

# Thermal, Morphological, and Mechanical Characteristics of Polypropylene/Polybutylene Terephthalate Blends with a Liquid Crystalline Polymer or Ionomer

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Received 21 December 2005; accepted 13 May 2006

DOI 10.1002/app.24797

Published online in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** Thermotropic side-chain liquid crystalline polymer (SLCP) and corresponding side-chain liquid crystalline ionomer (SLCI) containing sulfonate acid were used in the blends of polypropylene (PP) and polybutylene terephthalate (PBT) by melt-mixing respectively, and thermal behavior, morphological, and mechanical properties of two series of blends were investigated by differential scanning calorimetry, Fourier transforms infrared spectroscopy (FTIR), scanning electron microscopy, and tensile measurement. Compared with the immiscible phase behavior of PP/PBT/SLCP blends, SLCI containing sulfonate acid groups act as a

physical compatibilizer along the interface and compatibilize PP/PBT blends. FTIR analyses identify specific intermolecular interaction between sulfonate acid groups and PBT, and then result in stronger interfacial adhesion between these phases and much finer dispersion of minor PBT phase in PP matrix. The mechanical property of the blend containing 4.0 wt % SLCI was better than that of the other blends. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 4712–4719, 2006

**Key words:** liquid crystalline ionomer; blend; compatibilization

## INTRODUCTION

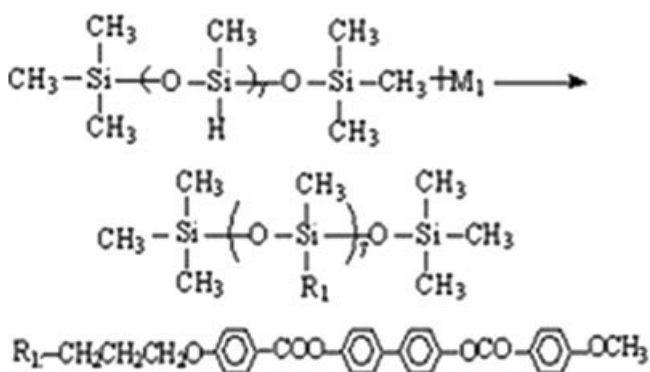
Polymer blending is a useful and attractive approach to produce new polymer materials because of their simplicity and comparatively low cost. Among these polymer blends, the blends of thermotropic liquid crystalline polymer (LCP) with other thermoplastic polymers have been a popular research topic. LCP with high strength and stiffness, high chemical resistance, good dimensional stability, and low linear thermal expansion coefficient are attractive engineering materials with high performance.<sup>1–4</sup> LCP consist of repeating stiff mesogenic (liquid crystalline) monomer units that are incorporated either into the main-chain or into the side chain of a polymer backbone, and now the literature on the blends of the main-chain LCP and other thermoplastic polymers are quite extensive.<sup>5</sup> However, because of poor compatibility between dispersed LCP phase and thermoplastic matrix, the expected mechanical properties based on the rule of mixtures is difficult and impossible (in most cases) to

obtain.<sup>6,7</sup> To increase the compatibility between them, graft polymers or copolymers have been used to improve the interfacial adhesion between the LCP and matrix phases. One approach is to introduce a third component containing the ionic groups that should promote the interchain interactions as a compatibilizer. The lightly sulfonated polystyrene ionomers containing sulfonate acid or metal salt were used as compatibilizer, for example, zinc salts of lightly sulfonated polystyrene (Zn-SPS) was reported to compatibilize blends of a LCP with nylon 66 (PA66) and bisphenol polycarbonate (PC) as a result of intramolecular interactions between the molecules.<sup>8</sup> Other block and graft copolymers have also been used to promote compatibility between LCP and various thermoplastic polymers;<sup>6,9–15</sup> O'Donnell<sup>9</sup> and Datta et al.<sup>6</sup> used a small amount of maleic anhydride-grafted polypropylene (PP-MA) as a compatibilizer to improve the adhesion of LCP and polypropylene (PP). More recently, Weiss and coworkers reported that the sodium salt of a poly(ethylene-*co*-acrylic acid) ionomer (EAA-Na) could be used to compatibilize the Vectra A-950/polyethylene (PE) blends.<sup>16,17</sup>

Thermotropic liquid crystalline ionomer (LCI) is the LCPs containing ionic groups; they would offer the possibility for promoting intermolecular interaction through ion–dipole association and improvement of the interfacial adhesion between the phases in blends. As a novel compatibilizer it has attracted more and more attention during recent years owing to their

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Contract grant sponsors: National Natural Science Fundamental Committee of China, Hi-Tech Research and Development Program (863) of China, and Science and Technology Research Major Project of Ministry of Education of China.



