

Compatibilization of polycarbonate/poly (ethylene terephthalate) blends by addition of their trans-esterification product

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Received: 2 August 2018, Accepted: 2 September 2018

ABSTRACT

In this study, poly carbonate (PC) and poly (ethylene terephthalate) (PET) were reactive melt-blended under two different conditions to produce PC/PET copolymers. For each condition, samples were taken at specified mixing times representative a specific structure of copolymers and each one employed to physically compatibilize a PC/PET blend with a fixed composition. Reactive blending and copolymer structure are described by solubility analysis results. Continues declining and going through a minimum are two trends of solubility versus mixing time depending on reactive blending condition. Decreasing and increasing patterns of solubility curves were attributed to the formation of copolymers with longer and shorter block lengths, respectively, and the level of solubility was related to the amount of produced copolymers. Differential scanning calorimetry (DSC) and scanning electron microscopy (SEM) techniques were employed to investigate blend compatibility. The content and structure of copolymers showed favorable correlation of T_g differences of blend components and PET crystallinity. As expected, T_g of blend components approached to each other by the addition of copolymers, and the copolymers with longer block length caused less T_g differences. The melting point and crystallinity of PET were affected by introducing the copolymers too. In addition to the main melting endotherm, melting endotherm peaks of compatibilized blends had a shoulder that its corresponding melting point and crystallinity are related to the copolymer structure so that the longer length of block copolymer or higher its amount leads to the higher melting points. The SEM micrographs showed that, after the addition of the copolymer, smaller PET particles formed and uniformly dispersed in the PC matrix. A strong correlation between the blend morphology and the level of blend compatibility was demonstrated. The more compatibilized PC/PET blend, the better dispersion of PET particles in the PC matrix was obtained. The results of this study could be a basis for designing and production of compatibilizers suitable to achieve a desired level of compatibility in PC and polyester blends, specially in PC/PET blend. **Polyolefins J (2019) 6: 75-83**

Keywords: PET; PC; trans-esterification; compatibility; crystallinity.

INTRODUCTION

Blending methods for preparation of new materials have attracted more attention compared to synthesizing new materials owing to their convenience and affordability. Due to this aim, different blends with various types have been prepared for diverse applications like automotive industry [1], electrical industry [2], packaging [3] and biomedical applications[4]. PET has been introduced as a proper candidate for packaging industry thanks to good mechanical properties, high chemical resistance and high transparency [5,6]. However, some

drawbacks, such as low heat deflection temperature and impact strength have hampered its usage [7]. PC as an engineering polymer has excellent transparency and toughness, whereas it suffers from weak chemical resistance. Blending has been utilized for compensating the PC weaknesses to achieve high performance PC-based blend [8]. PC and PET are immiscible and PC/PET blend is an excellent material, that their immiscibility may be improved by incorporating nanomaterials and modifiers [9]. Moreover, blending method such as reactive blending can improve the miscibility of PC/PET blend. In this method, a catalyst

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triggers and progresses trans-esterification reaction in which copolymers composed of PC and PET were produced [10]. This subject has undergone extensive studies usually aimed to find parameters in relation to an effective reaction. In this regards, different types of freshly added catalysts [11] or PET contained catalysts [12], catalyst content [13], blend composition [14], mixing time [15] and mixing temperature [16] could be taken into account. Researchers have studied this reaction in the solid state of PC and PET blends [17] too. Rheology [18], spectroscopy [19], microscopy [20], thermal [11] and solubility [21] techniques have been employed to evaluate the reaction and microstructure of the corresponding copolymers.

The in-situ produced copolymers are responsible for the in-situ blend compatibilization. In such compatibilization method, the reactions are not fully controlled and as a result unavoidable side reactions occur through which degradation of blend components adversely affects the blend properties. To overcome these drawbacks, the copolymer was prepared and added to the blend in order to achieve compatibility [22].

