

# **PROJECT REPORT**

**On**

**“SYNTHESIS AND CHARACTERIZATION OF BIOPLASTIC  
FROM CORN STARCH: A COMPARATIVE STUDY OF BIO AND  
NANO REINFORCEMENT FILLERS”**

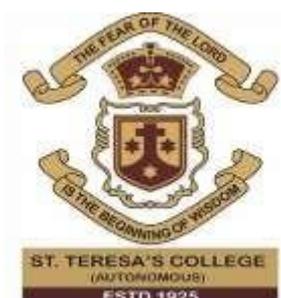
Submitted by

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**(AM20CHE007)**

*In partial fulfillment for the award of the*

**Postgraduate degree in chemistry**



**DEPARTMENT OF CHEMISTRY**

**AND**

**CENTRE FOR RESEARCH**

**ST TERESAS COLLEGE (AUTONOMOUS)**

**ERNAKULAM**

**2021-2022**

DEPARTMENT OF CHEMISTRY AND  
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ST TERESAS COLLEGE (AUTONOMOUS)  
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This is to certify that the project "SYNTHESIS AND CHARACTERIZATION OF BIOPLASTIC FROM CORN STARCH: A COMPARATIVE STUDY OF BIO AND NANO REINFORCEMENT FILLERS" is the work done by FASEELA N. F.

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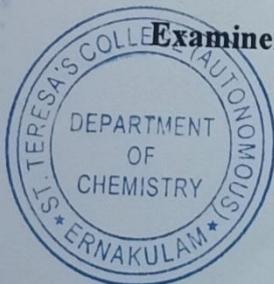
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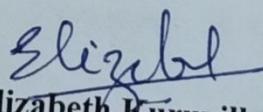


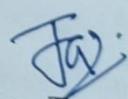
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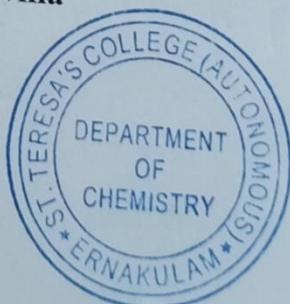


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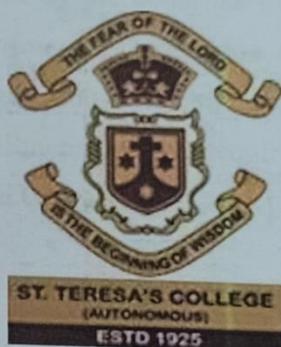
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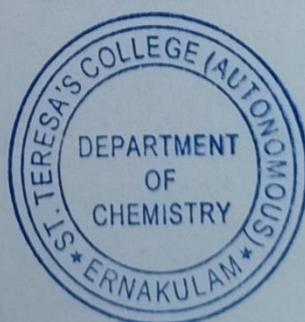


CERTIFICATE

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## **DECLARATION**

I hereby declare that the project work entitled “**SYNTHESIS AND CHARACTERIZATION OF BIOPLASTIC FROM CORN STARCH : A COMPARATIVE STUDY OF BIO AND NANO REINFORCEMENT FILLERS**” submitted to Department of Chemistry and Centre for Research, St. Teresa’s College affiliated to Mahatma Gandhi University, Kottayam, Kerala is a record of an original work done by me under the guidance of **Dr. ELIZABETH KURUVILLA**, Assistant Professor, Department of Chemistry and Centre for Research, St. Teresa’s College (Autonomous), Ernakulam. This project work is submitted in the partial fulfillments of the requirements for the award of the Degree of Master of Science in Chemistry.

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FASEELA N. F

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## LIST OF ABBREVIATIONS

|                                    |   |
|------------------------------------|---|
| GO                                 | Graphene Oxide  |
| CESP                               | Chicken Egg Shell Powder                                    |
| CSA                                | Corn Starch + Acetic Acid                                   |
| CS <sub>1</sub>                    | Corn Starch + Acetic Acid + Sorbitol                        |
| CS <sub>2</sub>                    | Corn Starch + Acetic Acid + Glycerol                        |
| CS1a                               | Corn Starch + Acetic Acid + sorbitol + 1g egg shell powder  |
| CS1b                               | Corn Starch + Acetic Acid + sorbitol + 2g egg shell powder  |
| CS1c                               | Corn Starch + Acetic Acid + sorbitol + 3g egg shell powder  |
| CS <sub>1</sub> (GO <sub>1</sub> ) | Corn Starch + Acetic Acid + sorbitol + 0.1 g graphene oxide |
| CS <sub>1</sub> (GO <sub>2</sub> ) | Corn Starch + Acetic Acid + sorbitol + 0.5g graphene oxide  |
| SEM                                | Scanning Electron Microscopy                                |

|     |                            |
|-----|----------------------------|
| XRD | X – Ray Diffraction        |
| TGA | Thermogravimetric analysis |

# Chapter 1

## INTRODUCTION

### ABSTRACT

Plastic wastes cause severe issues to environment since it does not undergo any bio-degradation process. Development of bio-degradable plastics is an alternative to this problem. In this thesis we report the synthesis of starch based biodegradable polymer. We synthesized the starch based bio-plastic from corn. A comparative study with different plasticizers and reinforcement fillers were carried out. Chicken Egg Shell Powder (CESP) and Graphene Oxide (GO) were studied as Bio and Nano reinforcement fillers and sorbitol and glycerol were used as plasticizers. The properties of the synthesized bio-plastics were studied using techniques such as XRD, SEM, TGA and Tensile strength using UTM. The percentage of degradation for each sample in air and soil were also studied.

### 1.1 PLASTIC

Plastic is an important part of everyday life. Plastic is any synthetic or semisynthetic organic polymer. Bags, bottles, containers, food wrappings etc. that we use every day is made up of plastic. We cannot imagine a world without plastic. Importance of plastic is increasing day by day. But at the same time it is harmful to environment, aquatic and terrestrial organisms. Generally, polyethylene plastic films, such as low-density polyethylene

(LDPE) and high-density polyethylene (HDPE), are being used to produce a variety of polyethylene plastic films. The main drawback of this plastic is its non-degradability. Over 1000 million tons of plastic were predisposed as unwanted elements, and they might take several hundreds of years to decay <sup>[1]</sup>. The annual production has increased nearly 200-fold, reaching 381 million tonnes in 2015 <sup>[2]</sup>. The percentage of plastics in municipal solid waste continues to grow rapidly. When plastic wastes are dumped in landfills, they interact with water and form hazardous chemicals, and the quality of drinking water may also be affected <sup>[3]</sup>. We can reduce these problems associated with this material by introducing biodegradability to plastic.

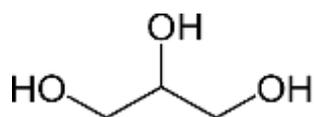
## **1.2 BIODEGRADABLE PLASTIC**

Biodegradation is the property of a material that can be completely converted into water, CO<sub>2</sub>, and biomass through the action of microorganisms such as fungi and bacteria. Bio-plastic is the plastic which can be degraded by the microorganisms present in the earth. Bio-plastics have the ability to be degraded by microorganisms present in the environment by entering the microbial food chain. <sup>[4]</sup> It can be easily decomposed and it reduces hazards associated with the plastic. There are two main types of bio-plastics:

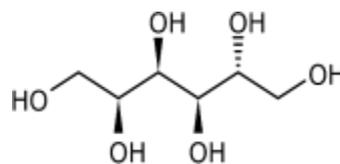
1. Oxo-biodegradable plastic (OBP)
2. Hydro-biodegradable plastic (HBP)

OBP was made by adding a small portion of fatty acid compounds of specific transition metals to traditional plastics. HBP was made from bio- based sources such as corn, wheat, sugar-cane, petroleum-based sources or

a blend of the two. HBP degrade more quickly than OBP <sup>[5]</sup>. Poly lactic acid (PLA), poly glycolic acid (PGA) etc. is examples of biodegradable polymers. To improve the characteristics of bio-plastic, it needs some other materials such as plasticizers <sup>[6]</sup>. Plasticizers are an organic material which has a low molecular weight which is added to weaken the inflexible character of the plastic. It increases the flexibility and extensibility of the bio-plastic <sup>[7]</sup>. Plasticizer is dissolved in each polymer chains, so it will facilitate the movements of polymer molecule and makes the glass transition temperature lower <sup>[8]</sup>. The purpose of the addition of plasticizer is to produce a film that is more flexible and not brittle <sup>[9]</sup>. In the present work we use sorbitol and glycerol as plasticizer. Structures of plasticizers are given below:



*Fig 1(a): Glycerol*



*Fig 1(b): Sorbitol*

Bio-plastics may be openly taken out from natural resources like lignin, proteins, lipids, and polysaccharides (e.g., starch, chitin, and cellulose). Approximately 50% of the bio-plastics which are used commercially are prepared from starch. The production of starch-based bio-plastics is very simple and they are widely used for packaging applications. The tensile properties of starch are suitable for the production of packing materials. The required characteristics of the bio-plastics are achieved by fine-tuning the

quantities of the additives. For trade applications, the starch-based plastics are regularly mixed with eco-friendly polyesters <sup>[10]</sup>.