Scheme 1 Synthetic route of the SLCP.

special excellent mechanical rheological processing and adhesive properties in blends.<sup>18,19</sup> Meanwhile, PP/polybutylene terephthalate (PBT) blends is one of the attractive engineering polymer blends for commercial application. In this particular blend, PP provides hydrophobicity, while PBT is the hydrophilic component; therefore PP and PBT are immiscible. In the previous articles, we have reported the compatibilization of main-chain thermotropic LCIs containing sulfonate groups in PP and PBT blends.<sup>20,21</sup> In this article, we prepared PP/PBT/SLCP blends based on PP/PBT with a thermotropic side-chain LCP (SLCP) and PP/PBT/SLCI blends based on PP/PBT with a side-chain LCI (SLCI) containing sulfonate acid groups. Their structure, thermal behavior, morphological, and mechanical properties were investigated. Moreover, we tried to evaluate the compatibilization of the SLCI as a compatibilizer in PP and PBT blends, compared with the blends of PP/PBT/SLCP.

## EXPERIMENTAL

### Materials

PBT is obtained from PBT Engineering Plastic Plants of YIZHENG Chemical Fiber Group. Its intrinsic viscosity is  $0.75 \pm 0.02$  dL/g and MFI is 90–120 g/10 min. PP (F401, 97%) is supplied by Liaoning Huijin Chemical Group (China) and its MFI is 1.7–3.1 g/10 min. Poly (methyl hydrosiloxane) (PMHS) (weight-average molecular weight = 700–800) is provided by Jilin Chemical Industry (Jilin City, Jilin Province, China). The thermotropic SLCP and ionomer were synthesized by the procedure described by Hu and Zhang,<sup>22</sup> the mesogenic monomer  $M_1$  is 4-allyloxybenzoyloxy-4'-methoxybenzoyloxy-p-biphenyl and the ionic monomer  $M_2$  is 4-undecyl-enoxy-4'-phenylazobenzene sulfonic acid. The SLCI was synthesized by copolymerization of  $M_1$  and  $M_2$  with PMHS. The PMHS/ $M_1$ / $M_2$  molar ratio was 1 : 6 : 1.  $M_1$  and  $M_2$  were dissolved in freshly distilled chloroform. The mixture was heated to 65°C under nitrogen and anhydrous conditions, and

then 2 mL THF solution of hexachloroplatinate (IV) catalyst (5 mg/mL) was injected with a syringe. The progress of the reaction was monitored by IR spectra until the Si—H absorption peak of PMHS at  $2160\text{ cm}^{-1}$  disappeared. The crude polymer was purified by precipitation with ethanol and dried under vacuum. The yield of the SLCP and SLCI were 92 and 76%, respectively. The synthetic routes are outlined in Schemes 1 and 2.

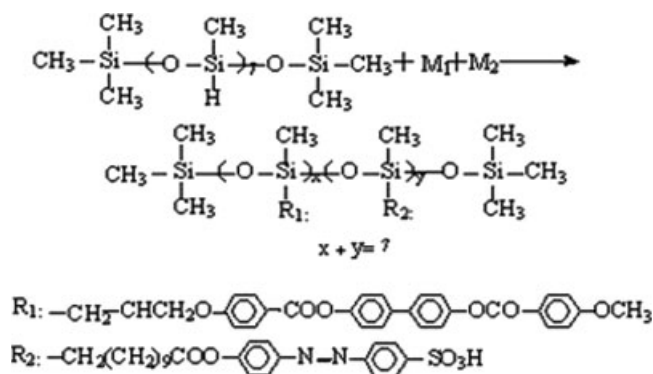
### Preparation of the blends

Blends of PP/PBT/SLCP and PP/PBT/SLCI were prepared by melt-mixing. Before blending, all materials were dried at 120°C under vacuum for at least 12 h, and then were mixed in an XSS-30 Torque Rheogoniometer with a mixing chamber of  $60\text{ cm}^2$  at 230°C at a rotor speed of 32 rpm for 10 min. All the blends had a fixed ratio of PP/PBT 1 : 4 by weight, with different amounts of SLCP and SLCI added. Compositions of all the blends were listed in Table I.

### Materials characterization

The crystallization behaviors and miscibility of the blends were measured with a Netzsch DSC 204 (Netzsch, Germany) equipped with a liquid nitrogen cooling system. Operated at nitrogen flow, specimens were first heated to 200°C at 20°C/min heating rate to eliminate heating history, then cooled to 20°C at 10°C/min cooling rate; The second heating rate was the same as the first. The glass-transition temperature ( $T_g$ ) was defined at the midpoint of the change in the special heat and melting transition temperature ( $T_m$ ) was determined as the maximum of the crystallization peak. The miscibility of a blend was inferred from the observation of a single  $T_g$  (or  $T_m$ ); multiple  $T_g$ s (or  $T_m$ s) indicated phase separation.

Fourier transform infrared spectroscopy (FTIR) of the blends in solid state was obtained by the diffuse reflection method performed on a Nicolet 510P spectrophotometer.



Scheme 2 Synthetic route of the SLCI.