Oxazoline in reaction with polypropylene was used by Jeziorska [23] as a compatibilizer for PC/PET blends. He reported that the compatibilized PC/PET blend had one T_g between those of the neat components. Moreover, PET crystallinity and melting temperature were raised by compatibilization. The solubility analysis and measurement of the intrinsic viscosity indicated that the produced PC and PET block copolymers successfully compatibilized the blend [23].

Ma et al. [24] produced PC and PET random copolymers by trans-esterification reaction and the achieved copolymers were added into PC/PET blends to compatibilize them in the melt state. Glass transition temperature of PC/PET blends with various amounts of a compatibilizer was studied. Results revealed that, T_g difference of the components decreased with increasing the content of copolymer, so that at 60 wt% concentration only one T_g was observed indicating the blend miscibility.

Xue et al. [25] employed a bifunctional epoxy to compatibilize PC and poly(trimethylene terephthalate) (PTT) blends. T_g of the polyester in the compatibilized PC/PTT blend was higher than that of the uncompatibilized blend. Moreover, crystallization of epoxy in compatibilized PC/PTT blends was studied [26]. PTT crystallization behavior was disrupted by the addition of PC, resulting in lower melting temperature and crystallinity content.

In this work, PC and PET copolymers were produced by reactive blending in the presence of a freshly added catalyst. After that, the copolymers were added to the PC/PET blend with a predetermined composition. The compatibility obtained due to the addition of the copolymer was evaluated by DSC and SEM techniques and the results were related to the content and microstructure of copolymers. The findings were correlated with the data of solubility analysis, a traditional method to qualitatively evaluate the copolymer microstructure in PC and PET reactive blending.

EXPERIMENTAL

Materials and methods

PC (grade 712) with an MFI of 9.5 g (10 min)⁻¹ (250°C and 10 kg, measured by a Gottferd MFI instrument (model MI-4, Germany) located in Research and Technology of Iranian National Petrochemical Company) was obtained from Khuzestan Petrochemical Company of Iran. PC (grade SC1100) with an MFI of 9.0 g (10 min)⁻¹ (250°C and 10 kg) as reported by the manufacturer was prepared from Samsung Company of South Korea. PET (grade 821) with an intrinsic viscosity of 0.82 dLg⁻¹ (reported by the manufacturer) was obtained from Tondgooyan Petrochemical Company of Iran. The catalyst, lanthanum acetylacetonate, was purchased from Merck (Germany), for use in the trans-esterification reaction. Triphenyl phosphate was purchased from Merck (Germany) as an inhibitor of trans-esterification reaction.

At first, PC 712 and PET were dried at 100°C for 24 h. The blends were prepared in a 50 millilitre Brabender internal mixer. A blend of PC and PET with a 80:20 wt% composition was melt blended for 15 min in the absence of freshly added catalyst. After that, known as premixing, acetylacetonate lanthanum catalyst was added into the mixture and mixing was continued for 20 min according to Table 1.

The criterion to choose condition is formation of different and specified copolymer structures with varied block lengths. The lower temperature and lower catalyst content provided in the C2 condition are suitable to produce copolymers with relatively long block length. On the other hand, the higher temperature and larger catalyst content provided in the

Table 1. PC and PET Reactive blending conditions.

Sample code	T (°C)	Premixing time (min)	Rotor speed (rpm)	Catalyst content (wt %)
C1	280	15	50	0.20
C2	270	15	30	0.15

C1 condition can produce not only copolymers with long block length but also with shorter block length at longer reaction times.

Table 2 represents sampling times during reactive blending of PC and PET to prepare the copolymers.

The sample codes represented in Table 2 are formed by combining the mixing condition (C1 or C2) followed by sampling time (the number after R). For example, C1R5 represents the sample prepared under C1 condition and taken at 5th min of the mixing. PC 1100, PET and the products of C1 or C2 series were dry blended and then melt mixed for 5 min at 270°C for a fixed composition of 66.5/28.5/5 wt%. In the beginning of the mixing process, 1 wt% of triphenyl phosphate was added to the blend to prevent the reaction during the mixing stage.

