

Rheological and Thermal Properties of Blends of Modified Poly(ethylene terephthalate) with *p*-Acetoxybenzoic acid and Poly(butylene terephthalate)

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ABSTRACT: Blending of thermotropic liquid crystalline polyesters (LCPs) with conventional polymers could result in materials that can be used as an alternative for short fiber-reinforced thermoplastic composites, because of their low melt viscosity as well as their inherent high stiffness and strength, high use temperature, and excellent chemical resistance and low coefficient of expansion. In most of the blends was used LCP of 40 mol % of poly(ethylene terephthalate) (PET) and 60 mol % of *p*-acetoxybenzoic acid (PABA). In this work, blends of several copolyesters having various PABA compositions from 10 to 70 mol % and poly(butylene terephthalate) (PBT) were prepared and their rheological and thermal properties were investigated. For convenience, the copolyesters were designated as PETA-*x*, where *x* is the mol % of PABA. It was found that PET-60 and PET-70 copolyesters decreased the melt viscosity of PBT in the blends and those PBT/PETA-60 and PBT/PETA-70 blends showed different melt viscosity behaviors with the change in shear rate, while blends of PBT and PET-*x* having less than 50 mol % of PABA exhibited totally different rheological behaviors. The blends of PBT with PETA-50, PETA-60, and PETA-70 showed the morphology of multiple layers of fibers. © 1999 John Wiley & Sons, Inc. *J Appl Polym Sci* 74: 1797–1806, 1999

Key words: modified poly(ethylene terephthalate); *p*-acetoxybenzoic acid; poly(butylene terephthalate); thermotropic liquid crystalline polyester; blends

INTRODUCTION

Thermotropic liquid crystalline polyesters (LCPs) are reputed to be the second-generation engineering plastics and many kinds of LCPs have been reported.^{1–5} The molecules of LCPs are easily oriented by shear strain and elongational flow during processing and form multilayer structures whose layers have various orientation states.^{6,7}

These polyesters can be prepared by conventional techniques, that is, solution-condensation, interfacial-condensation, or melt-condensation processes. For example, one of the well-known methods to prepare such thermotropic polyesters is to modify poly(ethylene terephthalate) (PET) by reaction with *p*-acetoxybenzoic acid (PABA).

The first indication of a liquid crystalline character in copolyesters came from Jackson and Kufuss at Tennessee Eastman.^{8–10} The first patent was issued in 1973 and the liquid crystalline character was indicated two decades ago.^{11–14} McFarlane and Davis were the first to recognize that these polymers have liquid crystalline characteristics.¹⁵ To improve the mechanical properties of

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some polymers, generally, inorganic fillers or reinforcements are compounded with the polymers. But the presence of solid fillers results in a dramatic increase in the melt viscosity. LCPs have been intensively studied because of their inherent high stiffness and strength, high use temperature, excellent chemical resistance, low melt viscosity, and low coefficient of expansion. Because of these excellent properties, the blending of LCP with conventional polymers could result in materials that can be used as an alternative for short-fiber-reinforced thermoplastic composites; the *in situ* composites have many potential advantages as compared with the short-fiber-reinforced polymers.¹⁶ Kiss¹⁷ reported that the final injection pressure was reduced to about 50% when 30% of an LCP polyester with naphthalene moieties of unknown composition was added to the polyether-sulfone matrix. Usually, LCPs having a formulation of 40 mol % PET and 60 mol % PABA are used in blends and composites with engineering thermoplastics and polyolefins such as nylon 6, polyetherimide, and polypropylene.^{18–24} In addition, LCPs can be used as processing aids in blends.^{20,21,25}

Chang et al. studied the blends of poly(butylene terephthalate) (PBT) with three different LCPs having different mesogen units.²⁶ Paci et al. discussed the calorimetric data for the blends of PBT with a commercial wholly aromatic liquid crystal polyester (Vectra B950) and compared them with PBT/poly(biphenyl-4,4'-ylene sebacate).²⁷ Zalduo et al.²⁸ presented some results of the rheology of blends of PBT and PET/60PABA and interpreted their results in the light of the phase behavior and compatibility by differential scanning calorimetry and dynamic viscoelasticity. Joseph et al.²⁹ studied the thermal behavior of blends of PET and PET/60PABA. The rate of crystallization increased as the LCP content increased in the blend, which was attributed to the nucleation of the PET crystallization by the LCP. The degree of crystallinity also increased with the addition of the LCP. Isothermal crystallization kinetics studies of blends of PBT and a liquid crystalline poly(biphenyl-4,4'-ylene)sebacate were reported by Paci et al.³⁰ They found that the two polymers were miscible in the isotropic state, and they showed that the degree of crystallinity of PBT increases on addition of the liquid crystalline component up to about 35–50% by weight.³⁰ Metha and Deopura³¹ reported the calorimetric data for the blends of PBT and LCP (PET/60PABA) in the form of as spun and drawn fibers.

DSC melting and crystallization results showed that PBT is compatible with the LCP and the crystallization of PBT decreases by the addition of LCP in the matrix. It was believed that this could be associated with the dissolution of the PBT phase in the PET-rich phase of the PET/60PABA blend.^{32,33,34} The object of the present work was to prepare blends of several copolyesters having various PABA compositions from 10 to 70 mol % and PBT and to investigate the rheological and thermal behavior of the blends.

EXPERIMENTAL

Materials

The modified polyesters were prepared by the reaction of PABA and PET as described elsewhere.³⁵ The modified polyesters possess several different PET and PABA compositions and are designated as PETA-x, where x is the mol % of PABA. PET (T_m : 250°C; M_w : 52,000; M_n : 26,000; IV: 0.82) and PBT (T_m : 225°C; M_w : 68,300; M_n : 31,400; IV: 1.256) of commercial grade were supplied by Sunkyung Industry Co. (Suwon, South Korea). Thermal properties were measured by Perkin–Elmer differential scanning calorimetry (DSC). Inherent viscosity (IV) was obtained by an Ubbelohde viscometer at 150°C in *ortho*-chlorophenol at the concentration of 0.5 g/dL.