### **1.3 STARCH**

Most of the green plants produce polysaccharides as an energy store. Starch is a carbohydrate that contains greater amount of glucose units, combined through glycosidic linkages. Pure starch is white in colour. The starch powder does not possess any specific taste or odour. Furthermore, pure starch cannot be dissolved in cold water or alcohol. It is non-toxic, biologically absorbable, and semi-permeable to carbon dioxide. The linear and helical amylose and the branched amylopectin are the two types of molecules present in starch. The amylose content may vary from 20 to 25%, while the amylopectin content varies from 75 to 80% by weight, depending on the type of plant. Amylopectin is a far greater molecule than amylose. If heated, starch would become soluble in water, and the grains swell and burst. Due to this, the semi-crystalline arrangement is also lost, and the minor amylose particles begin percolating out of the granule forming a network. This network compresses water and increases the viscosity of the mixture. This procedure is known as starch gelatinization and amylose shows an imperative part through the initial stages of corn starch gelatinization <sup>[11]</sup>.

While heating the starch, it becomes a paste and the viscosity is also increased. High amylose starch is a smart reserve to use as an obstruction in packing materials. Due to the low price, renewability, and having decent mechanical properties, it was used to produce decomposable films to partly or else completely substitute the plastic polymers. The percentage of amylose and amylopectin content in various starches is shown in Table 1.

| <b>Source</b> | <b>Amylose (in %)</b> | <b>Amylopectin<br/>(in %)</b> |
|---------------|-----------------------|-------------------------------|
| Arrowroot     | 20.5                  | 79.5                          |
| Banana        | 17                    | 83                            |
| Cassava       | 18.6                  | 81.4                          |
| Corn          | 28                    | 72                            |
| Potato        | 17.8                  | 82.2                          |
| Rice          | 35                    | 65                            |
| Tapioca       | 16.7                  | 83.3                          |
| Wheat         | 20                    | 80                            |

*Table 1: Amylose and amylopectin concentration in various natural sources*

The tensile properties of the bio-plastics would rise when the amylose content was increased <sup>[12]</sup>. From the above table we can understand that the corn is a material which possesses high content of amylose. So polymer made from corn starch will have high tensile properties. So we chose corn starch in our present work.

## 1.4 CORN

Corn (*Zea mays*), also called as **Indian corn** or **maize** is a cereal plant which belongs to a grass family, *poaceae*. It is the edible part of the plant. The domesticated crop originated in the America and is one of the most widely distributed of the world's food crops. Corn is used as livestock feed, as human food, as biofuel, and as raw material in industry. Corn was first domesticated by native peoples in southern Mexico about 10,000 years ago. Modern corn is believed to have been derived from the Balsas teosinte (*Zea mays parviglumis*), a wild grass. Its culture had spread as far north as southern Maine by the time of European settlement of North America, and Native Americans taught European colonists to grow the indigenous grains. Since its introduction into Europe was by Christopher Columbus and other explorers and colonizers, corn has spread to all areas of the world suitable to its cultivation. It is the most important crop in the United States and is a staple food in many places<sup>[14]</sup>.



*Fig 2: Corn plant*

Composition of the corn is given in the Table 2 below:

|         | <b>Average (%)</b> | <b>Range</b> |
|---------|--------------------|--------------|
| Protein | 7.7                | 5.7-9.7      |
| Oil     | 3.3                | 2.6-4.9      |
| Starch  | 61.7               | 59.9-64.8    |

*Table 2: Composition of the corn*

## **1.5 REINFORCEMENT FILLERS**

However, starch biopolymers are dissolvable in water and have low mechanical strength, which has proved to be a major negative aspect. Researchers have attempted various methods to enhance the properties of starch biopolymers, such as using reinforcement fillers. One of the strategic ways for improving the mechanical properties of bio-plastic is the filler addition in bio-plastic matrix. Many different types of natural, synthetic, organic and inorganic materials can be used as reinforcement fillers, such as talc, glass fibres, corn waste lignocellulose fibre, cocoa pod husk, eggshells, rice husk etc. These agricultural waste materials can be powdered into fine or coarse particles and used as fillers, reducing the problems associated with the disposal of agricultural residues <sup>[15]</sup>. In the present work we made use of Chicken Egg Shell Powder and Graphene oxide (GO) as bio and nano reinforcement fillers.

### **1.5.1 Chicken Egg Shell as Bio reinforcement filler**

Chicken Egg shell is promising bio filler for further development of corn starch based bio-plastics. Chicken Egg shells are composed of 96% calcium carbonate ( $\text{CaCO}_3$ ) by weight, which is an amorphous crystal. Other than  $\text{CaCO}_3$ , eggshells also contain small amounts of magnesium carbonate ( $\text{MgCO}_3$ ), calcium phosphate ( $\text{Ca}_3(\text{PO}_4)_2$ ), organic components and water. Its disposal constitutes serious environmental hazards. Thus, finding a use for waste eggshells can decrease the problem of their disposal and can help achieve sustainable development. Local restaurants and bakeries, as well as the food manufacturing industry, such as biscuit and bakery factories produce waste eggshells in huge quantities and are thus a good source to obtain this residue <sup>[16]</sup>.

### **1.5.2 Graphene oxide as Nano reinforcement filler**

Graphene Oxide (GO) is promising nano filler for further development of corn starch based bio-plastics <sup>[17]</sup>. The properties of starch based bio-plastic have been successfully enhanced by addition of graphene oxide (GO) filler. GO is one layer of oxidized graphite obtained via chemical synthesis or through others graphite oxidation methods. There are many advantages of the use of GO as filler including - it is easy to dissolve in matrix of bio- plastic, it has high surface area and biocompatibility and it still inherit the graphene characters. This work tries to understand the influence of graphene oxide addition to the properties of corn starch based bio-plastics.

## **1.6 APPLICATIONS OF BIO-PLASTIC**

Biodegradable polymers have been at the forefront of research for biomedical applications in the last 50 years. The advancements have been seen in the areas of using biodegradable polymers as delivery vehicles for controlled drug release and development of therapeutic devices including implants and three-dimensional scaffolds for tissue engineering.

In tissue engineering applications, biopolymers have proven useful in replacing biogenic materials that could induce an immunogenic reaction due to non-specific host response <sup>[22]</sup>. Nontoxic bio-plastics sutures, commonly referred to as stitches, are now being used by medical professionals in hospitals and surgeries. They are easy to sterilise, robust and remain in place until the tissue has healed at which time they are dissolved by the body leaving no marks behind. Dentist are also getting on board and are using bio-plastics for dental implants that fill in the hole that remains after a tooth has been extracted.

They can use to make shopping bags, compostable waste collection bags and trays. In relation to the production of packaging, conventional plastics are being replaced by bio-plastics at a rapid pace. There's a big demand for bio-plastic packaging and it is the largest segment of the European bio-plastic market – estimated at around 44% of 2.05 million tonnes in 2017.

Bio-plastic packaging options include bags for compost, agricultural foils, horticultural products, nursery products, toys and textiles. They are also often used for disposable cups, salad bowls, plates, and Clingfilm and food containers <sup>[20]</sup>.

The cosmetics industry is another big producer of packaging for its products. Many of these products have a short life span once disposed of they end up in landfills. When you consider the impact disposable products such as toothbrushes, hairbrushes, cotton buds and razors are having – it's no wonder that brands are starting to look towards alternatives. This market is heavily consumer focused and today's shoppers expect certain standards from the brands they use. Bio-plastic alternatives enable brands to satisfy this requirement without impacting on product quality .Even small parts of cosmetic products such as bio-plastic caps can be injection moulded<sup>[23]</sup>.

# Chapter 2

## MATERIALS AND METHODS

This chapter gives a brief description of the materials and the experimental procedures adopted for the present investigation.

