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European Polymer Journal 43 (2007) 3826–3837

EUROPEAN POLYMER JOURNAL

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Miscibility and compatibilization of poly(trimethylene terephthalate)/acrylonitrile–butadiene–styrene blends

Mei-Ling Xue [a,b], Yong-Liang Yu [b], Hoe H. Chuah [c], John M. Rhee [a],
Nam Hoon Kim [d], Joong Hee Lee [a,d,*]

[a] *Polymer BIN Fusion Research Team, School of Advanced Materials Engineering, Chonbuk National University, Duckjin-Dong 1Ga 664-14, Jeonju, Jeonbuk 561-756, Republic of Korea*
[b] *Key Laboratory of Rubber-Plastics, Ministry of Education, Qingdao University of Science and Technology, Qingdao 266042, PR China*
[c] *Shell Global Solutions, Westhollow Technology Center, P.O. Box 1380, Houston, TX 77251-1380, USA*
[d] *Dept. of Hydrogen and Fuel Cell Engineering, Chonbuk National University, Jeonju, Jeonbuk 561-756, Korea*

Received 19 April 2007; received in revised form 25 June 2007; accepted 28 June 2007
Available online 18 July 2007

Abstract

Poly(trimethylene terephthalate)/acrylonitrile–butadiene–styrene (PTT/ABS) blends were prepared by melt processing with and without epoxy or styrene–butadiene–maleic anhydride copolymer (SBM) as a reactive compatibilizer. The miscibility and compatibilization of the PTT/ABS blends were investigated by differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA), capillary rheometer and scanning electron microscopy (SEM). The existence of two separate composition-dependent glass transition temperatures (T_gs) indicates that PTT is partially miscible with ABS over the entire composition range. In the presence of the compatibilizer, both the cold crystallization and glass transition temperatures of the PTT phase shifted to higher temperatures, indicating their compatibilization effects on the blends.

The PTT/ABS blends exhibited typical pseudoplastic flow behavior. The rheological behavior of the epoxy compatibilized PTT/ABS blends showed an epoxy content-dependence. In contrast, when the SBM content was increased from 1 wt% to 5 wt%, the shear viscosities of the PTT/ABS blends increased and exhibited much clearer shear thinning behavior at higher shear rates. The SEM micrographs of the epoxy or SBM compatibilized PTT/ABS blends showed a finer morphology and better adhesion between the phases.

Keywords: Poly(trimethylene terephthalate); Polymer blends; Miscibility; Compatibilization; Rheology

1. Introduction

Poly(trimethylene terephthalate) (PTT) is a newly commercialized crystalline polymer with growing applications in fibers, films, and engineering plastics [1–3]. This polymer was reported to have

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[*] Corresponding author. Present address: Polymer BIN Fusion Research Team, School of Advanced Materials Engineering, Chonbuk National University, Duckjin-Dong 1Ga 664-14, Jeonju, Jeonbuk 561-756, Republic of Korea. Tel.: +82 63 270 2342; fax: +82 63 270 2341.
E-mail address: jhl@chonbuk.ac.kr (J.H. Lee).

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doi:10.1016/j.eurpolymj.2007.06.048

outstanding tensile elastic recovery, good chemical resistance, a relative low melting temperature, and a rapid crystallization rate [4]. As an engineering thermoplastic, it was found to have mechanical properties similar to those of poly(ethylene terephthalate) (PET), while its processing characteristics are similar to those of poly(butylene terephthalate) (PBT). Thus, it combines some of the advantages of PET and PBT [4]. In addition, PTT possesses all of the advantages of thermoplastic polyesters, such as their dimensional stability, solvent resistance, and abrasion resistance, and can be employed under moist conditions where nylon cannot be used. The performance of glass fiber reinforced PTT was also found to be better than that of reinforced PET or PBT and some of its mechanical properties are comparable to those of glass fiber reinforced nylon [2]. Since its commercialization in 1998, PTT has been widely studied, especially with regard to its fiber properties [5–8], structure formation [1,9–11], crystal structure [12,13], and thermal and crystallization behaviors [14–20].

However, the low heat distortion temperature, low melt viscosity, poor optical properties, and pronounced brittleness of unreinforced PTT at low temperature have restricted its use as a desirable engineering plastic. Some of these deficiencies could be improved by developing PTT composites or blends with suitable polymers in which it retains its excellent properties. Polymer blending is a straightforward, versatile, and relatively inexpensive method of creating a new polymer material which has the desirable properties of all the constituent components. PTT blends are expected to possess a wide range of features that will broaden the applications of the homo-polymer. Recently, a considerable amount of research work pertinent to PTT blends was reported [21–32]. Guerrica-Echevarría et al. [22] found that the toughness of PTT was improved by blending it with poly(ethylene-octene). Run et al. and Krutphun et al. reported that PTT was miscible with poly(ethylene naphthalate) (PTN) in the amorphous phase, and that the variation in the glass transition temperature with the blend composition fit the Gordon–Taylor equation well [23,24]. The correlation of the morphology and rheological response of PTT/m-LLDPE blends was investigated by Jafari et al. [25]. Compatibilization studies of PTT/EPDM [26], PTT/PS [27] and PTT/PP [32] blends were recently reported. The miscibility, melting and crystallization behaviors of PTT/PEI [28], PTT/PTN [30] and PTT/PC [31] blends were also reported.

ABS, an extensively used commercial polymer, is associated with good processability, dimensional stability and higher impact strength at lower temperatures. ABS is a feasible choice for blending with PTT, because of their potential combination of good impact strength, modulus, heat and chemical resistance, and abrasion resistance. However, up to now, no commercial blend products based on PTT and ABS, or even fundamental studies of such blends, have been reported. Since the tailor-made properties of the final blend products depend on the miscibility between the components and the phase structures of the blends, miscibility and compatibilization investigations are required to obtain a blend with the desired properties. In this study, the miscibility of PTT/ABS blends were investigated by using differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA). Then, based on the miscibility result, epoxy and styrene–butadiene–maleic anhydride (SBM) were selected as reactive compatibilizers, and their compatibilization effects on the PTT/ABS blends were studied by DSC, SEM and rheological analysis.

