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# Material characterization of starch derived bio degradable plastics and its mechanical property estimation

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## Abstract

The extraordinary versatility and manufacturability of plastics has tremendously benefited the society in terms of economics, however, this prosperity paid a price in the form of depleting fossil fuels and adverse effects on the environment. In an attempt to minimize these undesirable consequences, researchers all over the world focused on establishing alternative sources that are renewable, sustainable and biodegradable which led to the development of bioplastics, where 100% of the carbon is derived from renewable agricultural sources. Bio plastics are considered to be superior to fossil fuel derived conventional plastics in terms of energy efficiency, petroleum consumption and carbon emission, but inferior in applicability. Any breakthrough in improving the manufacturability of bio plastics can help reduce dependency on petroleum-based polymers, accumulation of persistent plastic waste and better control over CO<sub>2</sub> emissions in the environment. This paper investigates starch bioplastics for biodegradability and mechanical properties like hardness and impact strength. It also attempts to characterize bioplastics through techniques like thermo gravimetric analysis and Fourier transform infrared spectroscopy analysis.

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*Keywords:* Bio plastics; starch; Biodegradation; Material characterization; Mechanical property estimation

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## 1. Introduction

Growing concern about the challenges faced in plastic waste disposal and recycling clubbed with non-renewable nature of raw material (petroleum) behind its production has pushed researchers to explore the possibilities of

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transforming renewable biomass to commercially viable polymers known as bio plastics. Since the early 70's, there is an increasing awareness over environmental impact of plastic waste, and a steady parade of innovators focused on creating more eco-friendly alternatives [1]. Several of the successfully deemed materials failed to meet the demanding price and performance specifications of manufacturers. But now the scenario has reversed its course of direction and bio plastic materials and products reached a crescendo during 2010, as more mainstream companies have decided to use bio plastics derived from agricultural feed stock like molasses, sugarcane juice, extract from non-edible cashew nut shells, plant cellulose or corn starch [1,2].

Biodegradable plastics are a promising alternative to petroleum plastics and they undergo a composting process to form a stable compound, in the final stage of recycling. Starch-based bio plastics represent from 85% to 90% of bio plastics available in the market [2]. Starch based bio plastics are made from native starch or slightly modified starch, either isolated or blended with natural/synthetic molecules. Cellulose based plastic is another example of a bio plastic made out of a naturally occurring polymer. Cellulose and Starch do not need to be polymerized since they are already polymers. One among the most promising bio plastics is PLA (polylactic acid) which is a result of polymerization of lactic acid through the fermentation of starch. Bio plastics like bio-polyethylene are identical to its oil derived form with an exception of difference in source of monomers. But most are unique and have no petro-polymer equivalent. Similarly some bio plastics are biodegradable while others are not. This is also necessary, as many products need to be resilient and should not degrade in the environment very easily [3].

The feed stocks for bio plastics come from a variety of biological sources. Starch based bio plastics are usually made from wheat, corn, rice, potatoes or barley while cellulose based bio plastics are commonly made from wood. Natural oils from soy, palm and other plants have also been used while some others like hyaluronic acid (HA) which is an animal derived monomer rely on bacteria to supply raw materials. Starch based plastics are thermoplastics blended with plasticizers and additives like sorbitol or glycerin. Pure starch absorbs water and disintegrates in liquids and this fact makes it ideal to be used for drug capsules. Esters and Ethers of starch also behave as thermoplastics but have been too expensive to put into production [3,4].

At present, bio plastics can contaminate conventional plastics sent to recycling, and the low volume of bio plastics in use does not give recyclers much incentive to invest in new technologies for identifying and separating bio plastics. To put into perspective the global consumption of plastics is about 200 million tons, whereas the market share of bio plastic is only 5-10%. The efficient conversion of biomass in to bio plastics is reckoned to reduce the challenges faced in bio waste recycling if it could substitute petroleum derived plastics for various applications. The field of bio plastics is still in its infancy but is developing rapidly and bio plastics have yet to reach the performance and level of diversity of petroleum based synthetic plastics. This paper attempts to characterize starch based bio plastics indigenously prepared from wheat starch through techniques like thermo gravimetric analysis, Differential scanning calorimetry and Fourier transform infrared spectroscopy analysis. It also investigates on the bio degradability of bio plastic in real world conditions.