### 2.1 MATERIALS

- Corn starch
- 5% Acetic acid
- Sorbitol
- Distilled water
- Glycerol
- Chicken egg shell powder
- Graphene oxide

### 2.2 EXPERIMENTAL METHODS

#### 2.2.1 Extraction of starch from Corn

The following steps detail extracting the starch from corn by the manual method. Corn was obtained from the nearest supermarket. Take out the kernels of the corn and wash thoroughly. First, 100 g corn was soaked in pure water for 2 hours. Then corn was drained and grinded in a mortar with 100 mL purified water. The mixture was filtered and the remaining solid mass was put into the mortar. Repeat the procedure three more times. The blend was again filtered through a micro sieve. The filter was allowed to settle in the beaker for 3hours. The water was removed and the starch, white

in colour, was obtained. The starch was washed with distilled water and dried in the oven for 60°C. About 40 g of starch was obtained from 100 g of corn.



*Fig. 3. corn starch obtained by manual method*

### **2.2.2 Preparation of bio-plastic film**

Three variants of bio-plastics were produced from the starch solution, without any plasticizer, with plasticizers (glycerol and sorbitol) and the combination of both plasticizers. The general procedure for the production of corn starch bio-plastic was the following:

A 20% corn starch solution was prepared by dissolving 8.0 g of powder corn starch in 40 mL of distilled water. The corn starch solution was measured and transferred into a 100 mL beaker, followed by the addition of 5% acetic acid. The mixture was then heated with continuous stirring till the corn starch solution became a thick paste. The bio-plastic mixture was spread onto a glass Petri dish. The mixture was allowed to dry at room temperature for 24 hours until it no longer stuck to the surface. The bio-plastic sheet produced was then allowed to cool at room temperature, after which it was removed and placed into a zip lock bag for further characterization.



*Fig 4: bio-plastic obtained from corn starch + acetic acid*

In order to increase the flexibility of bio-plastics, plasticizers were added to the mixture along with corn starch solution and acetic acid. Sorbitol and glycerol were used as plasticizers. Different bio-plastics with 5g of sorbitol, 5 g of glycerol and combination of sorbitol and glycerol were made.

### **2.2.3 Incorporation of Nano and Bio reinforcement filler**

Each of these variants was then prepared with the addition CESP and GO as reinforcement filler to further investigate its effects in corn starch bio- plastic.

#### **2.2.3.1 Incorporation of Bio reinforcement filler**

Waste eggshells of chicken eggs were obtained from household wastes. The thin shell membrane of the eggshells was removed from the inside and they were thoroughly rinsed. The eggshells were then broken down into smaller pieces and left to dry in an oven at 85 °C for 5 hours. The dried eggshells were then converted into a fine powder using a blender. The powder was then sieved using a sieve of size 63  $\mu\text{m}$  [24].



(a) (b)  
*Fig5 (a): cleaned chicken egg shell: Fig5 (b): powdered chicken egg shell*

Powdered chicken egg shell was incorporated in the bio-plastic in various concentrations. Bio-plastic films were prepared by adding powdered chicken egg shell in different quantities of 1g, 2g, and 3g respectively.

#### **2.2.3.2 Incorporation of Nano reinforcement filler**

GO was synthesized by Hummer's method through oxidation of graphite. The stepwise preparation is given as follows,

Graphite flakes (1g) and  $\text{NaNO}_3$  (1g) were mixed in 25 mL of  $\text{H}_2\text{SO}_4$  (98%) in a 500 mL standard flask kept under at ice bath ( $0-5^\circ\text{C}$ ) with continuous stirring. The mixture was stirred for 2 hours at this temperature and potassium permanganate (3g) was added to the suspension very slowly. The rate of addition was carefully controlled to keep the reaction temperature lower than  $15^\circ\text{C}$ .

The ice bath was then removed, and the mixture was stirred at  $35^\circ\text{C}$  until it become pasty brownish and kept under stirring for 2 days. It is then diluted with slow addition of 50 mL water. The reaction temperature was rapidly increased to  $98^\circ\text{C}$  with effervescence, and the colour changed to brown

colour. Further this solution washed by rinsing and centrifugation with 10% HCl and then deionized water several times. After filtration and drying under vacuum at room temperature, GO was obtained as powder <sup>[19]</sup>.



*Fig 6: Graphene oxide prepared by Hummer's method*

GO was incorporated in the bio-plastic in various concentrations. Bio-plastic films were prepared by adding GO in different quantities of 0.1g and 0.5g respectively.

| Sample no                          | Starch (mL) | Acetic acid (mL) | Sorbitol (g) | Glycerol (mL) | CSEP (g) | GO (g) |
|------------------------------------|-------------|------------------|--------------|---------------|----------|--------|
| CSA                                | 40          | 5                | -            | -             | -        | -      |
| CS <sub>1</sub>                    | “           | “                | 5            | -             | -        | -      |
| CS <sub>2</sub>                    | “           | “                | -            | 5             | -        | -      |
| CSGS                               | “           | “                | 2.5          | 2.5           | -        | -      |
| CS <sub>1</sub> a                  | “           | “                | 5            | -             | 1        | -      |
| CS <sub>1</sub> b                  | “           | “                | 5            | -             | 2        | -      |
| CS <sub>1</sub> c                  | “           | “                | 5            | -             | 3        | -      |
| CS <sub>1</sub> (GO <sub>1</sub> ) | “           | “                | 5            | -             | -        | 0.1    |
| CS <sub>1</sub> (GO <sub>2</sub> ) | “           | “                | 5            | -             | -        | 0.5    |

*Table 3: Composition of bio-plastics based on starch, glycerol, sorbitol and filler*

## 2.3 ANALYSIS AND CHARACTERIZATION

### 2.3.1 Moisture content

Bio-plastic samples were cut into 1.5 x 1.5 cm<sup>2</sup> pieces, followed by determining their initial weight ( $W_i$ ) and then drying them in the oven at 90 °C for 24 hours. The dried samples were again weighed to find their final weight ( $W_f$ ) and the moisture content was calculated using the following equation:<sup>[6]</sup>

$$\text{Moisture content (\%)} = \frac{W_i - W_f}{W_i} \times 100$$

### 2.3.2 Water solubility Test

The film samples were cut into square sections of 2.0 cm<sup>2</sup>, and the dry film mass was weighed accurately and recorded. The samples remained immersed in 100 mL distilled water and fixed agitation at 180 rpm was carried out for 6 h at 25 °C <sup>[28]</sup>. The lasting portions of the film were filtered after 6 h. They were then dried in a hot air oven at 110 °C until an ultimate fixed weight was found. Glycerol has a good water solubility range from 18% to 25% <sup>[41]</sup>. The percentage of total soluble matter (% solubility) was calculated as <sup>[6]</sup>

$$W_s (\%) = \frac{W_0 - W_f}{W_0} \times 100,$$

Where  $W_s$  is solubility in water;  $W_0$  is the weight at the beginning of the bio-plastics; and  $W_f$  is the final weight of the bio-plastics

### 2.3.3 Soil biodegradability test

Soil was obtained from the garden. All bio-plastic samples were cut into 1.5 x 1.5 cm<sup>2</sup> pieces and their initial weight ( $W_i$ ) was noted. These samples were then buried under 10 cm of moist soil sealed. For air biodegradation

each piece was previously weighed and kept in the air. After 2 days, the residual samples were recovered and cleaned to find their final weight ( $W_f$ ) [7]. The percentage weight loss, that is, the biodegradability of the bio-plastics, was determined using the equation given below:

$$\text{Weight loss (\%)} = \frac{W_i - W_f}{W_i} \times 100$$

### **2.3.4 Air biodegradability test**

All bio-plastic samples were cut into 1.5 x 1.5 cm<sup>2</sup> pieces and their initial weight ( $W_i$ ) was noted. For air biodegradation each piece was previously weighed and kept in the air. After 2 days, the residual samples were recovered and cleaned to find their final weight ( $W_f$ ). It has been observed for 45 days from the formation day [6].

### **2.3.5 Mechanical properties**

Tensile strength and Young's modulus were determined by using a Universal testing machine. The specimens were cut into 5 cm long and 1 cm wide rectangular strips using a pair of scissors. The tensile strength value and Young's modulus of the samples were obtained from the instrumental data [6].

### **2.3.6 Thermo-gravimetric Analysis (TGA)**

Thermo-gravimetric Analysis (TGA) is a method of thermal analysis in which the mass of a sample of measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption, adsorption and desorption as well as chemical phenomena including chemisorption, thermal decomposition, and solid gas reactions. Thermal stability of bio- plastic film samples was characterized using a Thermo-gravimetric

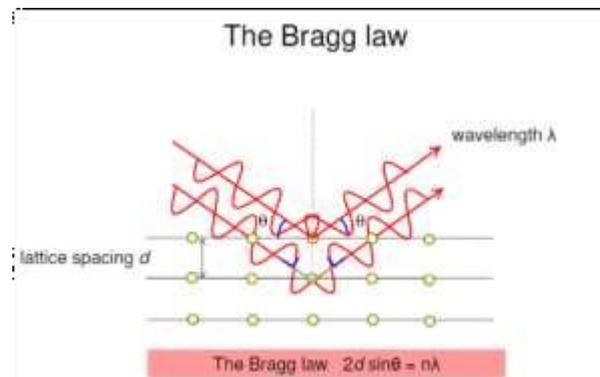
analyser. A selected sample was carried out at the rate of 20°C/min under room temperature, in the range of 1000-1500°C [6].



