

Synthesis, Characterization, and Application of Semi-Interpenetrating Liquid Crystalline Polymer Networks LCP/PS

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ABSTRACT: Attempts to extend the IPN technology to liquid crystalline polymer (LCP) systems have been made in search for a new approach for enhancing the compatibility of liquid crystalline polymer with engineering thermoplastics. A new type of interpenetrating polymer network based on liquid crystalline polymer : semi-interpenetrating liquid crystalline polymer network comprising liquid crystalline polymer PET/60PHB (LCP) and crosslinked polystyrene (PS) (for short: semi-ILCPN LCP/PS) has been successfully prepared. The compatibility and thermal properties of the semi-ILCPN LCP/PS with different amount of crosslinking agent were investigated by FTIR, SEM, DSC, and TGA, respectively. Furthermore, the possible application of the semi-ILCPN LCP/PS as a new kind of compatibilizer in PPO/LCP blends was also studied and discussed. Well-compatibilized PPO/LCP composites with considerably improved mechanical properties were obtained. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 76: 1141–1150, 2000

Key words: interpenetrating polymer networks; liquid crystalline polymer; PPO, polymer blends

INTRODUCTION

Polymer blends of thermotropic liquid crystalline polymer (TLCP) with engineering thermoplastics have been studied extensively for their great potentiality in two aspects. First, the low viscosity of the liquid crystalline polymers can reduce the overall viscosity of the blend.¹ Thus, molten-state TLCP can act as a lubricant enhancing the processability of polymers. Second, TLCP can be dispersed and oriented in the thermoplastics matrix to form a high aspect ratio microfibrillar structure

when the blends are processed under suitable conditions, which makes it possible to develop high-performance *in situ* composites of TLCP² with superior mechanical properties, good dimensional stability, and thermal stability. Unfortunately, the majority of thermoplastics are incompatible with common aromatic liquid crystalline polymers due to self-aggregation and phase separation,³ which thwarts the hope of achieving synergistic property interactions between blend components. To improve miscibility of the polymers, compatibilizers are used, which are designed to reduce the interfacial energy, improve the interfacial adhesion, and permit a finer dispersion during mixing of the blend components. In recent years, several different approaches have

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been used for enhancing the compatibility between TLCP and thermoplastic polymer matrixes including (1) adding the block copolymers consisting of the rigid-rod liquid crystalline polymer segments and flexible nonliquid crystalline polymer segments,^{4,5} (2) adding the graft copolymers with rigid-rod backbones and the flexible side chains,^{6,7} (3) adding ionomers,^{8,9} (4) adding multifunctional couplers,^{9,10} (5) introducing a long flexible spacer in the main chain of the TLCP,¹¹ (6) introducing some kind of interaction between TLCP and thermoplastic polymer matrixes,^{12,13} (7) selecting TLCP and polymer matrix pairs with active sites that can promote specific interactions, such as acid/base or hydrogen bonding,¹⁴ (8) *in situ* compatibilization by reactive extrusion.^{15,16}

Based on the extensive and systematic studies on the conventional interpenetrating polymer networks (IPNs),¹⁷ it is believed that interpenetrating liquid crystalline polymer networks might be also another promising effective method for enhancing the compatibility between TLCP and thermoplastics. In the last article, a kind of semi-interpenetrating liquid crystalline polymer networks (Semi-ILCPNs) based on a linear TLCP of PET/60PHB copolymer and partially crosslinked polyacrylonitrile was prepared.¹⁸ Such Semi-ILCPNs was formed to be melt processible, and the possibility of its use as compatibilizers was also discussed.

Poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) is one of the engineering plastics, which possesses excellent thermal stability, good mechanical properties, gas permeability, and electric properties, as well as better flame-retardant behavior and dimensional stability. Although PPO has a lot of the above-mentioned properties, the further applications in PPO as high-performance engineering plastics have been hampered by its poor processibility due to the high melt viscosity.

In this work, PPO/TLCP blend was prepared to get a kind of engineering plastic with better comprehensive properties and an acceptable cost. To improve the compatibility between PPO and TLCP, a new type of compatibilizer, semi-ILCPNs, based on TLCP and polystyrene, was designed and synthesized, whose liquid crystalline component is identical with that of the blend, and the second constituent component (PS) is completely miscible with PPO to be blended. Thus, it would be expected that this type of compatibilizer maybe exhibit good interfacial activity and reinforce the interface.