2. Experimental

2.1. Materials

Clear poly(trimethylene terephthalate), with the trade name Corterra CP509201, was supplied by the Shell Chemical Company, WV, USA. The polymer had an intrinsic viscosity of 0.92 dl/g, measured in a 60/40 mixture of phenol and tetrachloroethane at 30 °C. Acrylonitrile–butadiene–styrene (ABS), with the trade name PA-747S, was purchased from Chie Mei Enterprise Co. Ltd., Taiwan, China. Styrene–butadiene–maleic anhydride copolymer (SBM), with the trade name MPC 1545R, was purchased from Shanghai Sunny New Technology Development Co. Ltd., China. The maleic anhydride unit content was 7 wt%. Poly[(bisphenol A)-*co*-epichlorohydrin], with the trade name E-03 (609), a glycidyl end-capped epoxy resin with an epoxy equivalent weight of ~3000 g/equiv, was supplied by the Institute of Tianjin Synthetic Materials, China. Prior to melt processing, PTT was dried at 120 °C for 12 h in a vacuum oven to minimize the hydrolytic degradation of the melts. ABS, epoxy and SBM were dried at 85 °C for 2 h in a vacuum oven. All of the components were thoroughly mixed prior to extrusion.

2.2. Preparation of blends

The melt blending of the dried PTT and ABS with different compositions was carried out using a 35-mm twin-screw co-rotating extruder (Nanjing Giant, China). The barrel temperature ranged from 245 °C to 255 °C, and the screw speed was 144 rpm.

2.3. Measurements

The melt viscosities were measured using a Rosand Precision Capillary Rheometer (Bohlin Instrument Company, UK) at 250 °C. The capillary used had a length to diameter ratio (*L*/*D*) of 16/1 and a zero orifice length. The measurements were carried out at constant velocity. A double-bore experimental mode was used. The data were processed with the Bohlin Instrument Company's software, and were corrected for the capillary entrance elastic effect and the wall shear rate effect for a non-Newtonian fluid with the Bagley and Rabinowich corrections, respectively.

DSC measurements were carried out using a Netzsch differential scanning calorimeter, model DSC-204. The temperature was calibrated with an indium standard. The measurements were performed under a high purity nitrogen atmosphere to minimize the possibility of moisture regain and thermo-oxidative degradation. To avoid uneven thermal conduction through the samples, their weight was maintained at 7.5 ± 0.5 mg. The samples obtained at the same cooling stage during the extrusion process sealed in aluminum pans were heated from 20 °C to 270 °C at a heating rate of 10 °C/min to analyze the melting behavior of the blend.

The DMA measurements were conducted on a Netzsch DMA 242. The deformation mode was tensile with amplitude of 2.00 mm and a frequency of 5 Hz. The sample thickness was 0.5 mm and the heating rate was 0.5 °C/min.

The extruded bars were fractured in liquid nitrogen perpendicular to the direction of flow. The fracture surface of the blend was etched with toluene at 80 °C to remove the ABS phase, and then the rinsed and dried sample was gold-coated for SEM (JEOL JSM-6700F) examination.

3. Results and discussion

3.1. Miscibility

Fig. 1 shows the DSC heating scans for the PTT/ABS blends. Two separate T_gs were observed, indicating that the blends are phase separated in the amorphous phase. The lower temperature transitions observed between 40.7 and 46.1 °C were attributed to the T_gs of the PTT amorphous phase. The higher temperature transitions observed from 103.6 °C to 100.2 °C were the T_gs of the SAN phase in the ABS matrix. The variations of the two T_gs values as a function of the blend compositions are shown in Fig. 2. In this figure, it is noticed that the variations of the two T_gs are composition-dependent. When the ABS content was increased from 0 to 70 wt%, the T_g (PTT-phase) shifted from 40.7 °C to 46.1 °C and then remained at ~46 °C when the ABS content was further increased above 70 wt%. In contrast, the T_g (ABS-phase) decreased after the addition of 10 wt% PTT, and then remained unchanged with further PTT addition.

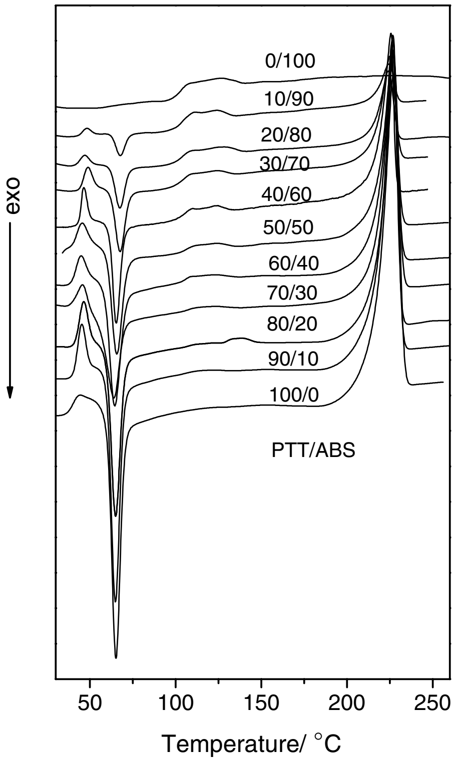


Fig. 1. The DSC thermograms for the PTT/ABS blends (heating rate, 10 °C/min).

