Effect of Ethylene–Butylacrylate–Glycidyl Methacrylate on Compatibility Properties of Poly (butylene terephthalate)/Thermoplastic Polyurethane Blends

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Abstract

Different proportions of poly (butylene terephthalate) (PBT)/thermoplastic polyurethane (TPU)/poly (ethylene–butylacrylate–glycidyl methacrylate) (PTW) blends were prepared by extrusion and injection molding. In this study, the PTW effect on compatibility properties of PBT/TPU blends was investigated from morphology, mechanical and thermal properties of the blends. Infrared spectroscopy indicated that there were reactions between the hydroxyl or carboxyl groups in PBT and a small portion of the epoxy groups in PTW during melt blending, and these reactions benefitted PTW and PBT to form grafted structures, which were conducive to viscosity increase and affected the crystallization of the blend. The morphological images obtained by scanning electron microscopy showed that in the PBT/TPU/PTW blend, the TPU particles, as the dispersed phase, were well distributed, and the phase interface between TPU and PBT became less and less obvious as the PTW content increased. It was proved that the melting point (T_m) and the crystallization temperature (T_c) of the blend decreased with the PTW content increase by the differential scanning calorimetry. It was shown by the dynamic mechanical thermal analysis spectra that the compatibility of PBT/TPU blends was enhanced by the addition of PTW. Mechanical property measurements indicated that PTW could result in a significant increase in impact strength by about 315%, while the flexural and tensile strength of PBT/TPU blends were decreased slightly.

Keywords: TPU; PBT; Compatibility; PTW copolymer; Blend.

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1. Introduction

As a recognized low-cost and efficient alternative method for developing new polymer materials, polymer blending has received more and more attention in recent years.^[1-6] Scientists have also carried out a lot of research in this area.^[7-11] As a semi-crystalline polymer, poly (butylene

terephthalate) (PBT) has been widely used due to its advantages of certain rigidity, high crystallization rate, fatigue resistance, heat resistance and solvent resistance.^[12-14] However, PBT has the disadvantage of insufficient toughness, especially the notched impact strength is not enough, and is limited in some applications. In order to improve its toughness, more attention has been paid in recent years. One of the methods to improve the toughness of PBT is usually blended with rubber or other elastomers.^[15,16] Thermoplastic polyurethanes (TPU) have been widely used due to their superior properties including high tensile, low temperature flexibility and good resistance to wear, tear, oil and solvent. As a linear copolymer, the microstructure of TPU is made up of hard segments and soft segments separated by microphase. Among them, the soft segments usually form an elastomer matrix to ensure the elasticity and low temperature performance of the TPU, while the hard segments serve as a

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multifunctional binding point for physical crosslinking and reinforcing fillers.^[17]

Poly (ethylene-butylacrylate-glycidyl methacrylate) (PTW) is a kind of terpolymer with epoxy groups. The epoxy groups in PTW can easily combine with hydroxyl and carboxyl groups in PBT when PTW is blended with PBT.^[18] The butyl acrylate segment in PTW can achieve better low temperature performance and good compatibility with TPU. As a result, PTW is a potential impact modifier for PBT/TPU due to its reactive ability with PBT and rubbery toughening with TPU.^[19]

In this study, the morphology, and the properties including thermal properties and mechanical properties of PBT/TPU blends with different contents of PTW were characterized. The results showed that only blending with TPU does not significantly improve the impact strength. In order to further toughen the PBT/TPU blend, PTW was added as a compatibilizer to the PBT/TPU blend without significantly impairing other properties and the compatibilizing effect of PTW was characterized.

Using a twin-screw extruder, all the components were melting blended together to prepare pellets, and then the specimens were prepared by injection molding. Infrared spectroscopy, scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) were employed to characterize the blend. The morphology of the blend ultimately determined its mechanical properties, which were mainly measured by tensile, bending and impact tests. It is a method to greatly toughen the PBT/TPU blend and improve its impact strength by modification with PTW, which provides a good idea for expanding the application of PBT in the engineering field. The reactive compatibilizing effect of PTW was the main reason for this improvement, which had been confirmed by the above characterization methods.

2. Experimental details

2.1 Materials

The PBT (named 1200-211M, Yizheng Chemical Fiber Group Corp., China) has a melt flow index (MFI) of 42-49 g/10min at 235 °C/2.16 kg. TPU was obtained from Wanhua Chemical Group Co., Ltd., China, its grade was WHT1195 with a density of 1.2 g/cm³. PTW, named Elvaloy® with ethylene/butylacrylate/glycidyl methacrylate (E/BA/GMA) monomer weight ratio of 66.75/28/5.25, was kindly provided

by DuPont Co., and its MI was 12 g/10 min at 190 °C and 2.16 kg.