TABLE I  
Phase Transition of the Blends

Sample	SLCP (wt %)	SLCI (wt %)	$T_m$ (°C)/heating		$T_c$ (°C)/cooling	
			PP	PBT	PP	PBT
B <sub>0</sub>	0.0	0.0	166.0	224.5	122.6	197.6
B <sub>1</sub>	4.0	0.0	166.2	224.5	122.0	197.3
B <sub>2</sub>	8.0	0.0	165.2	223.4	121.8	196.5
B <sub>3</sub>	12.0	0.0	165.5	223.0	121.6	195.4
B <sub>4</sub>	0.0	2.0	166.2	224.2	122.7	197.5
B <sub>5</sub>	0.0	4.0	165.1	225.4	122.0	196.7
B <sub>6</sub>	0.0	6.0	165.4	223.0	121.8	195.4
B <sub>7</sub>	0.0	8.0	166.2	222.0	122.3	193.9
B <sub>8</sub>	0.0	12.0	165.7	221.2	120.6	187.5

The blend morphology was characterized with a model 505 scanning electron microscopy (SEM) from Philips. The samples were freeze-fractured in liquid nitrogen, and the fracture surface was observed.

Tensile strengths and modulus of the blends were measured with a model 1000N Tensile Tester. The samples were injection-molded with a QLB-D350 Molder. Tensile tests at an elongation rate of 5 mm/min were performed by GB1040-92 on dumbbell samples with an overall length of 11.5 mm and a narrow-section diameter of 6.5 mm.

## RESULTS AND DISCUSSION

### Thermal properties

Detecting phase transition temperature of the components is one of the most effective methods to characterize the miscibility of blends when the components of the blends have a temperature difference larger than 20°C. DSC thermograms of PP/PBT/SLCP blends are given in Figures 1 and 2. Because of a small amount of the SLCP, we failed to detect its  $T_g$ . The melting temperature ( $T_m$ ) and crystallization temperature ( $T_c$ ) of PP and PBT phases, which are determined as the maximum of melting and crystallization peak from second heating and cooling DSC thermograms respectively,

are summarized in Table I. PP and PBT are known as crystalline polymers, and they exhibit two distinct melting temperatures ( $T_m$ ) at second heating DSC thermogram. One  $T_m$  is seen at 166.0°C, which is identical to that of PP phase, and the other  $T_m$  corresponding to PBT phase is also visible at about 224.5°C. This means that PP and PBT are immiscible and phase separated in PP/PBT blend (B<sub>0</sub>) because PP is nonpolar and PBT is polar. The  $T_m$ s of other PP/PBT/SLCP blends (B<sub>1</sub>, B<sub>2</sub>, and B<sub>3</sub>) show no significant shift compared with that of PP/PBT blend. Meanwhile, the crystallization temperature tends to shift to a lower temperature region at cooling course because of the supercooling, and the  $T_c$  of PBT decreased slightly compared with that of PP/PBT blend, while the  $T_c$  of PP show no significant shift. This means that the addition of SLCP have no visible influence on the phase behavior of PP and PBT in the blends.

DSC thermograms of PP/PBT/SLCI blends at second heating course are given in Figure 3. The melting temperatures determined from DSC thermogram are summarized in Table I. Because of a small amount of the SLCI chain, we failed to detect its  $T_g$ . However, we note that the  $T_m$  of PBT in ternary blends varies with the composition, compared with the binary blend. When the SLCI was added for about 2.0 wt %, the  $T_m$  of PBT was depressed by 0.3°C, and the  $T_m$  of PP had no

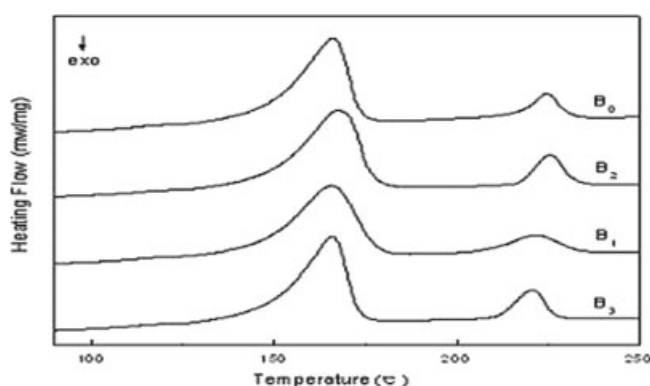


Figure 1 DSC thermograms at second heating of PP/PBT/SLCP blends.