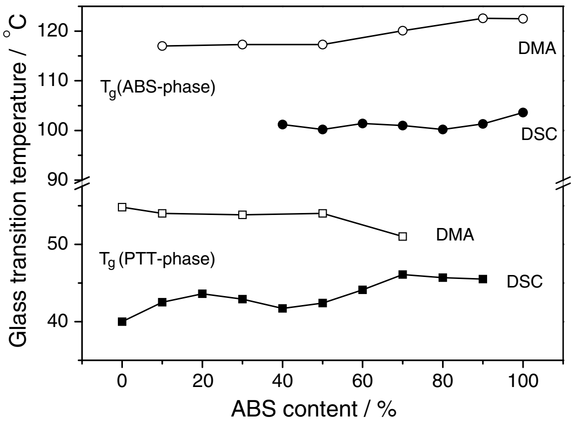


Fig. 2. Plots of glass transition temperatures as a function of ABS content of PTT/ABS blends.

These results are typical behaviors of a partially miscible system [33–35]. The results indicate that: (i) PTT is partially miscible with ABS; (ii) the miscibility of the PTT/ABS blends is improved slightly as the ABS content is increased; and (iii) assuming that the empirical Fox equation [36] is applicable to the PTT/ABS blends, the T_gs changes indicate that the solubility of ABS in the PTT-phase is greater than that of PTT in the ABS-phase.

The variations of the melting temperature (T_m) and cold crystallization temperature (T_{cc}) of PTT as a function of the blend compositions are shown in Fig. 3. The T_m value shifts to lower temperatures with increasing ABS content. In general, for a blend exhibiting some phase mixing, the presence of a second partially solubilized polymer will cause the melting temperature to decrease, due to the reduction in the chemical potential. This behavior is usually observed in the case of miscible or partially miscible blends [28,30,34,37,38]. Accordingly, the variation of T_m indicates that the solubility of ABS in the PTT-phase is slightly increased with increasing ABS content. On the other hand, it also implies that PTT and ABS are partially miscible. In contrast, the T_{cc} value shifts slightly to higher temperatures as the ABS content is increased, indicating that a much higher activation energy is needed for the organization of the neighboring PTT segments, due to the interference of the ABS segments. This behavior, in turn, is characteristic of partially miscible blends [34,35].

In order to confirm the DSC results, the miscibility was characterized by dynamic mechanical analysis (DMA). Fig. 4 shows the plots of tan δ vs. temperature for the pure PTT, ABS, and selected blends. Similar to the DSC results, each blend exhibits two separate glass transition relaxations corresponding to the PTT and ABS phases, respectively. Contrary to our expectations, the blend composition showed little influence on the relaxation temperature of the PTT-phase. The T_g (PTT-phase) in the blends stabilized at ~54 °C, and was 0.8 °C lower than that of pure PTT, except in the case of the 30/70 PTT/ABS blend, whose T_g was 3.8 °C lower than that of pure PTT. However, when the PTT content was increased from 0 to 50 wt%, the T_g (ABS-phase) decreased from 122.5 °C to 117 °C, implying an increase in the miscibility between PTT and ABS. These results confirmed that PTT was partially miscible with ABS.

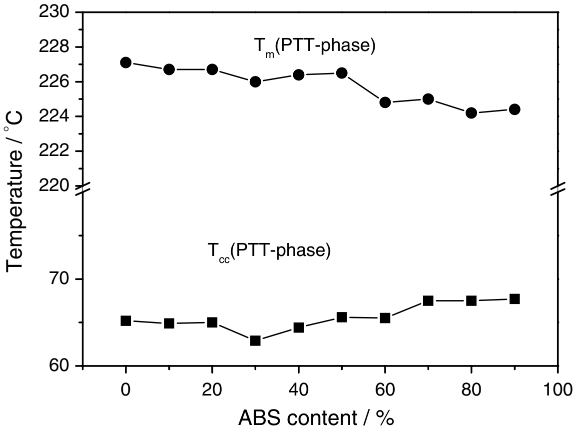


Fig. 3. Variations of the T_{cc} and T_m values of the PTT-phase with the blend composition.

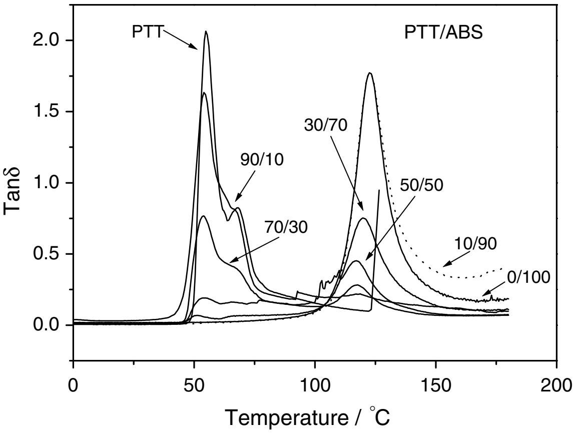


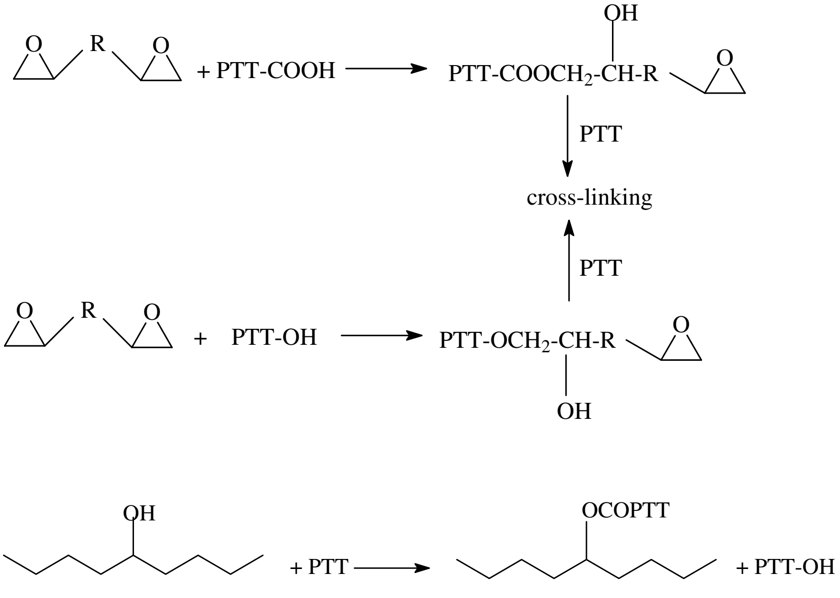
Fig. 4. The plot of tan δ vs. temperature for the PTT/ABS blends melt processed at 250 °C.

