

Material Characterisation

Thermal, crystallization, and rheological characteristics of poly(trimethylene terephthalate)/poly(butylene terephthalate) blends

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Abstract

Blends of poly(trimethylene terephthalate) (PTT) and poly(butylene terephthalate) (PBT) were miscible in all of the blend compositions studied, as evidenced by an observed single and composition-dependent glass transition temperature for each blend composition. The variation of the glass transition temperature with the blend composition was well predicted by the Gordon–Taylor equation, with fitting parameter being ca. 6.9. The cold crystallization (peak) temperature was found to increase, while the melt crystallization (peak) temperature was found to decrease, with increasing PTT content. The subsequent melting behavior for these blends (after cold crystallization) showed the melting point depression behavior, in that the melting (peak) temperature for each component was lowered with increasing content of the other component. During crystallization, the pure components crystallized simultaneously to form their own crystals. The blend having 60 percent by weight of PTT showed the lowest apparent degree of crystallinity. The steady shear viscosities for the pure components and the blends showed slight decrease with increasing shear rate (within the shear rate range of 0.25–25 s⁻¹), with those of the blends lying in between those of the pure components.
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1. Introduction

Poly(ethylene terephthalate) (PET) and poly(butylene terephthalate) (PBT) are linear aromatic polyesters which have been commercialized for a long time. Both PET and PBT are versatile polymeric commodities. Poly(trimethylene terephthalate) (PTT) has recently been introduced commercially by Shell Chemicals under the tradename Corterra™. PTT has properties in between those of PET and PBT, by taking an unusual combination of the outstanding properties of PET and pro-

cessing characteristics of PBT. These characteristics make PTT highly suitable for uses in fiber, film, and engineering thermoplastics applications.

Polymer blending is an attractive alternative for producing new polymeric materials with desirable properties without having to synthesize a totally new material. Other advantages for polymer blends are versatility, simplicity, and inexpensiveness. Numerous published articles related to various aspects of binary blends of polyesters are available in the open literature. Some of these are, for examples, blends of PET and PBT [1–8], PBT and an amorphous co-polyester of cyclohexane dimethanol, ethylene glycol, and terephthalic acid (PETG) [9], and PTT and poly(ether imide) (PEI) [10].

In PET/PBT blends, Escala and Stein [1] reported that the blends showed a single and composition-dependent

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glass transition temperature at all compositions, suggesting that PET and PBT were miscible in the amorphous state. Similar results were also reported by others [5,6]. Based on various experimental techniques, Escala and Stein [1] reported that, upon crystallization, PET and PBT did not co-crystallize. Avramova [5] confirmed such findings and added that, even though each component formed its own crystalline phase upon crystallization, both components could crystallize concurrently at all compositions of the blends and the presence of one crystalline phase did not deter or enhance the crystallization rates of the other.

In PBT/PETG blends, Nabi Saheb and Jog [9] reported that the blends exhibited a single and composition-dependent glass transition temperature at all compositions, suggesting miscibility between PBT and PETG molecules in the amorphous state. The miscibility of PBT and PETG molecules was confirmed by the negative value of the interaction parameter. They also reported that the blends exhibited composition-dependent melting point depression, a direct result of the presence of PETG. In addition, an observed decrease in the crystallization rates for the blends as evidenced by an increase in the crystallization half-time was attributed to either an increase in the glass transition temperature or to a decrease in the ability of PBT molecules to mobilize due to the presence of PETG molecules.

Recently, Huang and Chang [10] studied miscibility, melting, and crystallization behavior of PTT/PEI blends. They observed that the blends showed a single and composition-dependent glass transition temperature over the entire compositional range studied, indicating that the blends were fully miscible in the amorphous state. They also reported that recrystallization of PTT during a heating scan in a differential scanning calorimeter (DSC) was either retarded or fully inhibited by the presence of PEI component, a direct result of the decreased segmental diffusion of PTT molecules onto an existing growth face.

In the present contribution, blends of PTT and PBT were characterized for their thermal, crystallization, and rheological behavior. The objectives for this work are (1) to assess the miscibility of the blends in the amorphous state, (2) to investigate the effect of blend composition on non-isothermal cold and melt crystallization behavior, (3) to investigate the effect of blend composition on subsequent melting behavior (after non-isothermal cold crystallization), (4) to assess the effect of blend composition on the apparent degree of crystallinity obtained, and (5) to assess the effect of blend composition on the rheological behavior.

