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Investigation of Mechanical and Thermal Properties of Biodegradable Rod (Banana Waste/PLA) Developed from Extrusion Process

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Abstract

In this research paper, a novel idea of developing the biodegradable rod comprised of banana waste reinforcement in PLA matrix has been proposed. Rod is comprised of waste of banana leaves reinforced in PLA matrix with volume fraction of (0%, 15%, 25% and 35% respectively). Samples were developed using single screw extruder as per ASTM standards. During testing of the samples it has been observed that Mechanical Strength in terms of tension and flexural goes upto 23.41 and 21.05 MPa with 25% volume fraction respectively from 15.82 MPa found in case of pure PLA rod in terms of tension and 13.64 MPa in terms of flexural strength. Increase in reinforcement results into lack of strength due to imperfect bonding between reinforcement and matrix. Failure areas were explored with SEM analysis. Differential scanning calorimetry is done to check the thermal strength of the composite rod.

Keywords: *Single Screw Extruder, Polylactic Acid, Differential Scanning Calorimetry, Bannana Fibers, Mechanical and Thermal Strength.*

1. Introduction

In today’s scenario, due to the industrialization, the environmental degradation is attracting the people all over the globe to think about the future material that could be more sustainable and eco-friendly in nature. These biodegradable plastics and other bio based polymers when used as composites can replace the petro based polymers in the market up to the large extent. Composites are classified as non-biodegradable and biodegradable in nature. Biodegradable can be partially or fully depending upon the type of matrix and reinforcement.

1.1 Introduction to Polylactic Acid (PLA)

Polylactic acid (PLA) belongs to the group family of aliphatic polyesters that are usually made from hydroxy acids that comprises of polyglycolic acid that is mainly considered as biodegradable. PLA are high modulus polymers having high strength and of thermoplastic in nature that are usually derived from renewable resources on the earth for their application in various fields like industrial and medical sciences. Due to its biodegradable nature and having good properties, PLA has been widely used in different sectors of engineering industries like packaging (films and trays for biscuits, fruit, vegetables, and meat), agricultural (mulching films), furniture, electrical and electronic appliances (CD’s, computer keys, casings etc), houseware and other fibers or fabrics (t-shirts, socks, blankets, wipes, hygiene

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products, diapers) [17]. The reason is not only that PLA is biodegradable but also due to its performance and properties at a reasonable price. So its use is increasing at a very rapid rate. PLA also proved useful in biomedical science for making various internal components of the body like rods in bones, ankle screws, implants and drug delivery systems [8,6,10]. The main source of making PLA is lactic acid (monomer) produced by chemical synthesis or fermentation. Presently two types of configuration of PLA are there named L (+) and D (-) isomers which are produced from the carbohydrates by bacterial fermentation. Out of these above two methods of making PLA, chemical synthesis is not preferred in most of the cases specifically in industries due to the cost of the process and dependency of the process on the other one [3]. So homofermentative method consist of species of Lactobacillus is widely used due the advantages like lesser by products formation and greater lactic acid yielding. Pure L-Lactic acid is used in production of PLA maintaining the pH (5.6-6.4), temperature 38-42^{o}C and low oxygen concentration [9].

There are two main methods of synthesis of PLA from the lactic acid: Low Molecular Weight PLA by Condensation Polymerization and High Molecular Weight PLA by Ring-Opening Polymerization of Lactide. In medical field, PLA has been used as a drug delivery matrices and internal fixation of fractured bones [16]. In terms of agriculture, it has been used as growth promoter in plants, textiles and crop covers etc. Now a day's PLA has become an alternative to HIPS, PET, and PVC. This novel matrix has a forging role in containers and thermoformed cups. PLA has glass transition temperature ranging from 50-59^{0}C and melting point of 159-178^{0}C for its different process grades [4]. The properties of PLA as compare to other polymers in terms of mechanical strength are given in the table no 1**.** Due to the awareness and increasing attention towards the development of biodegradable products, the monomers are being manufactured from the various renewable feed stocks which inturn bring the PLA as a emerging solution to the plastic industry worldwide. The better methods of processing and improvement in the intrinsic properties of these polymers have increased the interest of commercial sector to make PLA products.