For solubility analysis, 1 g of PC/PET blend was dissolved in 50 mL of dichloromethane within 24 h under stirring at room temperature, then filtrated and weighed after drying at 80°C. The solubility was calculated through dividing the mass of soluble fraction by the initial mass of blend (1 gr). For thermal analysis, Perkin Elmer equipment (DSC882e model, USA) from Research and Technology of Iranian National Petrochemical Company was used. The analysis was conducted on the soluble and precipitated fractions. The temperature increased in the range of 25 to 280°C with the rate of 10°C/min and under nitrogen atmosphere to avoid thermal degradation.

SEM images (Vega XMU made by Tescan Company of Czech Republic) was utilized for morphology evaluation. Sheets of 2 mm thickness were prepared by a hot press at 260°C and cryogenically fractured under liquid nitrogen. The fractured cross-sections were etched by trifluoroacetic acid for 4 h to remove PET phase and then rinsed in water. The etched

Table 2. Sampling times during PC and PET reactive melt blending under C1 & C2 mixing conditions.

Condition Code	Mixing time (min)			
	5	10	15	20
C1	C1R5	C2R10	C1R15	C1R20
C2	–	–	–	C2R20

surfaces were dried overnight in an oven at 80°C and were coated by a thin layer of gold.

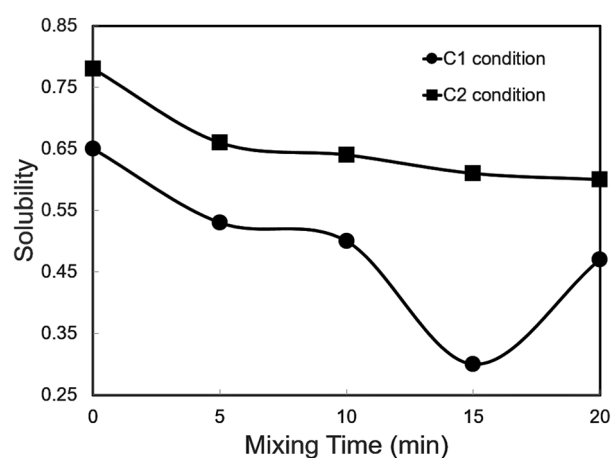
RESULTS AND DISCUSSION

Solubility

To prepare structurally different copolymers, PC and PET were melt blended under the C1 and C2 conditions (Table 1). Fig. 1 shows the curves of solubility versus mixing time, as a measure of trans-esterification progress.

With the C1 condition, a minimum centered after 15 min of mixing was developed. Mixing under the C2 condition resulted in reduction of solubility over the entire mixing time.

The solubility reduction was attributed to the formation of PC and PET block copolymers which were not soluble in dichloromethane. The pattern of solubility with mixing time under the C2 condition indicated that the production of PC and PET copolymers continued to the end of mixing time. In comparison to the C2 condition, the lower level of solubility which reduced with a steeper slope was achieved by the C1 condition. These differences represented the higher rate and more extensive reaction in the second condition. The reduction was followed by higher solubility after 15 min of mixing because of dissolving the earlier copolymers produced in dichloromethane. The copolymers participating in the reaction under the C1 condition made the block length shorter and this new chemical structure helped the copolymers to become soluble in dichloromethane. Considering the C1 con-

**Figure 1.** Solubility versus mixing time for the C1 and C2 processing conditions with dichloromethane as the solvent.

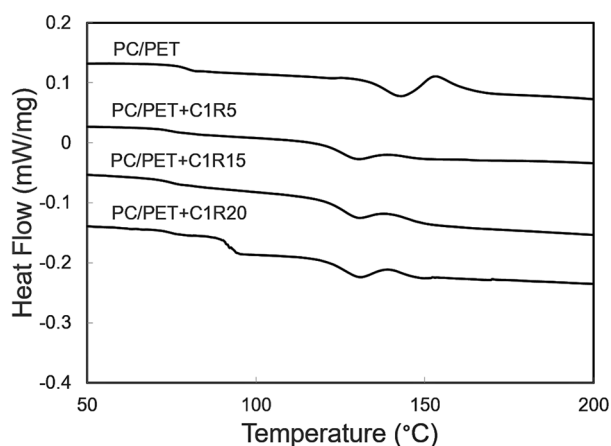


Figure 2. T_g of PC and PET components for blends with and without C1 series products.