## 2. Literature review

Biodegradable materials can be classified into three: [5]

- a) Agro based polymers blended with biodegradable synthetic polymers;
- b) Microbial polymers, made by fermentation of agricultural products. It includes Polyhydroxy Alkanoates, or PHA and PHBV (Polyhydroxybutyrate covalerate).
- c) Monomers or oligomers polymerized by means of conventional chemical processes and obtained from the fermentation of agricultural raw materials like PLA.

While PCL and PLA are the most used polymers in the market, other than starch derivatives, a fourth class is also added to the above-mentioned ones: [6]

- d) Derivatives of petrochemical origin by means of synthesis, representing several polymers and subgroups:
  - The polycaprolactones: PCL;
  - The polyesteramide: PEA;
  - The aliphatic copolyesters or Polybutylene Succinate Adipate: PBSA;
  - The aromatic copolyesters or Polybutylene adipate co-terephthalate: PBAT.

## 2.1 Starch-based plastics

Starch consists of the branched, polymerised amylopectin that surrounds the non branched amylose. For the production of bioplastics, the starch polymer and its monomer, glucose is used. Through biotechnical and/or chemical processes, this is converted into thermoplastic polyester and polyurethane. The milled products flour, as well as pellets or powder made from grain, potatoes or maize, are also economical raw materials. The by-products of the starch industry can be recycled as raw materials for fermentation processes [7].

Starch in its granular state has been used as a filling agent for polyolefin and a component in synthetic polymers blends. They have also been modified by means of grafting with vinyl monomers (e.g., methyl acrylate). A silane with a general formula of  $\text{CH}_3\text{-Si-O-(R}_1\text{,R}_2\text{,R}_3\text{)}$  can be added to a blend of Poly Ethylene to improve the compatibility of these two materials [8].

## 2.2. Thermoplastic-like Starch (TPS)

Even though starch is not a thermoplastic, starch based bio plastics melts at high temperatures (90–180°C) and tends to be fluidic under shearing. The presence of plasticizers like water, glycerin or sorbitol also contributes to this property which makes them fit to be used in injection, extrusion and blowing equipments. The semi-crystal granular structure of starch has to be retained so that it remains as a thermoplastic [9]. The water is a primary plasticizer and when added to the starch acts as an agent to break the structure of the native granule, i.e., breaking the bonds of hydrogen chains. However, additional plasticizers like Polyol are required, which are less influenced by atmospheric conditions, and that allows a melting phase at a temperature lower than that of starch degradation.

Table 1: Properties of wheat starch-based TPS, using different plasticizer levels

Sl. No:	Starch (%)	Moisture Content (%)	Glycerol content (% starch)	Density (Kg/m <sup>3</sup> )	Glass transition (°C)	Elastic Modulus (MPa)
1	74	16	14	1.39	43	997
2	70	12	26	1.37	8	52
3	67	9	36	1.35	-7	26
4	65	0	54	1.34	-20	2

The low resistance to water and the variations in mechanical properties under humid conditions restrict the wider utilization of starch as a bio plastic. Starch derivatives present a high permeability to moisture and degrade rapidly and have inconsistent mechanical properties that alter with time, apart from their low resistance to impacts. The plasticizer content in the sample affects the mechanical properties of the material (Table 1). Starch biodegradable plastics incorporated with biodegradable, renewable or synthetic polymers could improve the impermeability [8,10].

## 3. Experimental

The source of the raw starch is usually corn but could be any plant high in starch. This study used gluten-free wheat starch for preparing bio plastic.

### 3.1 Preparation of bio plastic

Wheat starch along with glycerol as a plasticizer and acetic acid is heated and agitated in a process to prepare bio plastics. Plasticizers are embedded between polymer chains, thus spacing them apart and lowering the glass transition temperature resulting in softening of plastic [3].

### 3.2 Thermogravimetry analysis

Thermogravimetric Analysis (TGA) measurements were carried out on approximately 3 mg of wheat starch based bio plastic powder samples at a heating rate of 100C/min using a Simultaneous Thermal Analyser (PERKIN

ELMER STA 6000). Figure 1 represents the TGA curves of boron treated and untreated Medium Density Coir Board samples. Thermal decomposition of each sample occurred in a programmed temperature range of 400C to 7000C. The continuous weight loss and temperature were recorded and analysed during Thermogravimetric analysis.