EXPERIMENTAL

Materials

Styrene(St) and crosslinker divinylbenzene(DVB) were purified by distillation under vacuum. Azobisisobutyronitrile (AIBN) was recrystallized from acetone and dried under vacuum. Poly-(2,6-dimethyl-1,4-phenylene oxide)(PPO) was obtained from GE Corporation. The antioxidant used in the preparation of LCP/PPO blend was B215 obtained from Beijing Plastic Corporation. PET/60PHB, where PET is poly(ethylene terephthalate), PHB is *p*-hydroxybenzoic acid, and 60 is the mol percent of the second (LC) component in the copolymer, was synthesized by *in situ* acetylation reaction described elsewhere.

Synthesis of Semi-ILCPN LCP/PS

The compositions of semi-ILCPNs with different content of the crosslinking agent are listed in Table I, along with the blends and PS homopolymers. Each sample is numbered, and the information such as the amount of each component in semi-ILCPNs and the crosslink level of the PS network is given in the abbreviations of the samples listed in the composition column. For instance, semi-ILCPN 1 is abbreviated as 39/61 PET/60PHB-PS(0.5%), where 39/61 is the weight ratio of the two components in the semi-ILCPNs, while 0.5% is the weight percentages of crosslinking agent DVB.

Sequential semi-ILCPNs preparation procedures utilized can be described as follows. To speed up the swelling process, the LCP PET/60PHB was first swollen in a solvent mixture, which was subsequently removed, then LCP was swollen with monomer St containing the crosslinking agent DVB and 0.8% AIBN as the initiator. The swollen LCP was removed from the remaining solution by filtering, and put into a glass ampule. Evacuated and flushed with nitrogen three times, the ampule was sealed under an N₂ atmosphere and then placed in an oven for polymerization *in situ* at 60°C for 24 h, 80°C for 12 h, and 110°C for 4 h, respectively. With the formation of the crosslinked PS network interlocked with LCP, a semi-ILCPNs sample was obtained. All semi-ILCPNs samples were dried at 70°C for 3 days under vacuum before characterization.

The pure PS samples with different amounts of crosslinking agent were prepared by the same

Table I Thermal Characteristics of Samples with Different Contents of DVB of PS from DSC and TGA Measurements

Samples	Compositions	DSC ^(a)		TGA ^(b)
		T_{g1} (°C)	T_{g2} (°C)	T_{id} (°C)
PS 1	Pure PS(0.5%)		104	404
PS 2	Pure PS(1.0%)		106	408
PS 3	Pure PS(1.5%)		109	409
LCP	PET/60PHB	64		434
BLEND 1	39/61 PET/60PHB-PS(0.0%)	64	101	406
BLEND 2	39/61 PET/60PHB-PS(0.5%)	64	104	406
Semi-ILCPN 1	39/61 PET/60PHB-PS(0.5%)	67	103	409
Semi-ILCPN 2	39/61 PET/60PHB-PS(1.0%)	68	100	415
Semi-ILCPN 3	39/61 PET/60PHB-PS(1.5%)	70	95	421

^a The half height temperature.

^b The initial decomposition temperature.

polymerization conditions as those in the synthesis of semi-ILCPNs. Blend 1 and 2 were prepared by the melt blending.

Preparation of PPO/LCP Blends Using the Semi-ILCPNs as a Compatibilizer

The weight ratio of PPO and PET/60PHB was fixed at 90/10. Semi-ILCPN 1 was taken as 0, 2, and 5% (w/w) with respect to PPO/LCP blends, respectively. All materials were dried at 110°C under vacuum for at least 24 h prior to mixing. The melt blending of the PPO, LCP, and semi-ILCPN 1 was performed with a SLF-35B corotating twin-screw extruder. The conditions chosen in melt blending are shown in Table II. The extrudates were cut into pellets by a pelletizer, then were injection molded into test specimens after drying at 110°C under vacuum for 24 h. Injection molding was carried out in a SZ-400ZT injection-molding machine. The injection temperature and injection pressure were 300°C and 5.0 MPa, respectively. The molded specimens consisted of standard test bars for tensile, bending, and impact tests. Five specimens of each composition were tested, and the average values reported.

Spectroscopic and Thermal Analysis

Fourier transform infrared (FTIR) spectra were collected on a Nicolet 750 FTIR spectrometer.

Samples for FTIR were prepared in the form of KBr pellets. Differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA) were performed on a Dupont 1090B thermal analyzer under a nitrogen flow at a heating rate of 20°C/min.

Morphology

A Hitachi S-450 scanning electron microscope (SEM) was used for the fractured surface observation of semi-ILCPN LCP/PS films obtained by hot compression at 220°C under an N₂ atmosphere. The fractured surfaces were sputtered with a 150 Å layer of evaporated gold before entering the SEM.