dition, sampling was conducted in 5, 15 and 20 min of mixing. These duration times corresponded to the earlier stage of reaction, the maximum amount of copolymers with long block length and formation of copolymers with short block length, respectively. The simpler solubility pattern achieved with the C2 condition limited the sampling times to 10 and 20 min of mixing corresponding to the middle and end of mixing times, respectively.

Thermal properties

Figure 2 and Tables 3 and 4 show the heat flow curves and the T_g of the blends obtained from the DSC analysis.

Figure 2 reveals that the neat PC/PET blend has two T_gs at 78°C and 138°C related to PET and PC, respectively, which indicates the immiscibility of PET and PC. By adding C1R5 to the immiscible PC/PET blend the T_g of PET increased to 80.1°C and that of PC reduced to 125°C. The difference between the T_g of blend components, hereafter named as T_g difference (ΔT_g), decreased from nearly 60°C for neat blend to 45°C after the addition of C1R5. C1R15 had the same effect on the T_g of blend components, but led to the smaller T_g difference of 36.7°C due to its greater effect on the blend. For C1R20, the T_g difference reached 46.8°C which was still lower than that of the neat blend but it was larger than those of the blends con-

taining C1R5 and C1R15.

Table 4 presents the T_g of the blends containing the products of reactive melt mixing of PC and PET under the C2 condition. The T_g difference of the blend containing C2R10, by 10°C lower than that of neat blend, is 50°C. The addition of C2R20 decreased the T_g difference of neat blend to 53°C that was similar to that caused by the addition of C2R10.

The solubility versus mixing time was utilized to compare the amount (or the extension of reaction) and chemical structure of the samples. The solubility of C1R5 and C1R15 was in the decreasing section of the overall pattern in which the second one was lower (Fig. 2). This means that both the samples were comprised of copolymers with the same chemical structure and with different contents. The structure included long block length copolymers and C1R15 caused higher amount of copolymers than C1R5. This chemical structure of copolymers was in favor of PC/PET blend compatibilization that was shown by lower T_g differences of the blends containing C1R5 and C1R15 compared with that of non-compatibilized blend. The lower differences can be achieved by increasing the copolymer content. C1R20 was sampled in a mixing time which corresponded to the raising section of Fig. 1; indicating to the shorter block length of copolymers rather than those produced earlier (C1R15 and C1R5). This enabled C1R20 to compatibilize PC/PET blend, but with lower level of compatibility in comparison to those reached by C1R5 or C1R15. This was understood by a drop in ΔT_g of neat blend after mixing it with C1R20 copolymer which had larger T_g difference than those compatibilized blends using C1R5 and C1R15.

The reactive melt mixing products prepared under the C1 and C2 conditions not only affected the T_g of the blend components but also influenced PET crystallization which was evaluated by the endothermic melting peaks depicted in Fig. 3 and related data are presented in Tables 3 and 4.

PET in the pure blend had a melting point centered at 234°C. For the products prepared under the C1 condi-

Table 3. Thermal properties of PC/PET blend components after compatibilization by C1 series products.

Sample code	T _g PET (°C)	T _g PC (°C)	ΔT_g (°C)	T _{m1} (°C)	T _{m2} (°C)	ΔH_1 (J/g)	ΔH_2 (J/g)	$\Delta H_2/\Delta H$ (%)
Neat PC/PET	78	138	60	234	-	8.12	-	0
PC/PET+C1R5	80	125	45	231	240	5.51	3.88	41
PC/PET+C1R15	82	119	37	232	243	4.48	5.19	54
PC/PET+C1R20	75	122	47	232	242	3.92	4.28	52

Table 4. Thermal properties of PC/PET blend components after compatibilization by C2 series products.