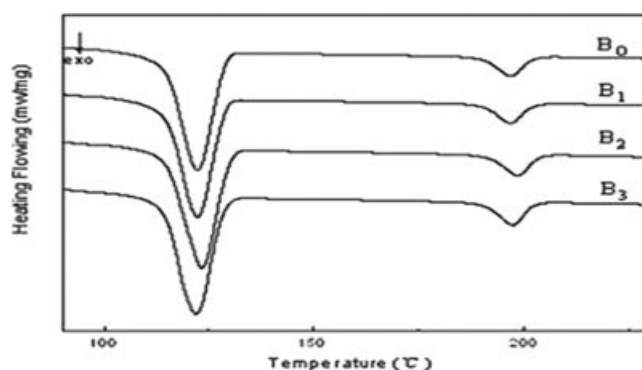
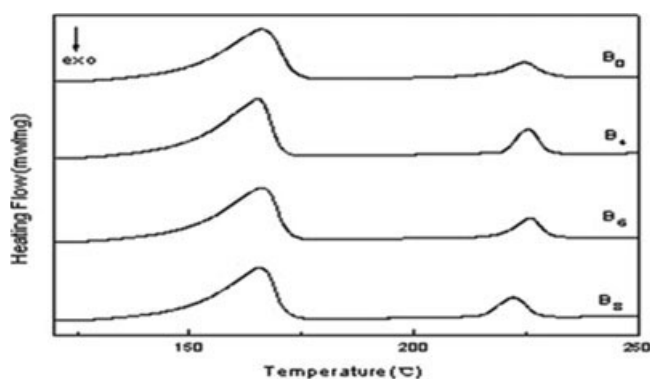


Figure 2 DSC thermograms at second cooling of PP/PBT/SLCP blends.



**Figure 3** DSC thermograms at second heating of PP/PBT/SLCI blends.

significant shifts. With increasing SLCI concentration in the blends, the  $T_m$  of PBT decreased much more; shifts in 4.0 wt % SLCI and 12.0 wt % SLCI were 1.2 and 3.2°C, respectively. In other words, the transition temperature of PP had no significant shifts, but the transition temperature of PBT decreased, indicating the presence of the interactions between PBT and SLCI.

Moreover, Figure 4 shows the cooling DSC thermogram of the PP/PBT and PP/PBT/SLCI blends, and the data are summarized in Table I. It is interesting to note that the crystallization temperatures of PP and PBT phases both decreased with increasing SLCI concentration in the blends, and the  $T_c$  of PBT obviously decreased 3.7°C when the SLCI concentration in the blends increased to 8.0 wt %. When the SLCI content increased to 12.0 wt %, the  $T_c$  of PBT was depressed by 10.1°C. These  $T_c$  shifts suggest that the compatibilization in PP/PBT blends is accomplished by the addition of the SLCI containing sulfonate acid, which promotes mixing through intermolecular interactions between SLCI and PP/PBT phases.<sup>23</sup> Moreover, SEM also observes this result.

### Morphology

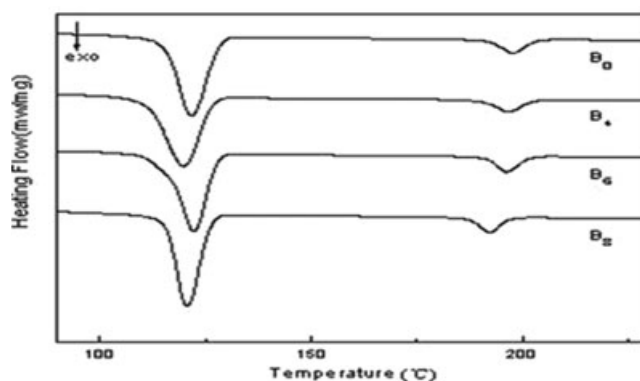
SEM micrographs of fractured PP/PBT/SLCP blend surfaces are shown in Figure 5. Because all the samples were obtained in a mixer, the lack of field effect made it difficult to generate LCP fibrillar in these blends. For the PP/PBT binary blend [Fig. 5(a)], this sample has a distinct two-phase morphology, i.e., a continuous PP phase with a dispersed PBT phase. The PBT particles in the form of spheres have diameters in the range of 5–15  $\mu\text{m}$ . All the spheres and holes have smooth surfaces, indicating poor interfacial adhesion between the PP and PBT phases, that is to say, PP and PBT phases in the blends are immiscible. Such a phenomenon is corresponded with the results in the literature.

With the addition of the SLCP, phase separation appeared more apparently, the big spheres became much more, compared with that of PBT in PP/PBT