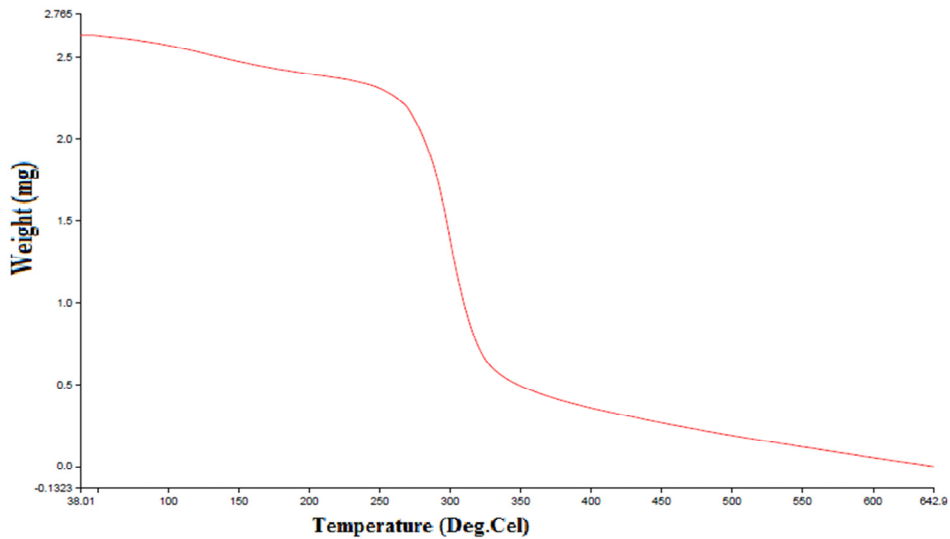


Fig. 1. Weight v/s Temperature graph

### 3.3 Differential thermal analysis

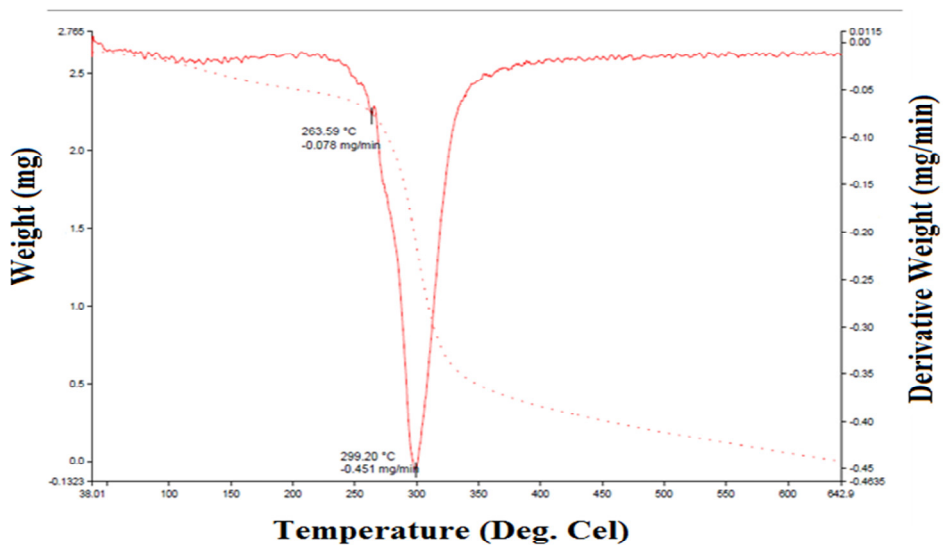


Fig. 2. Derivative weight vs Temperature graph

Differential thermal analysis was done on the same sample simultaneously to obtain the curves shown in figure 2. The measurements were carried out on wheat starch based bio plastic powder samples at a heating rate of 100C/min

using a Simultaneous Thermal Analyser (PERKIN ELMER STA 6000). The continuous weight loss and temperature were recorded and analysed during Differential thermal analysis.

### 3.4 Fourier transform infrared spectroscopy

The infrared spectra of wheat starch based bio plastic powdered samples were recorded on Fourier transform infrared spectroscopy (FTIR) Spectrophotometer. Infrared assignments for absorption peaks of samples are shown in Table 2.

