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Nilay Tuccar Kilic, Buse Nur Can, Mehmet Kodal, and Guralp Ozkoc

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Effects of Epoxy-POSS Nanoparticles on the Properties of PLA/TPU Blends

Nilay TUCCAR KILIC, Buse Nur CAN, Mehmet KODAL, Guralp OZKOC*

Department of Chemical Engineering, Kocaeli University, 41380, Kocaeli/Turkey

nilaytuccar@yahoo.com.tr

**guralp.ozkoc@kocaeli.edu.tr*

Abstract: In this study, the influence of epoxy functionalized polyhedral oligomeric silsesquioxanes (G-POSS) loading level on the mechanical, thermal and rheological properties of poly(lactic acid) (PLA)/thermoplastic polyurethane (TPU) blends were investigated. The blends were prepared in an Xplore Instruments MC15 twin-screw microcompounder at 200°C barrel temperature and 100 rpm screw speed. Mechanical properties were characterized by tensile and impact tests, thermal properties were determined via differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA). Rheological properties were examined by oscillatory rheometer. A decreasing trend in tensile strength and thermal degradation temperature were obtained for PLA/TPU blends at high G-POSS loading levels. G-POSS loading level did not exhibit significant effect on glass transition, crystallization and melting temperatures of the blends.

Keywords: polylactide, thermoplastic polyurethane, blends, POSS.

PACS: 61.25.hk

INTRODUCTION

Petrochemical based non-degradable polymers have negative affects to the ecological system. Biodegradable polymer usage is a solution to solve this problem [1]. Poly(lactic acid) (PLA) is a linear, semi crystalline, aliphatic, biodegradable polyester that can be produced from lactic acid by the fermentation of renewable sources such as whey, corn, potato, or molasses [2]. PLA has a relatively high strength and high modulus but its brittleness and low toughness confines its application. Blending PLA with flexible polymers is a practical and economical way to toughen PLA products. Elastomeric thermoplastic polyurethanes (TPUs) can be used to toughen PLA; since the soft segment of a TPU is mainly polyester, therefore; it is expected to have a good compatibility with PLA. Nanofillers can enhance the mechanical properties of toughened systems [3].

Polyhedral oligomeric silsesquioxanes (POSS) are organic/inorganic hybrid materials used as nanofillers with possibly reinforcement of polymers. The incorporation of POSS with reactive groups into a polymer matrix creates a possible nanoscale reinforcement, in which POSS molecules may link to polymer chains covalently [4].

In the current study, it was aimed to investigate the influence of epoxy functionalized POSS (G-POSS) loading level on mechanical, thermal, and rheological properties of PLA-TPU blends. The reason of choosing epoxy functionality is its potential of making covalent bonding with –COOH functional groups of PLA and –CNO functional groups of TPU.

EXPERIMENTAL

Materials

Poly(lactic acid) (PLI005, MFI: 10-30 g/10 min at 190°C/2.16 kg) was purchased from NaturePlast Company, France. Caprolactone diol based commercial thermoplastic polyurethane (TPU) (75 Shore A hardness) was obtained from Lubrizol, USA. Glycidylisobutyl-POSS (G-POSS) was purchased from Hybrid Plastics Company, USA. G-POSS can be dissolved in THF, chloroform and hexane as PLA and TPU does, which points out a possible compatibility. PLA and TPU are in pellet form; G-POSS is in crystalline white powder form.

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Sample Preparation

PLA/TPU/G-POSS blends having different POSS contents (0.25, 0.5, 1 and 3 wt%) were prepared via melt blending in a twin-screw microcompounder (15 mL Microcompounder, Xplore Instruments, The Netherlands) at a screw speed of 100 rpm. PLA/TPU ratio was kept constant as 90/10 by weight. Before compounding, PLA and TPU were dried in a vacuum oven at 65 °C for 24 h. Prior to processing, predefined amount of PLA, TPU and POSS were dry-mixed. The barrel temperature of microcompounder was set to 200 °C. The mixing time was kept constant at 3 min. In order to prevent thermo-oxidative degradation of PLA, the barrel was continuously purged with Argon. The melts were directly transferred with preheated transfer cylinder to DSM Xplore 12 mL laboratory injection-molding machine to obtain standard test bars according to ISO527/5a. The melt and mold temperatures were set 200°C and 30°C, respectively. The samples were abbreviated with respect to POSS loading level, for instance G-POSS 0.25 represents the composite including 0.25 wt % glycidylisobutyl-POSS.

