

Compatibilization of blends containing thermotropic liquid crystalline polymers with sulfonate ionomers

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Zinc salts of lightly sulfonated polystyrene ionomers (Zn-SPSs) are shown to be effective compatibilizers for blends of a wholly aromatic liquid crystalline polyester (LCP) with nylon 66 (PA66) and polycarbonate (PC). Zn-SPS is miscible with PC, PA66 and the LCP, though the origins of miscibility are different for the three binary blends. Zn-SPS and PA66 mix because of strong intermolecular attractive interactions between the sulfonate and amide groups, while the ionomer is miscible with PC and the LCP as a result of intramolecular repulsive interactions along the ionomer molecules. The addition of Zn-SPS to LCP/PA66 and LCP/PC blends reduces the dispersed phase size and improves the adhesion between the phases. The compatibilized blends have significantly higher tensile modulus and tensile stress at break than the blends without ionomer.

(Keywords: liquid crystalline polyester; sulfonate ionomers; compatibilization)

INTRODUCTION

Blends of liquid crystalline polymers (LCPs) with other thermoplastic polymers have been a popular research topic for the past decade, and the literature on the subject is now quite extensive¹⁻³. Most of the commercially available LCPs have been combined with nearly every conceivable thermoplastic resin. The majority of the published work has been concerned mainly with the selfreinforcement of such blends, which are often called in situ composites, i.e. blends in which a reinforcing LCP microfibrillar phase is formed during the processing step. The presumed advantage of these materials is the attainment of properties comparable to those of a composite of short glass fibres but with improved processability and dispersion of the reinforcing phase compared with more conventional composite materials. In practice, however, the properties of LCP/polymer blends fall far short of expectations, largely as a result of poor interfacial adhesion between the dispersed microfibrillar LCP phase and the thermoplastic matrix phase.

Several approaches have been used to enhance the interfacial bonding between the two phases. Rueda and Shaw⁴ and Bassett and Yee⁵ modified the geometry of the LCP fibrils during processing to provide mechanical interlocking between the LCP fibrils and the matrix. O'Donnell *et al.*⁶ and Datta *et al.*⁷ achieved substantial improvements in the mechanical properties of blends of a

copolyester LCP and polypropylene (PP) by adding a small amount of maleic anhydride grafted polypropylene (PP-MA) as a compatibilizer. The maleic anhydride, or succinic acid hydrolysis product, most likely reacted either with the amine end-groups of the LCP to form an amide linkage between the two polymers or participated in transesterification reactions with the polyester. Either reaction would improve interfacial adhesion by forming a covalent bond between the LCP and the PP.

Other researchers have improved the adhesion between liquid crystalline and flexible, thermoplastic polyesters by promoting transesterification reactions⁸⁻¹⁰ between the polymers during melt processing. For example, Lee and DiBenedetto⁸ used a melt reaction to form covalent bonds between a wholly aromatic polyester LCP, polycarbonate (PC) and a third, compatibilizing polymer. In that case, the compatibilizer, or more precisely the coupling agent, was a second polyester LCP. Interfacial adhesion was improved as a result of transesterification reactions between the LCP coupling agent, the reinforcing LCP and PC.

The objective of the present research was to evaluate the use of lightly sulfonated polystyrene ionomers (SPSs) as compatibilizers for LCP/polymer blends. In this case, compatibilization is accomplished by physical interactions, rather than covalent bonds, as was presumably the case in the studies cited above. The use of reversible, physical interactions to effect compatibilization may be preferable to the use of covalent bond, grafting reactions with respect to the control of the properties, processing

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and recycling of either the blend or the individual components.

Ionomers, i.e. polymers containing a small amount of bonded salt groups, are attractive compatibilizing agents because of the specific interactions that may develop between the ionic groups and complementary polar functional groups on another polymer. Examples of intermolecular interactions that have been documented to occur in ionomer/polymer blends include hydrogen bonding, ion-dipole interactions, acid-base interactions, charge transfer interactions and transition metal complexation^{11,12}. The extent of interaction may be controlled by varying the ionic group concentration and/ or the choice of the anion and cation. Alternatively, when specific, intermolecular interactions do not take place, ionomers may still be viable compatibilizers because of strong intramolecular repulsive interactions within the ionomer^{13,14}.

Carboxylate ionomers such as metal salts of poly(rethylene-co-r-methacrylic acid) (PEMA) are commonly used as compatibilizers in polymer blends containing non-LCPs¹⁵. However, carboxylate ionomers, especially the commercially available ones, are rarely fully neutralized, so both free acid and salt groups are usually present. This introduces considerable ambiguity as to the relative importance of the acid and salt groups for providing compatibilization, and the dominant compatibilization mechanism is often ambiguous. For example, for polyamide-containing blends that are compatibilized by carboxylate ionomers, for which the literature is rich¹⁵, compatibilization is most likely effected through an amidation reaction involving carboxylic acid groups and amine end-groups of the polyamide, as well as through ion-dipole interactions involving the metal carboxylate groups. Although there is considerable circumstantial evidence in the literature and in patents that specific ion effects also influence compatibilization with ionomers, documentation of this is scarce. Specific ion-dipole interactions between ionomers and nonionomers are easier to evaluate with sulfonate ionomers because the stronger sulfonic acid is easier to neutralize completely than the relatively weak carboxylic acid.