*Fig 7: Thermo gravimetric analyser*

### **2.3.7 X- Ray Diffraction (XRD)**

X-Ray diffraction technique is a precise and popular tool for determining the crystal structure of thin films. It yields complete information about the crystal structure, orientation, lattice constants, crystalline size and composition, defects, and stress in the thin film. From the position and shape of the lines, one can obtain information regarding the unit cell parameters and microstructural parameters (grain size, micro strain, etc.), respectively. It requires no sample preparation and is essentially non- destructive. The wave nature of X-Rays means that they are diffracted by the lattice of the crystal to give a unique pattern of peaks of reflections at differing angles and of different intensity, just as light can be diffracted by a grating of suitably spaced lines [21].



*Fig 8: Bragg's Law of X-Ray Diffraction*

The diffracted beam from atoms in successive planes cancels unless they are in phase, and the condition for this is given by Bragg's relationship.

$$n\lambda = 2d\sin\Theta$$

Here  $d$  is the inter-spacing,  $\Theta$  is the angle of diffraction,  $\lambda$  is the wavelength of the incident beam and  $n$  is the order of diffraction. Diffraction peak position is accurately measured with XRD, which makes it the best method for characterizing homogeneous and inhomogeneous strains. Homogeneous or uniform elastic strain shifts the diffraction peak position. From the shift in peak position, one can calculate the change in  $d$ -spacing, which is the result of the change of lattice constants under a strain. In homogeneous strains vary from crystallite to crystallite or within a single crystallite and this cause a broadening of the diffraction peaks that increase with  $\cos \Theta$ . The crystallite size,  $D$  can be determined using Debye- Scherrer formula

$$D = K\lambda / B\cos\Theta$$

Where,  $\lambda$  is the X-ray wavelength,  $K$  is the Scherrer constant,  $B$  is the full width of the height of a diffraction peak,  $\Theta$  is the diffraction angle. XRD is a non-destructive technique and does not require elaborate sample preparation, which partly explains the wide usage of XRD method in materials characterization. In addition, X-ray diffraction only provides the

collective information of the particle sizes and usually requires a sizeable amount of powder. It should be noted that since the estimation would work only for very small particles, this technique is very useful in characterizing nanoparticles.

### **2.3.8 Scanning Electron Microscope**

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. The electron beam is scanned in a raster pattern, and the position of the beam is combined with the intensity of the detected signal to produce an image. In the most common SEM mode, secondary electrons emitted by atoms excited by the electron beam are detected using a secondary electron detector. The number of secondary electrons that can be detected, and thus the signal intensity, depends, among other things, on specimen topography. Some SEMs can achieve resolutions better than 1 nm. Specimens are observed in high vacuum in a conventional SEM, or in low vacuum or wet conditions in a variable pressure or environmental SEM and at a wide range of cryogenic or elevated temperatures with specialized instruments.



*Fig 9: Scanning Electron Microscope*

# Chapter 3

## RESULTS AND DISCUSSION

### 3.1 PHYSICAL APPEARANCE

Physical appearance of different samples are given here as figures.



*Fig 10 (a): CSA (corn starch + acetic acid)*



*Fig 10 (b): CS<sub>1</sub> (corn starch + acetic acid + 5 g sorbitol)*



*Fig 10 (c): CS<sub>2</sub> (corn starch + acetic acid + 5ml glycerol)*



*Fig 10 (d): CSGS (corn starch + acetic acid + 2.5ml glycerol + 2.5 g sorbitol)*

| <b>SAMPLE</b>   | <b>PLASTIZICER</b>                    | <b>APPEARANCE OF FILMS</b>  |
|-----------------|---------------------------------------|---|
| CSA             | -                                     | Transparent, surface cracks, brittle and hard, difficult to peel                                      |
| CS <sub>1</sub> | Sorbitol (5g)                         | Crystal clear, rigid, non-sticky, not brittle and not fragile, flexible, peelable, sturdy             |
| CS <sub>2</sub> | Glycerol (5mL)                        | More transparent, more sticky, brittle, not fragile, flexible, difficult to peel                      |
| CSGS            | Sorbitol (2.5g)<br>+ Glycerol (2.5mL) | Transparent, less sticky than CS <sub>2</sub> , sturdy, rigid and not fragile, flexible, easy to peel |

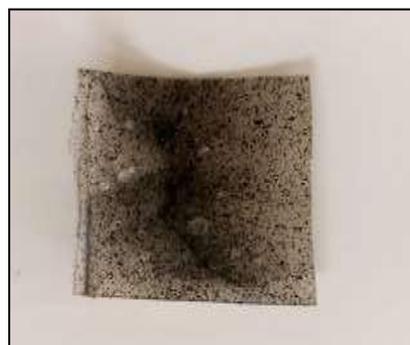
*Table 4: properties of the bio-plastic on preliminary observation*

From the preliminary observations, we found out that CS<sub>1</sub> forms better plastic than CS<sub>2</sub>. Because bio-plastic from CS<sub>2</sub> is stickier, brittle, not fragile, difficult to peel when compared to the bio-plastic from CS<sub>1</sub>. Then we can conclude that sorbitol is the best plasticizer for corn starch based films than the glycerol. So our next aim is to enhance the properties of CS<sub>1</sub> by the addition of reinforcement fillers. For the further experiment, we only consider the bio-plastic from CS<sub>2</sub>.

Reinforcement fillers were incorporated in to the bio-plastic in various concentrations to produce Bio and Nano composites.



*Fig 11 (a): CS<sub>1</sub>(GO<sub>1</sub>) {corn starch + acetic acid+ 5 g sorbitol + 0.1 g GO}*



*Fig 11 (b): CS<sub>1</sub>(GO<sub>2</sub>) {corn starch + acetic acid+ 5 g sorbitol + 0.5g GO}*

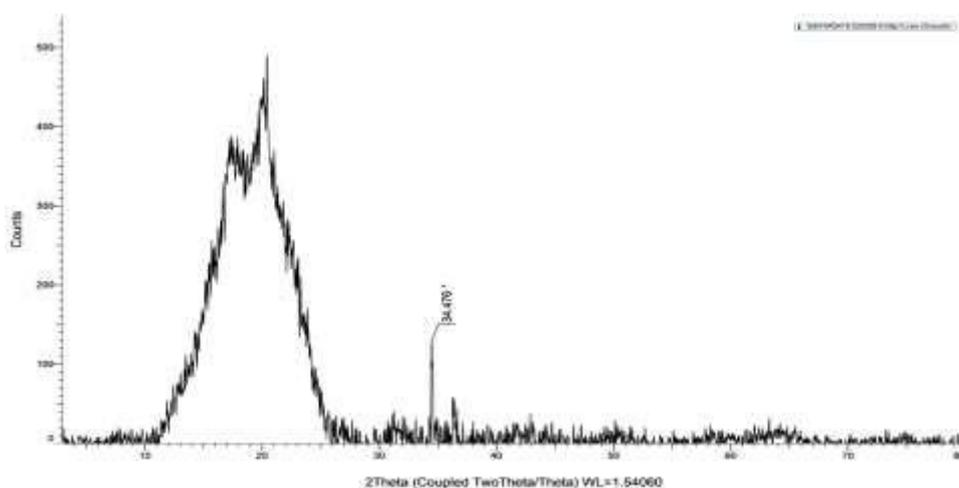


*Fig 11 (c):CS<sub>1</sub> b (Corn starch + sorbitol+ acetic acid + 2g CESP)*

## 3.2 X-RAY DIFFRACTION STUDIES

### 3.2.1 XRD Pattern of CSA

XRD Pattern of CSA is given below in the fig 12. It contains corn starch and acetic acid. It doesn't contain any plasticizers in it. A large amorphous region with crystalline peaks, as noted, was observed in the unplasticized bio-plastic, CSA [13].



*Fig 12: XRD pattern of CSA*

### 3.2.2 XRD Pattern of CS<sub>1</sub>

The XRD Pattern of CS<sub>1</sub> is shown in the fig: 13. When we introduced sorbitol, we can see some other peaks in the XRD. It contains some additional peaks other than 34.250° which arise due sorbitol molecules. X- ray diffraction pattern of CS<sub>1</sub> showed a significant increase in peak intensity at 17.164°. The addition of sorbitol has a major impact on the essential

nature of bio-plastic which is shown by its sharp, well-defined peaks combined with insignificant amorphous regions [13].

