Polymer Blends of Polyamide-6 and Poly(phenylene oxide) Compatibilized by Styrene-co-Glycidyl Methacrylate

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SYNOPSIS

Incompatible polymer blends between polyamide-6 (PA6) and poly(phenylene oxide) (PPO) have been compatibilized in situ by the styrene-glycidyl methacrylate (SG) reactive copolymers. The epoxy functional groups in SG copolymers can react with the PA6 amine and carboxylic endgroups at interface to form various SG-g-PA6 copolymers. These in situ-formed grafted copolymers tend to anchor along interface to function as compatibilizer of the blends. The styrene and the SG segments of the grafted copolymers are miscible (or near miscible) with PPO; whereas the PA6 segments are structurally identical with PA6 phase. The compatibilized blend, depending on quantity of the compatibilizer addition and the glycidyl methacrylate (GMA) content in the SG copolymer, results in smaller phase domain, higher viscosity, and improved mechanical properties. About 5% GMA is the optimum content in SG copolymer that produces the best compatibilization of the blends. This study demonstrates that SG reactive copolymers can be used effectively in compatibilizing polymer blends of PA6 and PPO. © 1996 John Wiley & Sons, Inc.

INTRODUCTION

Mixing two or more polymers together to produce blends or alloys is a well-established strategy for achieving a specified portfolio of physical properties, without the need to develop new polymer systems. Among the growing array of commercial polymer blends, the majority are immiscible mixtures in which the components reside in separate domains. Immiscible polymer blends often have poor mechanical properties relative to their respective components because of the unfavorable interaction between molecular segments at interface, leading to poor control of morphology and stress transfer under loading in the solid state.1-3

Liu and Baker,4 Xanthos,5 Xanthos and Degli,6 and Chang7 reviewed the recent developments on the compatibilization of incompatible polymer blends. The block or graft copolymers, with segments either miscible or identical to the respective matrix polymers, can act as compatibilizers of the respective polymer blends. These copolymers may be considered as interfacial agents of the blends because they tend to concentrate at the interface and act as emulsifier. Alternatively, an incompatible polymer pair A and B can be compatibilized by a reactive copolymer C-X (X represents reactive group) that can react with component B to form the C-X-B block or graft copolymer provided that the C segment and the component A are identical or miscible (or at least partially miscible). Before reaction, the copolymer C-X itself is not considered to be as a compatibilizer for a polymer blend of A and B, only the in situ-formed copolymer can act as a compatibilizer.7

The in situ compatibilizations have attracted great attention recently as an alternative to replace the conventional block or graft copolymers to compatibilize immiscible blends. Examples of in situ compatibilizations have been concentrated mainly in the polyblends between various nylons and maleic anhydride (MA) grafted polyolefins through the reaction between anhydride and the —NH2 end groups of nylons.8-12 Other than MA functional group, copolymers containing carboxylic acid,13 sulfonic acid,14 and oxazoline15,16 have also been used. Copolymers containing glycidyl methacrylate (GMA) monomer have attracted great attention.

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lately because of their versatile applications. Chung and Carter claimed a polymer composite with excellent low-temperature impact properties based on polycarbonate, poly(ethylene terephthalate) (PET), high butadiene content acrylonitrile-butadiene-styrene rubber, and a styrene-methyl methacrylate-glycidyl methacrylate copolymer. Dagli and Kamdar reported the use of ethylene-glycidyl methacrylate copolymer as a reactive compatibilizer in compatibilizing blends of high-density polyethylene with PET. Liu et al also found that the GMA grafted polypropylene (PP) is an effective compatibilizer in the blends between PP and acrylonitrile-co-butadiene-co-acrylic acid.

In a continuous program to investigate the in situ compatibilizations of polyblends, we reported a series of the in situ compatibilizations based on epoxy-containing copolymers or polymers.