3.2. Compatibilization strategy

The prerequisite for preparing PTT/ABS blends with the desired properties is to improve the compatibility of the components. Generally, their poor compatibility can be improved by incorporating a surface-active block or graft copolymer which preferentially resides at the polymer–polymer interfaces with different segments having affinity for one or other of the two blend components. Such compatibilizers are either added before blending (physical compatibilization) or formed in situ by interfacial reactions (reactive compatibilization) during melt processing. Reactive compatibilization consists in blending two immiscible or partially miscible polymers bearing functional groups. These functional groups can react with the compatibilizer during the limited time of melt processing. As a result, block or graft copolymers are formed at the interface.

PTT has carboxyl and/or hydroxyl terminal groups which can react with epoxide, amine, anhydride, and acrylic acid functional groups during melt blending, whereas ABS consists of SAN plastic and PB rubber phases without any active functional groups. Therefore, the most common compatibilization strategy is to incorporate a functional polymer that is capable of reacting with the carboxylic acid and/or hydroxyl terminal groups of PTT and is also miscible with ABS. Accordingly, epoxy and SBM can act as compatibilizers for PTT/ABS blends. The compatibilization mechanism of epoxy in PTT/ABS blends occurs via two main types of reactions (Scheme 1). One is the nucleophilic substitution between the epoxide and the carboxyl or hydroxyl functionalities of PTT (the ring opening reaction of epoxide). The reaction products have another epoxide end group, since epoxy is bi-functional, and this end group can further react with an objective functional group. As a result, cross-linking may occur. The other type of reaction is the alcoholysis reaction between the ester group and side hydroxyl group. The side hydroxyl groups include the hydroxyl groups in the structural unit of epoxy resin and those produced during the ring opening reactions.

Fig. 5 shows the effects of 1 wt% epoxy on the melting behavior of the PTT/ABS blends. It is clear that both the T_g and T_{cc} values of the PTT-phase are shifted to higher temperatures in the presence of epoxy. This indicates that excess energy is needed to initiate the movements or regular arrangement of the PTT segments, because of the epoxy's compatibilization effect. However, the melting temperature showed no discernable changes in the compatibilized and uncompatibilized blends. These results are consistent with those obtained for the PTT/PC blends compatibilized by epoxy [35]. Fig. 6 shows the influence of the epoxy content on the DSC heating thermograms of the PTT/ABS blends. The T_{cc}s of the compatibilized blends were higher than that of the uncompatibilized blend. As the epoxy content



Scheme 1. Chemical reactions between epoxy and PTT during melt blending.

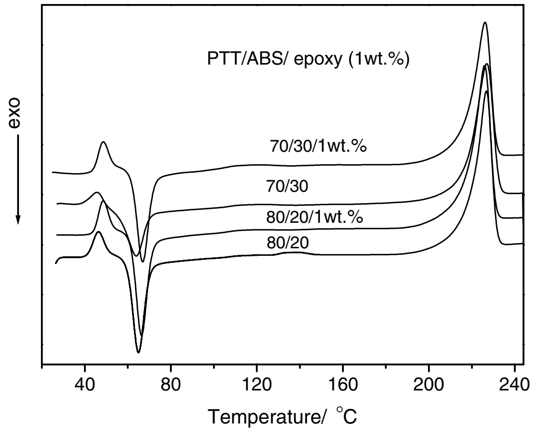


Fig. 5. Effects of epoxy content on the DSC thermograms of the PTT/ABS blends.

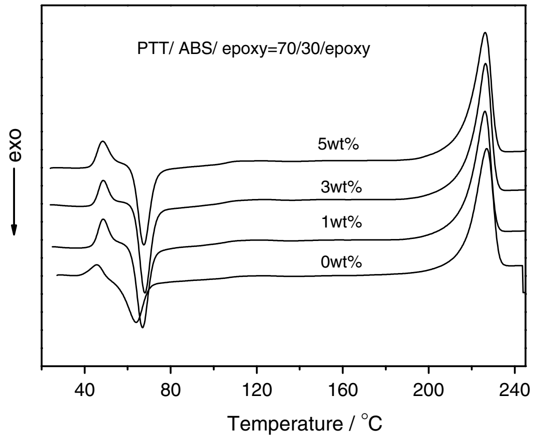


Fig. 6. Effects of epoxy content on the DSC thermograms of the PTT/ABS blends.

increased from 1 to 3 wt%, T_{cc} shifted to a higher temperature and then showed a slight decrease at an epoxy content of 5 wt%. This implies that 5 wt% epoxy is an excessive dosage and that the part of it which does not function as a compatibilizer may act as a plasticizer and/or nucleation agent, which causes cold crystallization to occur relatively easily. This epoxy content-dependent behavior is due to the complex reactions between PTT and epoxy. The nucleophilic substitutions between epoxide and PTT make positive contributions to the phase adhesion and the segments' organizational energy, whereas the alcoholysis between the ester and side hydroxyl groups has two different effects on the blends. One of these is the formation of cross-linking products, which not only improve the miscibility, but also reduce the melt viscosity difference between the PTT-phase and the ABS-phase. The other is the formation of low molecular weight products together with the excess epoxy, which do not act as a compatibilizer and have a negative effect on the blend properties, i.e., they act as a plasticizer. These results indicate that optimum epoxy content is required to balance these different aspects of the behavior.

