



Review on Characterization of Various Poly Lactic Acid based Biodegradable Composites



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Abstract

Composites materials are the material made up of two or more materials with different physical and chemical properties but the individual components remain separate and distinct in final product. Composites have become so vital due to their high effective stiffness and strength, availability, low cost, specific strength, better dimensional stability and mechanical properties, eco-friendly and biodegradable as compared to plastics and fibers. Among all the property now a day's researchers concern is more on the biodegradable properties due to strict environmental rules and regulations. However always the non-biodegradable composites have more strength and stability, researchers adding various additives, blending fibres and resins which are biodegradable to bring the strength and stability on par with non-biodegradable composites. The biodegradable poly lactic acid composites has satisfactory thermal stability and mechanical rigidity. It can be reused and recycled. The rate of Biodegradability of PLA biodegradable composite is assessed in natural conditions. The main aim of this paper is to study the effect of various Resins, Fabrics and Additives on PLA biodegradable composite based on mechanical tests, DSC test, and to identify the best suitable material for various application.

Keywords: Microscopic Test; PLA; Tensile Test; Thermal Test

Abbreviations: PLA: Poly Lactic Acid; WF: Wood Flour

Introduction

Researchers are more concern on the biodegradable thermoplastic fibre reinforced composites due to its reuse and recycled properties and biodegradability [1]. Poly lactic acid [PLA] is famous for its renewability and biodegradability when compared with other composite resins due to this PLA is gaining more attention from the researchers [2]. More over the poly lactic acid is derived from the natural resources which will reduce carbon foot print. Along with this PLA is also eco-friendly and

is compatible with other Fibres (Bamboo, Banana, Sisal, Basalt Fibers, jute), Resins (PBS, PLLA, PMMA) and additives (Talc, ENR, wood flour(WF), polyethylene waste and hazelnut shell, ultrafine bamboo-char, PEG, SiO₂, hemp hurd). In this study we see the use of PLA with above mentioned constituents. In this paper we study the effect on tensile properties, impact strength, flexural strength, DSC, FTIR, SEM, biodegradability and water absorption properties with different constituents.

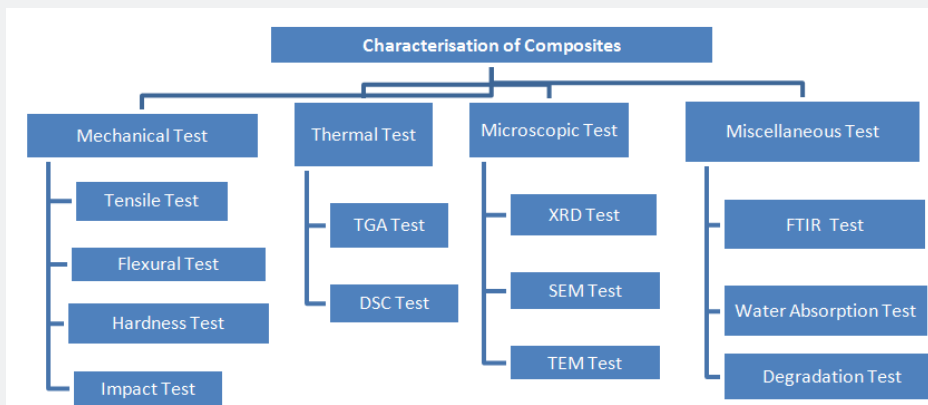


Figure 1: Various characterization methods.

The composites used for literature review in this paper are PLA/ PBS/WF with the composition of (70/30/5), PLLA/ PMMA with the composition of (20/80,30/70), PLA/BaF with the composition of (80/20), PLA/talc/ENR with the composition of (60/30/10) and BF/PLA, PLA/WF, PLA/ Natural rubber; PLA/NF/, PLA/ bamboo-char are taken with the composition of (70/30) and PLA/hemp, PLLA/JF, PLA/ SFare taken with the composition of (80/20). Figure 1 shows the various characterization techniques studied in the paper for biodegradable poly lactic acid composites. Depending on the application we can choose the characterization methods to understand the properties of the materials.