Polyamide-6 (PA6) is one of the important classes of engineering plastics with excellent solvent resistance and good processability. However, most polyamides have a number of deficiencies, including low heat deflection temperature, poor dimensional stability, and low impact strength. On the other hand, poly(phenylene oxide) (PPO) has maintained a key position within engineering plastics since its inception in the late 1960s. It exhibits high-dimensional stability and good thermal properties. However, deficiencies such as poor solvent resistance and difficulty to process prohibit it from a broad usage. Polymers produced by blending PA6 and PPO are certainly an ideal combination to create new products with balanced properties provided that the advantages of one component can compensate the deficiencies of the other. Polymer blends of PA and PPO have attracted great interest from both industries and academia. Synergetic PA/PPO/SEBS(styrene-ethylene, butylene-styrene) blends have been reported by Campbell et al where they used the anhydride-functionalized PPO as an effective compatibilizer to produce highly ductile materials. Hobbs et al studied the PA6,6/PPO blends using styrene-butadiene-styrene block copolymer as a impact modifier. Brown reported that the blend of aryloxytriazine-functionalized PPO with amine-terminated PA results in PPO-PA copolymer formation as an in situ-formed reactive compatibilizer of the blend. Gan and Paul reported that the blends of functionalized PPO with poly(ethylene terephthalate) (PBT) have also been reported by Hobbs et al. Liang and Pan reported SG copolymer as a reactive compatibilizer of the PPO/PET blends. Akkapeddi and VanBuskirk demonstrated that hydrogenated SEBS and styrene-ethylene-propylene triblock copolymers can be used to compatibilize and toughen the blends of PPO with PP.

In this study, copolymers of SG with various GMA contents have been synthesized and used to compatibilize the blends of PPO and PA6. We intend to report their specific miscibility and correlation with their resultant morphological, rheological, and mechanical properties.

**EXPERIMENTAL**

**Materials**

SG copolymers with various weight fractions of GMA in styrene were prepared by suspension polymerization at 80°C for 8 h using 0.5% by weight of benzoyl peroxide as initiator. The detailed procedures were described previously. PPO was the pilot plant product produced by China Technical Consultants Inc. with viscosity of 0.45 dL/g (in CHCl₃ solvent at 30°C). PA6 is NOVAMID 1010C2 produced by Mitsubishi Kasei Corporation of Japan.

**Melt Blending**

All blends were prepared on a corotating 30-mm twin-screw extruder (L/D = 36, Sino-Alloy Machinery Inc.) with a rotational speed of 280 rpm. The barrel temperatures were set from 250 to 280°C. The blended pellets were dried and molded into various ⅛-inch ASTM testing specimens on a Toshiba IS55EPN 1.5-oz injection molding machine.

**Rheological Properties**

Torque versus time measurements were carried out in a Brabender Plastic-Corder using 30 g of sample and a constant rotation speed of 30 rpm. The temperatures inside the mixing were controlled at 270°C for pure PPO and the mixture of PPO/SG5 at 240°C for other compositions. The capillary rheological measurements of the blends and matrices were carried out at 260°C using a Kayeness Galaxy X Capillary Rheometer with a die orifice radius of 0.04 inch and a die length of 0.8 inch. Melt flow rates (MFR) of the matrices and blends were measured at 260°C and 1.1 kg load.
Scanning Electron Microscopies (SEM)

The morphologies of selected blends were examined by SEM using a Hitachi S-570 microscopy at an accelerating voltage of 20 kV. The cryogenically fractured surfaces of the molded specimens were coated with gold before the SEM examinations.

Dynamic Mechanical Analyses (DMA)

The DMA were carried out by using a DMA Model 983 from Du Pont, by operating from −50 to 250°C under nitrogen atmosphere. The dimension of the specimens used is 50 × 12.7 × 3.2 mm.

Mechanical Properties

Tensile properties were performed by the ASTM D638 test method using an Instron universal testing machine Model 4201. The crosshead speed was 5 mm/min. The data were recorded and treated by a computer. Unnotched Izod impact strengths were measured on a pendulum type hammer (TMI 43-1) according to the ASTM D256 method at room temperature. The critical strain energy release rates (Gc) were measured using the method derived by Plati and Williams.40