Blends

Blends were prepared in a Rheometrics Mechanical Spectrometer (RMS) test pot. To minimize thermal degradation or oxidation during melt blending, tris(2,4-di-*t*-butylphenyl)phosphite as an antioxidant was added when blending. All the blended polymers were powdered using a freezer mill to have high efficiency in the dispersion for each of the blends. The fine-powdered PBT was blended with any of PETA-x, which were also in the powder state. The dry blending was performed in a glass tube agitating at 2000 rpm with a vibrator for about 5 min. The blend ratios were 80/20, 50/50, and 20/80 (PBT/PETA-x). All the blends contained 1000 ppm of antioxidant. These mixtures were poured into the RMS test pot, then melt mixing was performed in between the plates of that instrument. After the RMS test, the remaining samples were collected from the plate and were used for measurement of the thermal analysis and morphology.

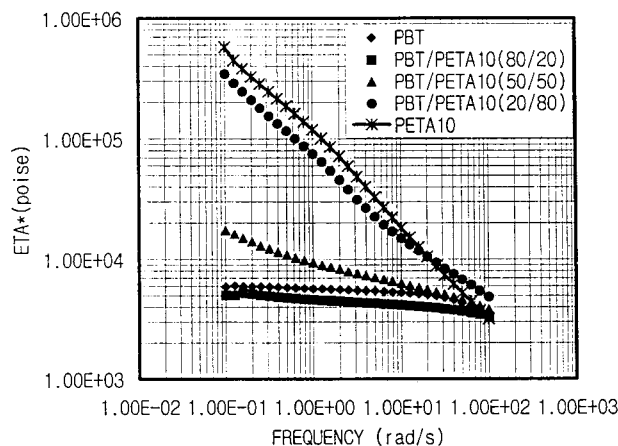


Figure 1 Complex viscosity behavior of PBT/PETA-10 blends as a function of blend composition at 240°C.

Properties of Blends

Dynamic Mechanical Measurement

Viscoelastic properties were measured on an RMS, using parallel plates at 240 and 280°C. Strain was fixed at 30%. Before the measurements, the powdery modified copolyesters were dried in a vacuum oven at 105°C for 24 h. The powder samples were directly poured on the disc in the test chamber. During the measurement, nitrogen flow was used to reduce sample oxidation.

Thermal Analysis

The thermal analysis was performed under nitrogen with a heating rate of 10°C/min using a Perkin-Elmer differential scanning calorimeter (DSC7).

Scanning Electron Microscopy

Scanning electron microscopy (SEM) was carried out using a Cambridge Instrument SEM (Stereoscan 240). Samples were cryogenically fractured in liquid nitrogen, followed by gold coating prior to their installation in the SEM chamber.

RESULTS AND DISCUSSION

Rheological Properties

There are some differences in the rheological properties when comparing the features of LCP with those of conventional thermoplastic poly-

mers. These thermoplastic polymer molecules are long and, in the melt form, in a random coil configuration. When extruded, injection-molded, and fiber spun, these long molecules tend to align, but the continuity of the chain is low and the chains remain partially coiled, so the molecules of these polymers have an alignment by tensile and shear forces. On the contrary, LCP molecules, under appropriate processing conditions, have the tendency to align and to remain in that orientation, thus presenting the extended-chain conformation. The willingness of the molecules of LCP to remain in alignment accounts for their fibrous structure and their excellent mechanical properties which are similar to those of fiber-reinforced thermoplastics, so LCPs are often referred to as self-reinforcing polymers. On the other hand, the

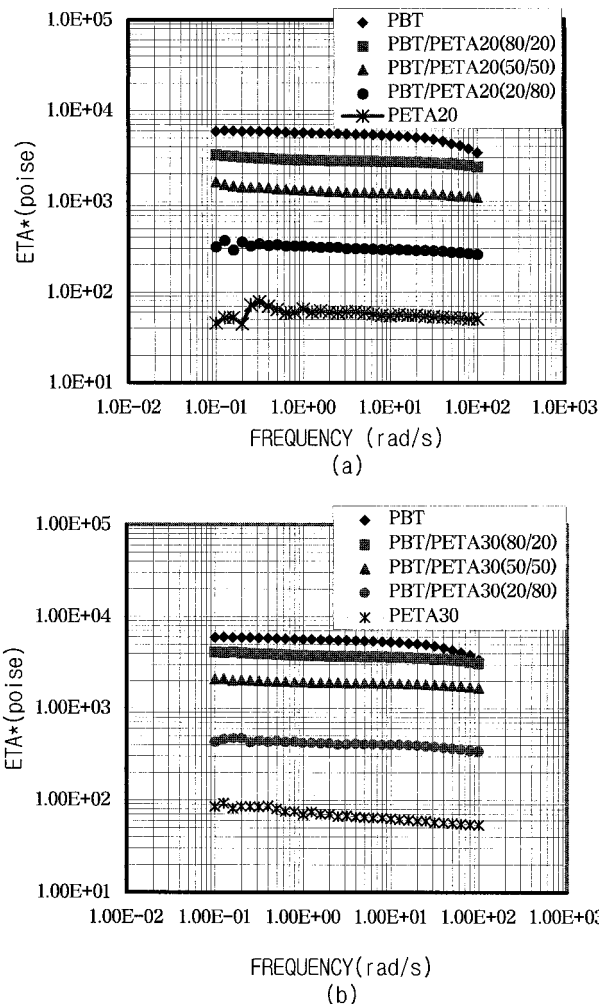


Figure 2 Complex viscosity behavior of (A) PBT/PETA-20 and (B) PBT/PETA-30 blends as a function of blend composition at 240°C.