Tensile Strength

Poly Lactic Acid is famous for high rigidity and tensile strength. Tensile strength decreased with increasing wood flour composition keeping the composition of PLA and PBS 70/30. The composition of 70/30/5 gave highest tensile property of 47.66 MPa [3]. The results confirm that pure PLA shows more rigidity of 3.2 GPA and 68 MPa of tensile strength. The addition of PBS modified the properties of poly lactic acid. On adding PBS ductility improved to 30-35% when compared with pure PLA but rigidity and yield stress decreased. PMMA/PBS binary blend showed brittle behavior with low rigidity of 1.6 GPA. The ductility of the blend not change with addition of PMMA up to 80 percentage by weight. The highest tensile strength of 53MPa was obtained for PLA30/PMMA70/PBS composite. The recycled bamboo fabric-reinforced composite showed a tensile strength of 74.64 MPa. The highest tensile strength was found to be of bamboo fabric-reinforced composite 80.64MPa. When compared to the BF-PLA composite, a decrease in tensile properties was observed in recycled BF-PLA composite [4]. With increasing talc content, the tensile property of PLA composite decreased steadily. With addition of 30% talc, Tensile strength of PLA and PLA/talc composite was found to be 62 MPa which decreased to 55 MPa. The major change was between 20-30%. The tensile strength decreased with increasing ENR content. It decreased from 55-16MPa. Tensile strength of PLA/Talc/ENR composites depend on ENR dispersion and PLA and ENR surface interaction. For every increase in percentage of basalt fiber content tensile properties increases [5]. For 0 wt. % TS is 39mpa. At 4wt%, 8 wt. % & 12wt% TS increases 42mpa, 43mpa & 45mpa resp. Tensile strength of PLA/talc/ENR was lower than that of virgin PLA when BC was added into PLA. With increasing BC loading, tensile strength increased to maximum value of 14.03 MPa, when the BC loading was 30% then it decreased with more adding of BC content. Tensile strength was highest for Ag-HH/GMA/PLA (HH content 20%) i.e. 60MPa. A significant improvement in the TS and TM of PLLA was obtained by the reinforcement with untreated WJF; in warp direction their respective values were increased to 102.5% and 211.1%. In weft direction, TS and TM were also found to be 77.5% and 105.6% higher than unreinforced PLLA samples, respectively [6]. The strains at maximum tensile stress

for untreated WJF/PLLA composite samples were found to be 3.8% in warp direction and 4.1% in weft direction. It has been noticed that tensile strength of untreated WJF/PLLA composite was more in warp direction. The tensile strength of NWJF/PLLA composite was 55MPa. The tensile strength of Treated WJF/PLLA composite was 87MPa.

Impact Strength

Impact strength decreased with increasing wood flour composition keeping the composition of PLA and PBS 70/30. The composition of 70/30/5 gave highest impact strength property 4.1 KJ/m². Highest impact stress was observed at PLA70/PMMA30/BS17% blend i.e. ~ 45KJ/M2. Pure PLA exhibited a low impact strength of 3.4 kJ/m². The addition of 17 wt. % of impact modifier to pure PLA resulted in improvement of toughness to 2460.1 kJ/m². PMMA-rich formulations displayed a dramatic reduction of the materials toughness. Impact strength of the recycled bamboo fibre reinforced composite is 103 J/m. impact strength of recycled BF-PLA is lesser than virgin BF-PLA composites because of the composites turned brittle during the recycling process [7]. The impact strength of PLA composite decreased with the addition of talc because it increase the resistance to deformation and made the composite more brittle. Impact strength of polylactic acid (PLA) and PLA/talc composite was found to be 27J/m which decreased to 22J/m with addition of 30% talc.

The impact strength of PLA/talc composite increased with the ENR addition. The impact strength of PLA composites with the composition with P50T30E20 was 159J/m. For every increase in wt. % of basalt fiber content impact properties increases. For 0 wt. % impact strength is 8.8mpa. At 4wt. %, 8 wt. % & 12wt. % impact strength increased to 11mpa, 12mpa & 12.3MPa respectively. The impact strength increased from pure PLA (2.65kJ/m²) to PLA/LNR (6.44kJ/m²) due to the addition of 10 percentage of LNR as toughening agent [8]. The impact strength further increased to 8.88kJ/m² with addition of only 1% of C30B concentration and then decreased gradually. Impact strength increased to a maximum value of 20.50 J/m with the increase in addition of BC. When the loading reached 30%, it improved by 160% as compared with pure PLA of 7.88 J/m. The impact strength of NWJF/PLLA composite was found to be 12.98KJ/m² [9]. The impact strength of Treated WJF/PLLA biodegradable composite was 18.1 KJ/m².