RESULTS AND DISCUSSION

Chemistry

A reactive compatibilizer is a copolymer containing reactive functional groups that are able to react with one (or both) of the blend component to form a copolymer and acts as a phase emulsifier of the blend. Reactive polymers undergo the usual chemical reactions seen in low-molecular-weight materials. It is common assumption in polymer chemistry that the reactivity of a functional group does not depend on the size of the molecule to which it is attached. However, the reactions involving the functional groups in polymers are more complex than those of the low-molecular-weight materials. The in situ compatibilizer formed varies with chemical structure, temperature, mixing efficiency, and the concentration of reactive groups. In this study, the epoxy groups of SG compatibilizer can react with amine or carboxylic acid end group of PA6 and the hydroxyl end group of PPO by the following simplified equations:

\[ \text{SG-CH-CH}_2 + \text{PA6-NH}_2 \rightarrow \]
\[ \text{SG-CH(OH)CH}_2-\text{NH-PA6} \quad (1) \]

\[ \text{SG-CH-CH}_2 + \text{PA6-COOH} \rightarrow \]
\[ \text{SG-CH(OH)CH}_2-\text{O-C-PA6} \quad (2) \]

\[ \text{SG-CH-CH}_2 + \text{PPO-OH} \rightarrow \]
\[ \text{SG-CH(OH)CH}_2-\text{O-PPO} \quad (3) \]

The above reactions have been well-documented previously.4-7,19,21,23,25 The in situ-formed SG-g-PA6 copolymers tend to concentrate along the interface between PA6 and PPO to act as an effective compatibilizer. On the contrary, the SG-g-PPO copolymers are most likely to reside in the PPO phase and therefore do not function as compatibilizers. However, certain mixed graft copolymers, SG-g-PPO-g-PA6, are also expected to be formed, which can also function as an effective compatibilizer.

Torque Versus Time

Torque measurements have been used successfully to obtain qualitative information concerning the chemical reactivity and the extent of reaction in a compatibilized blend.19,31 Figure 1 illustrates the torque versus time curves for PA6, SG5, and PA6/SG5 = 1/1 mixture. The measured torque of PA6 is unstable, but the average torque value remains fairly

![Figure 1](image-url)
constant. The torque value of SG5 remains almost constant except a slight drop after 400 s, which means the suspected self-curing of the SG5 does not occur. The torque versus time plot of the PA6/SG = 1/1 mixture shows the torque value decreases gradually but the average value is still higher than that of PA6 and SG5. This observed result is quite unexpected: the molecular weight increase from the expected grafting reactions between PA6 endgroups and the epoxy groups in SG5 should reflect in substantial increase of the resultant torque value. Figure 2 shows the torque versus time curves for PPO, SG5, and PPO/SG5 = 1/1 mixture. The torque value of pure PPO is fairly high and remains nearly constant after 300 s, an indication of good thermal stability. The mixture of PPO and SG5 shows a gradual torque increase with time after about 60 s, which can be attributed to the reaction between PPO hydroxyl terminal group and the epoxy functional groups of SG5.

**Capillary Rheometry**

The shear viscosity versus shear rate behaviors of the uncompatibilized and compatibilized PA6/PPO = 70/30 blends are shown in Figure 3. Without single exception, the apparent viscosity of this PA6-dominant blend system increases with the increase of GMA content in SG compatibilizer. Higher viscosity from the better compatibilized blend can be related to the expected higher molecular weight from grafting reactions and the increased interfacial friction of the grafted copolymers anchoring along the interface. Higher GMA content in SG is expected to produce more graft copolymers and greater numbers of grafts per chain of the grafted copolymers. Figure 4 illustrates the shear viscosity versus shear rate of the PA6/PPO = 50/50 blend system where the uncompatibilized blend has higher shear viscosity than the SG compatibilized blends (except that from SG0). This uncompatibilized blend is a cocontinuous structure, and the intrinsically high viscosity of PPO causes high viscosity of the blend. Good miscibility of polystyrene with PPO is well known. The SG0 (polystyrene containing no reactive group) is ex-
pected to be dissolved in PPO phase to act as a plasticizer to reduce the PPO phase viscosity and results in the low viscosity of the blends. Introduction of the lower GMA content compatibilizer SG2 to the PA6/PPO = 50/50 blend results in about the same viscosity as the previous SG0 blend. This result indicates that the grafting reaction takes place insignificantly in this SG2 compatibilized blend. The unreacted SG2 is similar to polystyrene, which can form miscible blend with PPO. Because SG2 has less GMA content in the main chain, it is expected to form only small amounts of the lightly grafted SG-g-PA6 copolymers. Therefore, a greater fraction of the SG2 (relative to SG10) is expected to remain in PPO phase either as free SG2 or as SG-g-PPO copolymer, both tend to reduce the viscosity of the PPO phase. The compatibilized blend with SG10 compatibilizer shows even higher viscosity than the uncompatibilized blend as shown in Figure 4. The higher epoxy content of the SG10 compatibilizer has relatively greater opportunity to make direct contact with PA6 endgroups during melt blending and tends to form the highly grafted SG-g-PA6 copolymers at interface. SG10 also tends to form more grafted SG-g-PPO copolymers within PPO phase and results in higher viscosity. If no grafting reaction takes place, the SG10 copolymer should act like polystyrene as a plasticizer for PPO and should reduce the PPO viscosity as polystyrene. Therefore, the observed highest viscosity from this SG10 compatibilized blend is indicative of greater extent of the graft reactions to form SG-g-PPO copolymers in PPO.