The influence of 3 wt% SBM on the melting behavior of the blends (Fig. 7) is similar to that of 1 wt% epoxy. Both the T_g and T_{cc} values of the PTT-phase in the SBM compatibilized blends shifted to higher temperatures as compared to the uncompatibilized blends, indicating the compatibilization effect of SBM on the blends. However, the changes in both the T_g and T_{cc} values of the PTT-phase (Fig. 8) indicated that the compatibilization

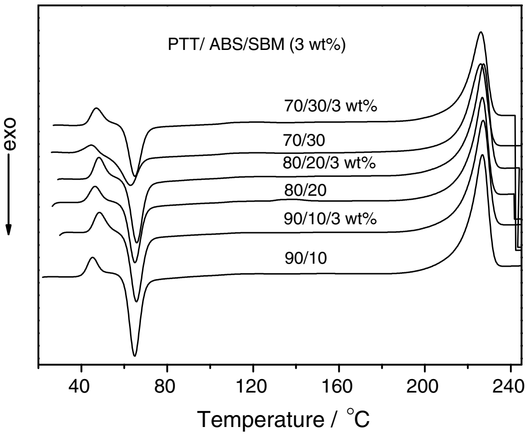


Fig. 7. Effects of SBM content on the DSC thermograms of the PTT/ABS blends.

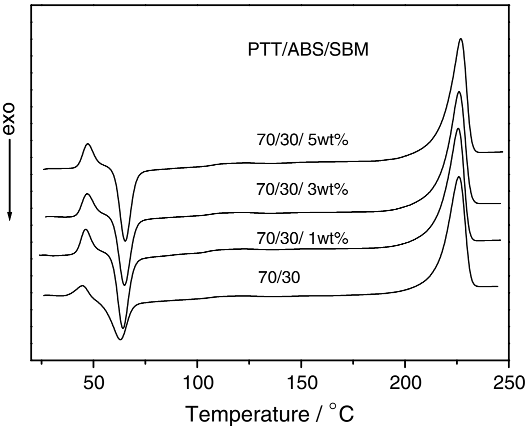


Fig. 8. Effects of SBM content on the DSC thermograms of the PTT/ABS blends.

effect of SBM did not show obvious dependence on its content (1–5 wt%), as did that of epoxy.

3.3. Rheology

The rheological properties of blends reveal some information on the compatibilization effect as well as the correlations among their rheological–morphological–mechanical properties [27,34,39]. To probe the compatibilization effect, the rheological properties of the epoxy or SBM compatibilized PTT/ABS blends were investigated.

Fig. 9 shows the variation of the shear viscosity as a function of the shear rate for PTT, ABS, and selected PTT/ABS blends at 250 °C. Obviously, the PTT melt exhibited near Newtonian flow behavior, whereas both the ABS melt and PTT/ABS blends showed typical pseudoplastic flow behaviors and a linear relationship between the apparent viscosity and the shear rate. Owing to the much more rigid structure of PTT than that of ABS, the ABS melt exhibited more remarkable shear thinning behavior.

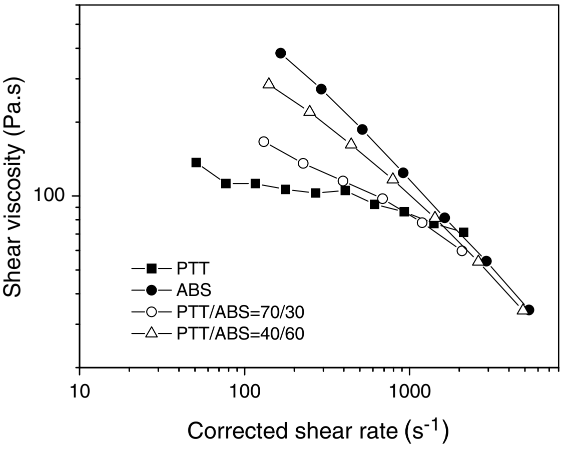


Fig. 9. Shear viscosities vs. shear rates for PTT, ABS and PTT/ABS blends.

Fig. 10 shows the comparisons of the shear viscosity vs. shear rate plots for the PTT/ABS blends at 250 °C with and without epoxy compatibilization. When the epoxy content was 1 wt%, the shear viscosity was increased compared with that of the corresponding uncompatibilized blend, implying an improvement in the adhesion between the phases. However, for the 3 wt% epoxy content blend, the viscosity was higher than that of the uncompatibilized blend when the shear rate was below 400 s^{-1}, whereas it was lower than that of the uncompatibilized blend when the shear rate was above 400 s^{-1}. These results indicate that the interactions between epoxy and PTT are different at different epoxy contents. Theoretically, there are two reactions between epoxy and PTT contributing to the increase in viscosity. One is the chemical interactions (nucleophilic substitutions) whose products are not destroyed at higher shear rates. The other is the physical interactions, i.e., hydrogen bonds, which are destroyed at higher shear rates; once the epoxy is free of such interactions, it will act as a plasticizer and make a negative contribution to the blend viscosity. Therefore, the above rheological behaviors indicate that: (i) when the epoxy con-

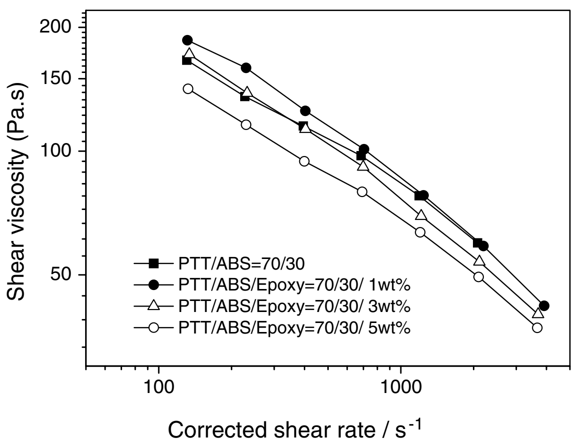


Fig. 10. Effects of epoxy content on the melt viscosities of the PTT/ABS blends.

