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Synthesis and Characterization of Biodegradable Polymer Nanocomposite

Dr. M.S.Murali[1]
Principal of ACS College of Enginnering, Bengaluru

Mr.Hareesh A[2]
Associate Professor Dept. of Mechanical Engineering, K .S .School of Engineering and Management, Bengaluru

Balaji.R[3], Deepak.M[4] , K.V.Dilip Kumar[5] , Manoj.V.Reddy[6]
[2-6]*Department of Mechanical Engineering, K .S .School of Engineering and Management, Visvesvaraya Technological University, Bengaluru, Karnataka, India*

Abstract: PLA(Poly lactic acid), well known biodegradable polymers, has been widely used in packaging, drug delivery, tissue engineering and human medicine. Due to its broadened applications, this processing would be potential substitute for petroleum products. Nanocomposites composed of organoclay and PLA and additives was prepared by solvent casting method. PLA is synthesized by ring opening polymerization/ polycondensation method. Mechanical, thermal and microstructure and FT-IR properties of the composites were studied.

Keywords: Polylactic acid, Biodegradable, Polymer, Nanocomposites, Organoclay.

1. INTRODUCTION

The impact of environment have provoked the need to substitute petro chemical based polymer with biodegradable ones .PLA is thermo plastic ,compostable and biocompatible ,polymer derived from renewable sources such as corn ,sugar beets, potato starch ,cheese whey ,generally ,polymers that are produced from petro chemical products have low recovery are reproduction rates and are not easily degraded in environment. the biodegradable polymers like PLA and PLGA are considered as the green eco friendly materials due to their biocompatibility and non toxic properties .fully biodegradable synthetic polymers have been available since many years .such as PLA ,poly glycolic acid poly caprolactone .Among all bio polymers PLA was extensively studied in food packaging and medical implants and drug delivery system due to its biodegradability PLA is becoming popular as a bio degradable plastic owing to its high mechanical strength and easy processibility compared to other bio polymers ,the limitations are poor thermal and mechanical resistance compared to petroleum based polymers which limit its application in packaging .

The above drawbacks could be overcome by enhancing their thermo mechanical properties through copolymerization, blending and filling techniques. The use of fillers with atleast nano scale dimension produces nano composites .

Tensile strength and elastic modulus of PLA nano composites increased significantly with clay and additive contents while the values of elongation percentage decreased drastically .Glass transient temperature (Tg)were determined by Differential Scanning Calorimeter(DSC),X-Ray Diffractometer (XRD)and Scanning Electron Microscopy (SEM) images reveals the difference layer of nano clay and additives .The formation of intercalated structure with good compatibility and homogeneously dispersed nano particles with clay concentrations.

There are few studies in which in the same condition of all properties of PLA and its nano composites have been evaluated ,such as above mentioned, have studied only about nanocomposites ,in this study, we have attempted with additives /fillers which influence good mechanical and thermal properties [1].

2. MATERIALS

PLA was obtained from sigma Aldrich with a density of 1350 kg/m³ , melting point of 160ºc and nano clay and additives and solvents for dissolving PLA

3. PREPARATION OF FILMS

PLA and PLA based nano composites films were prepared using solvent casting method.PLA is measured and dissolved in desired solvents stirred with magnetic stirrer for 5 hours .The dissolve solvents was poured on to greased glass moulds and then allowed to dry for about 24 hours at room temperature .

For preparation of PLA nano composite films ,a pre determined amount of clay and additives are added with PLA and dissolved with solvents in magnetic stirrer for 6 hours at 2000 rpm and poured on to greased glass moulds .

After drying at room temperature for 24 hours, all PLA films were dried at 60°c in a oven to remove remaining solvent to prevent plasticizing effect.

Figure 1 : Preparation of Films

4. MATERIAL PROPERTIES

4.1 Mechanical properties

Universal Testing Machine was applied to study the mechanical properties of the film samples upon the guidelines of ASTM standard method D882 . Initial grip separation was set at 200 mm and cross-head speed at 10 mm/min. Tensile strength (TS) was calculated by dividing the maximum load on the film before failure by the cross-sectional area of the initial specimen. Elastic modulus (E-M) was determined according to the slope of the stress /strain curve in the linear range. Percentage elongation (E) was defined as the percentage change in the length of the specimen to the original length between the grips. At least three replicates of each film were tested.