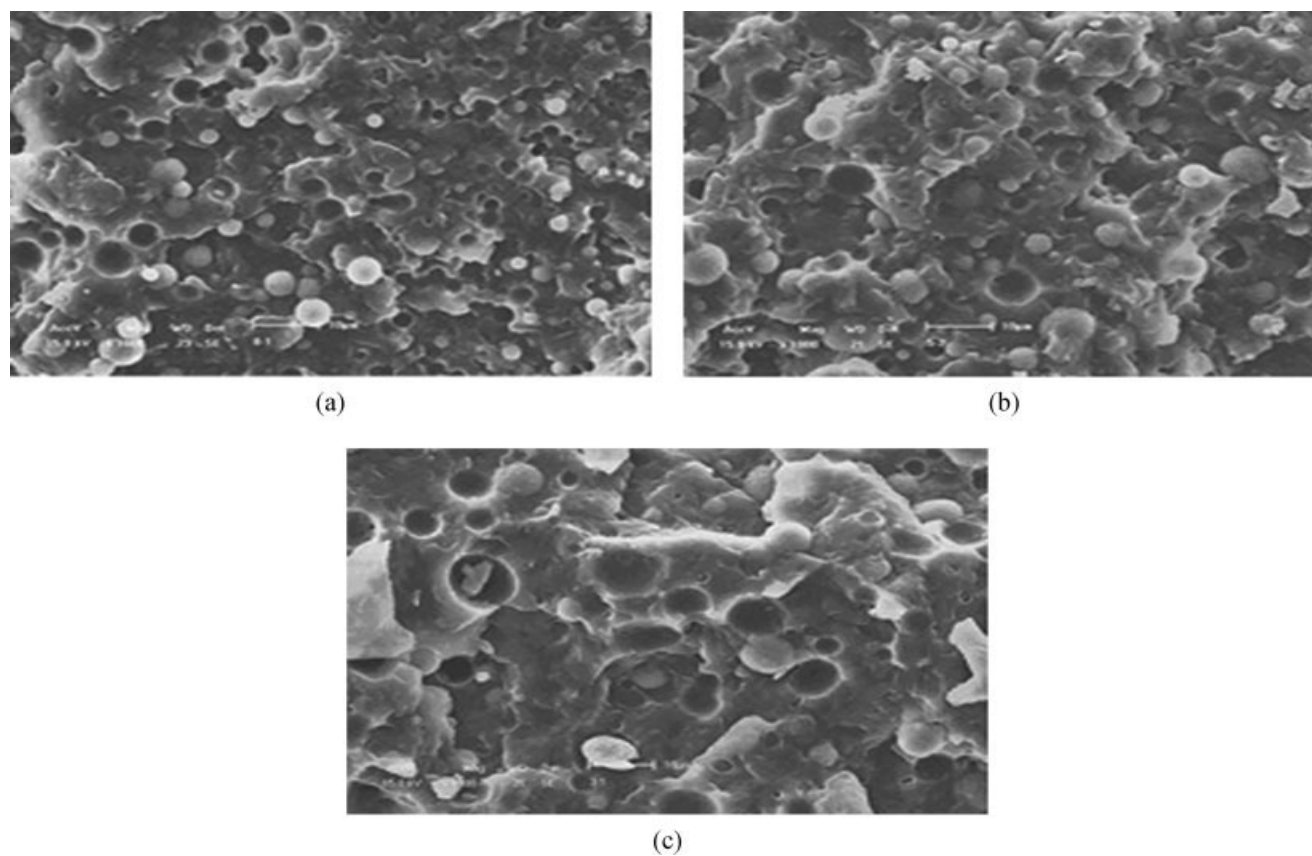
blend [Fig. 5(b,c)]. It is suggested that PP is nonpolar component, while PBT is polar component. The polarity of SLCP is similar to that of PBT because they both contain aromatic ring and ester groups, so the diameter of the dispersed phase became large because partially SLCP joined into the PBT phase. This meant that the addition of SLCP have no improvement on the immiscible structure of PP and PBT components.

Figure 6 shows SEM micrographs of fractured surfaces of the PP/PBT/SLCI blends. With the addition of the ionomer, the morphology of fractured surfaces changed dramatically. With 4.0 wt % SLCI in the ternary blend, the domain size of the PBT phase decreased significantly, and the diameter of the largest particle was smaller than 10  $\mu\text{m}$  [Fig. 6(a)]. With 8.0 wt % SLCI in the blend, The PBT particles became smaller and had uniform diameters below 5  $\mu\text{m}$  [Fig. 6(b)]. When the content of SLCI was 12.0 wt %, the interface between PP and PBT phase became more blurred, and the domain size of the PBT phase hardly disappeared and the PBT phase was well dispersed in the continuous PP phase [Fig. 6(c)]. From these phenomena, it is evident that the interfacial adhesion between two phases was improved because of specific interactions between the phases and the compatibilization of the blends was achieved. The SLCI containing sulfonate acid groups acted as physical crosslinking agent along the interface and compatibilized polymer blends to a finer dispersion.

In previous studies we used a main-chain LCIs (MLCIs) containing sulfonate acid groups for the compatibilization of PP blends with PBT. MLCI was a copolyester of 4,4'-dihydroxy- $\alpha,\alpha'$ -dimethyl benzalazine, 4-hydro-phenylazobenzene sulfonic acid, and sebacoyl dichlorides, 20 although their molecules possess liquid crystal behavior and the properties of ionomer, but the rigidity of the main chains was strong and sulfonate groups located on the terminal unit of the main chain. As a result, the compatibilization of ionic groups was limited. However, mesogenic segments and ionic groups of the SLCI all located on the



**Figure 4** DSC thermograms at second cooling of PP/PBT/SLCI blends.



**Figure 5** SEM micrographs of blends: (a) PP/PBT, (b) PP/PBT/4% SLCP, (c) PP/PBT/8% SLCP.

side-chain with a flexible polymethylhydrosiloxane main-chain, especially sulfonate groups on the terminal of the long and flexible spacer. So the compatibilization of SLCI was obviously superior to that of the MLCI.