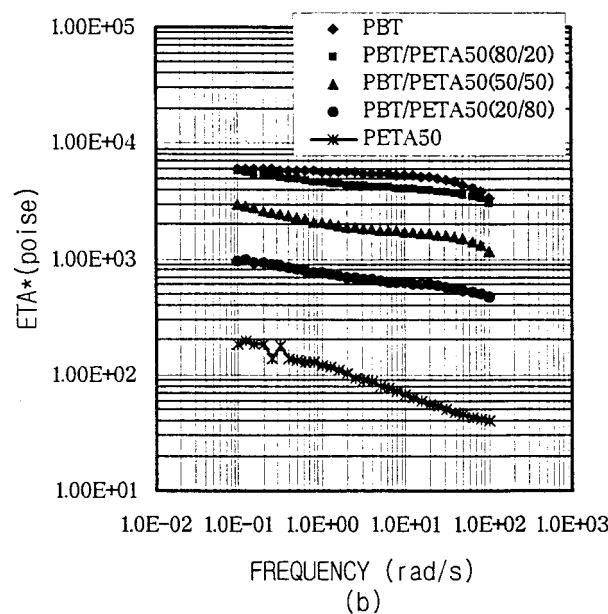
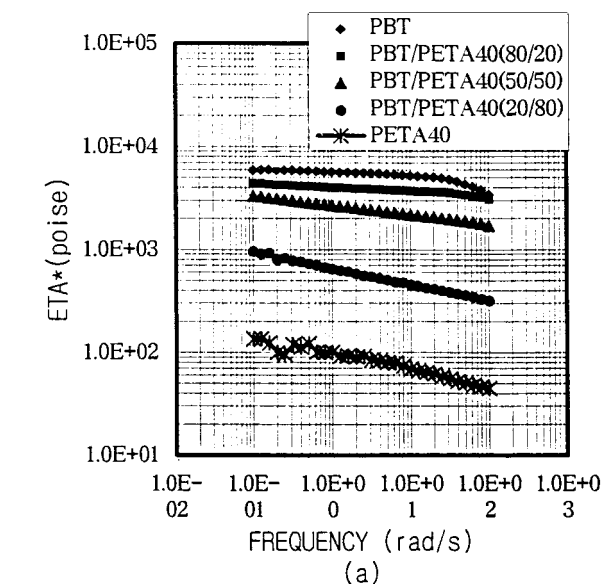


Figure 3 Complex viscosity behavior of (A) PBT/PETA-40 and (B) PBT/PETA-50 blends as a function of blend composition at 240°C.

viscosity in the steady-shear flow is the most investigated rheological property of blends with thermotropic LCPs.

In this work, the rheological properties of those blends were measured using a parallel plate mode by the RMS. The melt-viscosity behaviors of the blends of PBT with PABA-modified PET were studied as a function of the mol % of PABA. Figures 1–4 show the results. It was reported in our previous work that the modified PET having more than 50

mol % exhibited liquid crystalline behavior.³⁵ In Figure 1, PETA-10, the modified copolyester that did not show a liquid crystalline behavior, did not lower the viscosity of the blends. In the case of blends of PBT and PETA-20, or PETA30, shown in Figure 2, the melt viscosities of those blends were lowered much more than that of PBT. But it cannot be said that those materials, PETA-20 and PETA-30, are LCPs because the two PETA copolyesters have inherently low melt viscosity and the two materials did not show the behavior of LCPs in our previous analyses such as polarizing micrograph and rheological measurements.³⁵ The shear-rate dependence of the viscosity of those blends could be ignored because the shapes of the plotted curve are nearly linear. In other words, they exhibit Newtonian behavior.

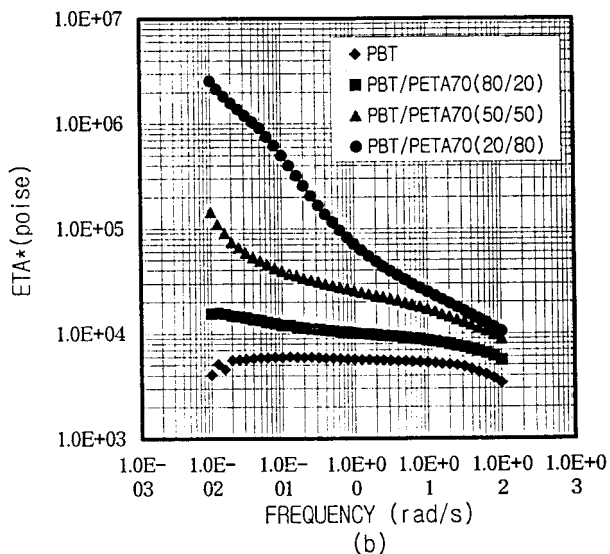
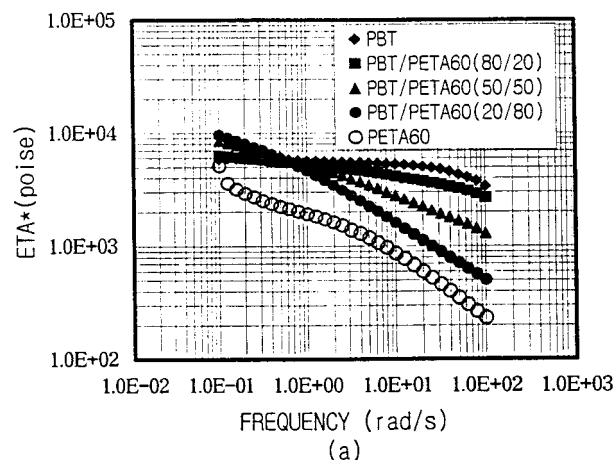


Figure 4 Complex viscosity behavior of (A) PBT/PETA-60 and (B) PBT/PETA-70 blends as a function of blend composition at 240°C.