2. Experimental details

2.1. Materials

Poly(trimethylene terephthalate) (PTT) was supplied in pellet form by Shell Chemicals (USA) (Corterra CP509201). The weight- and number-average molecular weights for this resin were determined to be ca. 78,100 and 34,700 Da, respectively. Poly(butylene terephthalate) (PBT) was supplied in pellet form by LG Chem (Korea) (LUPOX GP-2000). The weight- and number-average molecular weights for this resin were determined to be ca. 71,500 and 36,300 Da, respectively. Molecular weight characterization for these resins was carried out by size-exclusion chromatography (SEC).

2.2. Sample preparation

Both PTT and PBT resins were dried in a vacuum oven at 140 °C for 5 h and then were pre-mixed in a dry mixer to produce PTT/PBT pre-blends of 10, 25, 40, 50, 60, and 75% w/w of PTT, respectively. The pre-blends were then melt-mixed in a self-wiping, co-rotating twin-screw extruder (Collin, ZK 25), operating at a screw speed of 70 rpm and the die temperature of 260 °C. The extrudate was cooled in water and were palletized using a pelletizer (Planetrol, 075D2). The resulting blends were hereafter denoted x PTT/(1- x)PBT, where x is the weight percentage of PTT in the blends. Pure PTT, PBT, and each blend were later melt-pressed at 260 °C in a compression press (Wabash, V50H) under an applied pressure of 4.62×10^2 MN·m⁻² for 5 min before cooling down to room temperature to produce films of approximately 200 μm in thickness.

2.3. Differential scanning calorimetry measurements

A DSC (Perkin–Elmer, DSC-7) was used to record non-isothermal cold and melt crystallization exotherms and subsequent melting endotherms (after non-isothermal cold crystallization) for PTT, PBT and their blends. Calibration for the temperature scale was carried out using a pure indium standard ($T_m^0 = 156.6$ °C and $\Delta H_f^0 = 28.5$ J g⁻¹) on every other run to ensure accuracy and reliability of the data obtained. To minimize thermal lag between the polymer sample and the DSC furnace, each sample holder was loaded with a disc-shape sample weighing around 8.0 ± 0.5 mg which was cut from the as-prepared films. It is worth noting that each sample was used only once and all the runs were carried out under nitrogen atmosphere to prevent extensive thermal degradation.

The experiments started with heating PTT, PBT and their blends from 40 °C to a fusion temperature of 260 °C at a rate of 80 °C·min⁻¹ for a melt-annealing period

of 5 min in order to remove previous thermal histories, after which the samples were taken out and immediately quenched in liquid nitrogen to attain the completely amorphous state of the samples. In order to observe the non-isothermal cold crystallization and subsequent melting behavior, each sample was reheated again in DSC from 25 to 260 °C at a rate of 10 °C min⁻¹. After a melt-annealing period of 5 min at 260 °C, the sample was cooled down to 30 °C at a cooling rate of 10 °C min⁻¹ in order to observe the non-isothermal melt crystallization behavior. These experiments allow one to obtain values for the glass transition temperature T_g , cold crystallization peak temperature T_{cc} , apparent melting peak temperature T_m , and melt crystallization peak temperature T_c , respectively.

2.4. Crystal structure and crystallinity

Wide-angle X-ray diffraction (WAXD) was used to determine the crystal modification and apparent degree of crystallinity for PTT, PBT, and their blends which had been prepared in DSC by cooling from 260 to 30 °C at a cooling rate of 10 °C·min⁻¹. Each sample, after being taken out of the DSC sample holder, was pasted onto a glass sample holder using vaseline as adhesive. The WAXD intensity pattern for each sample was collected on an X-ray diffractometer (Rigaku, Rint2000), equipped with computerized data collection and analytical tools. The X-ray source (CuK α radiation, $\lambda = 1.54$ Å) was generated using an applied voltage of 40 kV and a filament current of 30 mA.

