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Effect of Polyethylene glycol on Mechanical, Thermal, and Morphological Properties of Talc Reinforced Polylactic Acid Composites

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Abstract

Poly lactic acid (PLA) was reinforced with various compositions of talc with plasticizers polyethylene glycol 1500 (PEG1500) and polyethylene glycol 6000 (PEG6000). These reinforced PLA were prepared by melt mixing technique using twin screw extruder. The prepared composite plastic materials were characterized by using mechanical, morphology, and thermal properties. The polylactic acid-talc-PEG1500 composites (PTP1500) show significant increase in flexural strength from 80MPa to 140MPa. Further the impact strength was also increased to the maximum of 23KJ/m². PTP1500 showed a better result when compared with Poly lactic acid reinforced with talc and PEG6000 (PTP6000).

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1. Introduction

Over the years plastics are being manufactured from petroleum sources due to which they have a few drawbacks. The major issues are global warming, landfill, and pollution of water because of white pollution. To overcome these problems, bioplastics may be desirable. There are various biodegradable plastics like Polylactic acid (PLA), polycaprolactone (PCL), Polyhydroxy alkonate (PHA), Polyhydroxy butyrate co-valerate (PHBV). During biodegradation micro organisms consume the host material and liberate the carbon di-oxide which serves as an indicator for degradation [1]. Bioplastics could easily be compostable, whereas the other plastics do not undergo biodegradation and are not compostable.

Among biodegradable products polylactic acid (PLA) linear aliphatic thermoplastic polyester of general formula $(C_3H_4O_2)_n$ is prepared from natural and renewable resources like corn, sugar beet, and sugarcane [2] and its life cycle analysis is low when compared with other products [3]. It has high strength, good transparency, thermo plasticity, and biocompatibility. The degradation of PLA occurs by enzymatic action of living organism. It has been used in numerous applications such as packaging, gardening, catering products, electronic goods, medical products, and automobile industry. There are various shortcomings while choosing PLA as a starting material but it has to improve in certain areas. It has slow crystallization rate, low glass transition temperature, and very poor processability [4]. Addition of filler or fiber to PLA it overcomes the drawback like brittleness and heat distortion temperature. PLA can be reinforced by natural fibres since they are obtainable and renewable resources. PLA has high strength and stiffness, low weight, recyclable and biodegradable. Fibres like hemp, flax, kenaf, jute, wheat straw, rice hulls, pineapple leaf, ramie, newspaper and banana have been studied as reinforcements for PLA [5-8]. The main drawback of these composite are moisture uptake and low thermal stability.

Filler addition to polymers will improve properties such as stiffness, strength, gas barrier properties; melt strength, and thermal stability. Mica, kaolin, calcium carbonate, talc are most often used as fillers to alter the properties of PLA. Based on aspect ratio the reinforcing fillers are characterized and defined as ratio of diameter over thickness for platelets and flakes types of fillers. Among fillers, mica, and calcium carbonate are more effective than talc due to increase in stiffness and impact strength. Talc is the only filler which improves the PLA crystallization and also acts as nucleating agent. Loading of talc at 10-40% can reinforce PLA and increases its tensile modulus and stiffness [9-11]. PLA-talc composites are compatibilized with maleic anhydride to increase the strength of the polymer-filler interface [12]. Various compatibilizers or plasticizers can be used to increase the adhesion of the matrix to fibres.

Plasticizer acts as an internal lubricant for the polymer. Plasticizers like glycerol, oleic acid, acetyl triethyl citrate, polyethylene glycol and ricinoleic acid [13-15] are used for this. Among the plasticizers polyethylene glycol (PEG) can be effective in reinforcement by talc. PEG enhances the crystallization rate of PLA. The role of plasticizer is to improve the flexibility and impact strength and act as a bicompatiblizer with the polymer. However, talc acts as nucleating agent at low concentration (less than 3 wt %) and with plasticizers help to improve the rate of crystallization [10]. In this current research work it was investigated how plasticizer at low loading (1 to 3 wt %) and talc at higher loading (10 to 40 wt %) can affect

the crystallinity and mechanical properties of the composites. Melt blending of the materials was done using a twin screw extruder.

2. Materials and Methods

2.1 Materials

PLA (Nature works, grade 3052D) was procured from Natur-Tec Pvt Ltd., Chennai, India. Its glass transition temperature and melting temperature are 55-65 °C and 145-155 °C. Its specific gravity is 1.24. Talc was procured from 20 Microns Pvt. Ltd., Chennai, India. Its size is 2 µm. Polyethylene glycol with molecular weight 1500 and 6000 was supplied from Subra Scientific Company, Chennai, India.