Sample code	T _g PET (°C)	T _g PC (°C)	ΔT _g (°C)	T _{m1} (°C)	T _{m2} (°C)	ΔH ₁ (J/g)	ΔH ₂ (J/g)	ΔH ₂ /ΔH (%)
Pure PC/PET	78	138	60	234	-	8.12	-	0
PC/PET+C2R10	71	122	50	236	244	5.79	5.56	49
PC/PET+C2R20	70	123	53	235	244	6.32	5.93	48

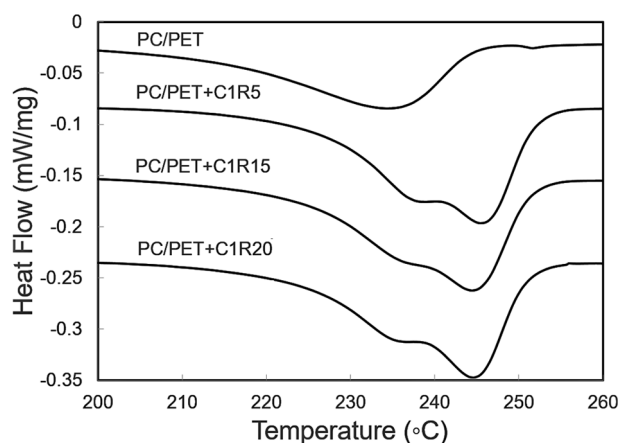
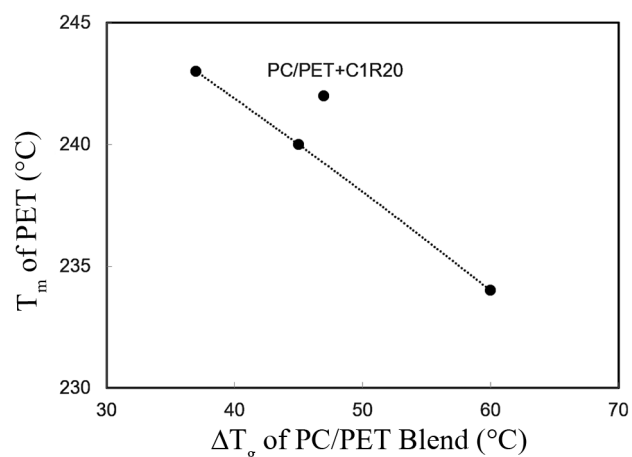
tion, a shoulder appeared higher than 234°C. The temperature corresponding to the shoulder changed in the narrow range of 231 to 232°C for all C1 series but the main peak temperature appeared at 240, 243 and 242°C, for C1R5, C1R15 and C1R20 in the order given.

Inclusion of the products of C2 series into PC/PET blends resulted in a main endothermic melting peak with a shoulder at lower temperature (not shown here). The melting temperature of PET which was centered at 234°C for the PC/PET blend, appeared at 244°C for the blends containing C2 series. The shoulder was centered at 236°C for C2R10 and at 235°C for C2R20.

PET crystallization in PC/PET blend was hindered by PC component which led to lower melting point in comparison to that of PET alone [27]. Mixing the blend with the products of C1 series led to crystallization of a portion of PET molecules, with a mean melting point slightly lower than that of PET in the neat blend, while the remaining PET molecules took part in crystallization with a higher melting point. The copolymers within C1 series, as mentioned before, were responsible for compatibilization of PC/PET blends relating to the structure and content of copolymers. The blend compatibilization in which PC is miscible with PET may result in the latter molecules having lower melting points with respect to their neat blend. Kong et al. [28] studied crystallization of PC and PET reac-

tive blending by the Hoffman-Week model in which they attributed the reduction of PET equilibrium melting point (T_m^0) to the miscibility of PC in PET. Beside T_m^0 , a similar behavior including lower melting point of shoulder in comparison to that of PET in neat blend was expected and it was observed in this work. PET molecules, which were relieved of PC effects, formed the main endothermic melting peak at higher temperatures. Fig. 4 shows the upper melting temperature of PET versus T_g difference between PC/PET blends as an index of the required compatibility.