2.2 Preparation of the blends

In order to avoid possible degradation before processing, PBT particles were placed in an air oven at 120°C for 4 hours, and TPU and PTW particles were placed in an air oven at 70°C for 4 hours for drying. The content of PTW was 0 to 7.0% by weight relative to a 100% PBT/TPU blend. PBT/TPU/PTW (70/30/7) represents a 70/30 blend with 7.0 wt% PTW. Table 1 lists the composition of the samples investigated in this study.

The blends of PBT/TPU/PTW, with a fixed PBT/TPU ratio (70/30) and different contents of PTW (0, 1, 3, 5, and 7 wt%), were fabricated in a twin-screw extruder (Bernstorff, 25A, L type) at the speed 200 r/min (rpm). From the die to the hopper, the barrel temperature was set to 250-250-250-250-250-250-230-60°C. The rod of the blends extruded by the extruder was immediately cooled by a water bath, and then cut into pellets, followed by drying in an oven at 120°C for 4 h. The final blend particles were injected into an impact and tensile samples by an injection molding machine (HTB110X/1, Guangzhou Borch Machinery Co. Ltd. China) for experimental observation. The injection temperature was set to 240-245-245-250-50 °C from the hopper to the mold.

2.3 Chemical structural analysis

Infrared (IR) spectroscopy of the blends and pure PBT was conducted with a Nexus 670, Nicolet, (USA) Fourier-transform infrared (FTIR) spectroscopy instrument. The specimens were hot press to thin films of 0.5 mm at 250 °C before testing.

2.4 Melt flow index (MFI)

MFI measurements of MTS ZRZ2452 were employed to characterize the melt viscosity according to the standard of ASTM D1238-04. During test, the temperature was set at 230 °C and the load was 2.16 kg. The following formula can be used to calculate the MFI value.

$$MFI = \frac{m \times 600}{t} (g/10min)$$

where m and t are the average mass and time needed for each cut segment, respectively.

Table 1. Components of different samples.

Component Sample	PBT (wt%)	TPU (wt%)	PTW (pph)
1	100	—	—
2	70	30	—
3	70	30	1
4	70	30	3
5	70	30	5
6	70	30	7

Fig. 1. Reactions between PTW and PBT during melt blending

2.5 SEM

A scanning electronic microscope (SEM, model name: S-3700N, HITACHI) was used to image the fracture surface of the blends. After immersing it in liquid nitrogen for about 15 minutes, the cryo-fracture samples (4 mm thick) were obtained. The specimen surface was sprayed with gold to enhance conductivity during the observation.

2.5 DSC

A differential scanning calorimetry (DSC, model 204c, Netzsch, Germany), which had liquid nitrogen cooling accessories, was used to perform DSC evaluation. The samples were scanned twice. First, at a rate of $10^{\circ}\text{C}/\text{min}$, the samples were heated from room temperature to 260°C , held there for 3 minutes, then under nitrogen decreased to a 30°C at a rate of $10^{\circ}\text{C}/\text{min}$. The second time, at a rate of $10^{\circ}\text{C}/\text{min}$, the samples were scanned from 30 to 260°C . The second heating and first cooling scans were recorded. All thermal parameters provided were averages of three repeat measurements.

2.6 DMA

A Netzsch DMA242c instrument was used for dynamic mechanical analysis (DMA) at 1 Hz frequency, 0.15 mm

oscillation amplitude and heating rate of $3^{\circ}\text{C}/\text{min}$. The temperature range of the blends and pure PBT were -180°C to $+150^{\circ}\text{C}$ and -100°C to $+100^{\circ}\text{C}$, respectively. The samples' dimensions were $10\text{mm} \times 4\text{mm} \times 1\text{mm}$.

2.7 Mechanical test

A tensile test was performed at room temperature in the tensile mode by a USA Instron machine (model 5566) according to GBT 1447-2005 to measure the elongation at break and yield strength. The impact strength test was performed by a USA Instron pendulum impact tester (model POE2000). All the values were average attained from five independent tests.

3. Results and discussion

3.1 IR spectra

PTW was used as a modifier to toughen the PBT/TPU in reference.^[18] The study showed that the hydroxyl and carboxylic group in PBT could easily react with the epoxy groups in the PTW. Fig. 1 illustrates the main chemical reaction in the PBT/TPU/PTW blends.