Characterization

A universal tester (Instron Universal Testing Machine) was used to analyze tensile behavior of the blends. The load cell used was 5 kN and the crosshead speed for the measurements was fixed at 10 mm/min. Measurements were performed, at room temperature, on dog-bone shaped tensile bars having dimensions according to ISO 527 standard. Notched Izod impact tests were performed based on ISO 180 standard with an Instron CEAST 9050 model impact testing machine. Differential scanning calorimeter (DSC) analyses were performed using Mettler Toledo DSC1 Star System in nitrogen atmosphere. The analysis was conducted from -90°C with a heating rate of 10°C/min to 200°C with a heating rate of 10°C/min. Thermogravimetric Analysis (TGA) were performed Mettler Toledo TGA1 Star system. Samples were heated from room temperature to 600°C in nitrogen atmosphere. Rheological tests were performed on Anton Paar Modular Compact Rheometer MCR 102 with plate geometry of 25 mm in diameter. Frequency sweep measurements were carried out at 190°C, 0.1-600 rad/s angular frequency, 1% shear strain and 1mm gap.

RESULTS

Izod impact strength, energy absorbed prior to failure, and elongation at break of PLA/TPU and PLA/TPU/G-POSS blends were higher than that of neat PLA because of the higher intrinsic flexibility of TPU. The impact strength of PLA/TPU blends first lowered by the addition of G-POSS; then started to increase by further addition of G-POSS. In addition, increasing G-POSS content demonstrated a decreasing trend in tensile strength in the presence of G-POSS.

TABLE 1. Mechanical properties of PLA, TPU, PLA/TPU, PLA/TPU/G-POSS blends.

Material	Izod Impact Strength (kJ/m^2)	Tensile Strength (MPa)	Energy absorbed in tensile test prior to failure (J)	Elongation at break (%)
Neat PLA	2.4 ±0.3	55.4 ±3.8	1.1 ±0.1	9.1 ±1.6
Neat TPU	27.7 ±1.0	37.5 ±2.8	49.2 ± 3.8	857.0 ±20.2
PLA/TPU	4.5 ±0.3	52.8 ±1.7	3.0 ±0.1	22.9 ±4.7
PLA/TPU/G-POSS0.25	4.0 ±0.3	49.9 ±0.7	2.1 ±0.3	23.4 ±7.5
PLA/TPU/G-POSS0.5	4.1 ±0.2	48.8 ±0.8	3.6 ±0.5	27.5 ±8.6
PLA/TPU/G-POSS3	4.4 ±0.2	45.6 ±0.8	3.7 ±0.4	28.9 ±3.3

DSC thermograms (Figure 1) showed that there was no significant effect of GPOSS addition on glass transition, cold crystallization and melting temperatures of PLA/TPU/G-POSS blends. Thermal decomposition temperatures of PLA/TPU and PLA/TPU/G-POSS blends were higher than that of neat PLA (Figure 2). TPU addition to PLA provided better thermal stability (Table 2).

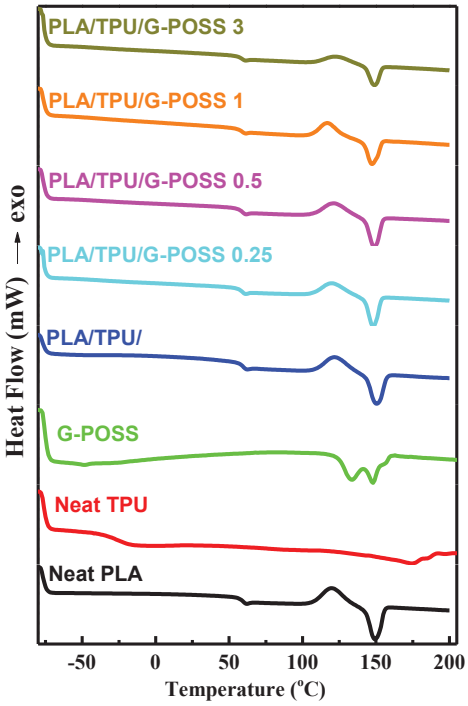


FIGURE 1. DSC thermograms of PLA, TPU, PLA/TPU, PLA/TPU/G-POSS blends.