PET melting point of the blends was lowered by higher difference in T_g . The reduction in melting point was smaller for C1R5 than that for C1R15 or C1R20 which has relatively the same melting point. In other words, more compatibilization of the blends resulted in higher melting points although there was no difference between C1R15 and C1R20. With C1R20, a less blend compatibility was achieved but the melting temperature of PET component, based on its T_g difference, was higher than expected. This may be attributed to the different structure of the copolymer in C1R20 with shorter block length than that in C1R5 or C1R15. The share of higher melting temperature crystals from the whole PET endothermic melting peak is represented by their corresponding enthalpy

**Figure 3.** Endothermic melting peaks from DSC analysis of PC/PET blends with and without C1 series products.**Figure 4.** Melting temperature corresponding to the second endothermic peak of PET versus T_g difference of PC/PET blend.

ratio ($\Delta H_2/\Delta H$). As Figure 5 shows, the lower T_g difference has given higher enthalpy ratios.

Mixing the blend with C1R15 instead of C1R5 decreased the T_g difference of the blend which resulted in higher enthalpy ratio. For C1R20, this ratio was somewhat lower than that for C1R15 but it was still higher than that for C1R5. Similar to the main melting peak temperature, the corresponding fraction of its crystals showed a good dependency on the amount and not on the type of PC and PET copolymers.

C1R15 corresponded to the minimum in solubility diagram (Figure 1) and is among C1 product series led to the minimum T_g difference and the maximum melting point and enthalpy ratio of the second endothermic melting peak. In this sample, the high content of copolymers with long block length is the responsible for these behaviors.

In opposite to C1 series products, here for C2 series there was an increase for shoulder melting points with respect to that of PET in the neat blend. This may be related to the lower compatibilizing ability of C2 in comparison to that of C1 series which resulted in lower miscibility of PC in PET, suppressing the impact on melting temperature, and therefore, higher melting point is expected. The ratios of $\Delta H_2/\Delta H$ were the same for C2R10 and C2R20. Table 4 presents the melting temperatures and the relevant enthalpies.

The copolymers of C2 series had somewhat the same structure and quantities. The identical ability of C2 series, based on relatively the same T_g differences, to compatibilize PC/PET blend resulted in the same effects on PET crystallinity, that have been shown by the same melting temperatures and enthalpy ratios of

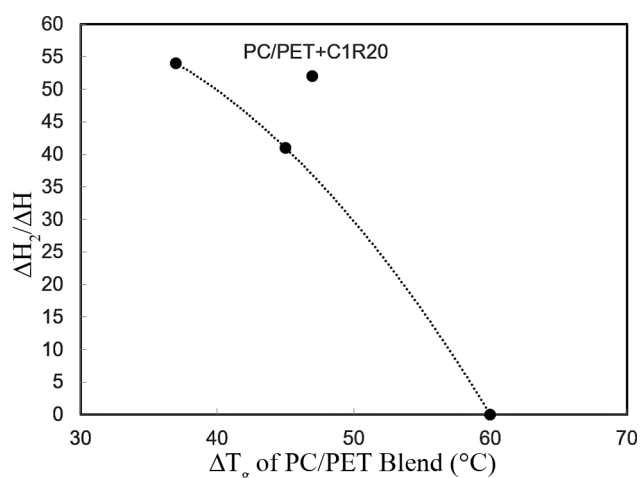


Figure 5. Enthalpy ratio of higher melting temperature peak to total PET enthalpy versus T_g difference of PC/PET blend.

PET component after addition of C2 series products into the blend.

The presence of C1 and C2 series products in PC/PET blends affected thermal properties, including T_g difference, T_m and crystallinity of PET phase depending on copolymer content and structures. Higher content and longer block length of copolymers, which come from the decreasing part of solubility pattern versus mixing time, decreased T_g difference and at the same time increased T_m and enthalpy ratio of the shoulder in the melting endothermic peak. On the other words, the higher level of compatibility between PC and PET phases increases the portion of PET molecules could take part formation of thicker crystals. In comparison of copolymer structure and its content, the effect of the latter is greater.