Flexural Strength

Flexural strength decreased with increasing wood flour composition keeping the composition of PLA and PBS 70/30. The composition of 70/30/5 gave highest flexural strength 70.2 MPa. Flexural strength of recycled bamboo fibre reinforced biodegradable composite was 156 MPa, which is higher compared to virgin BF-PLA composite. Increase in flexural strength is due to shortening and dispersed fabric yarn in composite during injection molding [9]. The flexural strength of Poly lactic acid with

and without addition of 30% talc was decreased from 95 MPa to 70 MPa. The flexural strength decreased ENR content increased. It decreased from 65-19MPa. For pure PLA composite flexural strength is the highest with 94MPa. Flexural strength of PLA/LNR is lower than virgin PLA. Flexural strength decreases with increasing addition of C30B. The highest flexural strength was observed for PLA/LNR/C30B-3 i.e. 37.2MPa. For every increase in wt% of basalt fiber content flexural properties increases [10]. For 0 wt% TS is 67mpa. At 4wt%, 8 wt% & 12wt% TS increases 75mpa, 76mpa & 77mpa resp.

Thermal Analysis

The glass transition temperature (T_g) decreased with increasing the wood flour composition. The maximum glass transition temperature was given by the composition 70/30/5 (w/w/phr) i.e. 57.5oC. The melting temperature (T_m) increased with increasing the wood flour composition [11]. The T_m was highest for the composition 70/30/30 (w/w/phr) is 156.3oC. The glass transition temperature (T_g) showed decreasing behavior as the PLA content decreased and PMMA content increased which varied from Pure PLA (61oC) to pure PMMA (116oC). It was observed that the maximum T_g was for the blend PLA20/PMMA80/BS is 97oC. In this test pure virgin PLA, glass transition temperature is 63c and melting temperature is 153oC. And for UT BSF/PLA composite temperatures are 61 and 151oC. For BP-T BSF/PLA composite temp are 63oC and 153oC respectively [12]. In this test, PLA composite shows the highest glass transition temperature, cold crystallization, melting temperature of 62oC, 86oC and 175oC respectively.

PLA/LNR/C30B-1 composite showed the highest temperature ranges for T_g , T_c and T_m of 55.5, 100.8 and 154.7oC respectively. The glass transition temperature (T_g) both for the composite mixtures and the polymer that occurred at -50 and -40°C. T_g for the sample OL42 was found to be -43oC. The maximum T_g temp was observed for the compound AgNP-HH/PLA i.e. 61.0oC and T_m was 166.0oC. With the increase in PBS content thermal degradation of PLA/PBS blends all increased significantly. This occurred because of the thermal stability of PLA is much lower than that of PBS. The absorption of Wood Flour slightly decreased the thermal stability. The addition of bamboo fibre in PLA increase the melting temperature from 151.70oC to 154.42oC and recycled bamboo fibre is 155.56oC. The glass transition temperature of PLA with addition of bamboo fibre ranges from 58–58.65oC and with recycled bamboo fibre 58.65oC [13]. With the addition of ENR, the decomposition temperature of PLA decreased as ENR caused poor distribution and dispersion of talc in PLA matrix, which lead to an increase in clusters of talc. The max T_{10} temperature was observed for the composite is 233oC. PLA/BC Bio composites showed the maximum T_g for the 30 percentage bamboo char was 51.2oC and the melting temperature range (T_m) was highest for (PLA70/BC30) i.e. 141-150(C) [14].

Microscopic Test

Smooth surface with a brittle fracture behavior was observed during SEM analysis of pure PLA whereas pure PBS showed rough surface behavior with ductile fracture behavior. The flexible behavior was predicted when PLA/PBS blend showed rough surface behavior. This took place due to high flexibility behavior of PBS which toughened PLA compound. Clear boundaries and spaces between the WF and PLA/PBS70/30 by weight blend matrix were observed in SEM images which indicated the strong hydrophilic WF and the weak hydrophilic polyesters which further indicated non-wettability behavior. Pure PLA exhibited brittle morphology and smooth surface after fracture. Homogenous morphology of PLA70/PMMA30 without BS was observed in SEM micrograph [15]. The mean droplet diameter for the blend PLA20/PMMA80/BS showed the highest diameter i.e. $D_v \sim 1200\text{nm}$ and $D_n \sim 800\text{nm}$. Talc particles formed big clusters of talc and had poor scattering in PLA matrix. The addition of 10 to 20percentage by weight of ENR into PLA/talc (70/30) composites led to formation of clusters in PLA matrix. The addition of C30B stabilized the morphology [16]. With addition 1% and 3% of C30B a high impact strength was indorsed by the stabilization of morphology in immiscible polymer blend.

