A Review on various characterisation of PLA based biodegradable composites

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Abstract

Plastic sheets are becoming very popular and have wide applications in domestic and industrial sector. The usage of biopolymers has increased rapidly at higher rate due to the economic effects. Because of very good degradation property, the biopolymers can be used anywhere and many researches have been going on to improve the mechanical properties of the biopolymers to make it usable in various applications. There are numerous natural fibres available like bamboo fibres, jute fibres, sisal fibre that show very good mechanical properties over the synthetic fibres. Modification is a key tool for improving the property of the biopolymer-natural fibre reinforced plastics material. In this Paper, we are discuss about the various several standard characterization methods such as Mechanical test, Thermal test, microscopic test and degradation test and miscellaneous test for PLA based biodegradable composites. The study reveals that PLA has significant variations in the characterizations when it is blended with various fibers and fillers.

I. Introduction

Due to significant considerations, the plastic industry has started doing research on various alternative raw materials in the last few years and giving more importance to natural and renewable materials. One of the fast developing alternatives is Bio-based polymer [1]. The usage of biopolymers has increased rapidly at higher rate due to the economic effects. Because of very good degradation property, the biopolymers can be used anywhere and many researches have been going on to increase the mechanical property of the biopolymers to make it usable in various applications. Modification is a key tool for improving the property of the biopolymer-natural fiber reinforced plastics material. Poly Lactic Acid (PLA) is one of the biopolymer which poses very good biodegradable properties. The objective of the paper is to understand the various characterisation techniques used to evaluate the PLA based bio composites. Characterisation techniques are falls in five different types they are Mechanical test, Thermal test, microscopic test and degradation test and miscellaneous test. The mechanical test further classified into tensile test, creep test, fatigue test, flexural test, hardness test and impact test. The thermal test is classified in the Thermo gravimetric test and Differential scanning calorimetry test. The microscopic test is classified test is classified into SEM Test, TEM Test and XRD Test. Degradation test is classified into degradation due to normal condition and with specific environmental Condition. Miscellaneous test are classified into FTIR Test and water absorption test.

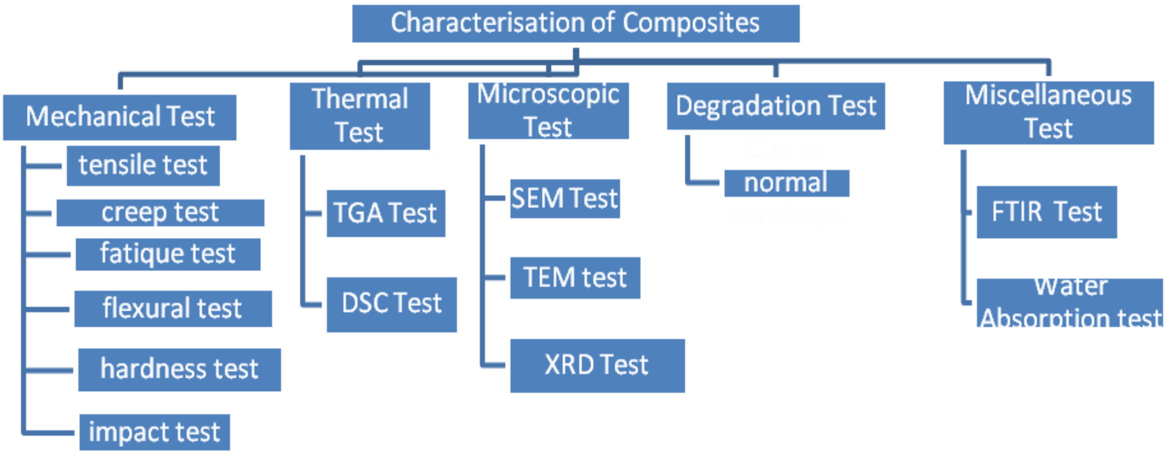

Figure 1: Characterization of Composites

II. Tensile strength

There are various standards used for conducting the Tensile strength tensile strength of the composites there are European standard EN1607 and ASTM Standards D638. According to EN 1607 European standard, $5 \times 5 \times t\ cm^3$ specimens were used [2]. The specimens were attached to 2 steel plates with a thickness of 20 mm using an epoxy resin and allow the specimens for 24 hours curing period. Then the specimens were inserted into the self-aligning pulling apparatus to the loading machine for confirming the load distribution were even throughout the specimen. Amsler Wolpert loading machine can be used with a maximum load 10 kN at a constant displacement rate of 10 mm/min for applying tensile load on the specimen. According to ASTM D638, 50×13×4 mm gauge dimensions flat specimens were used. The specimen were prepared either dog bone shape or dumb dell shape depending on the mould availability. The test can be carried out using universal testing machine with a maximum load 10 kN at a crosshead speed of 5 mm/min. The tensile strength of Treated WJF/PLLA composite (at wrap direction) was found to be 87MPa among all composites carried out in this paper[2].

