at the wavenumber range of 450-4500 cm<sup>-1</sup>. Based on the spectra, the peaks obtained for all the biodegradable plastic films were similar to each other irrespective of the pH conditions. The major peak determined from the biodegradable polymeric films prepared in acidic, basic and neutral medium, was due to the stretching of C-O-H groups which were found at 1043.97cm<sup>-1</sup>, 1044.03cm<sup>-1</sup> and 1044.19 cm<sup>-1</sup> respectively[37]. This was a clear evidence for the hydrolysis of starch which involves the breakdown of the glycosidic linkage at  $\alpha$  (1, 6) position of amylopectin, resulting in the formation of polymeric chains of amylose. The peaks observed between 3100 to 3700cm<sup>-1</sup> in all the spectras corresponds to the hydrogen bonded hydroxyl group (O-H) due to the complex vibrational stretching that naturally occurs in the carbohydrate structure [37]. The band at 1640 cm<sup>-1</sup>

was a result of adsorbed water molecules which again substantiate the formation of biopolymer which involves the acid modification reactions occurring in the structure of amylopectin polymers, indicating its conversion to the amylose chain [38].

From scanning electron microscope (SEM) image of biodegradable polymeric film prepared using glycerol, it was found that the film has a rough, irregular and uneven external morphology. Starch granules shows different shapes such as spherical, oval, cylindrical and oblong shapes but oval, cylindrical and irregular shapes were predominant [39]. The native banana starch granules show higher proportion of medium size granules. After acid modification the granule size get increased. The morphological properties provide clear evidence for the hydrolysis reactions of starch granules. Due to the attack of acid on amorphous regions, amylopectin chains comes in contact with the acid leading to the fusion of starch granules and results in the formation of linear amylose chains. After acid modification, starch granules tended to appear fused and less smooth than the native starch granules.

The conversion being considered was not the chemical conversion of the process but a relation of the net mass of polymer obtained per unit mass of the reaction mixture fed into the oven. These conversions were almost constant for all the samples and were independent of the pH and the type of plasticizer used. From this it can be inferred that the conversion was predominantly a result of the water losses which take place from the samples.

# Chapter 4

## Conclusions

Green chemical approach towards the synthesis of plastics has many advantages such as, ease with which the process can be scaled up and economic sustainability. The method we have adopted is fast, eco-friendly and convenient for the synthesis of biodegradable plastic films. We prepared plastic films from banana peels using three different sets of plasticizers by keeping the parameters baking temperature, pH and residence time constant. The three different sets of plasticizers chosen for the synthesis are Glycerol, Polyethylene Glycol (PEG) and Polyvinyl Alcohol (PVA). Sodium Metabisulphite was added as an antioxidant in order to improve the shelf life of plastic films. No hazardous substances were used in this method, which consequently enables the biosynthesis with the advantage of being environmental friendly. The biodegradability of the plastic films was detected by soil burial degradation test. The plastic strength of the films was examined by using tensile strength experiment. Hydrolysis reaction occurs during the synthesis of plastic films with the hydrochloric acid added, followed by the blending of plasticizers results in the formation of biodegradable plastic films which have plentiful of large sized starch granules which was confirmed by FTIR analysis and SEM studies. The conversion of the process which gives a relation between the net mass of polymer obtained per unit mass of the reaction mixture fed into the oven was also calculated from the trials performed during the experiment.

The soil burial degradation test discloses that, the film prepared from banana peels degrades at a rapid rate compared to synthetic plastic (polythene film), which did not degrade at all.

The tensile strength analysis reveals that among the films prepared neutral samples shows maximum tensile strength in which the neutral sample prepared using PEG as the plasticizer exhibits a maximum tensile strength followed by PVA and glycerol. The neutral sample which was prepared using PEG as the plasticizer exhibits maximum tensile strength of 50.8881 N/mm<sup>2</sup> and Young's modulus of elasticity value as 3.0198 N/mm<sup>2</sup>.

The FTIR study reveals the hydrolysis reaction occurring in starch in all the three cases. The band at 1640 cm<sup>-1</sup> represents adsorbed water molecules, which clearly provides evidence for the conversion of one of the polymeric component amylopectin to the other component of the starch, amylose due to the hydrolysis reaction. This conversion results in the formation of a better polymeric film. The peak obtained approximately at 1044 cm<sup>-1</sup> to be due to the stretching of C-O-H groups. Both of the above mentioned peaks substantiate the formation of biopolymer which involves the acid modification reactions occurring in the structure of amylopectin polymers. Based on the spectra obtained, the peaks obtained for all the biodegradable plastic films were similar to each other irrespective of the pH of the medium.

SEM images of biodegradable polymeric films indicate that all the films prepared have a rough, irregular and uneven external morphology. These images also provide clear evidence for the acid hydrolysis reactions. Starch granules get increase in size after acid modification reactions.

The conversion being considered from the combined plot of samples was not the chemical conversion of the process but a relation of the net mass of polymer obtained per unit mass of the reaction mixture fed into the oven. These conversions were almost constant for all the samples and are independent of the pH, and the type of plasticizer used.

The improved plasticity and flexibility exhibited by the films confirm the copolymerization reactions of the starch with the plasticizers.

Biodegradable polymers are predominantly used in sectors where the property may be a technical advantage during their use or their end of life like agriculture, horticulture, and flexible packaging.

The banana peels consists of many different components apart from starch. Currently only the reaction with starch has been considered. The interaction of all the other components with the reagents may also have an effect which must also be quantified. Thus, new formulations can be developed in the future to achieve the standard bioplastic requirements and this may led the world into another revolution.

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