A homogeneous dispersion of SF in the PLA matrix was observed before the water uptake behavior through SEM micro graphs. In SEM micro graphs of alkali and NaOH+silane treated fibers pulled-out fibers were rarely observed, which was suggestive of coating of polymer on SF which further improved fiber/PLA adhesion. The exfoliated structure could be due to a more heterogeneous structure of the Nano PLA composite; the reason behind this is the high content of Nano fillers which couldn't get homogenous structure. The amorphous behavior of PLA-PEG-PLA and PLA-PEG-PLA/SiO₂ hybrid material, implied that on adding SiO₂ no change was observed in PLA-PEG-PLA. A huge amount of slots and pores were appeared, when PEG fragments were dispersed in filth solution. The degradation of PLA after 12weeks, slots and pores enlarged and expanded to form 3-D porous scaffold. In PLA/LNR matrix, C30B Nano clay dispersed inn matrix. At 1% Nano clay got elongated and separated. But at 3% different structure was observed.

Miscellaneous Test

The amount of water absorbed after 30 days was more for PLA/PBS/WF (70/30/15) composite than for PLA, PBS, and PLA/PBS (70/30) blend. The reason for this was hydrophilic WF in composite absorbed water during storage. On first 3 days, there was rapid increase in water absorption but thereafter it became constant. After 28days of soaking, the composite sizes changed by 0.3-2.9%, with the highest changes recorded in the composite containing 42 vol. % hazelnut shell flour. With the 40 percentage addition of bamboo char having the water absorption very high. PLA absorbed higher amount of water

more when compared with PP or PE. The higher hydrophilic character of lingo cellulose fibers led to an increase in the water uptake values of combination of treated and untreated sisal fibers into the PLA matrix [17]. The decrease in tensile strength was observed when the composite was immersed in distilled water for 209 days at room temperature. This took place because water molecules changed the properties and structure of matrix and fiber. For untreated sample, $\phi_f = 0.156$, tensile strength was 53.8MPa. For silane sample, $\phi_f = 0.375$, tensile strength was 61.8MPa. For NaOH sample, $\phi_f = 0.341$, tensile strength was 81.5MPa. For NaOH + silane sample, $\phi_f = 0.345$, tensile strength was 78.6MPa. For untreated sample, $\phi_f = 0.156$, tensile strength was 48.1MPa. For silane sample, $\phi_f = 0.248$, tensile strength was 34.5MPa. For NaOH sample, $\phi_f = 0.171$, tensile strength was 45.4MPa. For NaOH+silane sample, $\phi_f = 0.174$, tensile strength was 42.3MPa. After 90 days of burial, the tensile strength of the PLA with addition of PBS decreased to a larger extent from 90 to 70 MPa. The PLA/PBS/WF (70/30/15) composite decreased to smaller extent i.e. from 50-45MPa [18].

In controlled composting conditions, the degradation rate of Virgin PLA was very much higher when compared with BF-PLA. After 30 days of burial, 45% of weight loss was observed in virgin PLA. At 70th day, BF-PLA composite showed a weight loss of 62%. Ester and Methyl groups of PLA were detected in the region 1,800 and 1,300cm⁻¹. BP-TBSF/PLA composite showed more intensity for the C=O stretch and -C-O- groups of ester bonds, at 1,730cm⁻¹ when compared with FTIR of PLA in the region of ester and methyl groups. When compared with untreated BF, the new peaks appeared in spectra of treated BF, These Peaks were introduced by the silane [19]. The results indicated that the surface of the BF was coated with the silane layer. FTIR spectra of bio composites were stable with that of PLA and no transfer of absorption took place, which showed physical interlocks between two phases.

Conclusion

The tensile strength of Treated WJF/PLLA composite (at wrap direction) was found to be 87MPa among all composites carried out in this paper. Highest impact stress was observed for PLA70/PMMA30/BS17% ble

nd is 45KJ/m². Flexural strength of the recycled bamboo fabric-reinforced composite was determined to be 156 MPa. For BP-TBSF/PLA composite glass transition temp is 63oC. And for the compound AgNP-HH/PLA melting temperature T_m is 166.0oC. In TGA analysis the melting temperature of recycled BF-PLA was found to be 155.56oC. This study conclude that by blending the PLA with various resins and reinforced with various fibres and adding fillers, we can achieve the required property for the particular applications. Addition of fillers increase the mechanical properties of the biodegradable composites significantly. The compatibility between the PLA resin with other resins are higher and shows significant improvement in the properties.

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