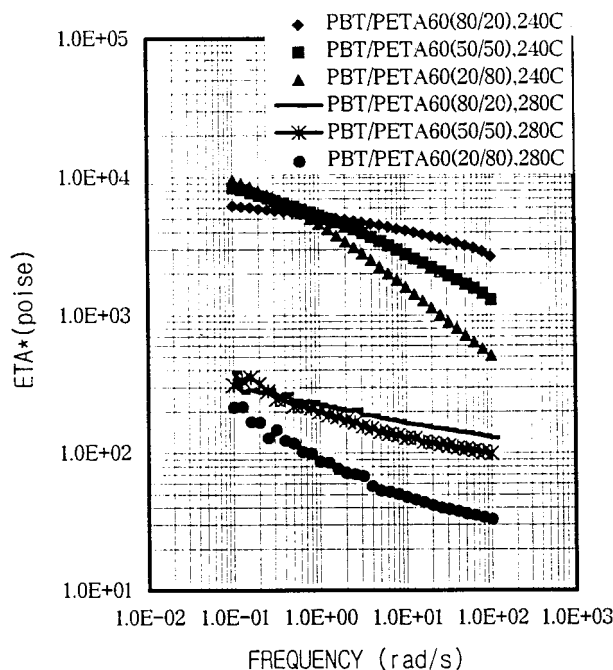


Figure 5 Complex viscosity behavior of (A) PBT/PETA-60 blends as a function of blend composition at 240 and 280°C.

Among the blends of PBT with PETA-*x* exhibiting liquid crystalline behavior, PETA-40 and PETA-50 showed some change of viscosity (Fig. 3). In Figure 3, the slope of the curve of the viscosity versus shear rate is increased slightly with increasing PETA content. This means that PETAs having liquid crystalline behavior have two functions: one is lowering the viscosity of the blend and the other is giving a non-Newtonian behavior to the blend systems by the incorporated PABA unit in PETA copolyesters. Also, in most studies on the blend of thermoplastics with an LCP, a drastic reduction of the viscosity was reported due to the presence of a liquid crystalline polyester, even at low content of the LCP. It should be noted, however, that noticeably exceptional flow curves were reported by Acierno et al.^{36,37} for the blends of polycarbonate (PC) and LCPs. They reported that at low shear rate the viscosity of blends exceeds that of the pure PC and increases with the LCP content. It was also reported, that both at low shear rates and at high shear rates, the viscosity values of all the blends lie within those of the pure components.^{38,39}

In our studies, similar results were observed, as shown in Figure 4(A). The decrease of melt viscosity for the blend of PBT and PETA60 are due to the liquid crystalline behavior of the co-

polyester. It should be noted, however, that the viscosity of PBT increased when adding PETA70 to PBT [Fig. 4(B)], although PETA70 containing 70 mol % PABA showed a clear typical liquid crystalline texture by polarizing microscopy in our previous work.³⁵ No studies have been made, however, on the rheological behavior of PET modified with 70 mol % PABA and its blend with PBT.

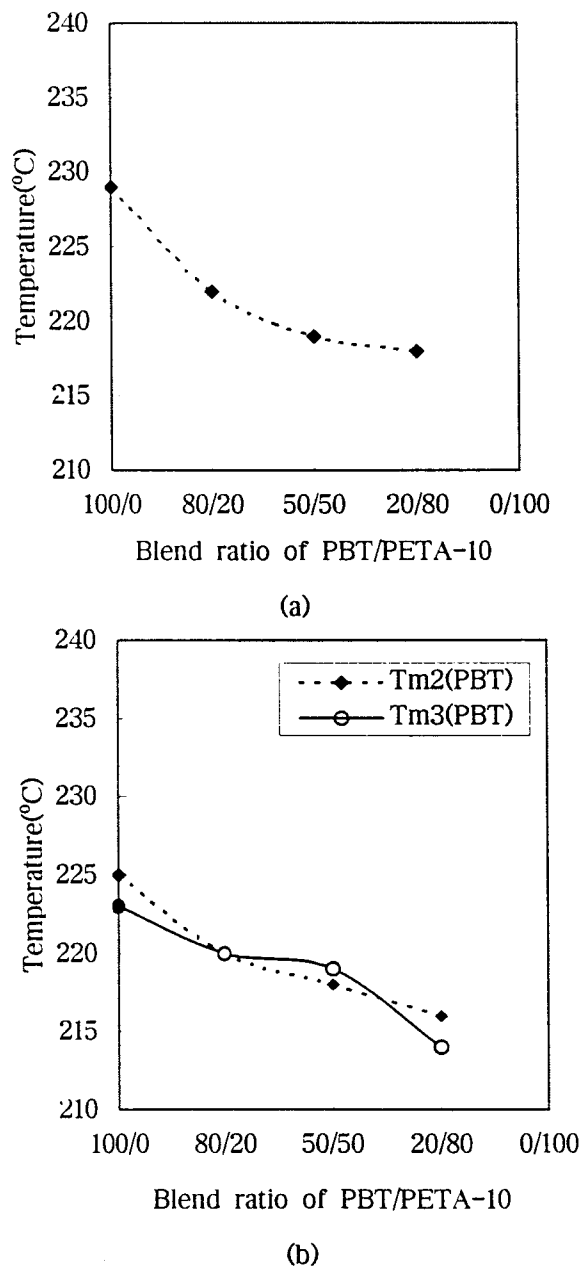


Figure 6 Melting temperature (A) on first DSC scan (T_{m1}) and (B) on second (T_{m2}) and third DSC scan (T_{m3}) of PBT in the PBT/PETA-10 blends as a function of blend ratio.

In Figure 4(B), as PETA70 was added to PBT, non-Newtonian behavior was observed. But the melt viscosity was increased by increasing ratio of PETA70. This means that in the blend of the LCP which has, especially, a higher melt viscosity than that of the other component polymer, even though it is an LCP, the melt viscosity of that blend was not lowered. The result is not in accord with the general rheological behavior of blends containing LCP as one component. The large difference in the melt viscosity at low shear rate could be explained by the same reason as in the case of the PBT/PETA60 blend. But the tendency to divide into two regions is reduced at high temperature, as shown in Figure 5. Thus, it may be concluded that the behavior of an LCP in blend systems is dependent on the combined factors of shear rate, composition, and temperature, etc.