Table 1 Properties of polymers [18]

Properties	PLA	PP	PET	Nylon	Cellophane
Density (g/cc)	1.25	0.9	1.4	1.2	1.45
Haze (%)	2.1	1-4	2-5	2-3	1-2
Tensile strength (psi)	15950	27550	29725	36250	13050
Tensile modulus (psi)	478500	348000	551000	264625	594500
Ultimate elongation (%)	160	110	140	125	23

1.2 Introduction to Reinforcements

Reinforcement is defined as an incorporation of a woven or non woven structure of fibrous material which is used to increase the physical properties of the composite that in-turn increase the strength of the neat resin. Different reinforcements have different properties that affect the final properties of the composites. Therefore the selection of reinforcement plays a major role in the making of the composite and its desired properties. Reinforcements could be particles of fabrics, fibers or whiskers as shown in figure no 1. Fibers are characterized as long axis having near circular cross-section. Particles orientation is not fixed in particular direction but on the other hand whiskers have defined shape having very less dimensions (diameter and length) as compare to the fibers. Flakes are used instead of fibers due to their dense packed structure and also they are less costly and can be produced in an economic way

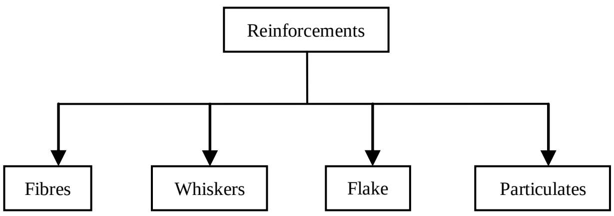

Figure 1: Common forms of reinforcements

1.2.1 Reinforcement and Treatment of Natural Fibers

Artificial fibers like aramid, glass and carbon are being used from a long time but the natural fibers have attracted the researchers due to their availability, less cost etc. due to the low density of these natural fibers (jute, kenaf, flax, sisal etc.), they are highly recommended in automotive industries. Reinforcements again could be biodegradable or non biodegradable. They may be metallic, wood based, plant and animal based. Mostly all the fibers are being used as the reinforcement in the matrix. Reinforcement may be in the form of yarn form, continuous, discontinuous, chopped form etc. The natural fibers extracted from the plants, animals are not being directly used for the purpose of reinforcements as they have impurities and the cellulose etc. which decreases the bonding strength of the fiber with the matrix which inturn affect the properties of the final composite. Lignin, cellulose, waxes, hemi-cellulose are the main components of natural fibers. Some selected fibers' components are given in table no 2.

Table 2 Chemical composition of natural fibers [11,12]

Fiber	Lignin (%)	Cellulose (%)	Hemi-cellulose (%)	Pectin (%)	Ash (%)
Kenaf	15-19	31-57	21.5-23	-----	2-5
Jute	12-26	45-71.5	13.6-21	0.2	0.5-2
Hemp	3.7-13	57-77	14-22.4	0.9	0.8
Sisal	7-11	47-78	10-24	10	0.6-1
Abaca	7-9	56-63	15-17	-----	3

Cellulose being a semi crystalline polysaccharide comprised of large hydroxyl group which gives hydrophilic properties to natural fibers that result into very poor interface and in-turn leads to moisture absorption [2]. Due to the open structure of hemi-cellulose having acetyl and hydroxyl groups, it is hygroscopic and little soluble in water. On the other hand Lignin being amorphous has least water sorption of Natural composites [5]. So to increase the bond strength of the composites, the natural fibers are being chemically treated. So there are various methods of treating these fibers like alkaline treatment, silane treatment, acetylation, benzoylation etc. Alkaline treatment is being commonly used by the researchers when reinforcement is done to either thermoplastics or thermosets. The important function of alkaline treatment is to disrupt the hydrogen bonding in the structure thereby increasing the roughness of the surface of fibers. In this treatment wax, lignin, oils etc covering the outer portion of the fiber has been removed up to certain extent by immersing the fibers into solution of NaOH for the required period of time which in turn increase the strength of the fibers when reinforced into the matrix [11,14]. Silane (SiH_4) makes the glass fibers properly adhere with the polymer matrix which makes the composite stable. It forms the stable covalent bond by reducing the hydroxyl group of cellulose at the interface of fiber and matrix [1]. It was been confirmed that silane treated fibers are much stronger than the alkaline treatment which leads to the higher tensile strength [15]. Acetylation is an etherification process results into plasticization which involves generation of acetic acid as a byproduct that must be removed from fiber to make it hydrophobic [7]. Benzoylation improve the interaction with hydrophobic by treating the fibers with the benzoyl chloride [13]. Benzoylation improves adhesion of fiber and matrix that increase the strength of the composite by decreasing the water content and improving the thermal stability of the fiber in the matrix.