Fig. 2 shows the IR spectra of PBT, PTW, TPU and PBT/TPU/PTW blends in the range from 800 to 950 cm^{-1} . In Fig. 2, there are two absorption peaks at 843 and 911 cm^{-1} in the IR spectra of PTW, which are characteristics of the unique

terminal epoxy groups in PTW.^[20] There are not similar peaks at 843 and 911 cm^{-1} in the IR spectra of the PBT/TPU/PTW blends and PBT compared with that of the PTW. After melt blending with the PBT/TPU, the epoxy groups disappeared, which provided evidence that there were the ring-opening reactions between the PBT and PTW.

Fig. 2. IR spectra of (a) pure PTW, (b) pure PBT, (c) PBT/TPU/PTW (70/30/3), (d) PBT/TPU/PTW (70/30/5), and (e) pure TPU.

Fig. 3. IR spectra of different samples: (a) pure PTW, (b) pure PBT, (c) PBT/TPU/PTW (70/30/3), (d) PBT/TPU/PTW (70/30/5), (e) pure TPU.

From Fig. 3, it can be seen that there exist two small peaks at 2900 and 2875 cm^{-1} in the 2800 - 3205 cm^{-1} range in the spectral curves of PTW, TPU and PBT/TPU/PTW blends, which indicates that the number of $-\text{CH}_3$ groups is much less than the number of $-\text{CH}_2$ groups. The main reason of the result above may be that there are long ethylene segments in TPU

and PTW. This structure in TPU and PTW is very similar to long-chain structure, which indicates that PTW is highly compatible with TPU. Therefore, IR spectrum analysis confirmed that PTW had a reactive compatibilization effect on PBT/TPU/PTW blends.

3.2 Melt flow index

Fig. 4 shows the melt flow index (MFI) values of PBT/TPU samples with different contents of PTW. It can be seen from Fig. 4 that the MFI of pure PBT is 47.5 g/10min. For PBT/TPU/PTW blends, the MFI value decreased with the increase of PTW content, which indicated that the melt viscosity of the blends increased with the increase of PTW. The decrease of MFI implied that the mobility of the molecular chain of the PBT/TPU/PTW blend was weakened, which is mainly due to the grafting induced by the reaction between PBT and PTW.

Fig. 4. The melt flow index (MFI) as a function of PTW content in the PBT/TPU (70/30) blends.

3.3 Morphology

Fig. 5 (a-d) shows the SEM images of pure PBT, PBT/TPU/PTW(70/30/0), PBT/TPU/PTW(70/30/3), PBT/TPU/PTW(70/30/7) blends. It can be observed from Fig. 5(b) that the TPU was poorly dispersed in the PBT matrix. The interfaces of the PBT and TPU were smooth and the two phases were independent of each other in Fig. 5(b), which implied that PBT had a poor compatibility with TPU and weak interaction with the TPU. This poor compatibility and weak interaction of these two phases would lead to poor toughness of the PBT/TPU blends. Meanwhile, with the addition of PTW, it can be seen from Fig. 5(c) and (d) that the size of the TPU particles became smaller and the distribution was more uniform. When 7wt% PTW was added, the phase state of TPU changed significantly. The particle distribution of TPU was more uniform, and the interface between TPU and PBT became more blurred, which indicated that the interaction between TPU and PBT was better. The increase in the interaction between PBT and TPU indicated that PTW had an

effective compatibilization effect on the PBT/TPU/PTW blend and lead to an increase in the mixing of components.

Fig. 5. SEM micrographs of fracture surfaces for (a) pure PBT, (b) PBT/TPU (70/30), (c) PBT/TPU/PTW (70/30/3), (d) PBT/TPU/PTW (70/30/7).

Fig. 6 DSC non-isothermal crystallization curves of PBT/TPU/PTW blends.

3.4 DSC analysis

It was investigated that the melting behavior and crystallization of the PBT/TPU/PTW and PBT/TPU blends via DSC. Fig. 6, the cooling curves of the blends, shows that the crystallization temperature (T_c) of the blends decreased with increase of the PTW. And this may be due to the chemical reaction between PTW and PBT, which prevented the accumulation of PBT macromolecular chains to form an orderly arrangement.

It can be seen that there are a main melting peak and a minor lower temperature peak in the melting curves in Fig. 7, which is the main feature of PBT.^[21-23] The small peak in Fig. 7 is

mainly due to the partial melting of the original imperfect crystal, which is the result of rapid crystallization rate of PBT.^[24] The main melting peak is mainly due to the recrystallized crystallites caused by the secondary filling crystallization during the heating process.^[25-27] As PTW content was increased, the melting point was lowered and the small peak disappeared. It is suggested that the addition of PTW caused a considerable improvement in the compatibility of the blend.