2.5. Shear viscosity measurements

Pure PTT, PBT, and PTT/PBT blend pellets were melt-pressed into circular disks of 1 mm in thickness and 25 mm in diameter. A rheometer (Rheometric Scientific, ARES RDA-II) with cone-and-plate geometry was used to study the steady and dynamic shear behavior of these materials. Before each measurement, the rheometer was heated up to 260 °C and the gap between the cone and plate was set at 0.052 mm. For a steady rate sweep test, the shear viscosity of the materials was determined as a function of shear rate. In the case of a dynamic measurement, the strain values were chosen such that the experiments could be performed in the linear viscoelastic region.

3. Results and discussion

3.1. Glass transition temperature of PTT/PBT blends

The presence of a single and composition-dependent glass transition temperature T_g , which was located between those of the pure components, is indicative of

miscibility between the two polymers in the amorphous state. Fig. 1a shows the DSC cold crystallization and melting thermograms for quenched PTT, PBT, and PTT/PBT blend samples recorded during heating at a heating rate of 10 °C·min⁻¹, Fig. 1b shows DSC melt crystallization exotherms for PTT, PBT, and PTT/PBT blend samples recorded during subsequent cooling at a cooling rate of 10 °C·min⁻¹. Careful analysis on each of the original cold crystallization and melting thermograms (such as those shown in Fig. 1a) using the DSC-7 software showed that a single T_g value was observed in each thermogram and the majority of the observed values for the blend samples were located between those of the pure components (i.e., $T_{g,PTT} = 43.1$ °C and $T_{g,PBT} = 34.8$ °C). This evidence suggests that PTT and PBT were miscible in the amorphous state. Even though the T_g s are not properly observed in Fig. 1a (which may be attri-

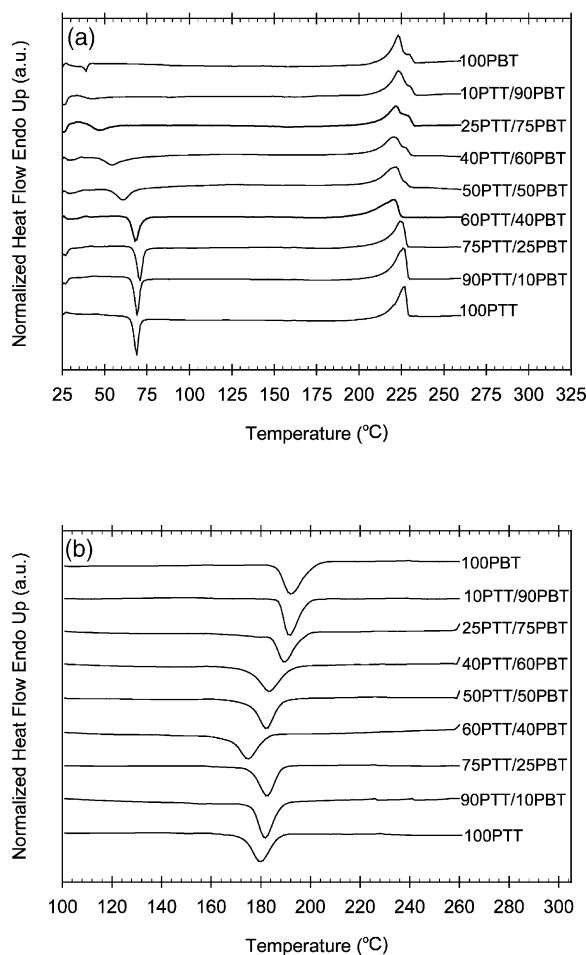


Fig. 1. (a) DSC cold crystallization and melting thermograms for quenched PTT, PBT, and PTT/PBT blend samples recorded during heating at 10 °C·min⁻¹, and (b) DSC melt crystallization exotherms for PTT, PBT, and PTT/PBT blend samples recorded during subsequent cooling at 10 °C·min⁻¹.

buted to the poor quality of the ascii data exported from the DSC-7 software and from the overlay plotting of these data in the same figure), the extracted values can be seen in Fig. 2.

A number of empirical models were proposed in order to predict the composition-dependence of T_g for a miscible polymer blend: some of these are the Gordon–Taylor [11], Fox [12], Couchman–Karasz [13], and Utracki [14] equations. These equations predict a monotonic dependence of the T_g value on the blend composition, without any cusp in the predicted curve. Among these, the Fox equation is widely used. In this model, the observed T_g value of the blend relates to the glass transition temperatures of the pure components 1 and 2 (i.e., T_{g1} and T_{g2}) and the blend composition according to the following equation [12]

$$\frac{1}{T_g} = \frac{w_1}{T_{g1}} + \frac{w_2}{T_{g2}} \quad (1)$$

where w_1 and w_2 are the weight fractions (in the amorphous state only) of components 1 and 2, respectively, and T_{g1} and T_{g2} are the respective T_g values of the pure components 1 and 2.