1.3 Methods of making composites

There are different manufacturing techniques of making the composites. For good processing of composite there must exist good bonding between fibers and matrix. Extrusion process is one common method of manufacturing composites. Extrusion process has been used to mix up the required ratio of fibers and matrix with the use of heat and forward rotating screw. Extrusion process is of two types: single screw extruder and twin screw extruder. Single screw extruder comprised of single screw rotating inside the periphery of barrel where barrel are covered up by the heaters to set the temperature range accordingly. The screw inside the extruder barrel is rotated at the predetermined speed with the help of motor that is a drive unit and die has been attached at the end of the barrel from where material has been pushed to the required shape as that of the die.

2. Processing of Materials

Treatment of natural fibers is very much required to avoid the weak bonding at the interface. In this experimentation fibers were crushed or chopped from leaves (shown in figure no 2) into required size (shown in figure no 3) and then to treat them perfectly, water is heated upto 60°C and then the fibers were dipped into that water and washed for

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around 1 hour. Thereafter they were dried for 2 days in open air. 10% NaOH sol is used to neutralize the fibers by removing excessive lignin form them and again washed with fresh distilled water. Again they were dried in sunlight for complete 1 day alongwith PLA pallets. PLA pallets were oven dried at 70°C for around 2 hours to completely remove the moisture from them.

3. Development of Rod

The mixing of PLA and banana waste has been done in the proper ratio in terms of volume percentage of fibers in the mixture. The ratio has been set in figures of 0%, 15%, 25%, and 35% respectively. To mix the PLA granules and banana waste fibers in chopped form, binder (coconut oil) has been used, so that fibers can stick properly to the granules. Thereafter the mixture is put into barrel of single screw extruder through the hooper. The speed of the screw is set to 30 rpm and temperature of the barrel ranges from 130^{o}C to 160^{o}C at three steps as shown in figure no 3.

(a) (b)

Figure 2: (a) banana leaves, (b) chopped fibers

Figure 3: Single screw extruder

Rods at different fiber ratios (0%, 15%, 25%, and 35%) were developed as shown in figure no 5 as per ASTM D7205. The diameter of the rod is 12.7 mm and length is 1143 mm.

(a) (b) (c) (d)

Figure 4: Composite rods comprised of (a) neat PLA, (b) 15% fiber fraction, (c) 25% fiber fraction and (c) 35% fiber fraction

4. Results and Discussion

Tensile testing (UTM of capacity 100 kN (Model HEICO- HLC 693.40) as shown in figure no 5(a) and flexural testing (UTM of capacity 10 kN (Model Shanta Engineering) as shown in figure no 5(b) has been done to check the strength of the composite rod.

(a) (b)

Figure 5: (a) Tensile and (b) flexural testing

The following results were obtained while checking the strength at the rod at different fiber fractions of 0%, 15%, 25% and 35% respectively as shown in table no 3.

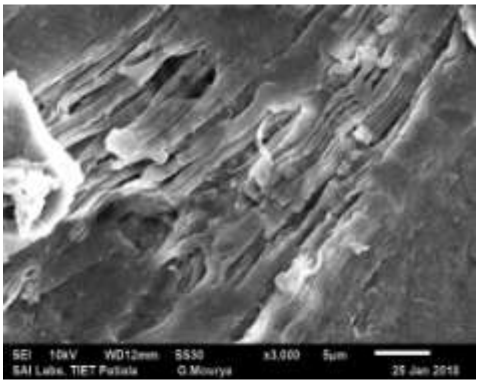
Table 3 Tensile and flexural strength of the composite rod.