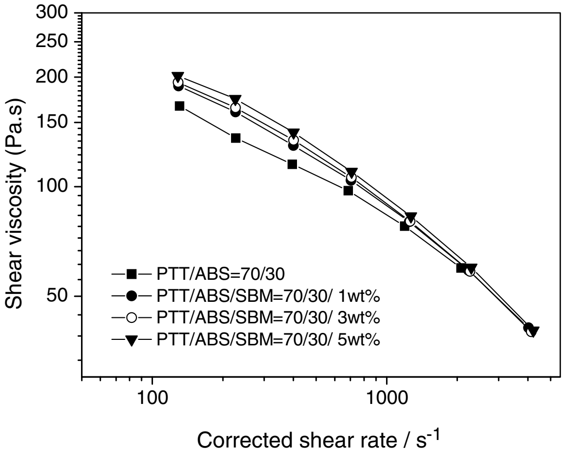
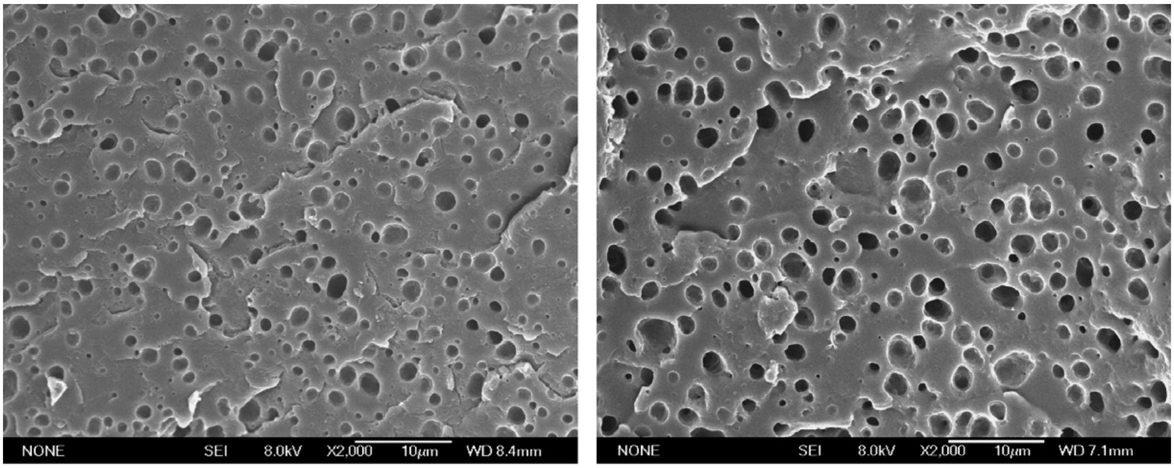
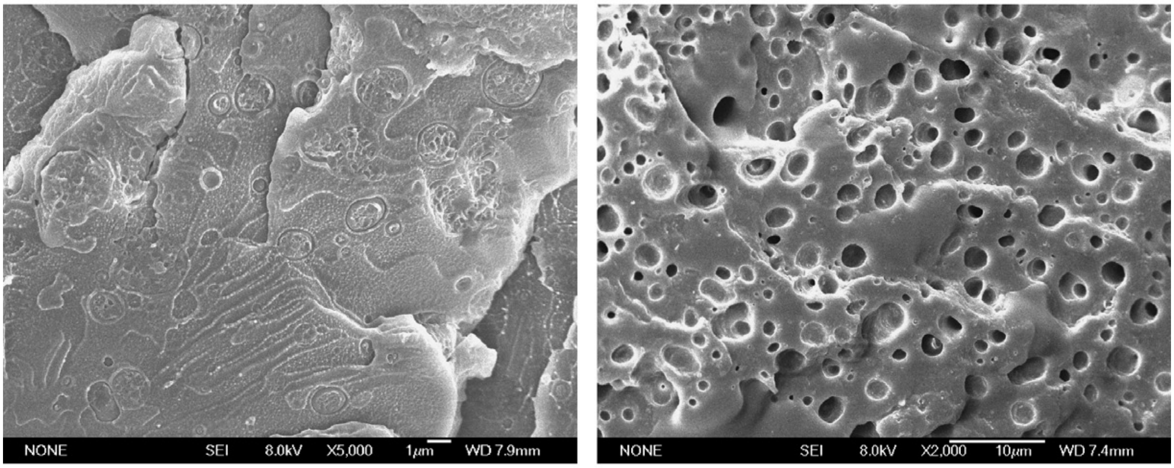


Fig. 11. Effects of SBM content on the melt viscosities of the PTT/ABS blends.

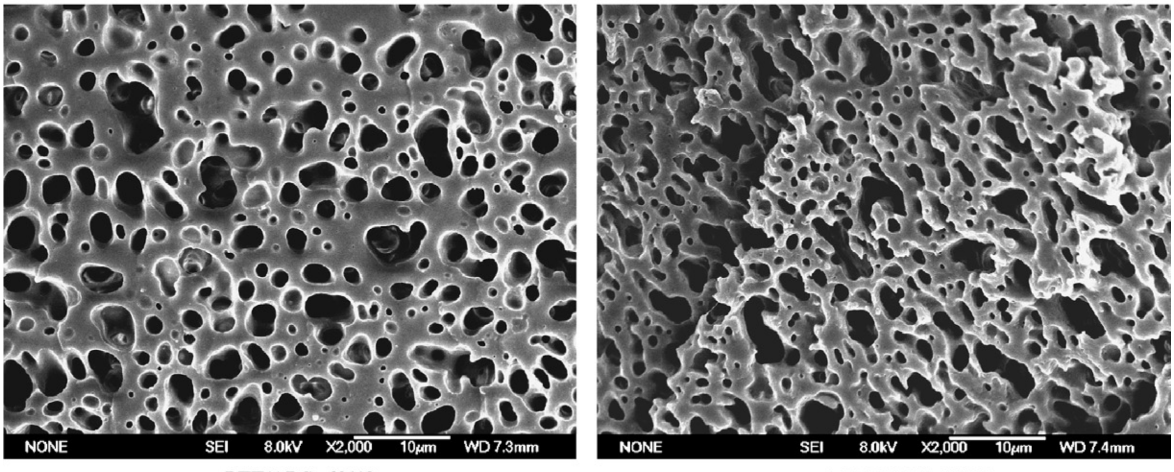
tent was 1 wt%, almost all of the epoxy was chemically bonded with PTT while only a small amount of physical interactions with PTT existed. The influence of the epoxy which was physically bonded with