Thermal Properties

The blends of PBT with PETA- x ($x = 10, 20, 40, 50,$ and 70) were studied in this work. The blend of PETA60 with PBT is exempted in this experimental work because of having been reported in many articles. In the blend of PBT with PETA-10, shown in Figure 6(A), decreases of the melting temperature in the PBT phase were observed. In this figure, T_{m1} means the melting temperature on the first heating scan. This result indicates that the interaction between PBT and the PET-rich phase of PETA-10 exists because of the partial compatibility of PBT

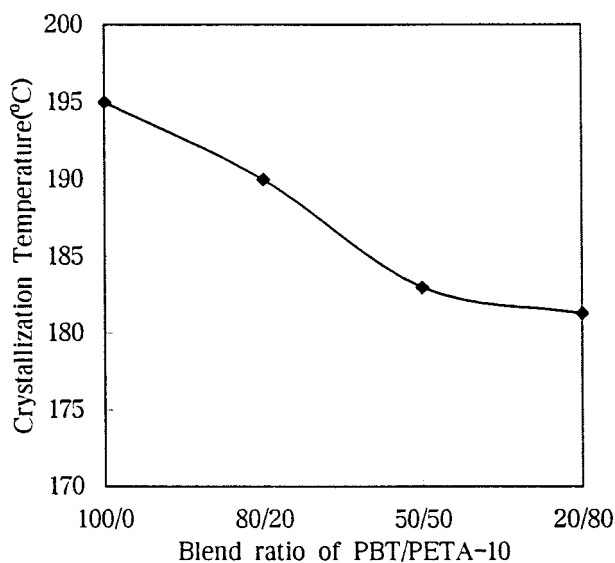


Figure 7 Crystallization temperature of the PBT/PETA-10 blends as a function of blend ratio.

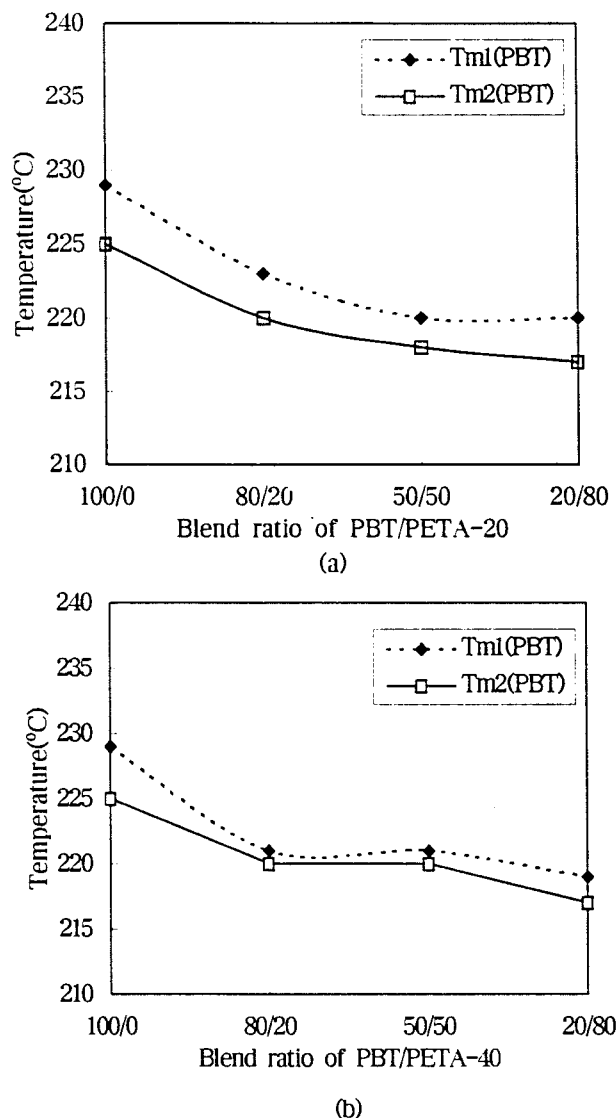


Figure 8 Melting temperature on first DSC scan (T_{m1}) and on second DSC scan (T_{m2}) of PBT in (a) the PBT/PETA-20 and (b) the PBT/PETA-40 blends as a function of blend ratio.

and PET. The thermal behavior of the blends at the second and third scans showed the same trend as the first scanning in that the melting temperatures were decreased by the addition of PETA-10 [Fig. 6(B)]. DSC cooling traces of the PBT/PETA-10 blend, shown in Figure 7, suggest that the crystallization temperature (T_c) of PBT was affected significantly by the addition of PETA-10. The crystallization was initiated at 195°C for PBT, but was delayed for the blends; the crystallization peak temperature was also lowered. It should be noted that the transesterification can be a factor in lowering melting points and impacting the crystallization

rate but no evidence of transesterification was observed in this work. In other blends of PBT with PETA-x copolyesters, there was a decrease of the melting temperature of PBT, regardless of the PABA mol % in PETA-x copolyesters (Figs. 8 and 9). In Figure 9, no significant difference in the trends of the melting temperature decrease was observed depending on the different DSC scanning. In case of