#### FTIR investigation of the blends

Figure 7 shows the FTIR spectral region for the carbonyl group in PP/PBT/SLCP blends. Because the structure of SLCP and PBT both contain the ester group, there are two characteristic absorbencies of the stretching vibration of the C=O band for SLCP and PP/PBT components, one is located at 1710, 1263  $\text{cm}^{-1}$  corresponding to the C=O band of PBT and the other is located at 1731 and 1259  $\text{cm}^{-1}$  corresponding to the C=O band of SLCP. For the PP/PBT/SLCP blends, the addition of SLCP had no influence on the position and shape of the C=O stretching vibration of PBT at 1710  $\text{cm}^{-1}$ . Moreover, when the content of SLCP increased to 8.0 wt %, the C=O absorbencies peak of SLCP at 1731  $\text{cm}^{-1}$  appeared and its intensity was strengthened with the increase of SLCP content.

It is interesting to note the change of the characteristic absorbencies of the stretching vibration of the C=O band at 1280–1250  $\text{cm}^{-1}$ . When the SLCP was added to the PP/PBT blend, two peaks appeared that corre-

sponded to 1274 and 1252  $\text{cm}^{-1}$ , and their positions have no change with the increase of SLCP content, but the intensity at 1274  $\text{cm}^{-1}$  decreased obviously. When the content of SLCP was 12 wt %, the intensity of the peak at 1274  $\text{cm}^{-1}$  was similarly equal to that of the peak at 1252  $\text{cm}^{-1}$ . These results indicate that no specific intermolecular interaction exists between the SLCP and PP/PBT.

Evidence for specific interaction between SLCI and PP, as well as PBT phases, was also confirmed by the Fourier transform infrared (FTIR) spectra. Figure 8 shows the IR spectra of B<sub>0</sub>, B<sub>5</sub>, B<sub>7</sub>, and B<sub>8</sub>. It was difficult to distinguish the SO<sub>3</sub>H absorption peak of SLCI molecule, which was weak due to quite a small amount of SLCI added. Figure 8 provides direct evidence that the interaction occurred between PBT and SLCI components in PP/PBT/SLCI blends.

The characteristic absorbencies of PBT were the stretching vibration of C=O band at 1710 and 1245  $\text{cm}^{-1}$ , the absorption peak of C=O from 1710  $\text{cm}^{-1}$  in PP/PBT slightly shifted to higher wave numbers in PP/PBT/SLCI blends, and the absorption peak at 1263  $\text{cm}^{-1}$  in PP/PBT shifted to lower wave numbers in PP/PBT/SLCI blends, i.e., 1263  $\text{cm}^{-1}$  in B<sub>0</sub>, 1261  $\text{cm}^{-1}$  in B<sub>5</sub>, 1258  $\text{cm}^{-1}$  in B<sub>7</sub>, and 1258  $\text{cm}^{-1}$  in B<sub>8</sub>. This IR result confirms an intermolecular interaction between PBT and SLCI, and the interaction comes from the