Recently, several groups have shown that specific ion effects profoundly influence the miscibility of SPS ionomers with polar polymers such as polyamides and polyethers^{16–26}. Moreover, strong repulsive interactions along the SPS chains are responsible for the miscibility of SPSs with polycarbonates and polyesters, and the efficacy of this effect is also influenced by the cation used¹³. Our research on mixtures of polyester and SPS ionomers has suggested that SPS ionomers might also work as compatibilizers for blends of polyester or poly(ester amide) LCPs with polyamide or polyester thermoplastic polymers. This paper describes the effect of adding the zinc salts of SPS ionomers (Zn-SPSs) on the morphology and mechanical properties of blends of a wholly aromatic polyester LCP with polycarbonate (PC) and nylon 66 (PA66).

EXPERIMENTAL

Materials

Lightly sulfonated polystyrene ionomers (SPSs)

containing 5 and 9 mol% sulfonic acid groups were prepared by sulfonating polystyrene (PS) with acetyl sulfate in a dichloroethane solution following the procedure of Makowski *et al.*²⁷. The starting polystyrene had $M_n = 106\,000$ and $M_w = 280\,000$, determined by gel permeation chromatography. The sulfonation level was determined by titrating a 90% toluene/10% methanol solution of each SPS to a phenolphthalein end-point with methanolic NaOH. The zinc salts, 5Zn-SPS and 9Zn-SPS, were prepared by neutralizing solutions of the two SPS samples with a 20% molar excess of zinc acetate dihydrate. The ionomers were then precipitated in a large excess of ethanol, filtered, washed several times with ethanol and dried at 70°C for several days under vacuum.

The LCP used was Vectra A950, a commercial, wholly aromatic copolyester of 73% hydroxybenzoate (HBA) and 27% hydroxynaphthanoate (HNA) from Hoechst-Celanese. Vectra A950, hereafter referred to as simply LCP, had a T_g of 100°C and a crystalline solid to nematic liquid transition ($T_{\rm KN}$) of 280°C, as determined by differential scanning calorimetry. No transition from the nematic to an isotropic fluid was observed below the degradation temperature of the polymer.

Bisphenol-A polycarbonate (PC) with $M_w = 48\,000$ and $T_g = 150^{\circ}$ C was obtained from General Electric.



Figure 1 D.s.c. heating thermograms of (a) 20/80 LCP/PC and (b) 18/ 72/10 LCP/PC/5Zn-SPS blends. For each blend, the LCP/PC ratio was 1:4



Figure 2 D.s.c. heating thermograms of (a) LCP, (b) 5Zn-SPS and (c) a 75/25 LCP/5Zn-SPS blend



Figure 3 Plots of E' and log (tan δ) at f = 1 Hz versus temperature for (a) LCP, (b) SZn-SPS and (c) a 75/25 LCP/5Zn-SPS blend

Nylon 66 (PA66) with $T_g = 63^{\circ}$ C and $T_m = 250^{\circ}$ C was obtained from Du Pont(Zytel 101NC10).

The LCP, PC and PA66 were dried at 100°C under vacuum for at least 24 h before melt blending. Binary and ternary blends were prepared by melt mixing under an inert nitrogen atmosphere in a Brabender mixer at 300° C using a 30 cm^3 mixing head with two counter-rotatory screws and a screw speed of 30 rev min^{-1} . Ternary blends were prepared by first fluxing the LCP and either the PC or PA66 together for several minutes and then adding the Zn-SPS. The ternary mixture was then mixed for 7 min. All blend compositions are expressed as weight percentages, e.g. 20/80 LCP/PC is a blend of 20 wt% LCP and 80 wt% PC.

Materials characterization

Thermal transitions were measured by differential scanning calorimetry (d.s.c.) using a Perkin–Elmer model DSC-7. The heating rate was 20°C min⁻¹. The T_g was defined as the midpoint of the change in specific heat, and T_m and T_{KN} were taken as the maxima in the respective transition endotherms. Dynamic mechanical behaviour was characterized with a Polymer Laboratories model MkII dynamic mechanical thermal analyser using a tensile fixture and a heating rate of 2°C min⁻¹. The transition temperatures were defined by the maxima in tan δ at a frequency of 1 Hz.

Tensile stress-strain measurements were made with an Instron model 1011 using either extruded fibres or 0.8-1.0 mm thick compression-moulded films and a cross-head speed of $5.08 \text{ mm} \text{ min}^{-1}$. Stress was calculated from the initial cross-sectional area of the sample, and the strain was defined as $\epsilon = (l - l_0)/l_0$, where l_0 is the initial gauge length (25.4 mm) and $l - l_0$ is the cross-head travel.

The blend morphology was characterized with an AMR model 1200 scanning electron microscope. Compression-moulded films and extruded fibre samples were freeze fractured in liquid nitrogen, and the fracture surfaces were imaged with the scanning electron microscope. Fracture surfaces from the tensile experiments were also sometimes used.