Table 2 Infrared assignments for absorption peaks of samples [11]

wave number ( $\text{cm}^{-1}$ )	Assignment
3500 - 3300	O–H stretching
2800 - 3000	C–H stretching in methyl and methylene groups
2920 - 2850	Aliphatic $\text{CH}_2$ asymmetric stretch
1750 - 1730	C=O stretching in carbonyl
1640 - 1618	C=C alkenes
1510 - 1504	C=C aromatic skeletal vibrations
1462 - 1425	$\text{CH}_2$ lignin
1350 - 1310	Phenol–borate B–O
1260 - 1234	C–O stretching vibration in lignin and hemicelluloses
1170 - 1153	C–O–C asymmetric bond
756	Out-of-plane ring deformation
691	COH out-of-plane ring bending

The chemical bonds in the sample after chemical treatment were investigated using FTIR spectroscopy. The FTIR spectrum of starch based bio plastic is shown in Figure 3. The transmittance range of scan was 500 to 4000  $\text{cm}^{-1}$ .

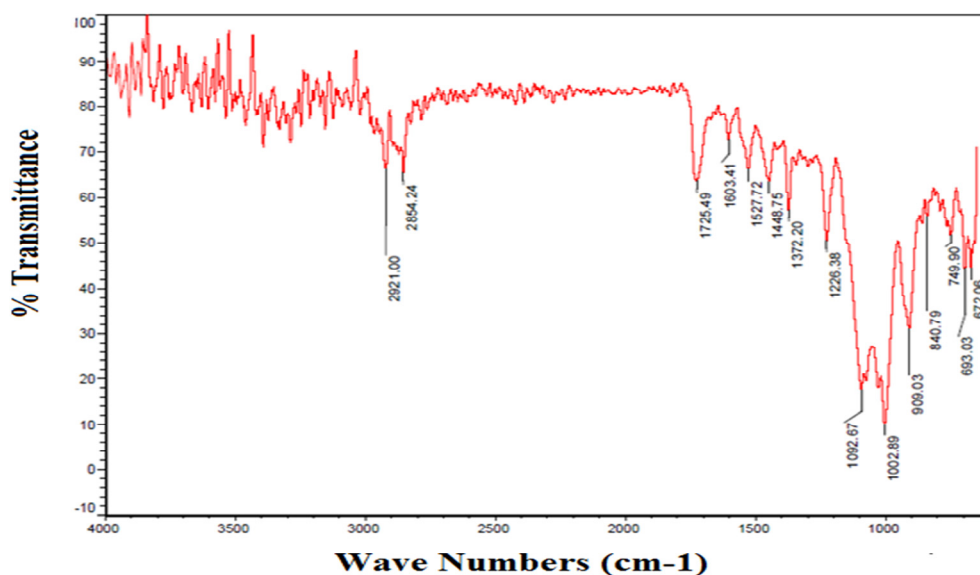


Fig 3. FTIR spectra (Percentage Transmittance vs Wave number)

From the spectrum it is inferred that there are (C–H) stretches, C=O stretch aldehyde, C=O stretch in carboxylic acid, aromatic combination, Methyl, C–C stretches, Alkyne C–H bond stretches present in the samples.

### 3.5 Mechanical property estimation

Bio plastic samples were subjected various mechanical testing as per ASTM standards to find the properties and were compared with other synthetic plastics. It is observed that the virgin bio plastic without added additives or impurities returned lower values when compared to synthetic plastics. Table 3 shows a comparison of some mechanical properties of common synthetic plastics [12] to that of the indigenously prepared bio plastic samples. The presence of agar in star based bio plastics had improved the properties.

Table 3. Comparison of mechanical properties of synthetic plastics to starch based TPS

	Tensile strength MPa	Izod impact J/cm	Flexural modulus GPa	Rockwell Hardness scale	Water absorption percentage Immersion of 24 hrs
Bioplastic	40.3	0.115	4	92.5 HRC	14.9
ABS	97.2	4.11016	2.09	102 HRC	0.30
Nylon	85.5	0.640544	2.82	121 HRC	1.20
PET	79.3	0.373651	2.76	125 HRC	0.10
Polypropylene	37.3	0.640544	1.55	95HRC	Slight
PTFE	18.7	1.86825	0.5	58 HRC	<0.01
PVC	51.7	0.533787	3.32	115 HRC	0.06

### 3.6 Biodegradability testing

The biodegradability of wheat starch based bio plastic samples was investigated in different controlled environments [13,14]. Three different samples of 50 gm weight were observed in conditions favouring accelerated disintegration. The first sample was buried under moist soil, the second sample was immersed in a jar of sea water and the third sample was buried in vermicompost.