4.2Thermal properties

Thermal behavior of the specimens was evaluated by DSC(Differential Scanning Calorimetry-DSC 200). Samples of 4-5 mg were sealed in standard aluminum dishes, using a sealed empty aluminum dish as the reference sample. Experiments were conducted from -10 to 200 °C, with a heating rate of 10 °C/min, on all of the samples. Degree of crystallinity (x%) was calculated by using a value of 93 J/g for the heat of fusion of the 100% crystalline PLA. And also thermal behavior of the specimens was evaluated by TGA (Thermal Gravetometric Analysis-TGA 50), Samples of 8-10 mg were sealed in platinum dishes. Experiments were conducted from 20°C to 835°C, with a heating rate of 10 °C/min, on all of the samples. Weight loss was determined with respect to temperature[2].

4.3 Film Microstructure

Film microstructure was determined by scanning electron microscopy (SEM) . Samples were prepared using standard techniques, mounted on aluminum tubs, and sputter coated with gold (100 Å) . The micrographs were collected using an accelerating voltage of 25-30 kV

4.4 XRD Pattern

XRD(X Ray Diffraction Calorimetry) patterns were obtained from X-ray diffractometer equipped with radiation Co Kα beam at a voltage of 40 kV and current of 30 mA. The relative intensity was recorded at ambient temperature over an angular range (2θ) of 2-45° at a rate of 1°/min and a step size of 0.02. The samples were cut in 3×3 mm^2 rectangles and placed on a glass plate, and the set was placed inside the chamber of the apparatus for measurement.

4.5 FT-IR Characterization

The interactions of polymer composites can be identified by means of FT-IR Spectra (Fourier Transform Infra Red Spectroscopy). If two Polymers form completely immiscible blends, then there are no appreciable changes in the FT-IR Spectra compared with those of each components. However, if two polymers are compatible, a distinct chemical interaction hydrogen bonding exists between their chains, causing the IR spectra of the composites change[3].

5. RESULTS AND DISCUSSION

5.1 Mechanical Properties

As Comparing Fig 2 & 3, in Fig 2 as adding clay and additives the tensile strength and elongation modulus increases and elongation decreases. Mechanical Properties implies the strength and durability of films. As concentration of clay increases tensile strength increases and elongation decreases.

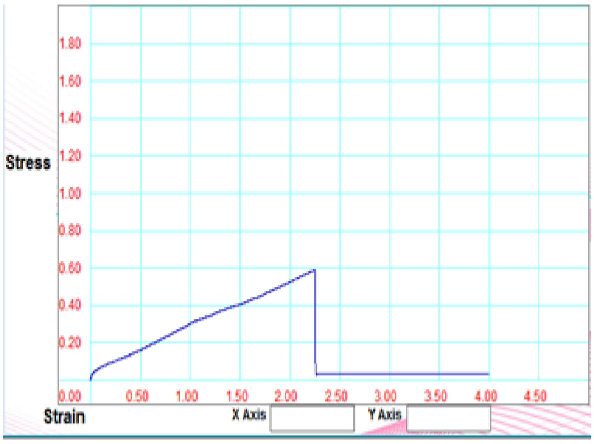

Figure 2: Stress-Strain curves for Pure PLA

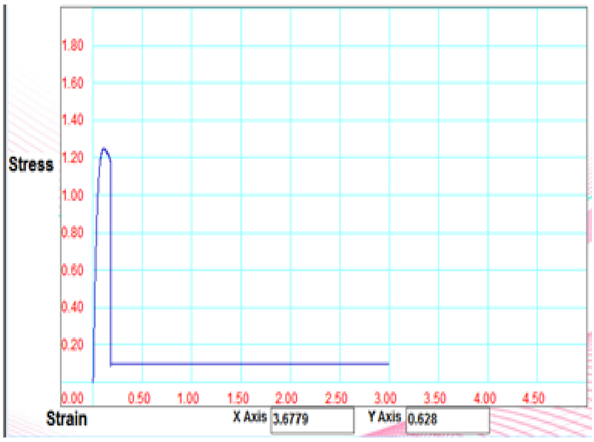

Figure 3: Stress-Strain curves for PLA Nanocomposites

Parameters	Pure PLA	PLA Nanocomposite
Peak Load (Kg)	2.943	6.25
Break Load (Kg)	0.149	0.501
Yield Load (Kg)	0.428	6.24
Elongation at Yield (mm)	8.47	5.55
Tensile Strength (N/Sq.mm)	5.774	12.243