Morphology

Figure 6. SEM micrographs of the etched cross-section of non-compatibilized PC/PET blend (a), blend compatibilized with C1R5 (b), blend compatibilized with C1R15 (c) and blend compatibilized with C1R20 (d).

The SEM image reveals the morphology of PC/PET blends consisted of a matrix-droplet type with large size of dispersed phase (Fig. 6a). The addition of C1R5 to the blend led to form a sponge-like structure. In comparison with the uneven dispersion of PET droplets in the pure blend, in the presence of C1R5 a more uniform dispersion with smaller PET particle size was created in the PC matrix. But the large PET particles could be still found (Fig. 6b). PET dispersed phase morphology was unified in the case of the compatibilized blend with C1R15 copolymer addition (Fig. 6c). The large particles which could be found in the presence of C1R5 were eliminated when C1R15 was added to the blend. With adding C1R20 the resultant morphology was unique and completely different (Fig. 6d). The morphology discrepancy observed for different samples may be related to their structural differences.

The SEM results are in agreement with the solubility and thermal properties from T_g difference and PET crystallinity; indications of different compatibilizing effects on the products of C1 series. PC/PET blend with 70:30 composition shows two T_g values corresponding to the T_g of its components (Figure 2 and Table 3). The SEM image of pure blend (Figure 6a) shows a coarse disperse-matrix morphology which is in agreement with the thermal analysis results. The