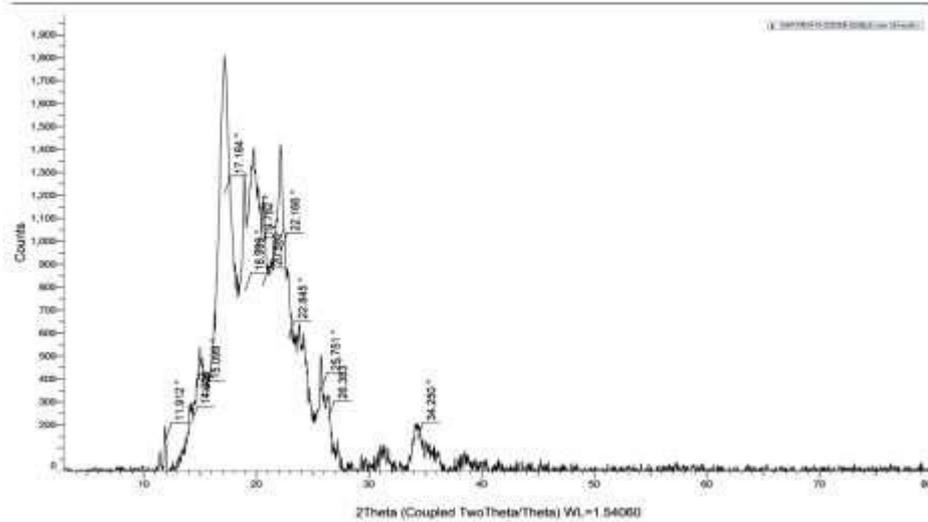


Fig13: XRD pattern of CS<sub>1</sub>

### 3.2.3 XRD Analysis of CS<sub>1b</sub>

#### 3.2.3.1 XRD Pattern of Chicken Egg Shell Powder

The XRD pattern of the CESP particles is displayed in Fig14 given below. The characteristic diffraction peaks of Chicken Egg Shell Powder appeared at  $2\theta = 23.04^\circ$ ,  $29.4^\circ$ ,  $31.44^\circ$ ,  $35.98^\circ$ ,  $39.40^\circ$ ,  $43.16^\circ$ ,  $47.54^\circ$ ,  $48.52^\circ$ , and the main peak at  $2\theta = 29.4^\circ$  demonstrated that the thermodynamically stable calcite crystal is a major phase of the CESP [18].

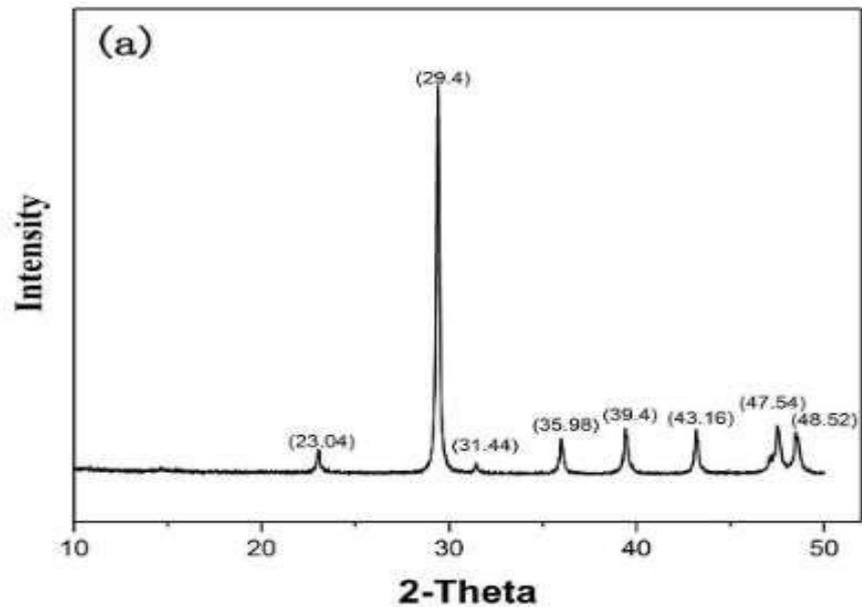


Fig 14: XRD pattern of the CESP <sup>[18]</sup>.

### 3.2.3.2 XRD pattern of CS<sub>1b</sub>

XRD pattern of CS<sub>1b</sub> is given below in the fig15. The diffraction peaks at  $2\theta = 23.266^\circ, 29.609^\circ, 36.234^\circ, 39.675^\circ, 43.433^\circ, 47.734^\circ$  and  $48.747^\circ$  shows the incorporation of CESP in the bio-plastic when compared with the XRD pattern of the CESP. This ensured the formation of a Biocomposite.

As can be seen from the diffraction pattern of the biocomposite, a new strong characteristic reflection appeared at  $2\theta = 29.6092^\circ$  compared with the XRD pattern of CS<sub>1</sub>, which leads to a semi-crystalline structure of the composite films. This indicated that the CESP was uniformly dispersed in

the CS matrix and built a strong interaction with corn. In contrast, the intensity of diffraction peak at  $2\theta = 29.6092^\circ$  increased significantly as the content of CESP is increased, which may be attributed to the calcite crystal and the agglomerates of CESP [18].

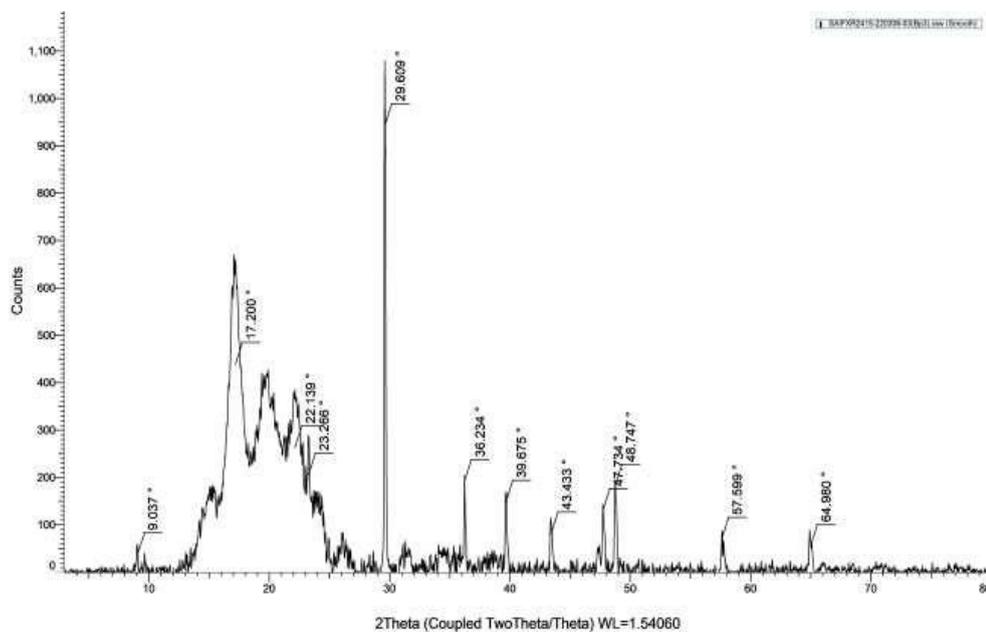


Fig15: XRD pattern of CS<sub>1b</sub>

### 3.2.4 XRD Analysis of CS<sub>1</sub>(GO<sub>2</sub>)

#### 3.2.4.1 XRD pattern of GO

Fig 16 shows XRD pattern of raw Graphite powder before process. From this XRD pattern it can be seen that the graphite has a diffraction peak at  $2\theta = 26.60^\circ$  and it is in accordance with which has been reported by many researchers [19].