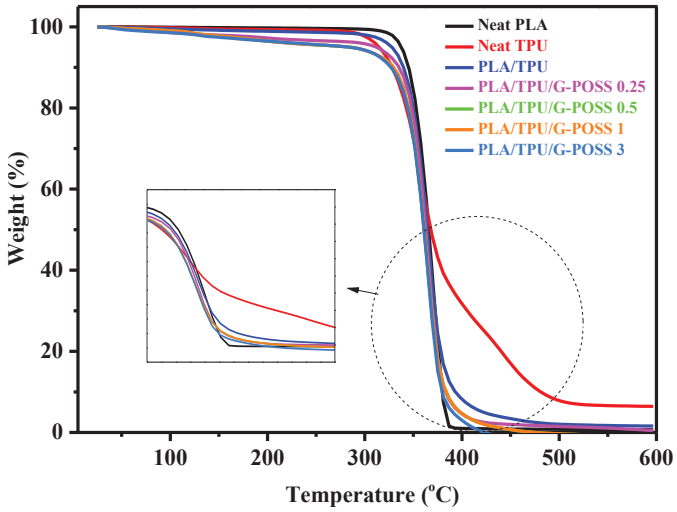


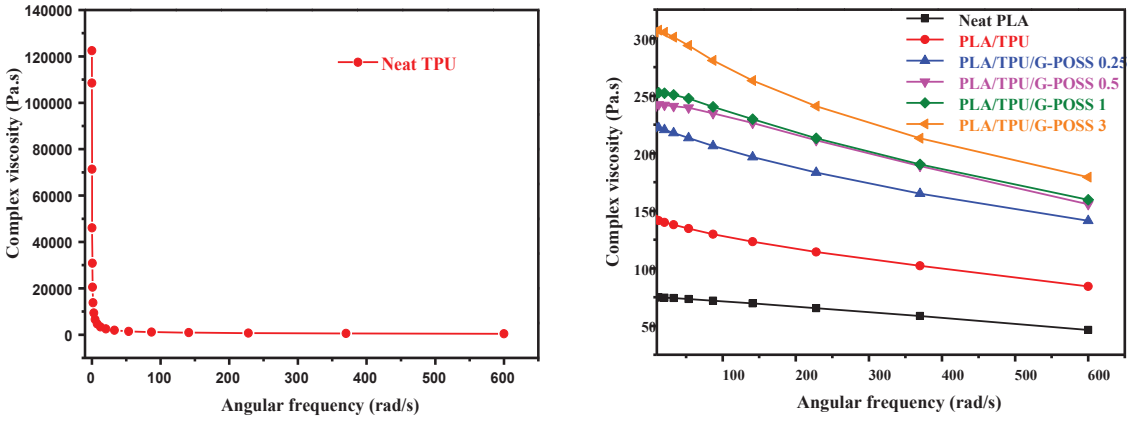
FIGURE 2. TGA thermograms of PLA, TPU, PLA/TPU, PLA/TPU/G-POSS blends.

TABLE 2. TGA results of PLA, PLA/TPU, PLA/TPU/G-POSS blends.

Material	T_{d10} (°C)	T_{d90} (°C)	$T_{decomposition}$ (°C)
Neat PLA	345.7	377.4	347.1
PLA/TPU	340.1	392.5	364.3
PLA/TPU/G-POSS0.25	333.8	383.2	362.6
PLA/TPU/G-POSS0.5	328.4	382.6	361.3
PLA/TPU/G-POSS1	329.6	381.9	361.4
PLA/TPU/G-POSS3	324.4	377.2	362.4

*T_{d10}, T_{d90} represent the decomposition temperature at which 10%, 90% weight loss occurs, respectively.

Rheological measurements demonstrated that both PLA/TPU and PLA/TPU/G-POSS blends had higher viscosities in comparison to neat PLA (Fig 3). The maximum viscosity at 190°C was obtained for neat TPU. The increasing content of G-POSS enhanced the melt viscosity of PLA/TPU. This can be attributed to a possible interaction of GPOSS with PLA or TPU via physical or chemical bonding which restricts the flow of polymer chains.



(a) (b)

FIGURE 3. Angular frequency vs. viscosity plot of (a). TPU, (b). PLA, TPU, PLA/TPU, PLA/TPU/G-POSS blends.

CONCLUSIONS

Mechanical, thermal, rheological properties of PLA were improved by melt blending with TPU. The incorporation of 0.25% GPOSS to the PLA/TPU blend decreased impact strength with respect to neat PLA/TPU, but further addition of GPOSS improved the impact strength. The tensile strength was decreased with GPOSS addition. The rheological properties were also affected by the addition of GPOSS to PLA/TPU system.

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