III. Creep tests

Creep is the property of material to move slowly or deform permanently due to mechanical stresses. Creep will occur due to the result of exposure to higher stress for prolonged time even its below the yield strength of material. For performing the creep tests, the specimens were prepared according to ASTM D638 and instrumented with a strain gauge protected with microcrystalline wax. Grip section were hold with a fiberglass to decrease stress concentrations. The specimens were loaded to 65 percentage ultimate tensile strength and the corresponding Strain where monitored with the help of computer controlled DAQ system. All the specimens were manufactured on same time and stored in same Environmental conditions. The Force can be applied for 2 hour time followed by 3 hour recovery period. For test stress levels were taken low to prevent non-linear creep. Non-linear creep creates high deformations, results in permanent change of structure of material [3]. The mechanical properties of 1%wt Nano diamond with octadecylamine /PLA composites yielded reduction of 35% in creep [4].

IV. Fatigue test

Fatigue test were done according to JSCE-E 535 standard. It is calculated using Instron testing machine which is equipped with extensometer and load cell and the testing is done using a 100 KN in monotonic and cyclic loads. The load is applied in between 50% and 90% of ultimate strength of the specimens. Ratio of the minimum to the maximum load should be 0.1 and the loading frequency is below 5 Hz. Frequency below 5 Hz will creates internal heating of biodegradable composites due to that there is no energy losses because of sinusoidal load. Regression method is used for evaluating the fatigue features of each biodegradable composite [5]. fatigue strength of jute with PLA at 10^6 cycles was 55% of the ultimate strength, which is identical percentage of GFRP [6].

V. Flexural test

For performing flexural test, there are three standards available, there are ASTM standard D790, European standard EN 12089, JIS K7171 standard. Out of that ASTM standard is the universally accepted standard. Flexural tests is conducted at ambient temperatures on Instron UTM machine which is computer controlled with ± 50 kN load cell and 2 mm/min ramp rate in room atmosphere. During experiment the specimen top layer is subjected to compression while specimen bottom layer is in tension and middle layer is in shear. Do to that flexural behaviour is analysed till failure of specimen due to bending and shear combination. The three point bending flexural test is carried out using Universal testing machine according to ASTM standard with an 80×13×4mm dimension specimens with 2 mm/min crosshead speed and 64 mm span length [7]. Flexural strength of the recycled bamboo fabric-reinforced composite was determined to be 156 MPa. An increase of flexural properties were observed in recycled BF-PLA composite, when compared to the BF-PLA composite [8].

VI. Fracture toughness testing

For utilizing the bio composite for some specific application, the main design considerations is the ability of the material to prevent crack propagation. The present understanding of biodegradable composites fracture toughness is very limited. For using the bio composite as a material for structures, the fracture toughness should be calculated. Usually the fracture toughness of the bio composite is lesser when compare with other structural materials. The fracture toughness depends on crack deflection and fibre debonding, fracture and pull-out. There are two methods to calculate the fracture toughness, there are Single-edge notch-bend tests and Double-notched four-point bend tests. Single-edge notch-bend tests were used to find values of fracture energy and fracture toughness of bio composites. Double notched four point bend tests were used to know the mechanisms that contribute the changes in fracture toughness. In Single-edge notch-bend tests, specimens is tapped by a cooled razor blade to get sharp cracks. The fracture energy is calculated by corrected beam theory in accordance with ASTM D5528 and the fracture toughness was calculated using the fracture load. In Double notched four point bend tests, two close identical cracks are made using razor blade by tapping in each machined notch in the specimen. Then the specimen were load in four-point bending test, which results in at the crack tips there are two close identical stress fields. One of the cracks will start propagating and it will leave a second crack tip that is loaded to a close critical fracture toughness of the material. The process zone region forward of this second crack tip will be investigated through SEM and TEM. Compare the fracture toughness values given by both tests[9]. Decrease in fracture toughness values for the hemp fibre reinforced PLA composites with respect to aging may be due to the formation of porous structure as a result of leaching of debonded fibres by the fibrillation process upon removal of lignin [10].

VII. Impact testing

The impact testing can be done by two test, there are izod test and charpy test. Izod test is carried out according to the ASTM D256 standard and Charpy impact tests according to the D638 ASTM standards. The dimensions of the specimen used for the Izod impact strength of the composites is 63.5×12.7×4 mm specimens with respect to ASTM standard. The dimensions of the specimen used for the charpy impact strength of the composites is 115×13×3 mm specimens with respect to ASTM standard. Both testing can be done in impact testing machine. Charpy impact strength is the energy required to create a fracture that goes on to break the specimen. Izod impact strength is the resistance offered by the composite specimen against fracture by applying heavy impact load [11]. Highest impact stress was observed for PLA70/PMMA30/BS17% blend with value 45KJ/m^2 [12].