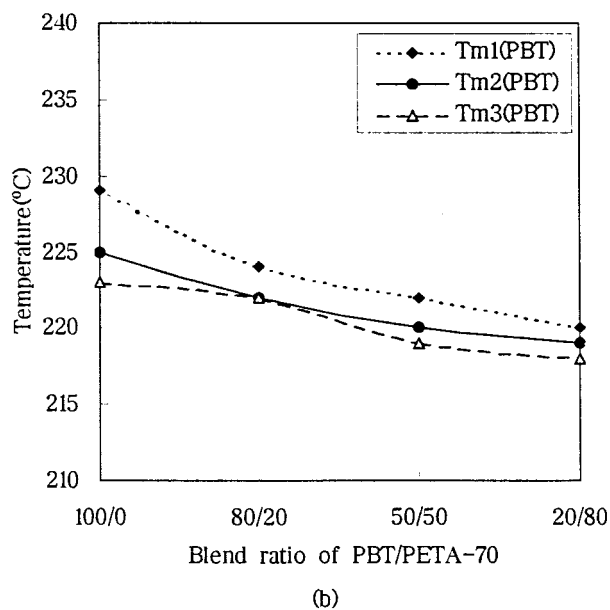
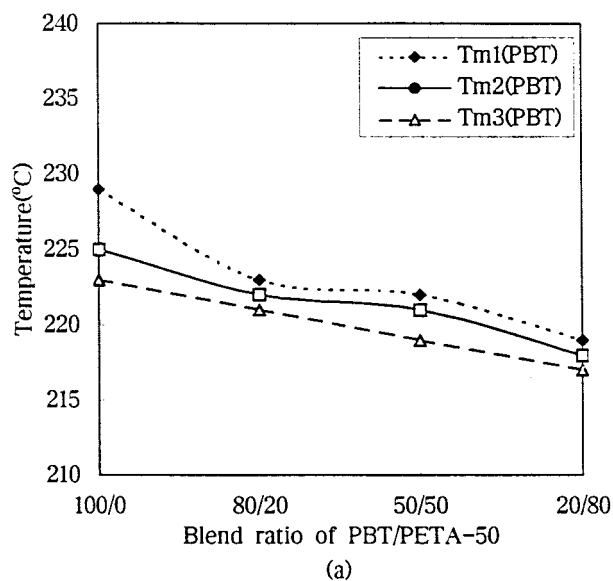


Figure 9 Melting temperature on first DSC scan (T_{m1}), on second DSC scan (T_{m2}), and on third DSC scan (T_{m3}) of PBT in (a) the PBT/PETA-50 and (b) the PBT/PETA-70 blends as a function of blend ratio.

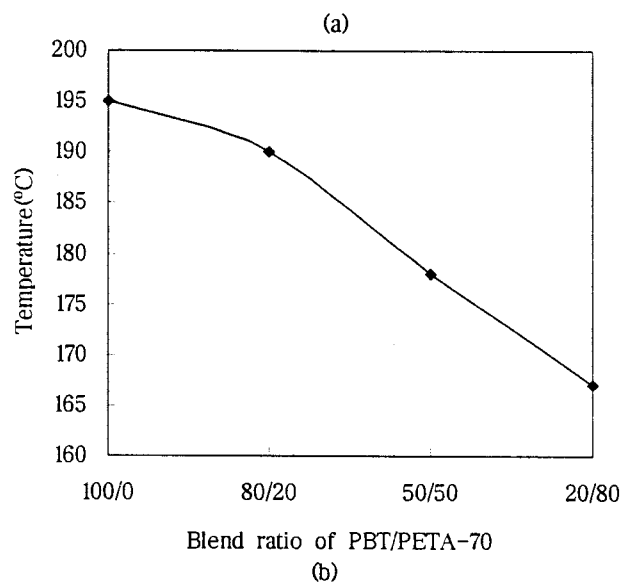
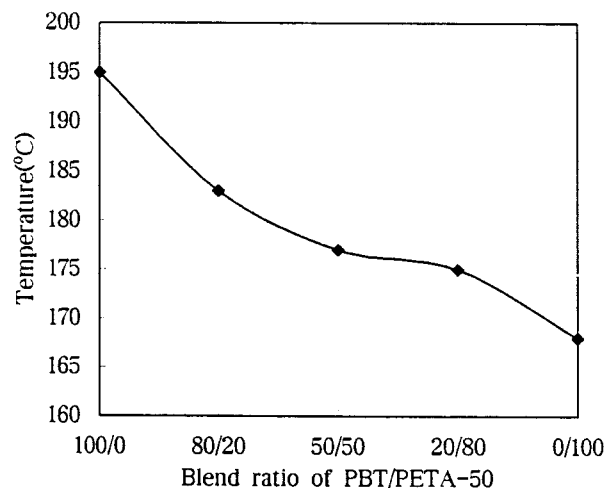


Figure 10 Crystallization temperature of (a) the PBT/PETA-50 blends and (b) the PBT/PETA-70 blends as a function of blend ratio.

PBT/PET-x blends ($x = 40, 50,$ and 70), as with the blend of PBT with PETA-10, the crystallization peak temperature is also lowered, as shown in Figure 10. This is because when PBT started to crystallize the PETA-x was in a supercooled mesophase state and it restricted the mobility of the PBT chains.

The dependence of both the heat of fusion (ΔH_m) and heat of crystallization (ΔH_c) on the composition of PETA-x copolyesters in blends are similar for the T_m and T_c behavior of the blends. For example, the ΔH_m of PBT and the ΔH_c of PBT in the blend of PBT with PETA-50 are shown in Figure 11. Both the ΔH_m of PBT in the blend and the ΔH_c of PBT in the blend decrease with increasing PETA-50 composi-