The Fox equation assumes random mixing between the two components, equal values of the enthalpic jump in the glass transition region between the two components (i.e., $\Delta C_{p1} = \Delta C_{p2}$), and no volume expansion between the two components during mixing. The dependence of the T_g value on the blend composition for PTT/PBT blends is illustrated in Fig. 2. The dotted line is the predicted composition-dependence of the T_g value for PTT/PBT blends according to the Fox equation. Apparently, the Fox equation overestimated the T_g value for the blends in all of the blend compositions.

Another popular equation used to predict composition-dependent behavior of T_g for a miscible polymer blend

is the Gordon–Taylor equation [11], which can be written as

$$T_g = \frac{w_1 T_{g1} + k w_2 T_{g2}}{w_1 + k w_2} \quad (2)$$

where k is an adjustable parameter. The solid line shown in Fig. 2 is the predicted composition-dependence of the T_g value for PTT/PBT blends according to the Gordon–Taylor equation, with the fitting k parameter being 6.89. Based on the predicted curve and the data shown in Fig. 2, good agreement between the observed T_g values and the prediction by the Gordon–Taylor equation was obtained only for blends having PTT content of 60 wt% or higher.

3.2. Crystallization behavior

From Fig. 1a, the cold crystallization (peak) temperature T_{cc} for PTT was observed at ca. 68.8 °C, while that for PBT was not observed (suggesting that PBT was able to crystallize fast enough during quenching the sample from a fusion temperature of 260 °C in liquid nitrogen). The observed T_{cc} values for PTT/PBT blends (taken from the thermograms shown in Fig. 1a) were plotted against the blend composition in Fig. 3. Obviously, a single and composition-dependent cold crystallization (peak) temperature was observed for most of the blends studied, except for the 10PTT/90PBT blend. The observed T_{cc} value for the blends was found to increase from ca. 47 °C in the 25PTT/75PBT blend to ca. 71 °C in the 75PTT/25PBT blend and level off to approach that of PTT. The results suggest that both PTT and PBT components in the blends crystallized simultaneously and that the presence of the more crystallizable PBT molecules in the blends having the PBT content of more than ca. 25 wt% helps promote the crystallizability of the blends.

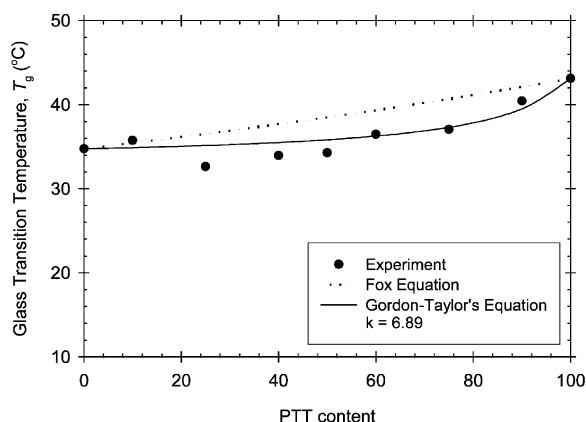


Fig. 2. Observed glass transition temperature T_g for quenched PTT, PBT, and PTT/PBT blend samples as a function of blend composition.

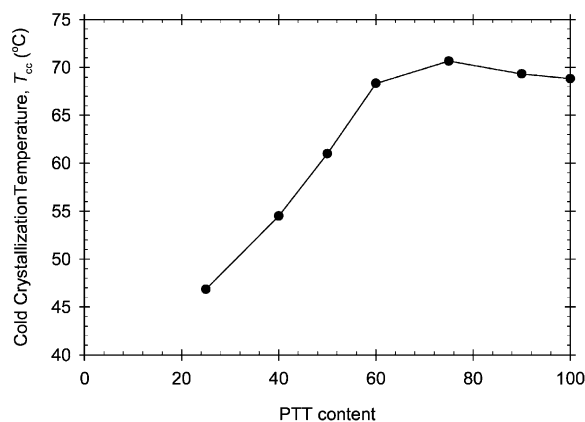


Fig. 3. Cold crystallization (peak) temperature T_{cc} for quenched PTT, PBT, and PTT/PBT blend samples as a function of blend composition.