The morphologies of the fractured surfaces of PPO/LCP melt blends at core and near skin regions parallel to the flow direction of the injection-molded specimens were also observed by the same SEM. The specimens used for SEM observation were taken from the middle part of the injection-molded tensile bars. The specimens were cryogenically fractured along the direction of flow in liquid nitrogen, and the fracture surfaces were coated with a thin layer of gold prior to the observation.

Mechanical and Rheological Testing

Tensile strength was measured according to ASTM D638M-84 method at ambient conditions

Table II Processing Conditions of PPO/LCP Blends with Different Contents of Semi-ILCPN

Zone	First Zone	Second Zone	Third Zone	Fourth Zone	Fifth Zone	Sixth Zone	Seventh Zone	Eighth Zone
T (°C)	260	270	280	280	280	270	270	270

using LJ-1000 tensile testing machine. The tensile modulus were obtained by a DSS-25T electric testing machine. The impact strength of notched test specimens was measured using a IZOD UJ-40 impact test machine. The bending strength of specimens was determined by a Rauenstein testing machine. Rheological properties of the LCP/PPO blends were measured using a RHEOGRAPH capillary rheometer (model 2001). A capillary of 1 mm in diameter and 20 mm in length ($L/D = 20$) was used.

RESULTS AND DISCUSSION

Characterization of Semi-ILCPN LCP/PS

FTIR is becoming one of the most useful tool to investigate specific interactions between polymer chains introduced into immiscible polymer blends for enhancing the miscibility of polymer pairs.^{19,20} By FTIR measurement some information can be obtained in the fundamental aspects, such as differentiating between miscible and immiscible blends and determining the presence and nature of the specific intermolecular interactions in polymer blends, etc.

Investigations on the polyblends reveal that if two polymers are immiscible, it is possible to obtain an unperturbed spectrum corresponding to one component polymer by subtracting the spectrum of the other component from the spectrum of the blend. On the other hand, if two polymers are miscible, molecular chains of one component polymer can be mixed closely to the other polymer chains to change their molecular environment, it results in IR bands shifts or broadening. In these cases, the FTIR difference spectrum become more complex, in other words, the unchanged spectrum of one polymer component cannot be obtained when the spectrum of the other component is subtracted from the spectrum of the blend. In this article, the FTIR difference spectroscopy technique was used to investigated the miscibility enhancement in semi-ILCPNs consisting of PET/60PHB and PS.

The FTIR difference spectra of the blend and semi-ILCPNs minus pure LCP [Fig. 1(b) and (c)] are shown in Figure 1, along with spectrum of the pure PS. From Figure 1, we could find that the difference spectrum of the blend sample minus pure LCP was identical to that of pure PS, which indicates the absence of interactions between LCP and PS molecular chains in PS/LCP blends.

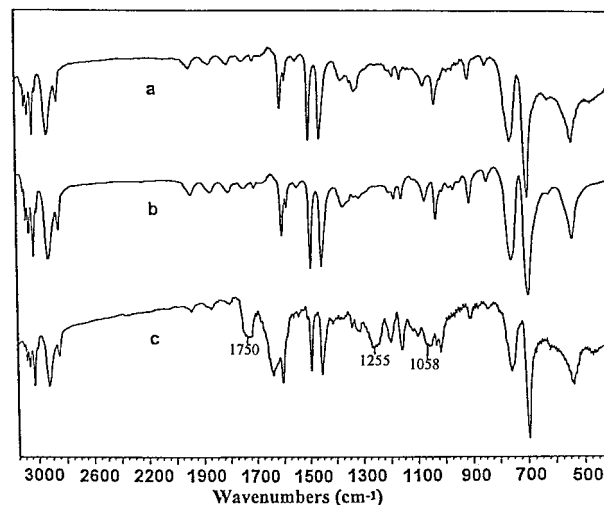


Figure 1 FTIR spectra of (a) PS 2, (b) difference spectrum of blend 2 minus LCP, (c) difference spectrum of semi-ILCPN 2 minus LCP (the compositions of samples are listed in Table I).

In other words, each polymer component keeps its individual properties, especially its own molecular environment, unaffected by the presence of the other polymer components in the polymer. In the case of the FTIR difference spectrum of semi-ILCPNs minus pure LCP, it was becoming more complex than the spectrum of pure PS. The characteristic absorption bands of corresponding to LCP were still remained in the difference spectrum, but compared with the spectrum of pure LCP, the absorption bands shifted to lower frequency. For example, in the difference spectrum a carbonyl absorption appears at 1750 cm^{-1} , whereas in the spectrum of pure LCP it appears at 1756 cm^{-1} . Furthermore, the absorption bands associated with the C—O stretching vibration at 1260 and 1060 cm^{-1} also shifted to lower wave numbers, 1255 and 1058 cm^{-1} , respectively. It is clear that an interaction caused by interpenetrating has, in fact, taken place between LCP and PS during the preparation of this semi-ILCPNs, which could result in enhancement of compatibility of LCP with PS.