Table 1 : Comparision of Mechanical Properties

5.2 Thermal Properties

5.2.1 DSC Property

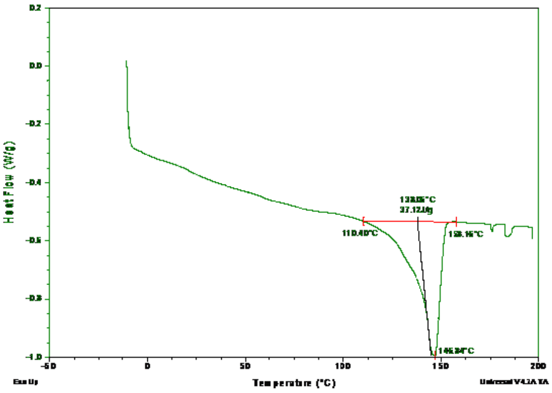

Figure 4: DSC graph for Pure PLA

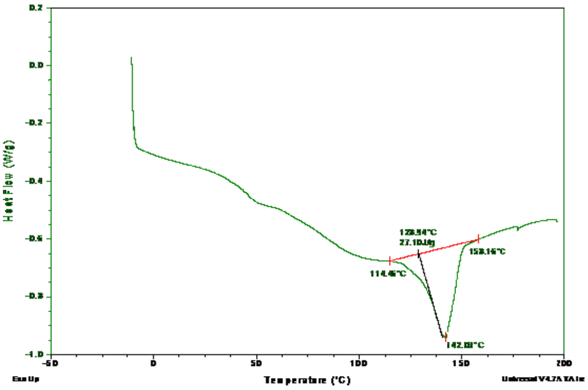

Figure 5: DSC graph for PLA Nanocomposites

Parameters	Pure PLA	PLA Nanocomposites
Heat Flow (J/g)	37.12	27.1
Tg (°C)	138.06	128.94
Tm (°C)	146.84	142.03

Table -2 : Comparision of DSC Property

Thermal analysis of PLA and its Nanocomposites are shown in Fig 4&5. PLA & its Nanocomposites undergoing heating up to 200°C. It is characterized by glass transition temperature and melting temperature. There is no significant change in Tg & Tm.

5.2.2 TGA Property

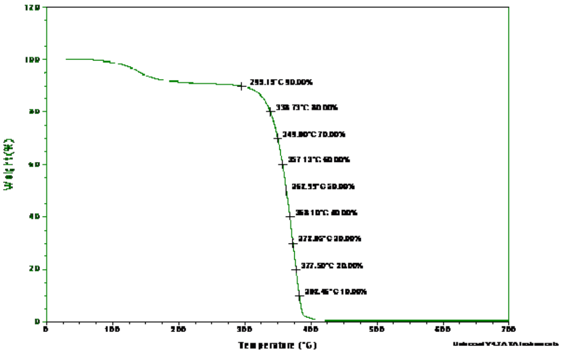

Figure 6: TGA graph for Pure PLA

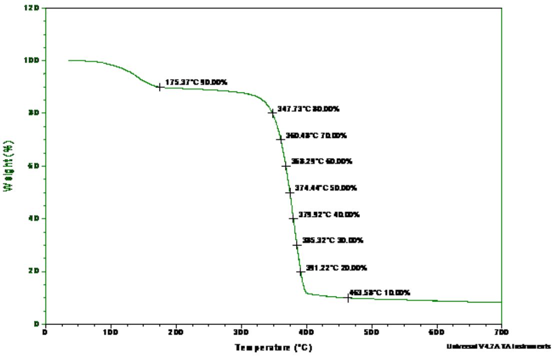

Figure 7: TGA graph for PLA Nanocomposites

TGA Analysis determines the thermal stability of films. Fig 6&7 shows the thermal stability of the films. Weight loss is determined w.r.t temperature. PLA with nanocomposites begins to degrade around 180°C & withstand a temperature upto 450ºC. Whereas, PLA will start deforming around 280°C & withstand a temperature upto 400°C. Therefore as clay and additive concentrations increases Thermal Stability Increases.

5.3 Film Microstructure

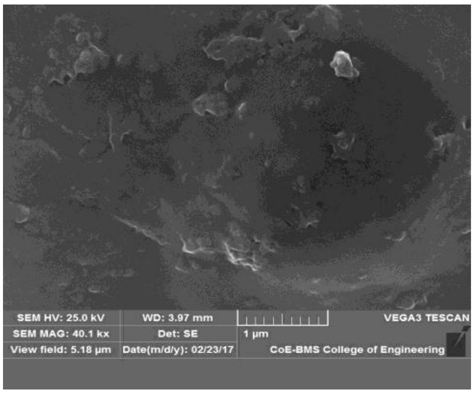

Figure 6: SEM images for Pure PLA

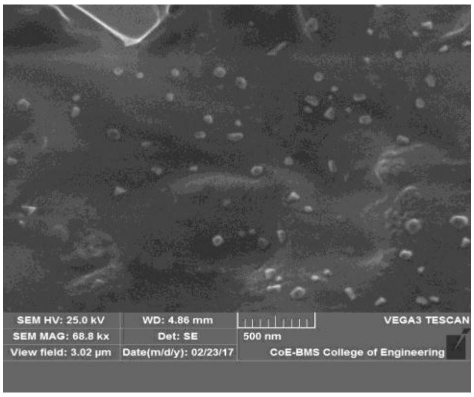

Figure 7: SEM images for PLA Nanocomposites

Pure PLA has uniform structure with holes and cracks, which confirms the WVP of this biopolymer. Whereas, Nanocomposites showed a good distribution and interfacial adhesion of nano particles in polymer matrix so, structure became more closed.