2.2 Preparation of Composites

All the raw material was predried for 2 hr at 80 °C to remove the moisture content present in the material. Mixing of polylactic acid, polyethylene glycol (with varying molecular weights) and talc were melt-blended by using a Berstorff High Performance co-rotating Twin screw Extruder. The screw speed was fixed at 280 rpm with barrel zone temperature 110 to 190 °C for melt mixing. The extrudate strands were pelletized for further processing. The materials were compounded for different proportions are given in Table 1.

Table 1: Formulation for the composites

Composition NO.	PLA (wt %)	Talc (wt %)	PEG1500 (wt %)	PEG6000 (wt %)
A	100	-	-	-
B	70	30	-	-
C	70	30	1	-
D	70	30	2	-
E	70	30	3	-
F	70	30	-	1
G	70	30	-	2
H	70	30	-	3

2.3 Preparation of test Specimens

Test specimen was prepared by using injection molding machine (L&T Demag with a tonnage of 60 tons capacity). The PLA was predried at 80 °C for 4 to 5 hr before processing to remove excess moisture in the material. The barrel temperature was fixed between 100 to

190 °C from feeding to nozzle zone. The standard specimen for tensile, flexural, and impact test were prepared as per ASTM standard.

2.4 Testing of the composites

Tensile and flexural properties of PLA composites were tested using Zwick universal testing machine. The tensile properties were tested as per ASTM D 638 (Type V) standard using a load of 250 kN at constant crosshead speed of 50 mm/min at room temperature. The flexural property was tested as per ASTM D 790 at a crosshead speed of 1mm/min. The impact property was tested using notch specimen as per ASTM D 256 using a striking pendulum machine Tinius Olsen, USA. Morphology of PLA composites were analyzed using fractured surfaces of the tensile specimens by scanning electron microscopy (SEM) Hitachi with an accelerating voltage of 30 kV. To avoid charging under the electron beam the samples were coated with gold. Differential Scanning Calorimetry of PLA composites was carried out using DSC 200F3 MAIA by Netzsch technologies. Nitrogen atmosphere is maintained to conduct the test at a temperature of 30 °C to 200 °C on heating and in reverse condition for cooling at the rate of 20 °C /min.

3. Results and Discussion

3.1 Tensile Strength

The tensile strength values of the PTP1500 and PTP6000 blends were shown in the Figure 1. The tensile strength of polylactic acid was 45 MPa. Generally, the tensile strength value is not increased due to the addition of talc. Even though the talc acts as a nucleating agent, the structure of the talc restricts the polymer from elongation may act as a point of stress concentration which reduces tensile strength [10]. Addition of increased filler content reduces the value of tensile strength. By adding the plasticizer PEG 1500 tensile strength is slightly reduces to 41 MPa for C composition. But, while considering the high molecular weight PEG 6000 it reduces to 38 MPa (F composition) compared to the A and B compositions. The elongation at break values of the PTP1500 and PTP6000 biocomposites were shown in the Figure 2. On addition of talc to the PLA the elongation at break value of the composites gets reduced due to the brittle nature of the talc. As the plasticizer loading increases the elongation at break was found to be increased. It confirms that the composite was changed from brittle nature to the ductile nature.