According to Fig. 1b, the melt crystallization (peak) temperatures T_c for PTT and PBT were observed at ca. 179.8 and 192.1 °C, respectively. The observed T_c values for PTT/PBT blends (taken from the exotherms shown in Fig. 1b) were plotted as a function of the blend composition in Fig. 4. Evidently, a single and composition-dependent melt crystallization (peak) temperature was observed for all of the blends studied. Generally, the observed T_c value of the blends was found to decrease from that of PBT to approach that of PTT with increasing PTT content, with an exception for that of the 60PTT/40PBT blend which exhibited much lower value than that of PTT. The results suggest that both PTT and PBT components in the blends crystallized concurrently and that the presence of the less crystallizable PTT molecules in the PBT-rich blends retards the crystallizability of the blends, while the presence of the more crystallizable PBT molecules in the PTT-rich blends helps promote the crystallizability of the blends, with an exception for the 60PTT/40PBT blend.

Based on the single T_g value observed, PTT and PBT molecules in the 60PTT/40PBT blend should be miscible in the amorphous state (i.e., melt state). However, since the WAXD results indicated that each of the PTT and PBT components in the blends formed their own crystals during simultaneous crystallization (see later) and since the molar ratio in the 60PTT/40PBT blend was comparable, during cooling PTT and PBT components must undergo phase separation in order for each component to form its own crystalline phase. The kinetics of the phase separation, therefore, might be responsible for the lowest T_c value observed for the 60PTT/40PBT blend.

3.3. Crystal structure and apparent degree of crystallinity

The crystal structure and the apparent degree of crystallinity for PTT, PBT, and PTT/PBT blend samples non-

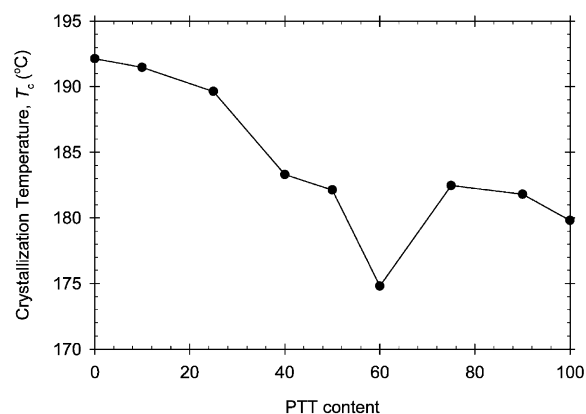


Fig. 4. Melt crystallization (peak) temperature T_c for melt-crystallized PTT, PBT, and PTT/PBT blend samples as a function of blend composition.

isothermally crystallized from the molten state at a cooling rate of 10 °C·min⁻¹ were observed using WAXD and the results are shown in Fig. 5. The characteristic X-ray peaks for PTT were observed at the scattering angles 2θ of ca. 15.3, 16.8, 19.4, 21.7, 23.6, 24.6 and 27.3°, which correspond to the reflection planes of (0 1 0), (0 $\bar{1}$ 2), (0 1 2), (1 0 $\bar{2}$), (1 0 2), (1 $\bar{1}$ 3), and (1 0 $\bar{4}$), respectively [15]. For PBT, the characteristic X-ray peaks were observed at the scattering angles 2θ of ca. 15.8, 17.0, 20.5, 23.2, and 25.0°, which correspond to the reflection planes of (0 $\bar{1}$ 1), (0 1 0), (0 1 1), (1 0 0), and (1 $\bar{1}$ 1), respectively [16]. Both PTT and PBT crystallize in a triclinic crystal structure, with the unit cell parameters being $a = 4.64$ Å, $b = 6.27$ Å, $c = 18.64$ Å, $\alpha = 98^\circ$, $\beta = 90^\circ$, and $\gamma = 111^\circ$ for PTT [17] and $a = 4.83$ Å, $b = 5.95$ Å, $c = 11.67$ Å, $\alpha = 100^\circ$, $\beta = 64^\circ$, and $\gamma = 69^\circ$ for PBT [18], respectively.