It is well known²¹ that the glass transition behavior of blends is mostly affected by the compatibility between polymers. Table I listed the glass transition temperature of the PS/LCP blends and semi-ILCPNs with different content of the crosslinking agent, along with PS homopolymers. The typical DSC thermograms of corresponding samples are shown in Figure 2. It can be seen that the blend exhibited two distinct T_g s

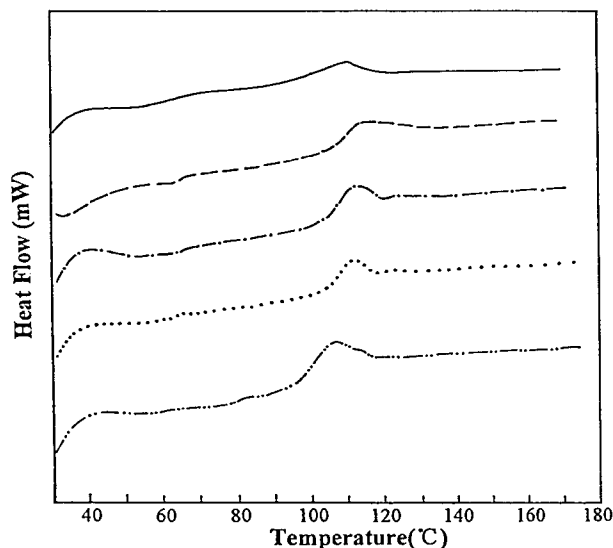


Figure 2 DSC thermograms of (—) blend 1, (---) blend 2, (- · -) semi-ILCPN 1, (· · ·) semi-ILCPN 2, (- · · -) semi-ILCPN 3 (the compositions of samples are listed in Table I).

corresponding to those of pure LCP and PS, and no any inward shifts happened, which indicated a complete immiscibility between the two components.

On the contrary, the semi-ILCPNs containing different content of DVB showed an obvious inward shift of the T_g s, and the greater the amount of DVB was added, the more the T_g s of the component polymers shifted towards each other, obviously revealing that the compatibility between LCP and PS has been improved by the forming of semi-ILCPNs in which enforced compatibility has taken place due to forming a “snake-cage” network structure,²² where the linear LCP phases and crosslinked PS networks were interlocked with each other. This agrees well with FTIR analysis discussed earlier.

To determine the thermal stability of the samples, thermogravimetric analysis of semi-ILCPNs, and also of the blend and the PS homopolymers, was carried out under an N_2 atmosphere at a scanning rate of $20^\circ\text{C}/\text{min}$; the results are shown in Table I and Figure 3. It is clear that the initial thermal decomposition temperature of all the semi-ILCPNs were higher than those of corresponding to pure crosslinked PS and PS/LCP blends. It means that the heat resistance of PS could be enhanced by forming semi-ILCPNs with LCP. In addition, the experimental results also show that the initial thermal decomposition temperature increases with increasing the content of

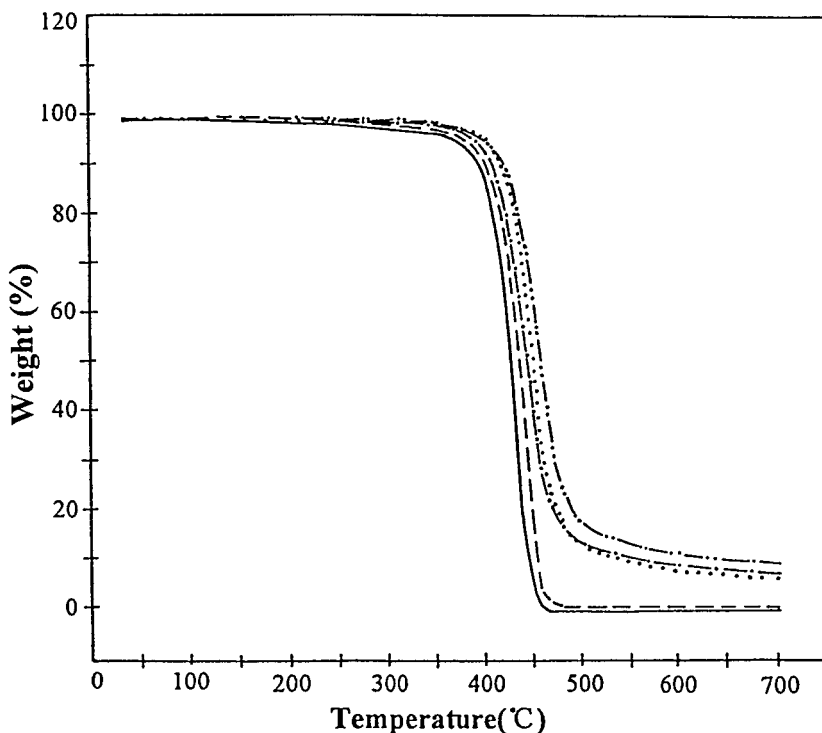


Figure 3 TGA thermograms of (—) blend 1, (---) blend 2, (- · -) semi-ILCPN 1, (· · ·) semi-ILCPN 2, (- · · -) semi-ILCPN 3 (the compositions of samples are listed in Table I).