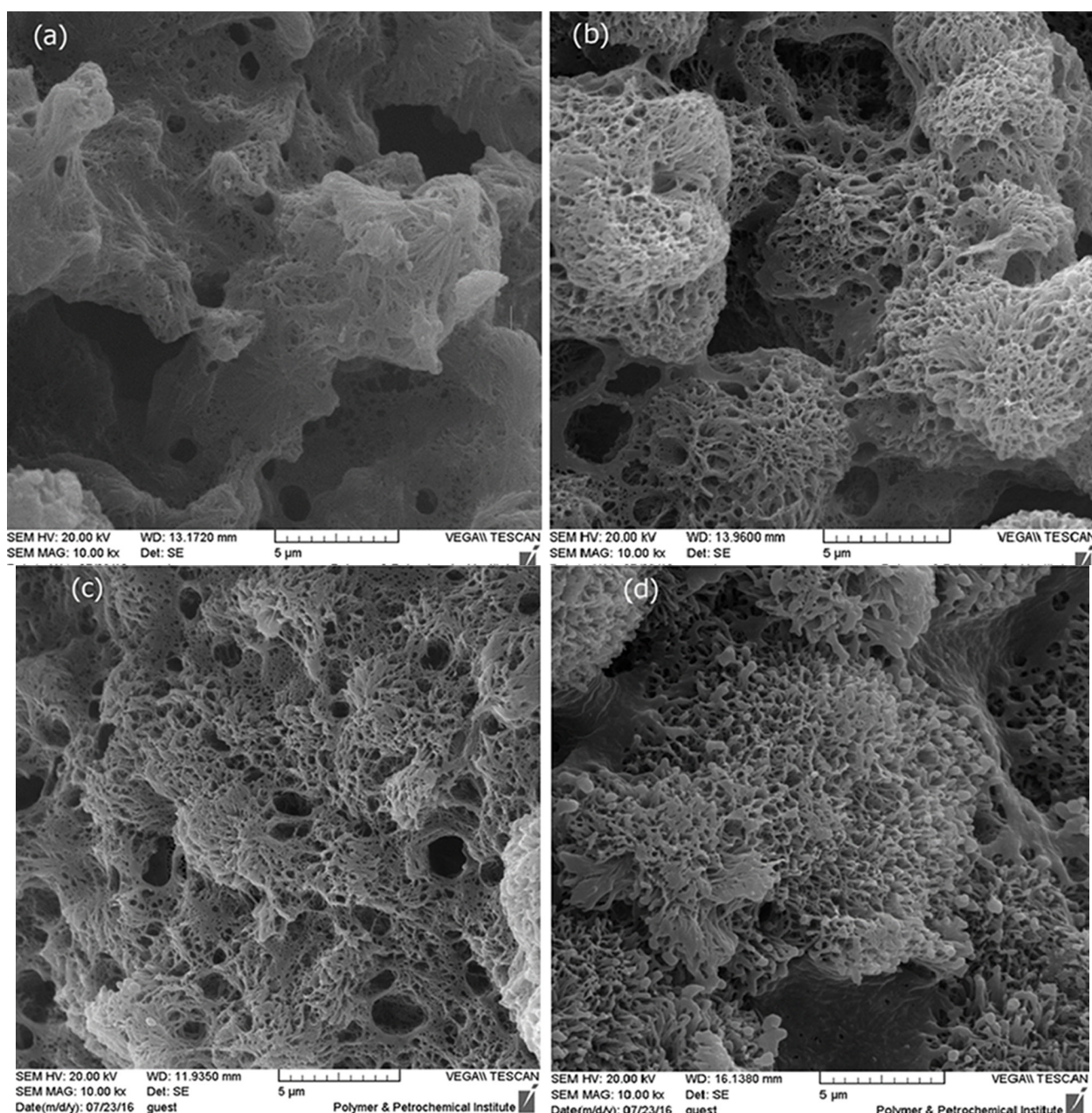


Figure 6. illustrates the SEM image of the sample used for evaluating the compatibilization effects.

addition of C1R5 to the copolymers of PC and PET with long block length made the blend partially miscible because of smaller T_g difference and formation of a shoulder in the melting endothermic peak of PET. In the SEM image, the better dispersion of PET particles in the PC matrix can be clearly observed (Figure 6b). In the blend containing C1R15, higher amount of copolymers made the blend more compatible than that containing C1R5 because of smaller T_g difference and more pronounced shoulder of melting peak. As Figure 6c shows, a more uniform morphology for the dispersed particles of PET is observed. The blend containing C1R20 has copolymers with short block length

and its morphology is somewhat similar to that of

C1R5-containing blend. In this case, the level of compatibility is lower than that for the case of C1R5-containing blend.

In this work, we developed a new strategy to enhance the miscibility of PC and PET with the addition of PC/PET copolymer using reactive melt blending. The introduced technique could help to produce suitable compounds made of PC and PET copolymers for compatibilization of PC/PET blends at desired levels. The presented correlation between solubility and thermal properties made this strategy easy to achieve.

CONCLUSION

In this study, we attempted to enhance the PC/PET blend miscibility with copolymer and reactive melt blending. 5 wt% of compatibilizers in C1 and C2 condition were able to compatibilize PC/PET blend with 70:30 composition ratio. The decreased T_g difference of the blend components and smaller PET particles distributed uniformly in the PC phase proved that the blend compatibilization was improved because of the simultaneous presence of PC and PET in the copolymer. The change in solubility with mixing time was related to the amount and structure of copolymers. T_g difference of the blend components was measured by DSC technique which showed a good correlation between them. For C1 series product, with the same copolymer structure, the higher copolymer content provided lower difference in T_g . By using copolymer with shorter block length, difference in T_g increased. For C2 series product, relatively the same amount of copolymers with identical structure resulted in similar T_g differences. PET crystallinity was affected by addition of C1 and C2 series, so that the main endothermic melting peak forms at higher temperatures than that of PET in the neat blend, besides its shoulder formation. In the shoulder region crystallization was affected by miscibility of PC and PET while the main endotherm showed crystallization under less negative effect of PC component. The melting temperature and enthalpy ratio of the main endotherm were mainly functions of copolymer content and not its structure. In actual fact, PET crystallization could not differentiate the reactive blending products during mixing, especially when the copolymer structure changed.

ACKNOWLEDGEMENTS

The authors wish to express their gratitude to the Research and Technology of National Petrochemical Company (NPC-R&T) for its financial support.

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