a: PTT/ABS=90/10 b: PTT/ABS=80/20



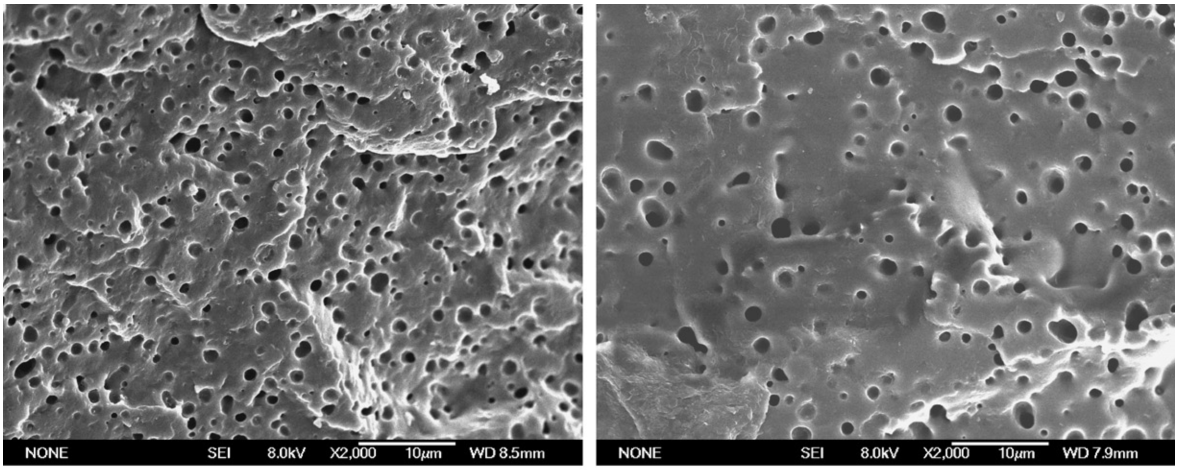
c: PTT/ABS=70/30 d: PTT/ABS=70/30



e: PTT/ABS=60/40 f: PTT/ABS=50/50

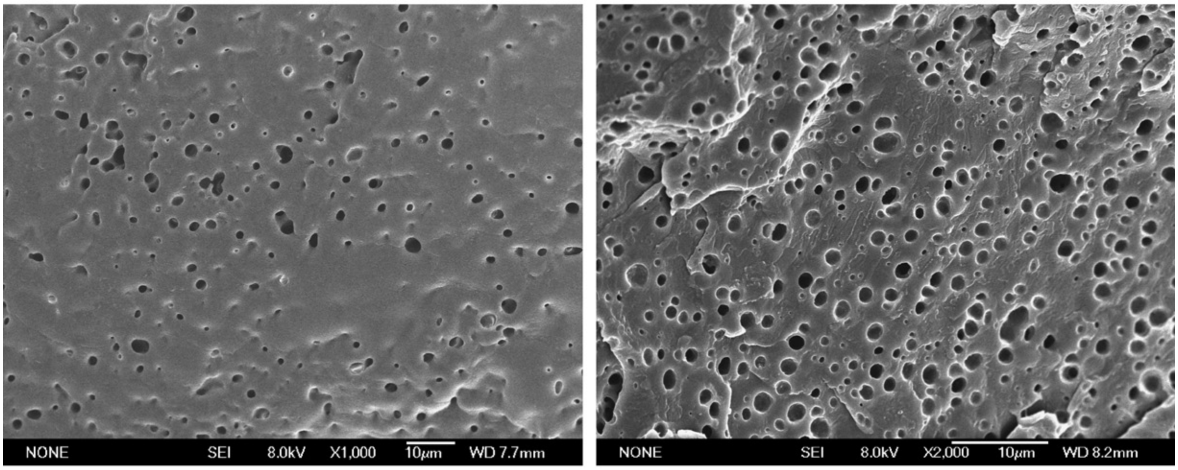
Fig. 12. SEM micrographs of PTT/ABS blends at different compositions (the ABS-phase was etched by toluene).

PTT on the viscosity was insignificant, even when the interactions were destroyed at higher shear rates and (ii) when the epoxy content was 3 wt%, the amount of epoxy which was physically bonded with