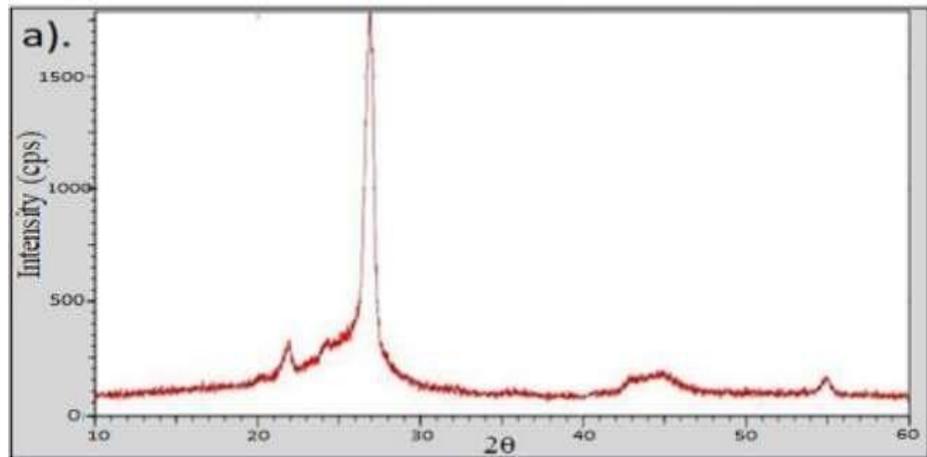


Fig 16: XRD pattern of raw Graphite powder before process <sup>[19]</sup>

XRD pattern of GO synthesized by Hummer's method is given below in the fig 17 .After the oxidation of graphite powder, the peak at  $2\theta = 26.60^\circ$  disappears and new peak appears at  $2\theta = 10.550^\circ$ . The diffraction peak of GO is broader which means that GO is a nanomaterial.

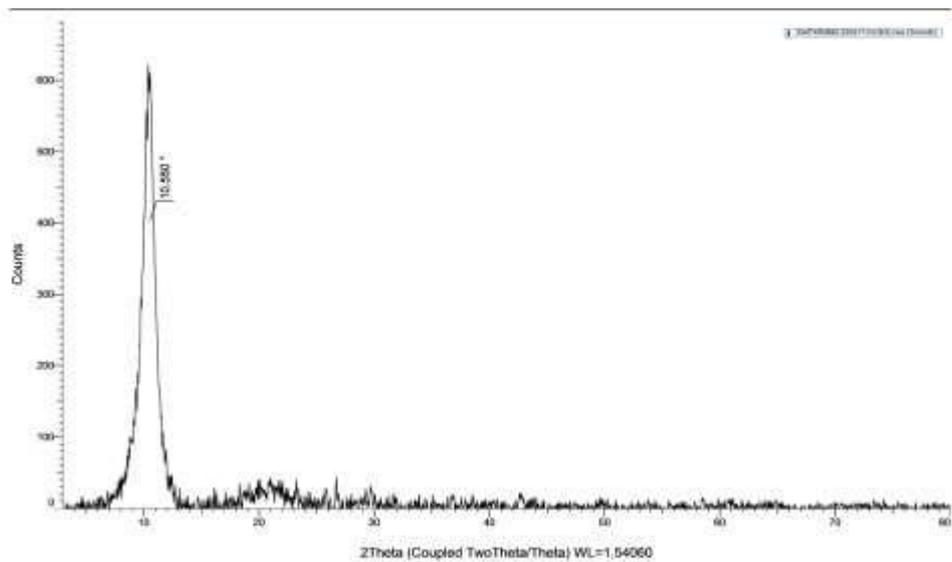


Fig 17: XRD pattern of GO synthesized by Hummer's method

### 3.2.4.2 XRD pattern of CS<sub>1</sub>(GO<sub>2</sub>)

The XRD pattern of Peak CS<sub>1</sub>(GO<sub>2</sub>) is given below in the fig 18. The diffraction peak at  $2\theta = 8.990^\circ$  shows incorporation of GO into the polymer. Thus we can ensure that the nanocomposite has been synthesized.

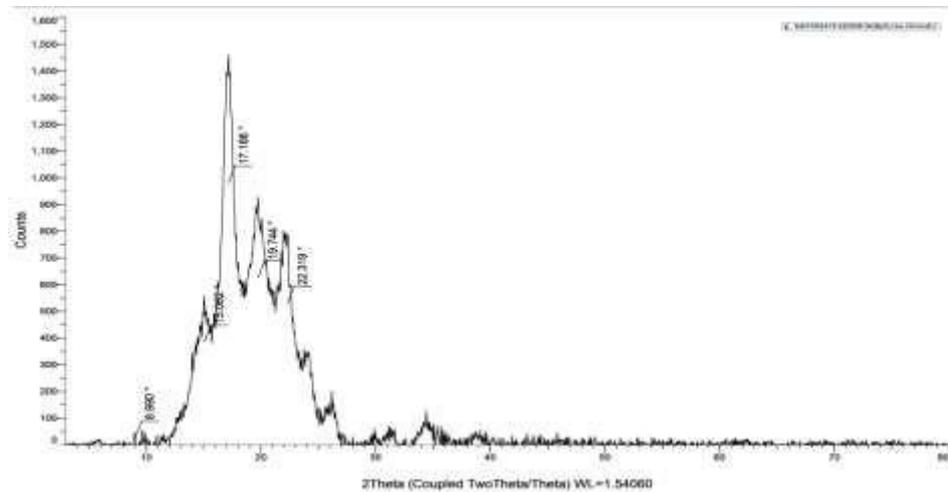


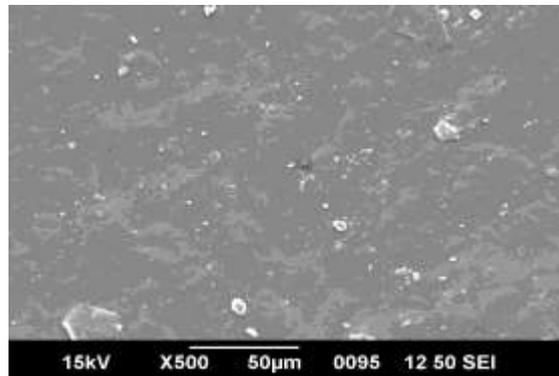
Fig18: XRD pattern of CS<sub>1</sub>(GO<sub>2</sub>)

### 3.3 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

SEM images of bio-plastics CS<sub>1</sub>, CS<sub>1</sub>b and CS<sub>1</sub>(GO<sub>2</sub>) were taken to study the surface change before and after the incorporation of reinforcement fillers. SEM images reveal that the incorporation does not cause any surface modification to the bio-plastic films. Surfaces should be found to be smooth and uniform before and after the incorporation.

### 3.3.1 SEM data of CS<sub>1</sub>

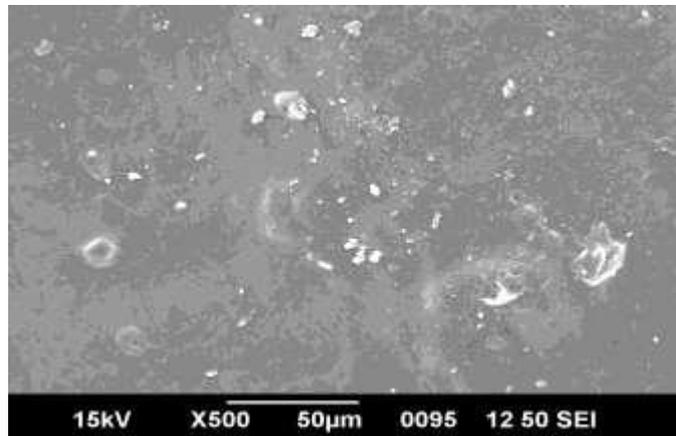
SEM image of CS<sub>1</sub> sample is given below in the fig 19. From the data, we can understand the surface morphologies of the CS<sub>1</sub> bio-plastic. Its surface is smooth and homogeneous. There is no cavities and cracks on surface. It indicates it is not brittle.



*Fig19: SEM image of CS<sub>1</sub>*

### 3.3.2 SEM data of CS<sub>1b</sub>

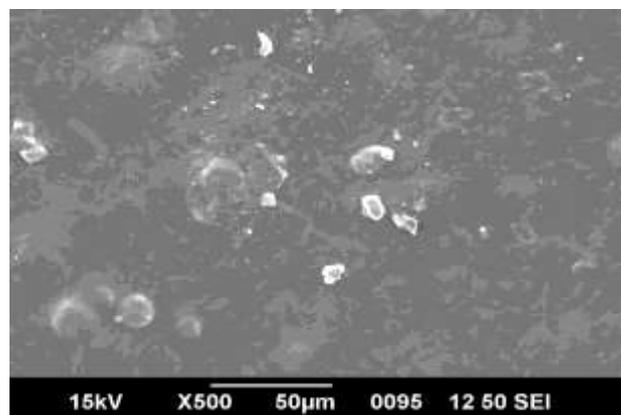
SEM image of CS<sub>1b</sub> sample is shown below in the fig 19. It exhibits the surface of CS<sub>1</sub>, which was observed a homogeneous and smooth structure with no cracks and obvious phase separation, and the CESP embedded in the starch matrix. This result demonstrated that the CESP particles are uniformly dispersed in the film matrix, and the CESP shows a better adhesion to the starch matrix caused by the organic components in the CESP <sup>[18]</sup>.