Figure 4 shows the relation of weight loss with respect to time required for decomposition of bio plastics in different controlled environments. From the graph we can infer that about 90% of starch derivative bio plastic could get degraded in 5-6 months.

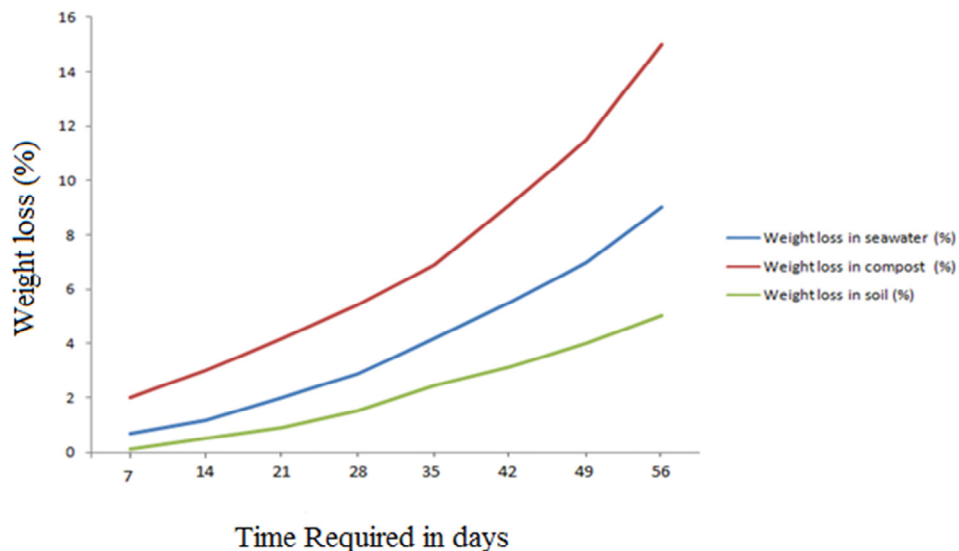


Fig. 4. Weight loss (in %) starch derivative bio plastic in sandy soil

Table 3. Percentage weight loss of samples under degradation

Time required in days	Weight loss in soil (%)	Weight loss in sea water (%)	Weight loss in compost (%)
7	0.1	0.7	2.0
14	0.5	1.2	3.0
21	0.9	2.0	4.2
28	1.5	2.9	5.4
35	2.4	4.2	6.9
42	3.1	5.5	9.1
49	4.0	7.0	11.5
56	5.0	9.0	15.0

The weight loss of samples in all the three conditions was recorded and it was clear that an accelerated weight loss occurred in the third sample where the sample exhibited more disintegration. Table 3 shows the weight loss of samples for a period of time in varied conditions.

#### 4. Results & Discussions

TGA analysis of the samples confirmed that there was only little moisture content present in the samples as evident from the curve. The sample showed no signs of degradation up to 60<sup>0</sup>C and thereafter succumbs to thermal degradation. A sudden dip is observed at around 250<sup>0</sup>C which indicates the maximum possible temperature before the material lost its form. After this point they showed significant degradation with a 80% loss in mass by around 330<sup>0</sup>C.

In DTA, the derivative weight and temperature shows the rate at which the weight is lost with the change in temperature. At a temperature of 263.59 <sup>0</sup>C derivative weight of the specimen decreases at a rate of 0.078 mg/min. With further increase in temperature derivative weight decreases and reaches its maximum value of 0.451 mg/min at 299.20 <sup>0</sup>C. It shows starch derivative bio plastic has thermal stability up to a temperature of 263.59 <sup>0</sup>C.

The FTIR spectra of bio plastic samples confirm that they could substitute for synthetic plastics like polyethylene and polystyrene in several applications. Mechanical property estimation confirmed that the virgin bio plastics produced from pure starch without impurities or added fibre/impurities lacks far behind that of synthetic plastics and the bio degradability tests confirmed that they disintegrate over a period of time.

#### 5. Conclusion

Wheat starch was used to prepare bio plastics and they were subjected to several tests to identify their mechanical and thermal characteristics. Presence of impurities in the form of fiber or blends of other polymers tends to improve the properties of starch based bio plastics. Adding nano materials to starch based TPS is a promising area to further research. They were found to be bio degradable while every bio plastic is not bio degradable. That offers a possible solution to the huge piles of plastic waste in our country. But bio plastics have still a long way to go before establishing it as a viable substitute for synthetic plastics. These materials are expected to be used to its maximum possible extent in the near future.

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