Sample No	Fiber fraction (%) v/v	Tensile strength (MPa)	Flexural Strength (MPa)
P1	0	15.82	13.64
P/B1	15	20.88	18.67
P/B2	25	23.41	21.05
P/B3	35	20.67	20.11

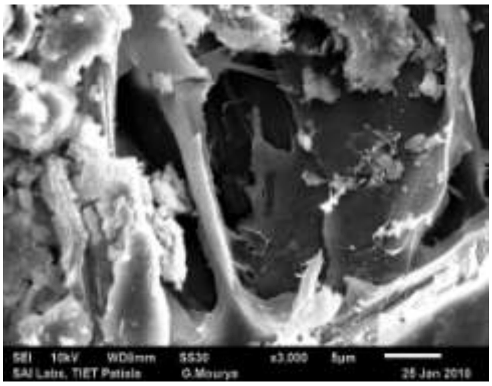
The results show that with neat PLA, rod's strength comes out to be 15.82 MPa in tension and 13.64 MPa in flexural. With the reinforcement the strength increases and goes upto 23.41 MPa in tension and 21.05 MPa in flexural with 25% reinforcement of fibers. With 35% reinforcement, strength reduces. The reason being improper mixing and binding of the fibers with PLA which results into porosity.

5. SEM Analysis

A scanning electron microscope (SEM) scan the surface with electron beam and produce the image of sample that produce the signals showing composition and surface topography. It has been done on SEM (Model SE 6510LV).



(a)



(b)

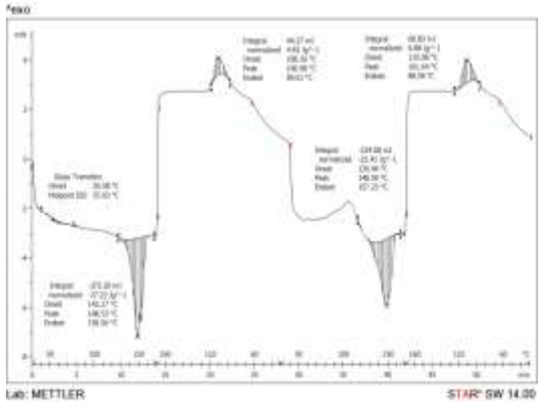
Figure 6: SEM images showing (a) uniform mixing of fibers and (b) fracture zone at 25% fiber fraction

The SEM images show the uniform presence of fibers in the composite rod as shown in figure no 6 (a) and possible failure zone after failure of the specimen as shown in figure 6 (b).

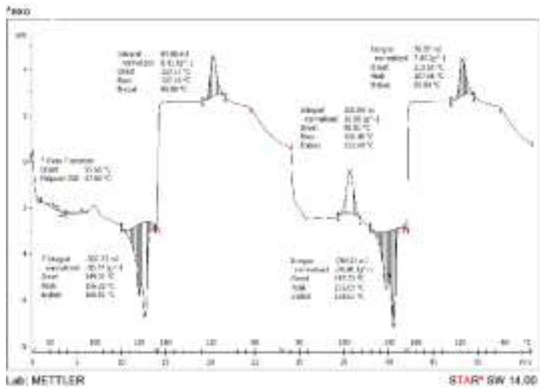
6. Thermal Analysis

Thermal analysis is a science where the properties of the materials are studied with the change in the temperature. DSC is one very common technique that is used to measure the energy absorbed or released by the sample when it is subjected to heating and cooling which provide the data related to exothermic and endothermic process. It has been done on DSC 3 star system by METTLER TOLEDO as shown in figure no 7.

Figure 7: DSC setup



(a)



(b)

Figure 8: DSC curves (a) Neat PLA rod and (b) 25% fiber fraction composite rod

DSC curves provide data about thermal changes in the material when 2 cycles are done. It has been shown that with the addition of fibers in the PLA, thermal stability improves. As shown in figure 8 (a), the melting starts from 142 °C and in reinforcement it starts at 149 °C as shown in figure 8 (b).

7. Conclusions

The tensile strength of neat PLA rod comes out to be 15.82 MPa while in case of composite rod comprising of chopped banana fibres and PLA, increase from 20.88 MPa to 23.41 MPa when fibre fraction increased from 15 to 25% by volume while in case of 35% fiber fraction, the values goes to down to 20.67 MPa and 20.11 MPa in case of tensile and flexural strength respectively. The reason being improper bonding and mixing of mixture (fibers and PLA). SEM micrographs validated the results of mechanical testing by showing surface morphology accordingly. DSC showed that the melting point of composite made by reinfrocment of 25% by volume fraction of fiber increased as compare to pure PLA sample which proved the result attained by reinforcement in terms of tensile and flexural strength. Tensile and flexural strength increaed by around 32% and 35% respectively when 25% reinforcement of fibers is there. So it paved way for the further research to be done by using such fibers as a reinforcment to make bioderadable composites.So these rods can be used in various applications like selfie sticks, reinforcement of these rods in concrete, table legs etc.

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