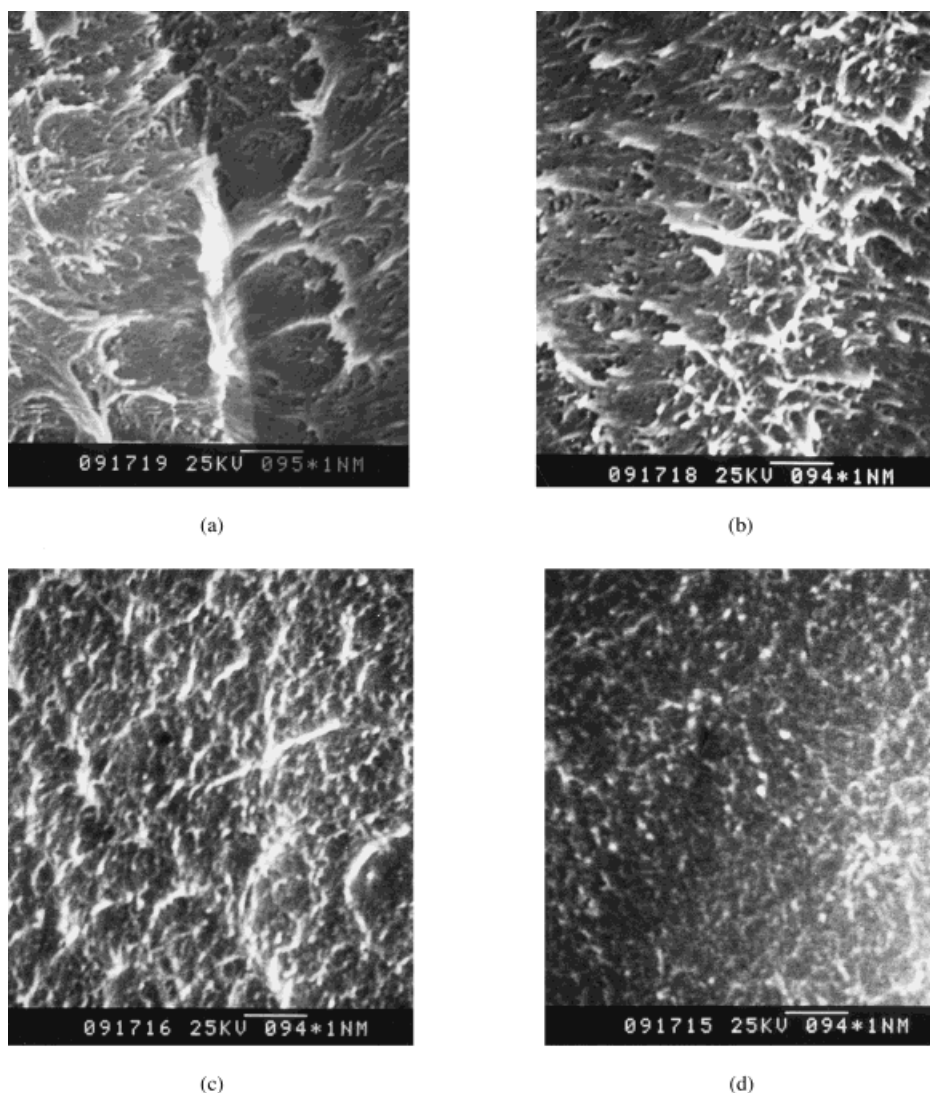


Figure 4 Scanning electron micrographs of fractured surfaces of (a) blend 2, (b) semi-ILCPN 1, (c) semi-ILCPN 2, (d) semi-ILCPN 3 (the compositions of samples are listed in Table I).

DVB; this is because the content of DVB directly affects the effective phase mixing,²³ semi-IPNs structure of PS with LCP, as well as the morphologies of the final products.