VIII. Hardness test

Hardness is a measure of resistance to different kinds of deformation, when a compressive force is applied. There are two types of hardness testing there are Rockwell hardness testing ASTM E384 standards and brinell hardness testing ASTM E10-14. The Rockwell Hardness Number is measured from the depth of permanent deformation of indenter in specimen. The indenter is either a conical diamond or a hard steel ball. The average diagonal lengths of indentation were measured and the unit was defined according to Vickers hardness. Rockwell Hardness ASTM D785, Hardness numbers have no units simply used as RHN, Higher numbers indicate harder materials. A standard specimen of 6.4mm thick is taken for testing. R and M scales are normally used with biodegradable composites [13]. For PLA with starch blends gives up to 25 VHN [14]. Rockwell hardness value of pure PLA is 88 RHN [15] and with 15% of Pine needle fibres the hardness reduced to 32 RHN [16]. The brinell hardness number of Recycled PLA reinforced with kenaf fiber with PES and sand has 298 BHN [17].

IX. Thermal Analysis

Differential scanning calorimeter and Thermo Gravimetric Analysis was used assess the thermal stability of biodegradable composites by using the standards of ASTM D7028, and ASTM D4065. TA-TGA Q500 thermal analyser can be used to carry out the Thermo Gravimetric Analysis and Pyris DSC6 thermal analyser is used to carry out the differential scanning calorimeter test. The analysis should be carry out in nitrogen atmosphere under dynamic mode. In Differential scanning calorimeter, heating and cooling rates were 10ºC per minute with nitrogen purge, and the sample weights were between 5 and 8mg. The composites were cut into pieces with the dimensions of 2 mm^2 and kept in 50 mL aluminium sealed crucibles. Glass transition temperature is the midpoint of the heat capacity increment. Melting temperature and crystallization temperature is the peak value of endotherm and exothermal phenomena in DSC curve respectively [18]. During multiple endotherms, the highest peak temperature is considered as Melting temperature. Heat of fusion and heat of crystallization of crystal phase is measured from total areas of DSC endotherm & exothermal respectively. In Thermo Gravimetric Analysis, the weight loss in the specimen due to volatilization of by-products degradation can be monitored using the same analysis. Thermo gravimetric curve will display a degradation process of single stage starting at ~100°C and ending at ~600°C and the starting and ending temperature depends on the polymers structural decomposition. After the polymer decomposition, there will char residue of 2–3 weight% approximately remaining. By using various points in the TGA curves we can study the decomposition temperature of biodegradable polymer composites [19]. the PLA with micro fibrillated cellulose composite shows the same trend as neat PLA, but the cold crystalline temperature of PLA with micro fibrillated cellulose composite (91.5 C) is lower than that of neat PLA (93.4 °C). the melt crystallization temperature of PLA with micro fibrillated cellulose composite occurs at a higher temperature (102.5 °C) than that of neat PLA (95.6 °C) [20]. The melting temperature of neat PLA and with recycled newspaper cellulose fiber is 54°C and 56 °C respectively. The glass transition temperature of neat PLA and with recycled newspaper cellulose fiber is 172°C and 170°C respectively [21]. The thermal degradation temperature at 10% and 75% weight loss for neat PLA is 323°C and 360°C respectively but for PLA with kenaf reinforced composite is 321°C and 357°C respectively and PLA with rice husk reinforced composite is 305°C and 340°C respectively[22].

X. Microscopy Test

The microstructure of the bio composites can be viewed by using scanning electron microscope and Transmission electron microscope. The microstructure of the bio composites is used to examine the scattering of fibre inside the resin and their nature of bonding between the resin and the fibre. Interfacing of resin with filler in the biodegradable composites cannot be revealed by lower magnifications images. We can able to understand the interfacing of resin with filler with higher magnifications provided by Transmission electron microscope and scanning electron microscope. The arrangement of fibres was observed using a scanning electron microscope of 20 kV. Before scanning, the specimen were coated with thin gold layer to escalate the conductivity of specimen to prevent electrostatic charging during specimen investigation [23]. To perform TEM Analysis, the specimens were cut and polished to rectangular sheets then embedded in epoxy resin and cure for 12 hours. ultra microtoming was performed with diamond knife generating foils with 50 X 500 μm^2 in crosssection and 50 nm in thickness and gathered on Cu grids. All specimens were stained with 2 wt% uranyl acetate for 2 min [24]. Fractured surface of cotton/PLA SEM analysis larger Delaminations. The fractured surface of the cotton/PLA composites treated with lignin showed fewer delaminations. In kenaf/PLA composites, no delaminations were recognisable. SEM analysis also showed that fibre pull-outs in untreated cotton/PLA composites are more than lignin-treated PLA composites [25]. To confirm the separation of individual crystallites or whiskers, the specimen was observed using TEM in nanometer scale. The size of the whiskers was estimated to be less than 10 nm in width and between 200 and 400 nm in length [26].