*Fig20: SEM image of CS<sub>1b</sub>*

### 3.3.3 SEM data of CS<sub>1</sub>(GO<sub>2</sub>)

From the fig 20, the morphology surface of CS<sub>1</sub>(GO<sub>2</sub>) synthesized. It can be seen that the surface of composite is relatively smooth with some blemishes. It indicated that the GO filler is capable to have good interaction in the composite, and the bio-plastic itself (without GO) also contributes to the formation of blemishes. There were no cracks in the surface and it indicates that the bio-plastic that was formed is note brittle.



*Fig21: SEM image of CS<sub>1</sub>(GO<sub>1</sub>)*

### 3.4 MOISTURE CONTENT

The moisture content for the various samples is calculated, and the results are shown in table 5.

| Sample                             | Initial weight , $W_i$<br>(grams) | Final weight , $W_f$<br>(grams) | Moisture<br>content<br>(%) |
|------------------------------------|-----------------------------------|---------------------------------|----------------------------|
| CSA                                | 0.2030                            | 0.1973                          | 2.8070                     |
| CS <sub>1</sub>                    | 0.3730                            | 0.3449                          | 7.4476                     |
| CS <sub>2</sub>                    | 0.4735                            | 0.4324                          | 8.6800                     |
| CS <sub>1</sub> b                  | 0.5992                            | 0.5793                          | 3.3210                     |
| CS <sub>1</sub> (GO <sub>1</sub> ) | 0.6314                            | 0.6100                          | 3.3892                     |
| CS <sub>1</sub> (GO <sub>2</sub> ) | 0.3633                            | 0.3518                          | 3.1654                     |

*Table 5: moisture content for the various samples*

The moisture content for corn starch samples was observed to increase with the addition of plasticizer [CS<sub>1</sub>, CS<sub>2</sub>, CS<sub>1</sub> b, CS<sub>1</sub> (GO<sub>1</sub>), CS<sub>1</sub>(GO<sub>2</sub>)], as unplasticized samples had the lowest moisture content. Of the plasticized samples, the moisture content was the highest for glycerol plasticized bio-plastics - CS<sub>2</sub>, while the lowest for sorbitol plasticized bio-plastic- CS<sub>1</sub>. As explained by Cerqueira et al., the probable reason for the obtained trend in moisture content can be the fact that glycerol contains hydroxyl groups that have strong attraction for water molecules, which allows them to form hydrogen bonds and hold water in their matrix. On the other hand, sorbitol

molecules have stronger hydrogen bonding with starch molecules; hence the attraction between sorbitol and water molecules is lesser, as compared to that between glycerol and water [1].

The addition of filler was seen to be inversely proportional to the moisture content, as increasing the amount of filler decreased the moisture content. The moisture content value is an important parameter for bio-plastics.

### 3.5 WATER SOLUBILITY

Water solubility of all samples was observed in Table 6.

| Sample                             | Initial weight,<br>$W_o$<br>(g) | Final weight,<br>$W_f$<br>(g) | Water<br>solubility in<br>(%) |
|------------------------------------|---------------------------------|-------------------------------|-------------------------------|
| CSA                                | 1.1923                          | 0.5343                        | 55.18                         |
| CS <sub>1</sub>                    | 0.1683                          | 0.1184                        | 29.64                         |
| CSGS                               | 0.9280                          | 0.4462                        | 51.91                         |
| CS <sub>1</sub> b                  | 0.6045                          | 0.3770                        | 37.63                         |
| CS <sub>1</sub> (GO <sub>1</sub> ) | 0.6430                          | 0.3720                        | 42.14                         |
| CS <sub>1</sub> (GO <sub>2</sub> ) | 0.3063                          | 0.1681                        | 45.11                         |

Table 6: Water solubility of all samples

Highest value of water solubility is for unplasticized sample CSA compared to the plasticized samples. Here also CSGS has higher water solubility than CS<sub>1</sub>. Reason is same that of moisture content analysis. The water solubility for CS<sub>1</sub> b, CS<sub>1</sub>(GO<sub>1</sub>), CS<sub>1</sub>(GO<sub>2</sub>) is very low when compared to CS<sub>1</sub> and

CSGS. In the case of  $CS_1(GO_1)$  and  $CS_1(GO_2)$ , the water solubility increases with the addition of the GO.

For a plastic to be good carrier, it should possess low water solubility. Otherwise the plastic will degrade easily when it comes in contact with water. So we can conclude that biocomposite  $CS_1 b$  is better for carrying.

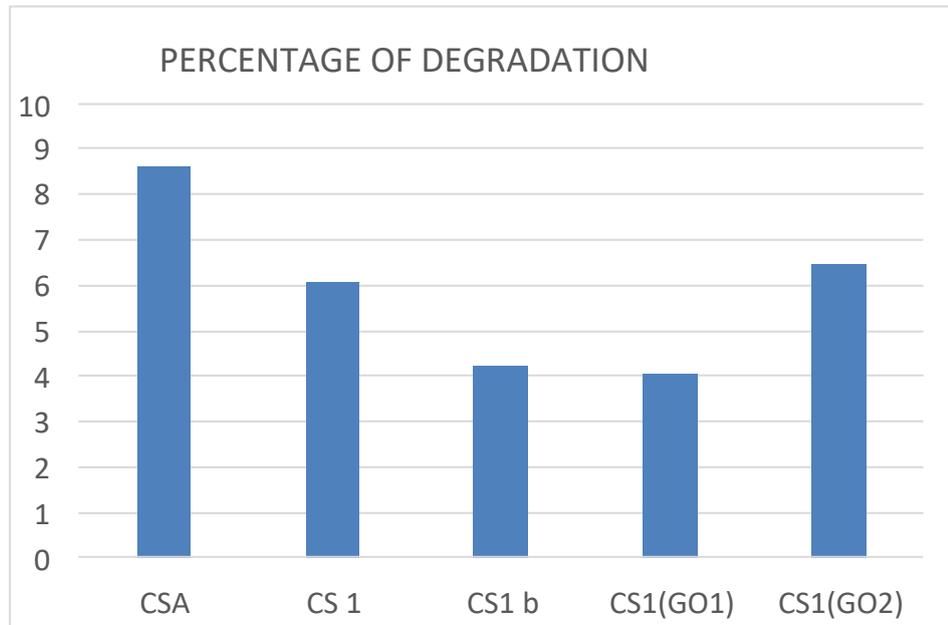
### 3.6 SOIL BIODEGRADABILITY TEST

Degradation of each sample in soil for first 14 days just after the synthesis is given below.

| Sample        | Initial weight | Final weight | Percentage of degradation |
|---------------|----------------|--------------|---------------------------|
| CSA           | 0.2800         | 0.2559       | 8.607                     |
| $CS_1$        | 0.3854         | 0.3605       | 6.460                     |
| $CS_1 b$      | 0.6428         | 0.6156       | 4.231                     |
| $CS_1 (GO_1)$ | 0.4119         | 0.3952       | 4.054                     |
| $CS_1 (GO_2)$ | 0.3511         | 0.3285       | 6.436                     |

Table 7: Degradation of each sample in first 14 days

Extent of biodegradability is given as graph below:



*Fig 22: Extend of biodegradability*

From this data we understand higher biodegradability rate in soil is for unplasticized bio-plastic CSA. A good bio-plastic means it has higher degradation rate in soil. From the above data it is clear that CSA is having good biodegradability. But its physical appearance is having surface cracks and it is so brittle. So we cannot say that CSA is good bio-plastic.

Other material with higher biodegradability rate is the nanocomposite.  $CS_1(GO_2)$  has higher biodegradation rate than  $CS_1(GO_1)$ . It means that when we increase the concentration of GO, it will easily undergo soil degradation. From the above data we can understand that the nano composite is better in the case of soil biodegradation.

### 3.7 AIR BIODEGRADABILITY TEST

Any of the samples do not degraded in the air in first 45 days just after the synthesis. It indicates the higher stability of synthesized bio-plastic in air. It means that we can use these materials as commercial packaging material. Figures of samples just after the synthesis and after 45 days are given below.