The morphologies of the fractured surfaces of the blend, as well as of semi-ILCPNs with different content of the crosslinking agent, were observed using SEM. The SEM micrographs in Figure 4 clearly show that the morphology of the blend [Fig. 4(a)] is typical for a phase-separated system characterized by rough dispersion of the LCP phase in the continuous PS matrix. The poor interfacial adhesion between LCP phase and PS matrix in the LCP/PS blend is also confirmed by

the nondeformed holes left after separation of the dispersed LCP particles from the continuous PS matrix on the fracture surface. In the case of semi-ILCPNs, however, the compatibility of PS with LCP was improved considerably, and the LCPs were well dispersed in the matrix. The micrographs of the semi-ILCPNs show a very smooth and homogeneous texture, and the diameters of dispersed particles are much smaller than those in the PS/LCP blends [Fig. 4(c) and (d)], which suggests that the semi-interpenetrating polymer networks have been formed between LCP and PS during the polymerization of St in the presence of LCP. This is in good agreement

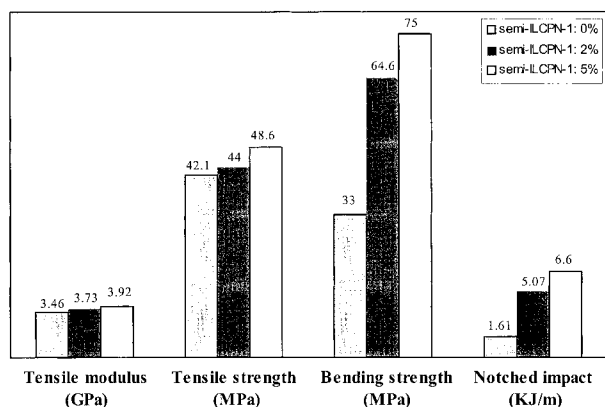


Figure 5 Mechanical properties of compatibilized PPO/LCP blends [PPO/LCP = 90/10 (w/w)].

with the results obtained by the DSC measurements.

Semi-ILCPN LCP/PS as Compatibilizer for PPO/LCP Blends

Mechanical Properties

It is understood that immiscible thermoplastic/LCP blends are characterized by a two-phase morphology and poor chemical/physical interactions across the phase boundaries. The poor interfacial adhesion between the LCP domains and the polymer matrix leads to the blends having very low mechanical properties. Vice versa, well-compatibilized thermoplastic/LCP blends usually result in lower interfacial tension in the melt, finer dispersed domains and considerably improved mechanical properties. Thus, the measurement of the mechanical properties of PPO/LCP blends can provide direct information about the compatibility of PPO with LCP.

The tensile properties, bending strength, and impact strength of the injection-molded blends based on PPO and LCP (90/10, w/w) are shown in Figure 5. It can be seen that the tensile properties of PPO/LCP blends are improved by addition of minor amounts of semi-ILCPNs. The tensile strength and tensile modulus of the uncompatibilized PPO/LCP blend are 42.1 MPa and 3.46 GPa, respectively, whereas those of compatibilized blends containing 5% semi-ILCPNs (with respect to PPO/LCP blend) are 48.6 MPa and 3.92 GPa, respectively. The enhancement in the tensile modulus of the compatibilized PPO/LCP blends indicates that the interfacial adhesion between the two phases has been improved. On the other hand, we also found that the tensile specimens of

compatibilized PPO/LCP blends showed ductile fracture behavior, while the uncompatibilized blends show fractured in brittle manner. This indicates that the miscibility and stress transfer between the two phases have been improved. It is also interesting to see that the bending strength of the compatibilized sample shows a large increase. Compared with the uncompatibilized PPO/LCP blend, the bending strength of the compatibilized sample with 5% semi-ILCPNs increase 2.3 times. This can be attributed to the improved compression resistance of the blend owing to the rigid semi-ILCPNs layer at the interface.

In addition to the enhancement of the tensile properties and bending strength, the Izod impact strength of the compatibilized blends was also improved dramatically. The Izod notched impact strength of uncompatibilized PPO/LCP blend is only 1.61 KJ/m, whereas for the compatibilized sample of the same composition but with 5% semi-ILCPNs, it is up to 6.60 KJ/m. As we know, the impact strength of the blend is ultimately dependent upon the dissipation capacity of the impact energy through the matrix and the delivery of the internal stress of the continuous phase to the dispersed phase. Therefore, this effect may be attributed to a combination of two factors. First, addition of the semi-ILCPNs can reduce the interfacial tension and improve interfacial adhesion between the two phases, which is favorable for the delivery of the internal stress.²⁴ Second, the semi-ILCPN has a semirigid network structure; thus, it can efficiently absorb and dissipate the impact energy of PPO/LCP blend system.