XI. Fourier Transforms Infrared Spectroscopy Test

Fourier transform infrared spectroscopy analysis is extensively performed on the biodegradable composites to detect the interaction and the phase performance of composites by classifying the functional groups existing in each specimen. To perform the Fourier transform infrared spectroscopy, the specimens were grounded finely by mixer mill. The specimens were mixed appropriately with 50 to 100mg approximately of potassium bromide powder and compelled in press to get thin translucent disc. FTIR spectra of specimens were documented in wavelength of range from 4,000 to 400 cm^2 through 50 scans by a resolution of 4 cm^2 in total reflectance mode. It is used to check the modifications done in bio composites has increase the properties, investigating the structure of polymer composites and to find the molecular interactions. If there is chemical interaction among the fillers and matrix, then there will be a corresponding change in the FTIR spectra bio composites. Lower the wavenumber of filler corresponding polymer indicates stronger interaction between the polymer matrix and the fillers [27]. The Fourier transform infrared spectra of BF both treated and untreated with the silicane coupling agent peaks appeared at 1372 cm–1, 1510 cm–1, 1733 cm–1 and 2869 cm–1 in the spectra of the treated BF, shows that chemical composition remain distinct after processing [28].

XII. Water absorption

Water absorption of the bio degradable is calculated using ASTM D570-98 standard. 100 x 10 x 4 mm^3 dimensions of the specimens is used for test according to the ASTM standard. Specimens were dried in an autoclave at 45.8 C for about 24 hours and submerged in fresh water and sea water at room temperature for a period of time usually 30 days. The weight of the specimen before the test and after the test were noted as Wt and Wo. The water absorption was calculated using the following formula

$$\text{Water absorption } (\%) = \frac{W_t - W_o}{W_o} \times 100$$

The bio composites usually shows the highest water absorption when compare with synthetic composites which is interpreted in terms of increasing of capillarity attraction attributed to an under cross-linked at particle vicinity. The degree of water absorption after 30 days was considerably greater for 70/30/15 PLA/PBS/WF biodegradable composite than pure PLA, pure PBS, and PLA/PBS blend. This is due to hydrophilic wood floor in the composite could absorb water throughout storage. Water absorption increased quickly on first day but after 3 days it showed constant absorption. After 28days of soaking, the composites' sizes changed by 0.3–2.9%, with the highest changes recorded in the composite containing 42 vol. % hazelnut shell flour. Bio composites with 40% BC loading showed the highest equilibrium water absorption, while the loading of 30 and 35% had the lower water absorptions [29].

XIII. Biodegradability Test

Biodegradability test for the biodegradable composites were done with respect to ASTM D5338-11 standard. For performing the biodegradable Composites, the specimens were buried in soil for the particular period of time (usually three months and depends on the Composites). The soil which is used should have 3.7 moisture content and 6.6% sand content with a 5.5 pH range. During testing the moisture content pH range and should be checked by moisture analyser and maintained by adding water every day for better result. After the particular period of time, the specimens were removed from the soil carefully without damaging and washed using distilled water and then dried with the temperature of 105°C for 6 hours and at normal room temperature for 24hours. Then the specimens were tested for the mechanical properties. In biodegradation tests, water penetrates through the cutting edges of the bio composites and As a result the specimen becomes loose, due to this mechanical properties of bio composites specimens falls considerably. Presence of mandelic acid in the nonwoven flax fiber reinforced poly lactic acid composites shows biodegradation with 20–25% loss in weight after 50–60 days. But in presence of dicumyl peroxide (as additive), biodegradation of the nonwoven flax fiber reinforced poly lactic acid composites was relatively slow (5–10% loss in weight even after 80–90 days) [30].

XIV. Conclusion

The growing awareness about sustainability, environmental considerations and reduction in the petroleum products has taken research to focus biodegradable fibres reinforced resin composites. The main advantage of the biodegradable fibre based composites materials being their low cost, low density, ease of separation, abundant availability, best energy recovery, C02 reduction, biodegradability and excellent recyclability in nature. In this article, we discussed various charactrisation techniques like Mechanical characterisation, thermal characterisation and morphological characterization of PLA based bio degradable composites. We can perform any of the above mentioned test to understand the particular properties of the PLA based biodegradable materials depending on the need.

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