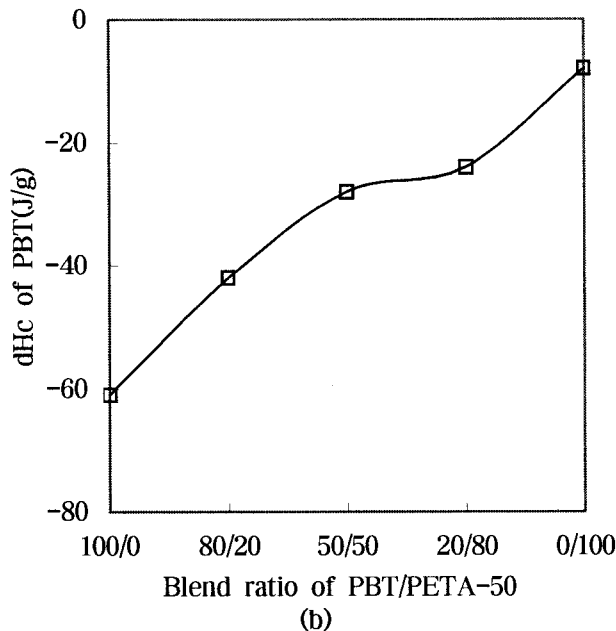
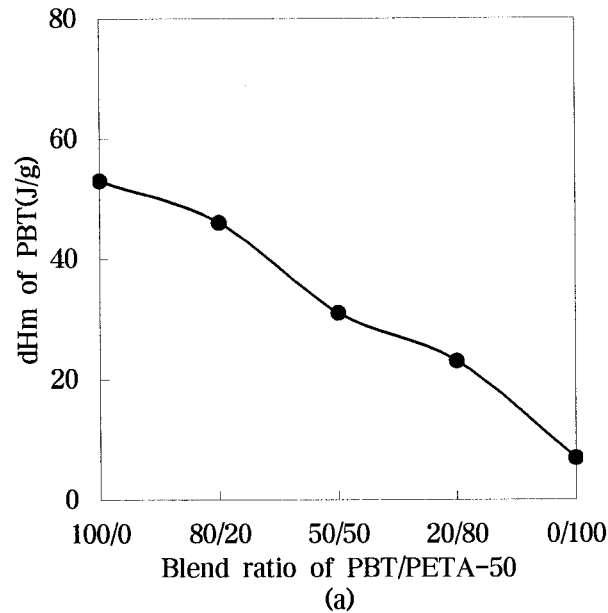


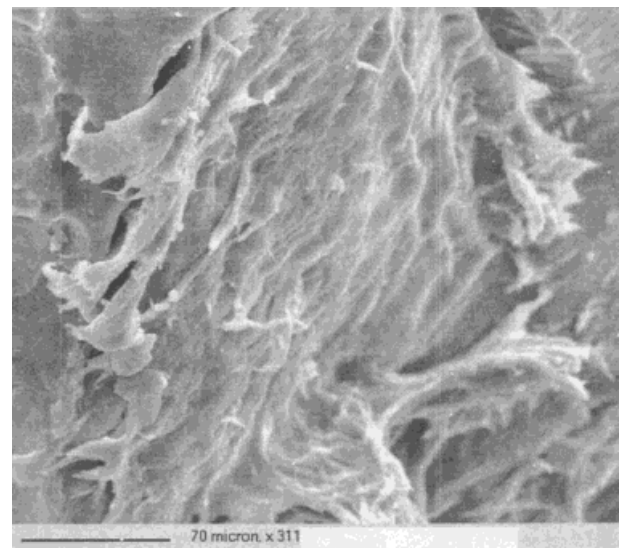
Figure 11 (a) Heat of fusion (ΔH_m) and (b) heat of crystallization of PBT in the PBT/PETA-50 blends as a function of blend ratio.

tion. This is in agreement with previous results for the blend of PBT with PET/60PHB in the literature.³¹ From the DSC results, it may be said that the modified PET restricts the crystallization of PBT.

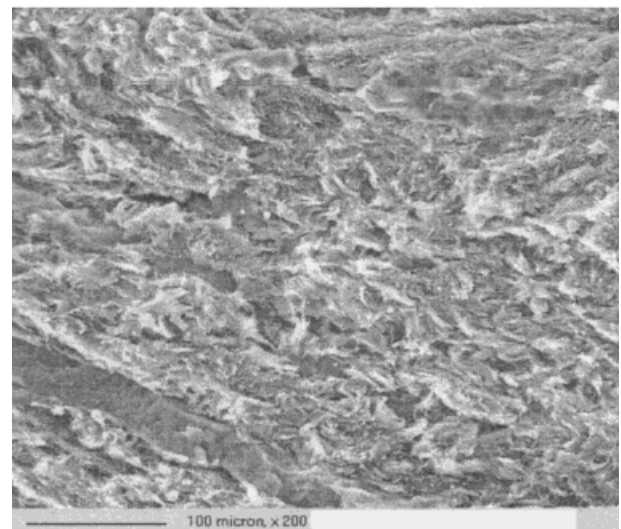
Morphology

The morphologies of the fractured surface of the blends of PBT and PETA-50 or PETA-60 were

characterized by SEM. SEMs of the PBT/PETA-60 blend are shown in Figure 12. The sample for this SEM measurement was prepared in a melt state in an RMS chamber without any shear to some direction. In general, the blends of LCP and thermoplastics are two-phase systems and they exhibit skin-core morphology. In the skin region, most of the fibrous LCP is oriented when sheared, while in the core, the LCP domains are less oriented or in spherical form. For the low content of PETA-60 in the blend, the morphology of the center region is shown as some cocontinuous phase between PETA-60 and PBT, but has



(a)

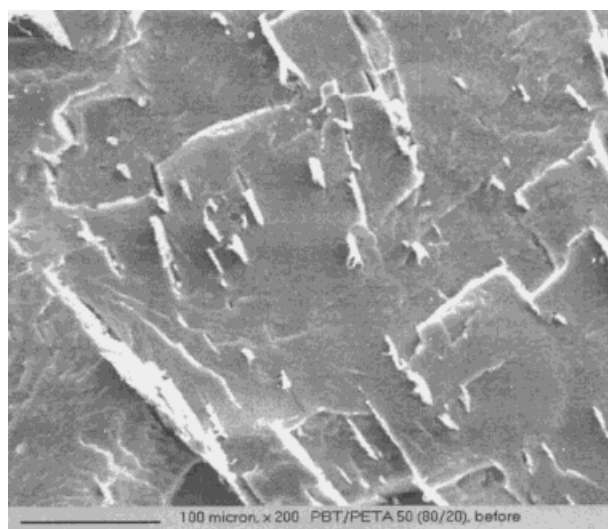


(b)