The characteristic X-ray peaks for all of the samples studied (taken from the diffractograms shown in Fig. 5) are summarized in Table 1. Apparently, apart from those of the pure components, no new peaks were observed in the diffraction patterns of the PTT/PBT blend samples, indicating that the PTT and PBT components in the blends crystallized separately, even though it was shown previously that both components crystallized simultaneously during non-isothermal crystallization from both the glassy and the molten states.

Further important information that can be obtained from the WAXD patterns is the apparent degree of crystallinity. The apparent degree of crystallinity χ_c^{WAXD} can be calculated from the ratio of the integrated intensities under the crystalline peaks A_c to the integrated total intensities A_t (i.e., $A_t = A_c + A_a$, where A_a is the integrated intensities under the amorphous halo). According to this procedure, the χ_c^{WAXD} values for PTT and PBT were found to be ca. 24.8 and 29.1%, respectively. Our χ_c^{WAXD} value obtained for PTT agreed well with the values of ca. 15–30%, reported by Chuah [19] for melt-crystallized PTT samples. The obtained χ_c^{WAXD} values of each component for PTT/PBT blends (calculated from the diffractograms shown in Fig. 6) were plotted as a function of the blend composition in Fig. 6, while the total χ_c^{WAXD} values were summarized in Table 1.

According to Fig. 6, the χ_c^{WAXD} values for both PTT and PBT components in the blends were found to decrease with increasing content of the other component. The total χ_c^{WAXD} value for the blends was, in general, found to decrease from that of PBT with increasing PTT content, reach a minimum value for the blend having the PTT content of 60 wt% and, finally, increase with further increase in the PTT content until approaching that of PTT (see Table 1). It should be noted that the total χ_c^{WAXD} values for the PBT-rich blends were, in general, higher than those for the PTT-rich blends, a direct result of the higher crystallizability of the PBT-rich blends. Similar behavior was also reported in blends of

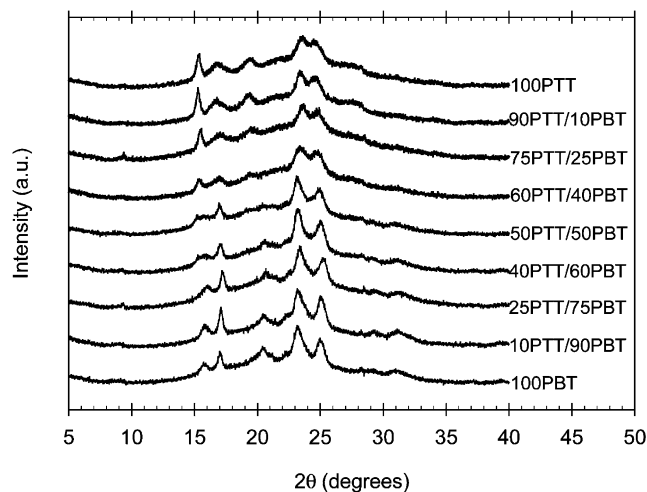


Fig. 5. Wide-angle X-ray diffractograms for PTT, PBT, and PTT/PBT blend samples after non-isothermal crystallization from the molten state in DSC cell at a cooling rate of $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$.

Table 1

Characteristic X-ray peaks and the total degree of crystallinity of PTT, PBT, and blends

Blend composition	Characteristic X-ray peaks (2θ)												Total degree of crystallinity		
PBT	–	15.8	–	17.0	–	20.5	–	23.2	–	–	25.0	–	29.3	31.2	29.1
10PTT/90PBT	–	15.8	–	17.1	–	20.5	–	23.2	–	–	25.0	–	29.3	31.2	31.0
25PTT/75PBT	–	16.0	–	17.2	19.5	20.7	21.6	23.4	–	–	25.3	27.8	29.3	31.4	27.0
40PTT/60PBT	15.3	15.9	–	17.1	19.4	20.6	21.2	23.2	–	–	25.1	27.4	29.1	31.4	28.2
50PTT/50PBT	15.2	15.8	–	17.0	19.1	20.4	21.2	23.1	–	–	25.0	27.6	29.1	31.1	25.4
60PTT/40PBT	15.4	–	16.8	–	19.4	20.6	21.7	–	23.5	24.7	–	27.4	29.5	31.2	21.6
75PTT/25PBT	15.5	–	17.1	–	19.5	–	21.8	–	23.6	24.9	–	27.3	29.3	31.1	22.8
90PTT/10PBT	15.3	–	16.7	–	19.3	–	21.8	–	23.4	24.7	–	27.5	–	31.3	24.5
PTT	15.3	–	16.8	–	19.4	–	21.7	–	23.6	24.6	–	27.3	–	–	24.8