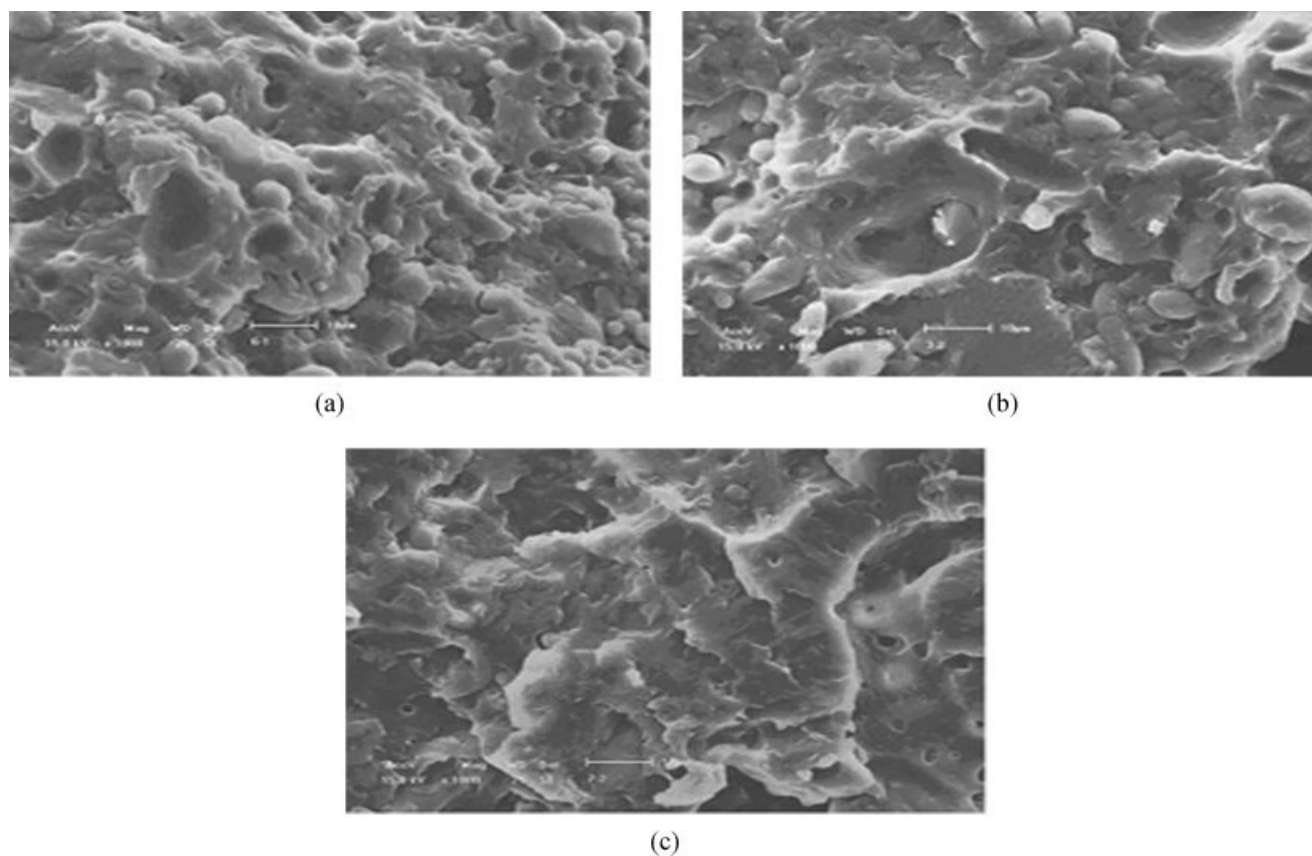


Figure 6 SEM micrographs of blends: (a) PP/PBT/4% SLCI, (b) PP/PBT/8% SLCI, (c) PP/PBT/12% SLCI.

ion–dipole interaction between the ionic groups of SLCI and the polar groups in PBT, meanwhile, the strength increases with the increase of SLCI component. According to the literature, if polymers are miscible, molecular chains of one component polymer can be mixed closely enough to the other component chains so that the intermolecular interaction will be strong enough to change their molecular environment, which results in IR peak shifts and broadening.<sup>23</sup> From these phenomena, it is evident that the interfacial adhe-

sion between PP and PBT phases is improved and the compatibility of the blends is achieved when the SLCI was present.

**Mechanical properties**

Mechanical properties of PP/PBT/SLCP and PP/PBT/SLCI blends were measured with a model 1000N Tensile Tester. The effect of the addition of SLCP or SLCI on the tensile properties of the corresponding

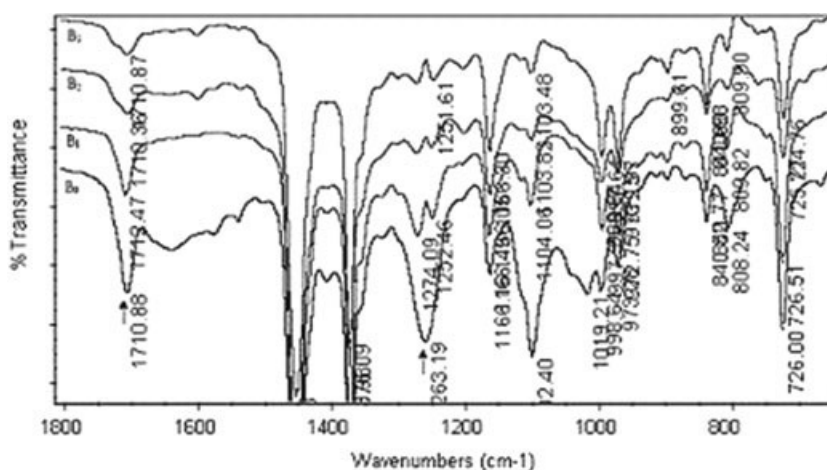


Figure 7 FTIR spectra of B<sub>0</sub>, B<sub>1</sub>, B<sub>2</sub>, and B<sub>3</sub> in PP/PBT/SLCP blends.