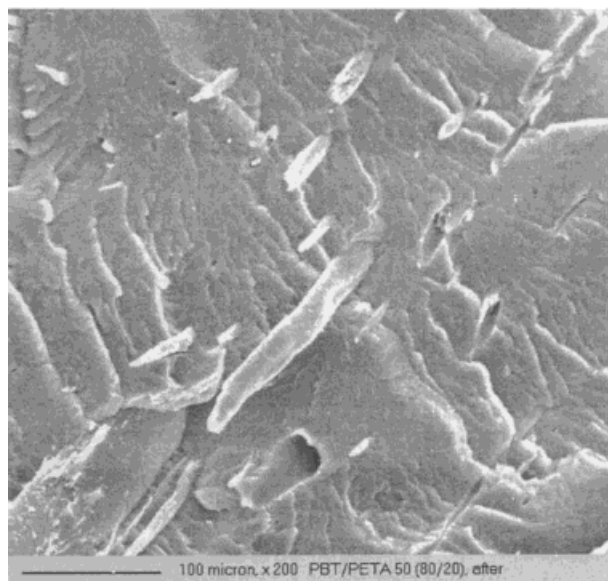
Figure 12 SEM micrographs of PBT/PETA-60 blends of (a) (80/20) and (b) (20/80) (w/w) composition.

little oriented PETA-60 phase [Fig. 12(a)], while at high PETA-60 contents, there are many short fibers that are oriented to the vertical direction from the matrix [Fig. 12(b)]. This implies that there is always a possibility to form fiber in the LCP blends when stress is applied to blend systems. This is proved by the morphology of the PBT and PETA-50 blend, as shown in Figure 13.

Figure 13(a) shows the morphology of the PBT/PETA-50 blend of 80/20 (w/w) without any directional stress. It is shown that there is no fibril for-

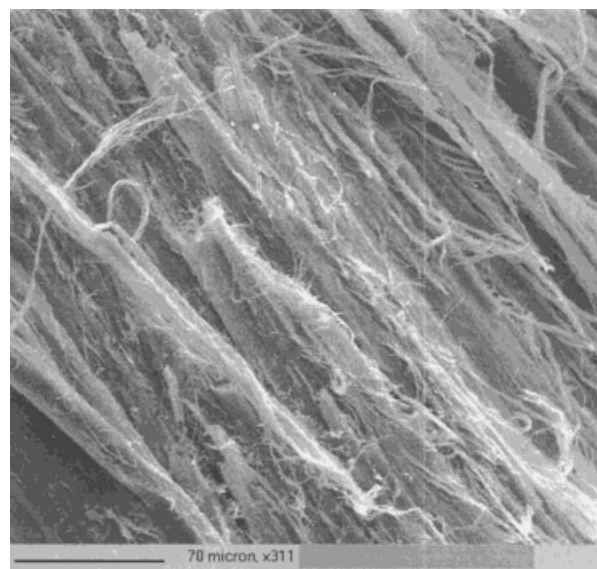


(a)

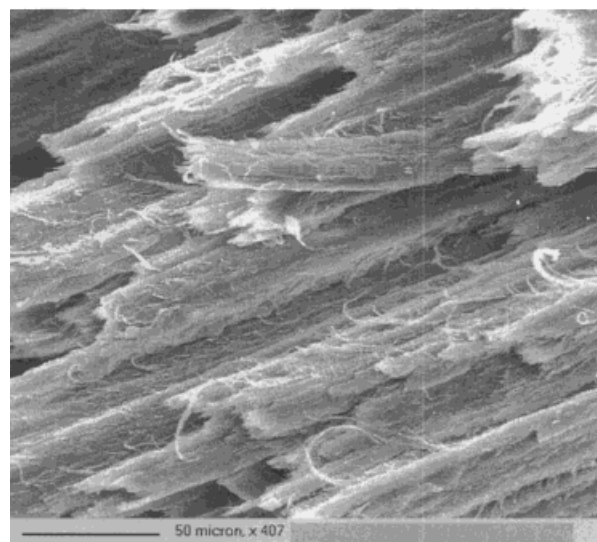


(b)

Figure 13 SEM micrographs of PBT/PETA-50 blends of (80/20) (w/w) composition: (a) before and (b) after shear.



(a)



(b)

Figure 14 SEM micrographs of PBT/PETA-50 blends of 20/80 (w/w) composition at (a) inner surface and (b) filament cut surface.

mation, and when some stress is forced in some direction, the LCP is oriented toward the stressed direction in Figure 13(b). As stated before, good reinforcement is achieved when LCP phases are deformed into fibers that are oriented in the flow direction. The degree of the fiber formation depends on the amount of LCP in the blend and also on the elongational forces affecting the molten material. At larger LCP contents, the LCP phase can form many

LCP fibers oriented in the matrix. Figure 14(a) shows a typical morphology of a blend of PBT and PETA-50 of 20/80 composition by weight, where the drawn fibers show the orientation. Figure 14(b) shows the typical morphology of the fiber cut during fracture for the blends of PBT with PETA-50 of the 20/80 composition by weight. It is seen that the blend system is composed of multiple layers of fibers. The same morphology was observed for the blends of PBT and PETA-60 and PETA-70, although the data are not shown here.

CONCLUSIONS

In this work, blends of several copolyesters having various PABA composition from 10 to 70 mol % with PBT were prepared. The modified PETA copolyesters were prepared for this work by the melt reaction of PABA with PET. Rheological and thermal properties of the blends were investigated. The properties of the blends of PBT and these modified copolyesters were different depending on the amount of PABA units in the copolyesters according to whether the modified copolyesters exhibit LCP behavior or not. Those liquid crystalline copolyesters decreased the melt viscosity of PBT in the blends, especially, PETA-60 and PETA-70 showed different melt viscosity behaviors with the change in shear rate. The morphology of the fibers cut during fracture for the blend of PBT with the thermotropic LCP, that is, PETA-50, PETA-60, and PETA-70, showed the multiple layers of fibers.

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