Table I Summarized Melt Flow and Mechanical Properties of PA6/PPO Blends

<table>
<thead>
<tr>
<th>Composition</th>
<th>MFR (g/10 min)</th>
<th>Tensile Strength (MPa)</th>
<th>Tensile Elongation (%)</th>
<th>Unnotched Izod Impact (J/M)</th>
<th>$G_c$ (J/M²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PA6/PPO = 50/50</td>
<td>0.18</td>
<td>24.2</td>
<td>2.4</td>
<td>79</td>
<td>690</td>
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<td>PA6/PPO/SG0 = 50/50/10</td>
<td>—</td>
<td>24.1</td>
<td>1.8</td>
<td>92</td>
<td>1070</td>
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<tr>
<td>PA6/PPO/SG2 = 50/50/2</td>
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<td>31.3</td>
<td>3.8</td>
<td>104</td>
<td>—</td>
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<tr>
<td>PA6/PPO/SG2 = 50/50/5</td>
<td>0.41</td>
<td>33.8</td>
<td>4.5</td>
<td>113</td>
<td>1690</td>
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<tr>
<td>PA6/PPO/SG2 = 50/50/10</td>
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<td>42.4</td>
<td>5.9</td>
<td>280</td>
<td>1920</td>
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<tr>
<td>PA6/PPO/SG5 = 50/50/2</td>
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<td>39.4</td>
<td>6.1</td>
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<td>PA6/PPO/SG5 = 50/50/5</td>
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<td>44.0</td>
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<td>2340</td>
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<td>2150</td>
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<td>PA6/PPO/SG10 = 50/50/5</td>
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<td>52.2</td>
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<td>3660</td>
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<td>PA6/PPO = 70/30</td>
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<td>25.0</td>
<td>7.5</td>
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<td>1920</td>
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<tr>
<td>PA6/PPO/SG2 = 70/30/2</td>
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<td>34.1</td>
<td>6.6</td>
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<td>—</td>
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<td>35.9</td>
<td>6.6</td>
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<td>—</td>
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<td>PA6/PPO/SG2 = 70/30/10</td>
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<td>38.4</td>
<td>7.2</td>
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<td>2600</td>
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<tr>
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<td>22.1</td>
<td>34.0</td>
<td>9.0</td>
<td>205</td>
<td>—</td>
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<tr>
<td>PA6/PPO/SG5 = 70/30/5</td>
<td>19.3</td>
<td>38.0</td>
<td>9.5</td>
<td>252</td>
<td>—</td>
</tr>
<tr>
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<td>17.3</td>
<td>41.0</td>
<td>10.1</td>
<td>312</td>
<td>3150</td>
</tr>
<tr>
<td>PA6/PPO/SG10 = 70/30/2</td>
<td>19.8</td>
<td>37.1</td>
<td>7.1</td>
<td>91</td>
<td>—</td>
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<tr>
<td>PA6/PPO/SG10 = 70/30/5</td>
<td>15.6</td>
<td>40.9</td>
<td>8.2</td>
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<tr>
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<td>41.8</td>
<td>8.6</td>
<td>134</td>
<td>3470</td>
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phase, whereas the SG-g-PA6 and SG-g-PPO-g-PA6 copolymers reside at interface.