*Fig 23(a): CSA on the first day*



*Fig 23(b): CSA on the 45<sup>th</sup>*



*Fig 24(a): CS<sub>1</sub> on the first*



*Fig 24(b): CS<sub>1</sub> on the 45<sup>th</sup>*



*Fig 25(a): CS<sub>1b</sub> on the first*



*Fig 25(b): CS<sub>1b</sub> on the 45<sup>th</sup>*



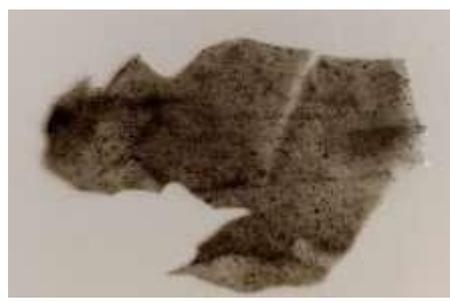
*Fig 26(a): CS<sub>1</sub>(GO<sub>1</sub>) on the first*



*Fig 26(b): CS<sub>1</sub>(GO<sub>1</sub>) on the 45<sup>th</sup>*



*Fig 27(a): CS<sub>1</sub>(GO<sub>2</sub>) on the first*



*Fig 27(b): CS<sub>1</sub>(GO<sub>2</sub>) on the 45<sup>th</sup>*

### 3.8 MECHANICAL PROPERTIES

Tensile strength is the amount of maximum strength needed to break the bio-plastics film. Tensile modulus is defined as the stress change divided by change in strain within the linear viscoelastic region of the stress v/s strain curves. Elongation at break is the indication of the amount of the variation of extreme film length while attaining tensile strength until the film breaks, related to the original length. Highest elongation break is for the sample CS<sub>1</sub>(GO<sub>2</sub>)

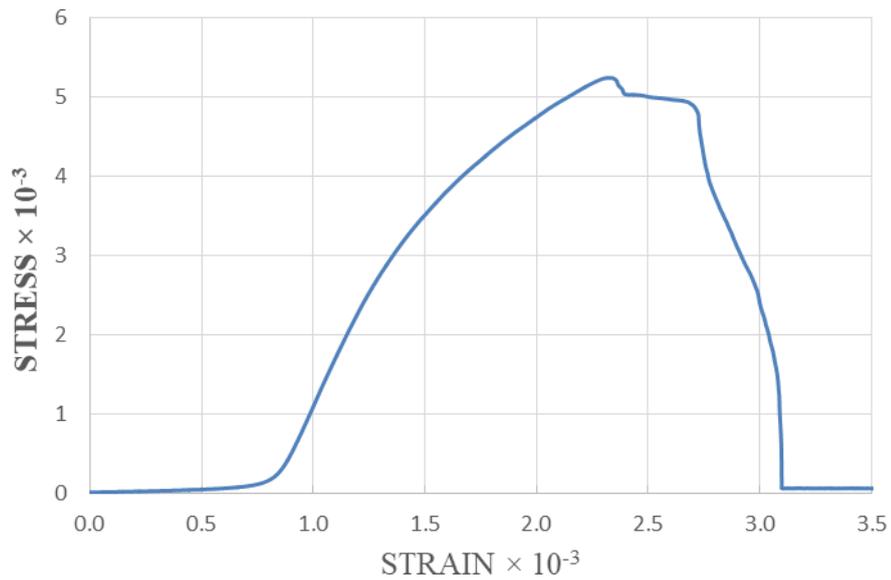


Fig 28: Stress-strain Graph for CS<sub>1</sub>b

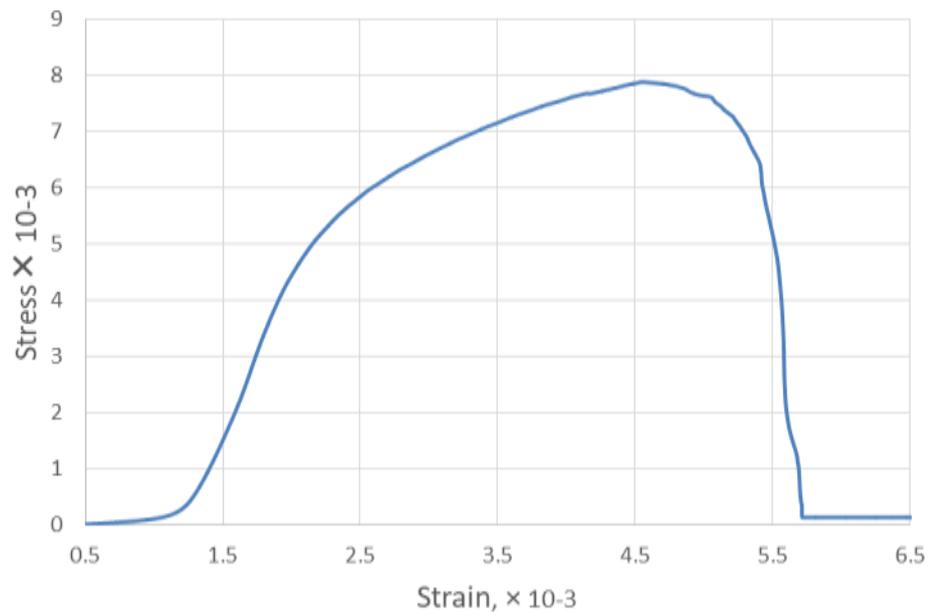


Fig 29: Stress-strain Graph for CS<sub>1</sub>(GO<sub>2</sub>)

The increase in tensile strength due to the addition of graphene oxide is caused by the strong interaction between the bio-plastic composite molecules with graphene oxide layers. In more detail, it is associated with the interaction between the hydroxyl (OH) group, either from the bio-plastic matrix or from the graphene oxide to form oxygen bridge. The stirring time affects the process of mixing between the composite matrix and the filler graphene oxide. This stirring time correspond to the filler particles distribution in the matrix cavity. The longer the stirring time (60 min.), the more GO particles will spread evenly within the matrix of bio-plastic. As the result, the better mechanical properties of bio-plastic will be gained.

### 3.9 THERMOGRAVIMETRIC ANALYSIS

The thermal degradation test for sample CS<sub>1b</sub> and GO added bio-plastic was performed and studied the weight loss, derivative weight % using TGA thermograms.

#### 3.9.1 Thermogravimetric data of CS<sub>1b</sub>

The thermogravimetric analysis graph of CS<sub>1b</sub> performed is given in the fig 29. The weight loss has been observed at a range, 100–200°C - no weight loss for the sample, in the range of 200–350°C- there is small weight loss up to 0.85 %, in the range of 600–800°C- no weight loss observed. Greater degree of weight loss was observed at 400°C. It indicated the evaporation of moisture from the bio-plastics. Major weight loss is in the range of 380 – 425°C. From this, we can say that the prepared samples can be used in the application operated at the elevated temperature.

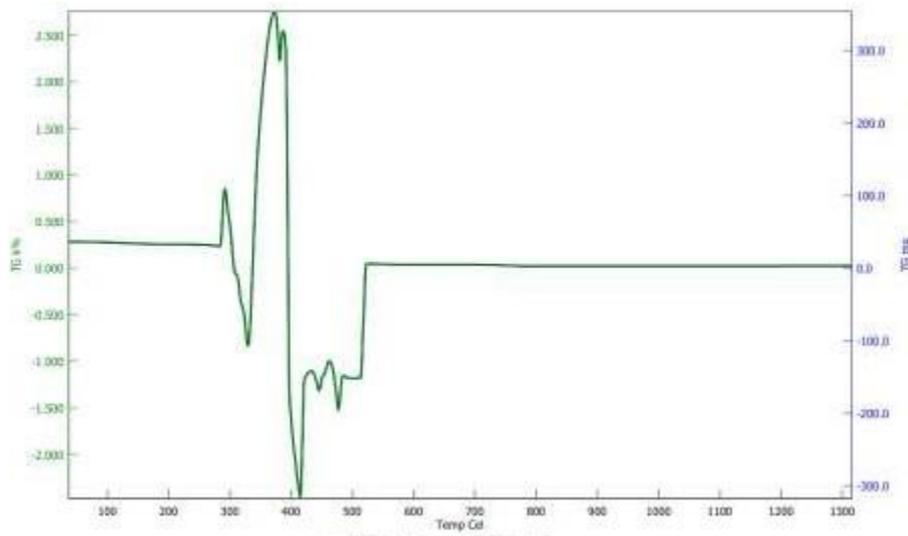


Fig 30: Thermogravimetric graph of CS<sub>1b</sub>

# Chapter 4

## CONCLUSION

Corn starch based biodegradable plastic was successfully synthesised. Comparative study of plasticizers showed that sorbitol is a better plasticizer for corn starch based bio-plastics. GO reinforced bio-plastic showed better mechanical properties compared to egg-shell reinforced bio- plastic. The relative moisture content was also higher for GO reinforced plastics which are an important parameter for biodegradability. All samples undergo degradation when it kept in soil by the microorganisms. It exists in air without any fast degradation. Because of the longer shelf life, it can be used as commercial packaging material. When we compare all of analysis results along with SEM images about morphology we reach a conclusion that polymer Nano composite is better plastic. It can be used as better packaging material in future. It provides solution for problems facing by our society because of plastic pollution.

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