Fig. 7. DSC melting curves of PBT/TPU/PTW blends.

3.5 DMA analysis

The DMA data of the blend can be used to characterize the glass transition temperature of the components of the blend and provide information for analyzing the phase structure and interphase mixing.^[28-30] Fig. 8 shows the relationship between the loss factor ($\tan\delta$) and temperature of PBT/TPU/PTW, PBT/TPU, PBT and the blends. In this experiment, PBT had a glass transition temperature at 72°C. It could be seen that there were two transition temperatures (T_{g1} and T_{g2}) in the PBT/TPU blends. The glass transition of the PBT component produced a large dynamic mechanical damping peak at about 65°C, and the relaxation of the glass transition temperature of the TPU in the blend induced the broad peak at about -37°C. When PTW content was 5 wt%, the high temperature peak representing the PBT component shifted to 66°C, and the peak corresponding to the TPU component increased to -38°C. When modified by PTW, the two peaks of TPU and PBT tended to be close. Because of low intensity, the PTW glass transition peak at ~ -30°C^[31] could not be determined. It can be clearly seen from the above results that the addition of PTW can improve the compatibility of the PBT/TPU blend, which is consistent with the SEM observation results.

Fig. 9 shows the relationship between the storage modulus (E') and temperature of PBT/TPU, pure PBT and two PBT/TPU/PTW blends. The E' of the PBT/TPU blend was decreased to 940 MPa at 50°C, while the E' of pure PBT was 1879 MPa, which was mainly due to flexible chains of TPU.

When the PTW content was 3 wt%, the E' was decreased from 940 to 843 MPa, which was mainly due to the soft segment of PTW. However, when PTW was added to a content of 5 wt%, E' was only further reduced by 3.68%. This can attribute to the fact that PTW will have more opportunities to react with PBT, therefore, creating greater friction forces with the chains of grafted PTW, which could compromise the decline brought by the soft segments of PTW.

Fig. 8 DMA loss factor ($\tan\delta$) versus temperature of pure PBT and the blends.

Fig. 9 DMA storage modulus (E') versus temperature of pure PBT and the blends.

3.6 Mechanical properties

The mechanical properties of polymer blends depend on the morphology and interfacial adhesion of the blends. The multiphase morphology lacking adhesion between the polymer components leads to premature failure, which results in poor mechanical strengths. In contrast, enhanced interfacial adhesion results in good mechanical strength. **Fig. 10** illustrates the relationship between the flexural strength and

tensile strength of the blend and the PTW content. In **Fig. 10**, it is observed that the tensile strength and flexural strength decreased monotonously with increasing the PTW content. When 7 wt% of PTW was added to PBT/TPU, the flexural strength and tensile strength of the blend were reduced by 18.3% and 17.2%, respectively.

Fig. 11 displays that the notched impact strength of the PBT/TPU (70/30) blend increases with the increase of PTW content. When the PTW content was 7 wt%, the impact strength was increased from 9.7 to 40.2 kJ/m², which was significantly higher than the impact strength of the PBT/TPU blend by about 314.4%. It can be seen that the impact strength could be remarkably improved by adding PTW into the blend. This was attributed to the existence of PTW, which increased the compatibility of PBT/TPU.

Fig. 10 Tensile strength and flexural strength of PBT/TPU/PTW blends.

Fig. 11 Impact strength of PBT/TPU/PTW blends.

4. Conclusions

Based on the above experimental results, it can be concluded that PTW can effectively compatibilize PBT/TPU blends. The

addition of PTW can significantly improve the impact strength. When the PTW content was 7 wt%, a super tough PBT/TPU blend was obtained. FTIR observations showed that there were reactions between terminal epoxy groups in PTW and the carboxylic acid or hydroxyl groups in PBT, resulting in a decrease in the MFI of the blends. Adding PTW can effectively increase the compatibility of PBT and TPU. SEM showed that the interaction between the TPU dispersed phase and the PBT matrix was enhanced. DSC analysis implied that the small molecules generated from the hydrolysis of PBT would act as “plasticizers” in the crystallization process, and thus led to a decrease of crystallization temperature. The compatibility of PBT and TPU was enhanced by the addition of PTW, which was shown by the loss factor ($\tan\delta$) analysis. The grafting reaction between the blend components was also indirectly proved by the storage modulus (E') diagram.

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Supporting information

Not applicable

Conflict of interest

There are no conflicts to declare.

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