The material mechanical properties can be roughly divided into two parts: strength (including tensile strength and modulus, bending strength, and modulus), and toughness (impact strength). Modification of a polymeric material often results in improving one property but affecting the other. It is very difficult for polymer materials to have both properties, strength and toughness, improved simultaneously. By now, only a few successful cases have been given.^{15,25,26} In the current blend system, considerably improved mechanical properties in both strength and toughness of the compatibilized PPO/LCP blends by semi-ILCPNs are achieved simultaneously. The formation of longer and finer LCP fibrils with a higher aspect ration due to compatibilization improvement between LCP phase and PPO matrix is ultimately responsible for the observed overall mechanical properties improve-

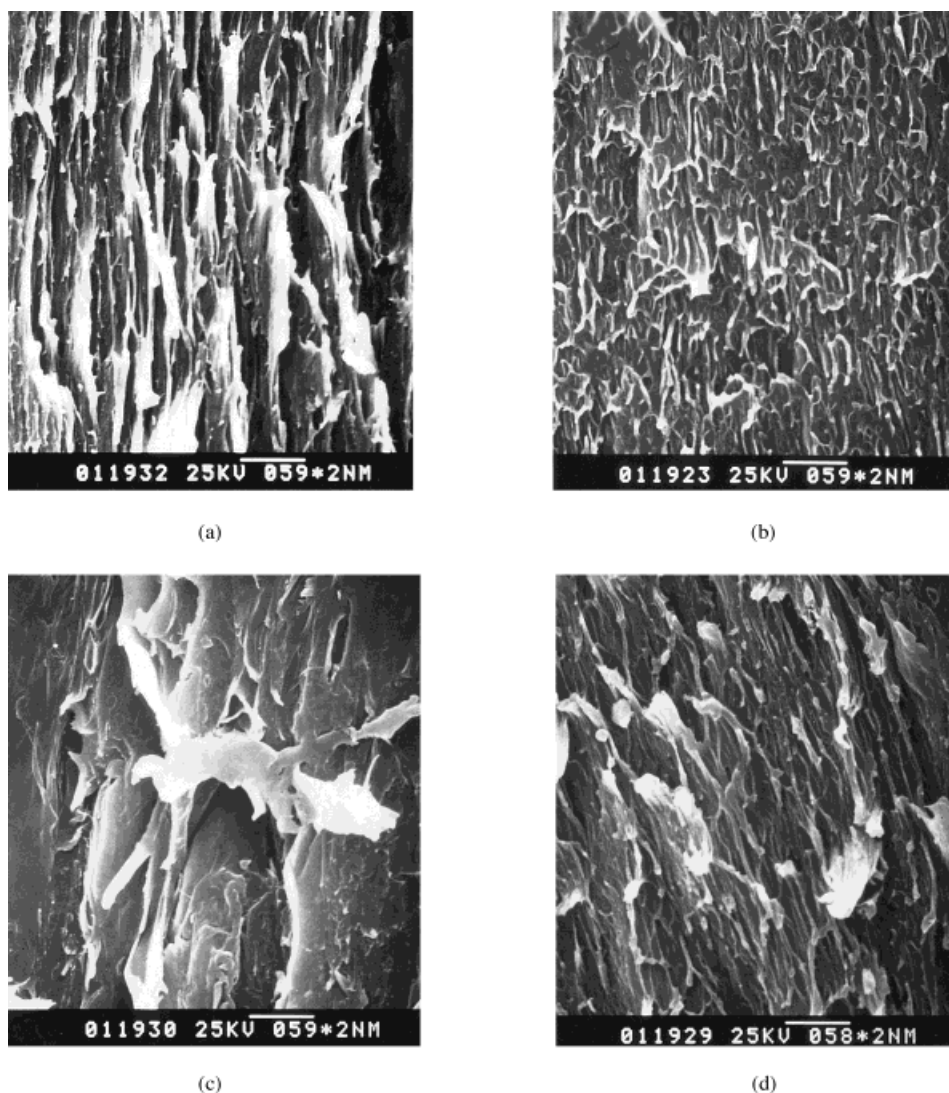


Figure 6 Scanning electron micrographs of compatibilized PPO/LCP blends near the skin regions: (a) 2% semi-ILCPNs, (b) 5% semi-ILCPNs and near the core regions: (c) 2% semi-ILCPNs, (d) 5% semi-ILCPNs.

ments, which could be further confirmed by SEM results for the fractured surface of the PPO/LCP blends containing different content of semi-ILCPNs.