5.4 XRD Pattern

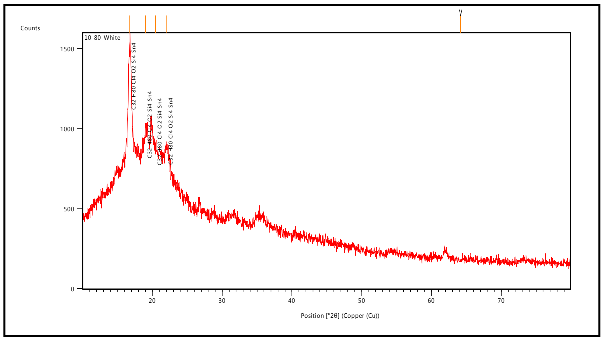

Figure 8: XRD Pattern for Pure PLA

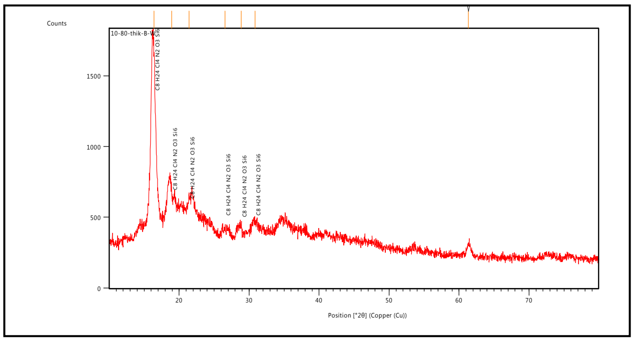

Figure 9: XRD Pattern for PLA Nanocomposites

5.5 FT-IR Properties

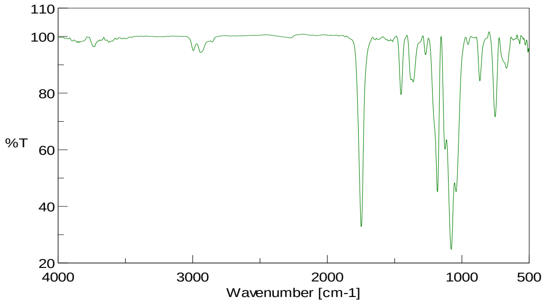

Figure 8: XRD Pattern for Pure PLA

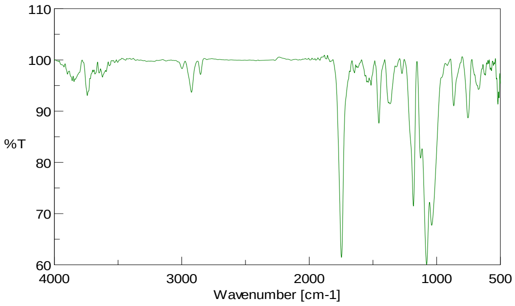

Figure 9: XRD Pattern for PLA Nanocomposites

FT-IR can be identified segment interactions and provide informations about phase behaviour of the polymer composite.

6. CONCLUSION

It is biodegradable, cheap and to be physical or chemical modified easily.The environmental problems will be no longer as severe as today.At present and in the near future, different Physical and chemical approaches are effective strategies to develop starch-based Completely biodegradable polymers of appropriate biocompatibility, degradation rate and physical properties for various applications.

6.1 Advantages

- PLA is derived from renewable resources such as corn, potato starch, Sugar Beets, Cheese Whey.
- The main advantage of bio-degradable materials are it breaks down naturally in the earth.
- PLA plastics are compostable, thermoplastic, biocompatible Polymer.
- PLA plastic is competitive with conventional plastic in terms of cost, since petroleum prices are only going up.
- PLA is more safe, since there is no danger of explosions in its production (as in the case with using petroleum products).
- Improperly disposed PLA plastics can contaminate recycling processes.

6.2 Applications

- PLA is widely used in Food Packaging, Tissue Engineering, Drug delivery systems and in Human medicine.

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