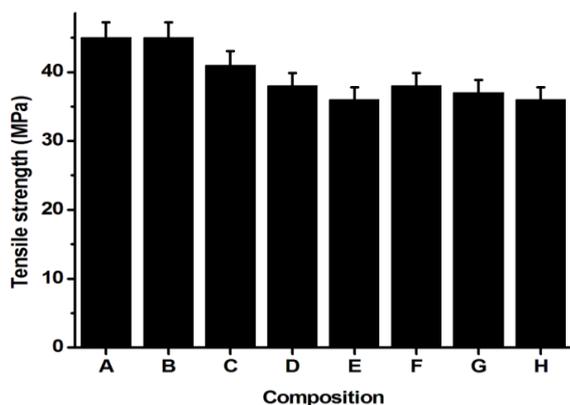


Figure-1 Tensile strength of the composites

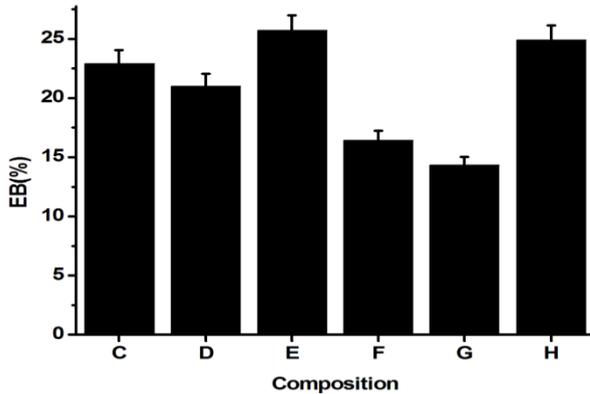


Figure-2 Elongation at break of the composites

3.2 Flexural Strength

The results of the flexural test for composites are given in the Figure 3. It was noted that there was a drastic increase upto 127% of flexural strength in the values when compared with that of the A composition. The incorporation of talc and PEG 6000 into the polylactic acid F composition initially increased and attained the peak value of 133 MPa. And during the D and E compositions it gradually reduced the flexural strength. Regarding the effects of PEG1500, we see a tremendous increase in the value of flexural strength. The flexural strength for composition C was found to be 140 MPa. On increasing the plasticizer loading the flexural strength gets reduced for the D and E composition. In D and E composition, the values decrease from 128 MPa to 125 MPa because high molecular weight plasticizer was used.

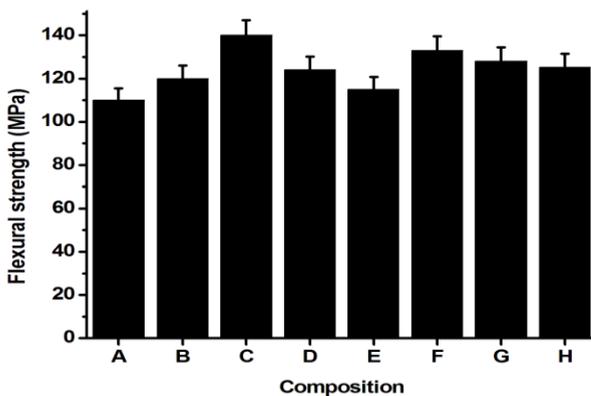


Figure-3 Flexural strength of the composites

3.3 Impact Strength

Figure 4, illustrates the notch izod impact test values for the composites. The impact strength of polylactic acid was 16 kJ/m². On addition of plasticizer with the polymer-filler matrix the interaction get modified results in change in the crystalline structure and prevents from the crack formation. This was confirmed by the increase in percentage of crystallization and from the SEM images. Effect of increased crystalline structure with favoured stress condition resulted in increase in the toughness. When PEG6000 was incorporated into the biocomposite, it significantly increased the impact strength. By adding the plasticizer PEG 1500 to the brittle polymer the values sweep up to 23 kJ/m². It was noted that in SEM images talc-rich phase was

observed by the addition of PEG1500. There was an increase in impact strength values of C composition, compared with that of A. The plasticizer helps by migrating into the PLA in a suitable manner and it changes the polymer from a brittle to a ductile one.

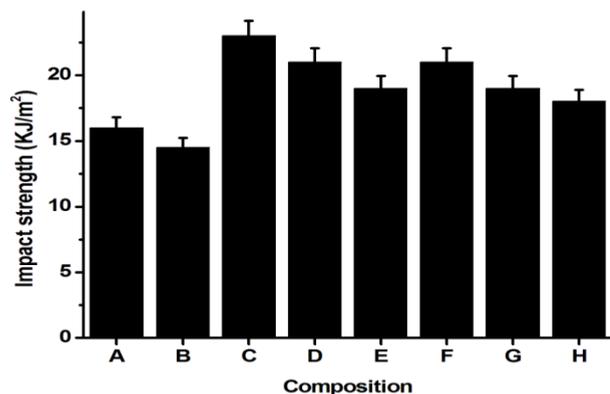


Figure-4 Impact strength of the composites

3.4 SEM

The fractured surfaces of the tensile failed specimens of the blends were examined by using SEM. Figure 5 a, b, and c shows the SEM images for neat PLA, PTP1500 (C composition) and PTP6000 (F composition) respectively. The SEM images of virgin PLA showed a thin layer of fractured surface which indicated a brittle failure. During the early stages of incorporation of talc without plasticizer it is not compatible with PLA so it can be easily pulled out and form a weak matrix [12]. On addition of plasticizer, which can also act as a lubricant for the PLA and talc will improve the dispersion of talc in PLA matrix may improve and the brittle nature changes to ductile one. This was confirmed by the increase in the flexural and impact properties of the composites in C and F compositions respectively. SEM images show a good dispersion without forming agglomerates of talc in the medium. When weighed against other combinations only C and F composition for both PEG1500 and PEG6000 gives good results in the previous tests. The SEM images are shown for these two compositions. The plasticizer has an excellent adhesion and also the drawability is increased. There is more consortium of phase separation in the PTP1500 when compared with PTP6000.