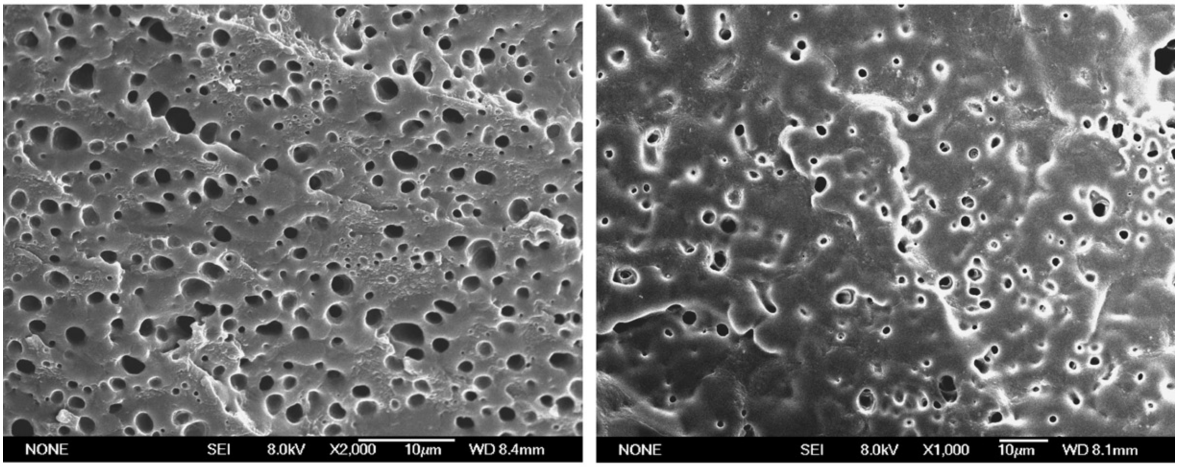
a: PTT/ABS/Epoxy=90/10/1wt.%

b: PTT/ABS/Epoxy=80/20/1wt.%



c: PTT/ABS/Epoxy=70/30/3wt.%

d: PTT/ABS/SBM=90/10/3wt.%



e: PTT/ABS/SBM=80/20/3wt.%

f: PTT/ABS/SBM=70/30/5wt.%

Fig. 13. SEM micrographs of compatibilized PTT/ABS blends (the ABS-phase was etched by toluene).

PTT was increased. It contributed to the increase in the viscosity at lower shear rates (<400 s^{-1}) and to the shear thinning behavior at higher shear rates. However, when the epoxy content was 5 wt%, the shear viscosity was lower than that of the uncompatibilized blend, indicating that 5 wt% epoxy was an excessive dosage, part of which acted as a plasticizer. These results confirm those obtained from the thermal analysis which indicates that there exists optimal epoxy content.

The influence of SBM on the blend viscosity (Fig. 11) differs from that of epoxy. The blend viscosity increased in the presence of SBM, indicating the formation of PTT-co-SBM copolymer which resulted in an increase in the flow resistance, due to the improved phase adhesion. When the SBM content was increased from 1 wt% to 5 wt%, the shear viscosity increased. It is noteworthy that, at higher shear rates, the blends compatibilized by SBM exhibited clearly shear thinning behavior, which led the viscosity to approach that of the uncompatibilized blends. This implies that the shear stresses can overcome the adhesion between the phases and result in a rapid decrease in the viscosity at high shear rates.

3.4. Phase morphology

The morphology of the PTT/ABS blends was investigated by SEM. Because of the weak phase contrast between PTT and ABS, the ABS-phase of the blend fracture surface was removed by hot toluene etching without affecting the PTT-phase, thereby allowing the morphological features of the blend to be better visualized. Fig. 12 shows the SEM images of the PTT/ABS blends with different compositions. The fracture surfaces in Fig. 12c and d are a pair of counterpart surfaces of the same sample. In the case of the sample without etching in Fig. 12c, the fracture surface did not show a clear morphology, due to the weak phase contrast and the biphasic morphology of ABS itself. Fig. 12d clearly shows a biphasic structure, because the fracture surface had been etched by toluene. For the uncompatibilized blends, the etched cavities formed sharp boundaries with the matrices, indicating that there was little or no adhesion between the PTT and ABS phases. The domain size of the dispersed ABS-phase increased with increasing ABS content, since the process of particle coalescence was favored by increasing the concentration. When the ABS content was between 40 and 50 wt%, pronounced phase coalescence occurred which resulted in a much coarser morphology.

Fig. 13 shows the SEM images of the compatibilized PTT/ABS blends. The 1 wt% epoxy incorporated blends showed finer morphologies. The boundaries of the cavities were obscure and seemed to be more difficult to etch, indicating that the interfacial adhesion was markedly improved; moreover, the morphology change was more pronounced when the epoxy content was increased to 3 wt%. Generally, the phase morphology of polymer blends is assumed to be governed by a number of factors, such as the blend composition, interfacial tension, and viscosity characteristics of the components. The reduction of the particle dimension can be attributed to the probable chain extension reactions that induce an increase in the matrix viscosity and the more effective break-up of the particles, which promote a more stable morphology and ensure improved mechanical properties. Therefore, the above SEM results clearly demonstrate that epoxy has a compatibilization effect on the PTT/ABS blends and that the compatibilization effect becomes more distinct as the epoxy content is increased.

Likewise, the SEM morphology shows better adhesion and dispersion with the addition of 3 wt% SBM, indicating the compatibilization effects of SBM on the blends. When the SBM content was 5 wt%, effective adhesion occurred between the phases, as shown in Fig. 13f. These results are consistent with those obtained from the thermal and rheological analysis.

4. Conclusions

PTT was partially miscible with ABS. Both epoxy and SBM showed compatibilization effects on the PTT/ABS blends, which led to a shift in the cold crystallization and glass transition temperatures of the PTT-phase to higher temperatures. The PTT/ABS blends exhibited typical pseudoplastic flow behavior. The rheological behavior of the epoxy compatibilized PTT/ABS blends showed an epoxy content-dependence. In contrast, when the SBM content was increased from 1 wt% to 5 wt%, the shear viscosities of the PTT/ABS blends increased and exhibited much clearer shear thinning behavior at higher shear rates. The SEM micrographs of the epoxy or SBM compatibilized PTT/ABS blends showed a finer morphology, which supports their compatibilization.

Acknowledgements

This work was supported by a Grant from the Post-Doctoral Program of the Chonbuk National University (2006). We also would like to thank the Ministry of Commerce, Industry and Energy (MOCIE) and Korea Industrial Technology Foundation (KOTEF) (the Human Resource Training Project for Regional Innovation), Republic of Korea, for financial support.

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