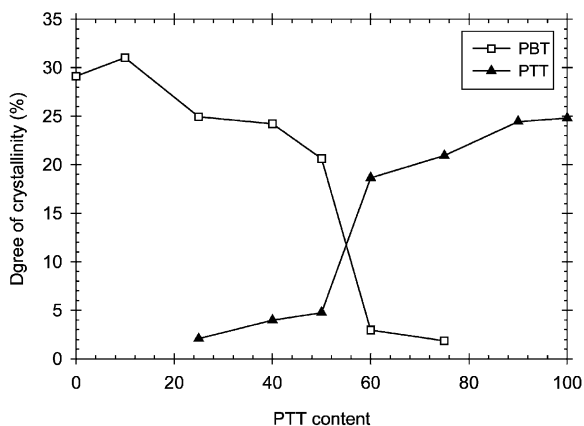


Fig. 6. Apparent degree of crystallinity for PTT and PBT components for both pure and blend samples as a function of blend composition.

poly(ethylene naphthalate) (PEN) and poly(butylene naphthalate) (PBN) [20].

3.4. Melting behavior

According to Fig. 1a, PTT exhibited a single melting endotherm, with the peak temperature T_m being observed at ca. $226.8\text{ }^{\circ}\text{C}$, while PBT exhibited double melting endotherms, with the peak temperatures of the major endotherm (T_{m1}) and the minor endotherm (T_{m2}) being observed at ca. 223.2 and $230.0\text{ }^{\circ}\text{C}$, respectively. The observed melting (peak) temperatures for PTT/PBT blends (taken from the thermograms shown in Fig. 1a) were plotted against the blend composition in Fig. 7. It should be noted that the assignment of the observed T_m to a given component in the 40PTT/60PBT, 50PTT/50PBT, and 60PTT/40PBT blends was quite difficult, since the melting range for both PTT and PBT components was in close proximity to each other.

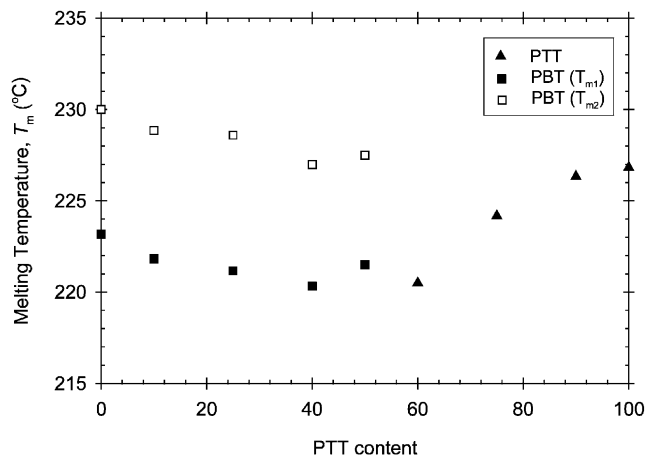


Fig. 7. Melting (peak) temperature T_m characterizing the melting of PTT and/or PBT crystallites for cold-crystallized PTT, PBT, and PTT/PBT blend samples as a function of blend composition.

According to Fig. 7, it is obvious that both PBT-rich and PTT-rich blends exhibited the melting point depression phenomenon, in that the observed T_m value of each component in the blends decreased with increasing content of the other minor component. The melting point depression can be used to study interactions between polymer molecules of two different species when one of the components in the miscible blends is partially crystalline. Due to the lowered chemical potentials of the molecules in the blends as compared to those of the pure state, the temperature at which the crystals of one component are in equilibrium with the miscible amorphous phase consisting of both components will be lower, hence the occurrence of the melt point depression phenomenon [21]. The melting point depression phenomenon for both of the pure components in the miscible blends of PET and PBT was also observed and reported [5,22].