Blend Morphology Observations

Investigations^{27,28} on the thermoplastic/LCP blends reveal that the interfacial adhesion and fibrillar morphology are greatly responsible for the reinforcement effects of thermoplastic/LCP blends and significantly affect the final mechanical properties, such as the modulus and strength. For fairly understanding the relationship between the morphologies of *in situ*-formed fibrils

and the mechanical properties of composites, SEM was used to examine the morphology of PPO/LCP blends. Injection-molded *in situ* composites of thermoplastic/LCP blends are known to reveal skin/core morphologies that were induced by the differences in the mold-filling flow kinematics and thermal histories experienced by the dispersed LCP phase between skin and core regions. Hence, separate morphological observations were carried out along both the skin and core regions to elucidate the influence of the semi-ILCPNs on the morphology of PPO/LCP blends.

Figure 6 shows the SEM micrographs along the skin and core regions, respectively, of the injec-

tion-molded PPO/LCP composite samples with different contents of semi-ILCPNs. It can be seen that well-developed and oriented fibrillation morphologies in the skin are formed in both sample 1 and sample 2 with 2 and 5% semi-ILCPNs, respectively [Fig. 6(a) (b)]. By comparing both samples, we can also find that the PPO/LCP blend with a higher semi-ILCPNs content exhibits fibrils with a higher aspect ratio and relatively more uniform length and diameter in the skin regions. Figure 6(c) and (d) show that a fibrous LCP phase was also formed in the core regions of injection-molded PPO/LCP blends. This can be attributed to the fact that the enhanced interfacial adhesion between the LCP phase and the PPO matrix can promote affine deformation of the LCP droplets during the injection-molded step via efficient transfer of forces between the two phase, which in turn, induce fibrillation of the LCP phase along the core regions in addition to fibrillation along the skin portions of the injection-molded specimens.²⁹ However, the fibrils formed in the core are much less uniform, and become thicker and shorter compared with those formed in the skin regions. It is, therefore, implied that the fibrils formed in the skin play a major role in the reinforcement of PPO/LCP blend because they have both a higher aspect ratio and a higher degree of orientation.³⁰

To develop an ideal fibril-reinforced composite based on thermoplastic and LCP, the formation of greater numbers and finer LCP fibrils and an increased interfacial adhesion are the two most important factors. The phase stabilization of PPO matrix with LCP phase can be improved by addition of small amount of semi-ILCPNs. However, most of the literature³¹⁻³³ reported compatibilized thermoplastic/LCP blends tend to decrease the number of fibrils or tend to convert LCP fibrils into droplet domains due to lower interfacial tension and finer dispersed phase domains, even though enhanced interfacial adhesion is obtained. In the present system, the compatibilized PPO/LCP blends without the loss of fibrous morphology of LCP were obtained.

Rheological Behavior

Figure 7 shows the effect of the semi-ILCPNs content on the apparent viscosity of PPO/LCP blends. It is noticed that the viscosity of PPO/LCP blends tends to increase with increasing semi-ILCPNs. This is a clear indication of the effective compatibilization of the PPO/PS blends using this semi-ILCPNs.^{26,27}

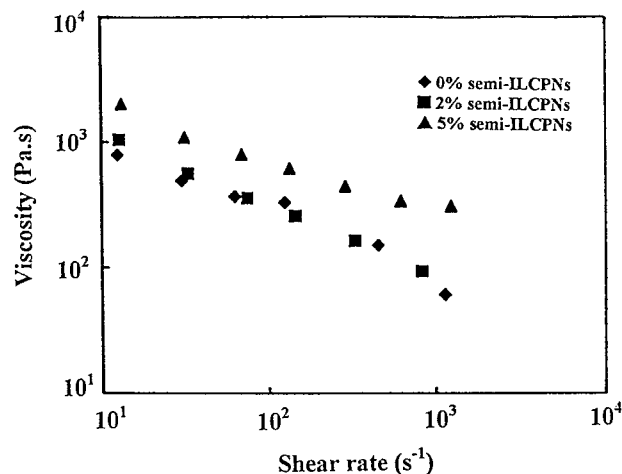


Figure 7 Plots of apparent viscosity vs. shear rate for (◆) 0% semi-ILCPNs, (■) 2% semi-ILCPN, and (▲) 5% semi-ILCPNs, PPO/LCP = 90/10 (w/w).

CONCLUSIONS

A new approach for enhancing the compatibility between LCP and thermoplastic matrix has been introduced, by which well-compatibilized PPO/LCP composites were obtained, which show considerably improved mechanical properties including both strength and toughness. In addition, addition of the semi-ILCPNs can enhance fibrillation of the LCP phase, which not only led to the formation of well-defined and oriented fibrils along the skin regions, but also induced fibrillation of the LCP phase in the core regions. More detailed studies relating to the effects of the weight ration of PPO to LCP and adding an amount of semi-ILCPNs on the thermal property of PPO/LCP blends are being undertaken in our laboratory.

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