**MFR**

The MFR from all the blends investigated are summarized in Table I and Figure 5. Figure 5 illustrates the effect of SG quantity and the GMA content in SG on the resultant MFRs of various PA6/PPO = 70/30 compatibilized blends. The trend of this PA6-dominant blend is similar to the results of Figure 3: the MFR of the blend decreases with the increase of SG quantity and the GMA content in SG. The MFR trend of the cocontinuous PA6/PPO...
\(= 50/50\) series blends is also similar to the results from Figure 4.

**SEM Morphologies**

SEM micrograph is the most convenient approach to differentiate the morphologies between a compatibilized and an uncompatibilized blends. An immiscible and incompatible blend usually results in coarser morphology than the corresponding compatibilized blend. The finer phase domains are the indication of better compatibilization of the blend. The morphologies of PA6/PPO = 70/30 blends, uncompatibilized and compatibilized, are given in Figure 6, where the large dispersed and spherical PPO particles with different dimensions can easily iden-

![SEM micrographs](image)

(A). PA6/PPO=50/50,  (B). PA6/PPO/SG0=50/50/10,  (C). PA6/PPO/SG2=50/50/10,


**Figure 7** SEM morphologies of fractured surfaces of the PA6/PPO = 50/50 blends. (A) PA6/PPO = 50/50; (B) PA6/PPO/SG0 = 50/50/10; (C) PA6/PPO/SG2 = 50/50/10; (D) PA6/PPO/SG5 = 50/50/10; (E) PA6/PPO/SG10 = 50/50/10.
Figure 8 DMA spectra of PA6/PPO = 50/50 uncompatibilized and compatibilized blends.

tified from the uncompatibilized blend [Fig. 6(A)]. The sizes of the PPO particles decrease with increasing amount of the SG2 (2% GMA and 98% styrene) compatibilizer [Fig. 6(B)–(D)]. Figure 7(A)–(E) demonstrates the effect of GMA contents in SG compatibilizers on the resultant morphologies of the PA6/PPO = 50/50 blend series. The blend with higher GMA content in SG results in finer PPO domains, as would be expected. These morphologies indicate that the in situ-formed compatibilizers reduce the interfacial tension in melt and results in smaller domain size of the dispersed-phase particles.

**DMA**

The loss modulus (E") versus temperature from the DMA of the PA6/PPO = 50/50 series blends are illustrated in Figure 8. The uncompatibilized PA6/PPO = 50/50 blend shows two distinct sharp transitions at 42.6°C (T_g1) and 231.9°C (T_g2), corresponding to T_g's of PA6 and PPO, respectively. The blend containing 10 phr polystyrene (SG0) also has a clear and sharp PPO T_g peak at 200.1°C, which is close to the predicted miscible value (203°C) calculated by Fox equation. However, the PA6 T_g peak remains essentially unchanged. That means the nonreactive SG0 is mostly distributed and miscible in PPO phase. The DMA behavior from the compatibilized blends is somewhat different. The addition of the reactive compatibilizers broadens the PPO peaks and shifts T_g's toward lower temperatures, and such a trend magnifies with increasing GMA content in SG copolymer. The shapes and T_g's of PA6 peaks of the compatibilized blends maintain about the same as the uncompatibilized blend. T_g's of various SG copolymers are between 101 and 105°C, very close to polystyrene. If no reaction occurs, those DMA curves (from SG0, SG2, SG5, and SG10) should have been nearly identical. The above observations indicate that the SG copolymers mainly reside in PPO phase and along the interface but virtually none in the PA6 phase. The SG with higher GMA content has more fraction residing at interface (as SG-g-PA6 and SG-g-PPO-g-PA6 copolymers) than the SG containing lower GMA. The dissolved SG and SG-g-PPO copolymers in PPO phase and the formation of an interphase by the SG-g-PA6 copolymer molecules between PPO and PA6 are both responsible for the PPO peak broadening and shifting toward lower temperature because PA6 has a significantly lower T_g. When the SG compatibilizer contains higher GMA content, the thick-
ness of the interphase is also expected to be increased and thus induces further broadening of the PPO $T_g$ peak.