3.5. Shear viscosity

Fig. 8 shows the steady shear viscosities η measured at 260 °C for PTT, PBT, and PTT/PBT blend samples as a function of shear rate. Within the shear rate range studied (i.e., 0.25–25 s^{-1}), it was found that the shear viscosities for these samples decreased very slightly with increasing shear rate, suggesting a slight shear-thinning behavior. Chuah [19] found that molten PTT behaved like a Newtonian fluid at low shear rates and like a shear thinning fluid at shear rates greater than 1000 s^{-1} . The shear viscosities for blend samples were found to locate between those of the two pure components.

The dynamic complex viscosities η^* measured at 260 °C for PTT, PBT, and PTT/PBT blend samples as a function of frequency are represented in Fig. 9. All of the observed η^* values for these samples were found to be

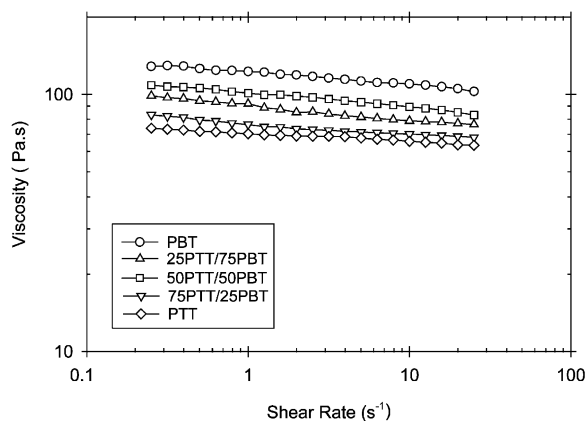


Fig. 8. The steady shear viscosity η measured at 260 °C for PTT, PBT, and PTT/PBT blend samples as a function of shear rate.

independent of changes in the frequency. For a given frequency, the η^* value for PBT was found to be higher than that for PTT. The η^* values for the 25PTT/75PBT and 50PTT/50PBT blends were found to be higher, only the value for the 75PTT/25PBT blend was lower, than those for both of the pure components (for a given frequency). The unusually high values of η^* observed in the 25PTT/75PBT blend may be a result of the high entanglement density of this particular blend [22].

4. Conclusions

Blends of poly(trimethylene terephthalate) (PTT) and poly(butylene terephthalate) (PBT) showed a single and composition-dependent glass transition temperature in all of the blend compositions studied, indicating that both

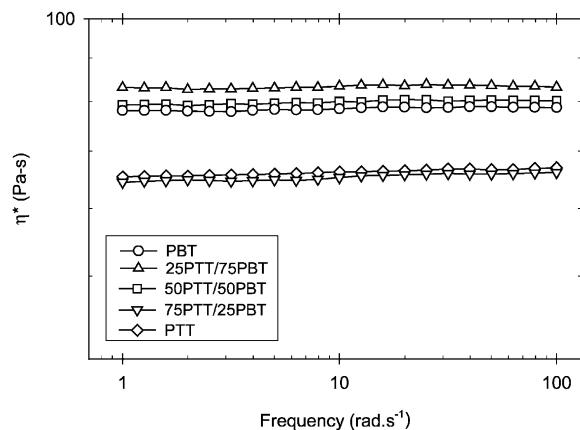


Fig. 9. The dynamic complex viscosity η^* measured at 260 °C for PTT, PBT, and PTT/PBT blend samples as a function of frequency.

components were miscible in the amorphous state. The observed glass transition temperature was found to increase with increasing PTT content and was fitted well to the Gordon–Taylor equation, with the fitting parameter k being ca. 6.9. The cold crystallization (peak) temperature was found to increase, while the melt crystallization (peak) temperature was found to decrease, with increasing PTT content. The subsequent melting behavior for these blends (after cold crystallization) showed the melting point depression behavior, in that the melting (peak) temperature for each component was lowered with increasing content of the other component. Wide-angle X-ray diffraction results showed that the pure components in the blends crystallized to form separate crystallites, even though the crystallization results suggested simultaneous crystallization of the two components during cooling. The minimum value of the total apparent degree of crystallinity were observed in the blend having 60 wt% of PTT. The apparent degree of crystallinity for each component was found to decrease with increasing content of the other component. The steady shear viscosities for the pure components and the blends showed slight decrease (within the shear rate range of 0.25–25 s⁻¹) with increasing shear rate, with those of the blends lying in between those of the pure components.

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