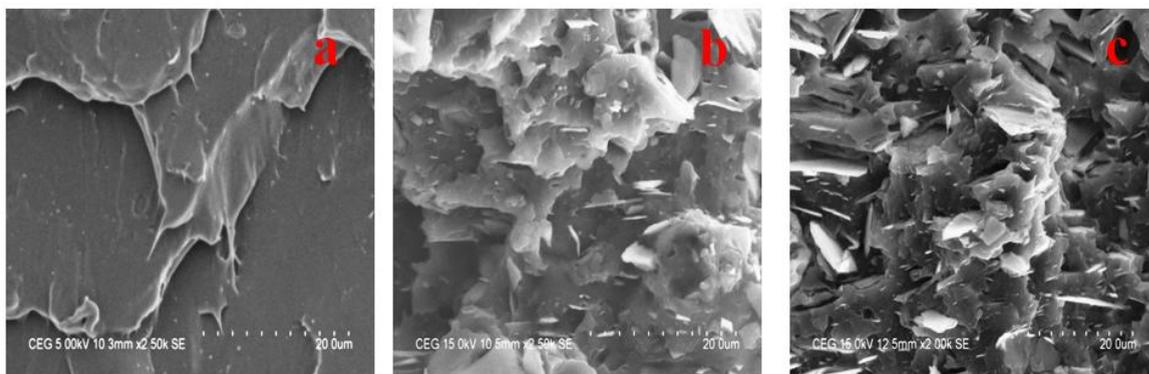


Figure-5 SEM Images of the composites a) neat PLA b) PTP1500 (C composition) c) PTP6000

(F composition)

3.5 DSC

The thermal properties of the neat PLA and for the composite (PTP1500 and PTP6000) were studied by Differential Scanning Calorimetry. The melting temperatures (T_m), crystallization temperature (T_c), and glass transition temperature (T_g) were determined for the composites. Figure 6 shows the DSC curves for the composites. Addition of talc restricts the free volume of polymer so it increases the T_g value whereas the incorporation of plasticizer into the PLA/Talc it shows decrease in T_g value. There was a correlation between the T_g and plasticizer because when the content of plasticizer got increased the T_g value got decreased. It showed good miscibility between the polymer and the plasticizer. T_c value showed a double peak due to the enhanced chain mobility, by the influence of plasticizer. Talc acts as nucleating agent so it reduces the T_m value and improves the crystalline percentage [16] but the plasticizer loading first increases and then decreases the T_c value and it limits the improvement in crystalline percentage. The percentage of crystallinity got increased from 42% to 45% in the addition of PEG1500. The melting temperature slowly decreased due to the effect of diffusion control growth process by the addition of plasticizer. There is no enormous change in T_m values when compared with PLA.

It is clear that talc acts as nucleating agent and it increases the mechanical property and crystallinity of the polymer [11]. Effect of plasticizer loading results in decrease in T_g , increase in T_m , and increased the tensile property based on the composite loading were reported [17]. Plasticizer plays an important role in the molecular mobility and it provide crystallization of the PLA in an enhanced result. The high molecular weight PEG1500 showed a better result than PEG6000.

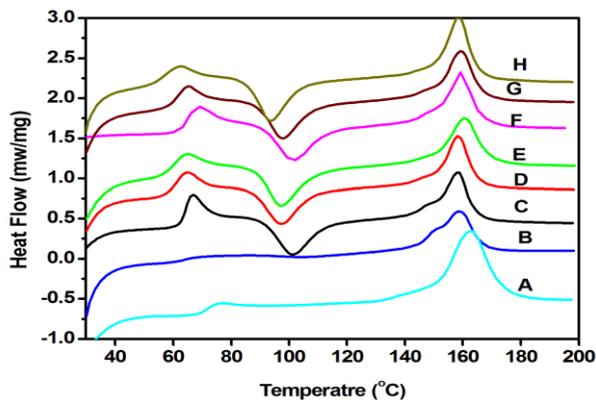


Figure-6 DSC curves of the composites

Conclusion

In the present study, PEG1500 and PEG6000 were added to PLA-Talc blends. Both act as a plasticizer between the polymer and filler. PEG1500 with PLA was more effective when compared with PEG6000. It was found that there was no increase in tensile test but there was a drastic increase in the flexural and impact strength test. This proved that the PLA changed from brittle material to ductile. On addition of talc and plasticizer there was an increase in crystallization and mechanical properties. It can be used in engineering applications where the strength is required. The addition of PEG1500 reduces the glass transition temperature and

increases the percentage of crystallinity. Addition of low molecular weight has high plasticizing effect in the composites. Blending of PLA, talc, and the plasticizer overcomes the problem of agglomeration and forms a strong matrix.

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