Mechanical Properties

A compatibilized polyblend, in general, has finer phase domain size, greater interfacial contact area, and interfacial adhesion than the corresponding uncompatibilized blend that is critical to stress transfer between phases. However, the finer domain size of the blend alone does not guarantee toughness improvement. The way the compatibilizer affects the inherent properties of the constituent matrices also needs to be taken into consideration. Table I summarizes all the mechanical properties including tensile, unnotched Izod impact, and strain energy release rate ($G_c$). Figures 9 and 10 show the effect of compatibilizer on tensile strength and elongation of the PA6/PPO = 50/50 blend series. Tensile strength increases with increasing of the quantity of compatibilizer and the GMA content in SG compatibilizer as shown in Figure 9. The SG5 compatibilizer gives the best tensile elongation improvement better than that from SG2 and SG10 (Fig. 10). A similar trend has also been found on the PA6/PPO = 70/30 series of blends shown in Table I.

Figure 11 shows the effect of compatibilizer on the unnotched impact strength of the PA6/PPO = 50/50 blend series, and the trend is exactly same as the ten-
sile strength shown in Figure 9. Again, the SG5 compatibilizer gives the best impact strength in the PA6/PPO = 70/30 series of blends shown in Table I.

Figure 12 illustrates the typical plots of notched impact strength versus BDO to determine the $G_c$ from the slopes of the lines. The $B$ is the specimen thickness, $D$ is the unnotched width, and $\theta$ is a geometrical factor.\(^4\) Figure 12 clearly shows that the blend has higher $G_c$ from the SG having higher GMA content in the PA6/PPO/SG = 50/50/10 blends. Same trend has also been observed for the PA6/PPO/SG = 70/30/10 blends as shown in Table I.

SG5 seems to be the optimum level of GMA in SG copolymer to improve the mechanical properties of the blends; further increase in GMA content does not show clear advantages. As we mentioned earlier,\(^1\) one or two grafts per chain of a reactive compatibilizer give the best reactive compatibilization. Higher grafted SG-g-PA6 copolymer tends to form pinning type conformation of the compatibilizer along the interface with relatively smaller styrene segments penetrating into the PPO phase. Such pinning conformation of the grafted copolymer results in less interfacial adhesion. Additionally, these copolymers may be forced back into PA6 phase under vigorous shear mixing conditions. Figure 13(A) shows the schematic diagram of the distribution and structure of the SG grafted copolymers containing small content of GMA where the SG-g-PPO and SG-g-PA6 are only lightly grafted. Figure 13(B) shows the formation of the pinning conformation of the highly grafted SG-g-PA6 copolymers along the interface from the SG copolymer containing higher content of GMA. Additionally, the SG-g-PPO grafted copolymers in PPO phase is also expected to be highly grafted. Therefore, if the GMA content in SG copolymer exceeds the optimum level (5\% in this

![Figure 13](attachment:image.png)

**Figure 13** Schematic diagrams illustrate the distribution and structure from the lower and higher GMA contents in SG copolymers. (A) Higher GMA content in SG copolymer; (B) lower GMA content in SG copolymer.
study), this compatibilized blend may actually result in inferior properties.

CONCLUSIONS

The compatibilizing effect of the *in situ* reactive compatibilizer (SG) on the immiscible and incompatible PPO/PA6 blends has been investigated. The SG copolymer itself in the blend does not function as a compatibilizer but will become one after reacting with PA6 endgroups to form various SG-g-PA6 and SG-g-PPO-g-PA6 copolymers at interface during the melt blending. The unreacted SG copolymers and the SG-g-PPO copolymers are believed mainly residing in the PPO phase. The epoxy groups of SG can react with amine or carboxylic acid endgroup of PA6 to form the SG-g-PPO copolymers and anchor at interface. The optimum GMA content in SG copolymer has been demonstrated as an effective reactive compatibilizer for the PA6/PPO blends.

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