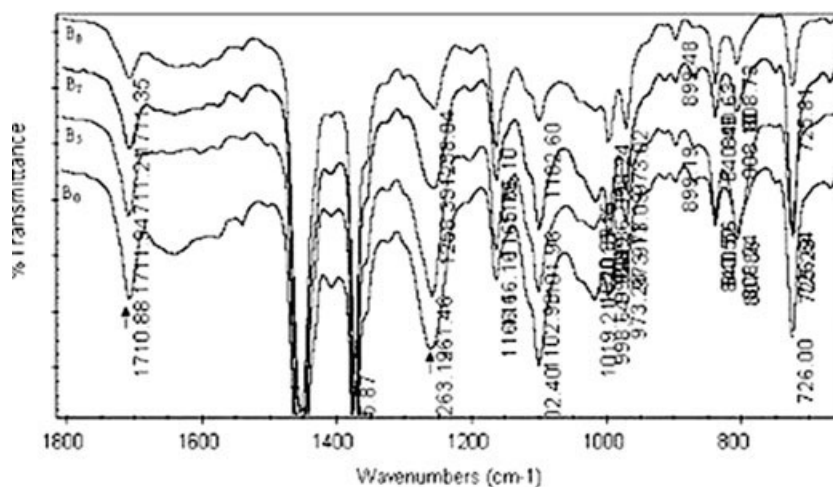


Figure 8 FTIR spectra of B<sub>0</sub>, B<sub>5</sub>, B<sub>7</sub>, and B<sub>8</sub> in PP/PBT/SLCI blends

blends is shown in Table II. Compared with the tensile behavior of PP/PBT blend, the SLCP had obvious effect on the tensile strength and ultimate elongation. The tensile strength of the blend B<sub>1</sub> increased from 5.428 to 8.831 MPa with addition of 4.0 wt % SLCP, however, when the content of SLCP was 8.0 wt %, the tensile strength began to decrease from 8.831 to 8.320 MPa and kept on decreasing to 5.704 MPa with the content of 12.0 wt % SLCP (B<sub>3</sub>). It indicated that a small amount of SLCP being added to the blends improved the mechanical strength of PP/PBT blend because of the orientation order of SLCP in liquid crystalline phase, but the addition of more than 4.0 wt % SLCP resulted the property of the blend to depress because of the incompatibility with PP and PBT.

It was interesting to note that the ultimate elongation of all PP/PBT/SLCP blends increased with the increase of SLCP content. It can be explained that the flexibility of polymethylhydrosiloxane main-chain made the ultimate elongation of the blends increase.

Compared with the tensile behavior of PP/PBT blend, the addition of SLCI to PP/PBT blends also increased the tensile strength of blends dramatically. When the content of SLCI increased to 4.0 wt %, the

tensile strength of the blend (B<sub>6</sub>) exhibited a maximum and it was 7.336 MPa, and then the tensile strength decreased slightly as the content of SLCI is more than 4.0 wt %. Meanwhile, we noticed that the tensile strength of PP/PBT/SLCI blends was slightly inferior to that of the corresponding PP/PBT/SLCP blends for the same content SLCP or SLCI blends. For example, the tensile strength of the blends B<sub>3</sub> and B<sub>5</sub> were 8.831 and 7.336 MPa, respectively. It is implied that enhancement function of SLCI in PP/PBT/SLCI blends were slightly decreased by the interaction between the sulfonic acid groups and PP or PBT phase but the compatibilization of the SLCI obviously increased, compared with PP/PBT/SLCP blends.

Moreover, the ultimate elongation of PP/PBT/SLCI blends also increased with the addition of SLCI to PP/PBT blends less than 4.0 wt %. When the content of SLCI was more than 4.0 wt %, the ultimate elongation of the blends began to decrease, and the result of the ultimate elongation of the blends corresponded with that of the tensile strength. The change of the ultimate elongation can be concluded from the structural difference between SLCI and SLCP molecules, when the content of SLCI was lower, the ultimate elongation increased because of a flexible polymethylhydrosiloxane main-chain and a long and flexible spacer between the main-chain and the sulfonate groups, but when the content of SLCI was higher, the ionic groups in SLCI molecule made the interaction between the components increase and resulted the flexibility of the blends to decrease, then the ultimate elongation decreased equally.

## CONCLUSIONS

Thermotropic SLCI containing sulfonate acid acts as a physical compatibilizer and compatibilized PP/PBT blends, compared with the corresponding LCP. FTIR

TABLE II  
Mechanical Properties of the Blends

Sample	SLCP (wt %)	SLCI (wt %)	Strength (MPa)	Ultimate elongation (%)
B <sub>0</sub>	0.0	0.0	5.428	0.22
B <sub>1</sub>	4.0	0.0	8.831	0.24
B <sub>2</sub>	8.0	0.0	8.320	0.26
B <sub>3</sub>	12.0	0.0	5.704	0.32
B <sub>4</sub>	0.0	2.0	6.687	0.25
B <sub>5</sub>	0.0	4.0	7.336	0.30
B <sub>6</sub>	0.0	6.0	7.304	0.29
B <sub>7</sub>	0.0	8.0	7.278	0.27
B <sub>8</sub>	0.0	12.0	7.175	0.26

and SEM analyses identified the intermolecular interaction between sulfonate acid groups and PBT phase, and the interaction resulted in a stronger interfacial adhesion between these phases and a much finer dispersion of the minor PBT phase in PP matrix. The mechanical properties of the blends containing 4.0 wt % SLCI contents were better than that of the other blends.

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