RESULTS AND DISCUSSION

LCP/PC blends

Figure 1 shows the d.s.c. thermograms between 30 and 160°C for 20/80 LCP/PC and 18/72/10 LCP/PC/5Zn-SPS blends. For each blend, the LCP/PC ratio was 1:4. For the LCP/PC blend, a T_g is seen at 150°C, which is identical to that of neat PC (ca. 150°C), and a weak T_g corresponding to a pure LCP phase is also visible at ca. 100°C. A previous study also showed that the two polymers were completely immiscible²⁸, and there was no indication that transesterification occurred between the two polymers. When the 5Zn-SPS $(T_g = 112^{\circ}C)$ was added, the T_g of the PC phase decreased by ca. 7°C and a T_g at ca. 118°C was also observed. One explanation of the latter T_g is that it represents an ionomer-rich phase. It is, however, about 6°C higher than that of the neat ionomer, which means that the 5Zn-SPS phase also contained some PC. The compositions of the two phases, assuming that neither phase contained any LCP, estimated from the Fox equation²⁹ were 83% PC/17% ionomer and 31% PC/69% ionomer. A T_g of a pure LCP phase was not resolved at 100°C, which might be a result of the crystallinity of the LCP and a relatively small volume fraction of amorphous LCP in the blend. An alternative explanation for the failure to resolve the T_{g} of the LCP in Figure 1 is that mixing of the LCP and the ionomer may have occurred, and the possibility of miscibility of the LCP and 5Zn-SPS is considered in the following paragraph.

The compatibility of PC and the ionomer is demonstrated by the partial miscibility indicated by the shift in the T_g of the PC-rich phase in Figure 1 when the ionomer is added to the blend. This result is consistent with a separate study of binary blends of PC and SPS ionomers which concluded that mixing occurred owing to strong intramolecular repulsive interactions within the ionomer^{13,14}. Because the T_g of the LCP was not resolved in Figure 1, it is not possible from those data to discern any information on the compatibility of the LCP with the other two polymers. To ascertain whether the ionomer interacts with the LCP, a 75/25 LCP/Zn-SPS binary blend was prepared, and the d.s.c. thermograms of that blend and the neat components are shown in Figure 2. In this case, resolution of the T_g of the LCP should be facilitated by the higher concentration of LCP. The LCP/ ionomer blend, however, exhibits only a single T_g at ca. 104°C, which agrees well with the Fox equation²⁹

System	Compatibilizer	Compatibilizer content (wt%)	Modulus (GPa)	Stress at break (MPa)	Strain to break (%)
PC		0	1.58	40	76
LCP/PC		0	$1.70 (0.16)^{b}$	49.0 (3.3)	10.0 (3.1)
LCP/PC	PS	10	1.70 (0.14)	48.0 (4.2)	7.1 (1.6)
LCP/PC	5Zn-SPS	10	1.90 (0.15)	58.5 (7.0)	4.8 (1.2)
PA66		0	1.05	62	220
LCP/PA66		0	1.20 (0.22)	81.0 (6.1)	92 (24)
LCP/PA66	9Zn-SPS	5	1.31 (0.21)	107.0 (13.0)	110 (42)

 Table 1
 Tensile properties of LCP/polymer blends^a

^a In all blends, the LCP/matrix polymer ratio was 1:4

^b The values in parentheses are the standard deviations of 5 measurements



Figure 4 Representative tensile stress-strain curves for compressionmoulded films of (a) 20/80 LCP/PC, (b) 18/72/10 LCP/PC/PS and (c) 18/72/10 LCP/PC/5Zn-SPS blends. The LCP/PC ratio in each blend was 1:4. Stress and strain are defined in the text

prediction of 103° C for a miscible blend. This is a truly remarkable result in that it is unusual to observe miscibility between a liquid crystalline polymer and a flexible coil polymer like Zn-SPS. One might suspect that miscibility could arise if strong associative interactions occurred between the LCP and the ionomer, but Fourier transform infra-red spectroscopic analysis of the carbonyl vibrations failed to detect a significant interaction between the two polymers. An alternative mechanism for miscibility is a repulsive interaction between the styrene and sulfonated styrene units on the ionomer, which favours mixing with the LCP in the same way that it does with PC^{13,14}.

Miscibility of the LCP and the Zn ionomer was confirmed by dynamic mechanical thermal analysis (d.m.t.a.) of the 75/25 LCP/5Zn-SPS blend (*Figure 3*). A single tan δ peak was observed for each of the component polymers and the blend, corresponding to T_g values of 105, 132 and 116°C, respectively. The blend T_g measured by d.m.t.a. was about 5°C higher than that predicted by the Fox equation. The blend tan δ peak in *Figure 3* is skewed on the high temperature side, which suggests that the peak may actually be a composite of two peaks, one for an LCP-rich phase and one for an ionomer-rich phase. No attempt was made to resolve the two peaks further, but it is clear from *Figures 2* and *3* that the two polymers are at least partially miscible.

Figure 4 compares the tensile stress-strain behaviour of a compression-moulded 20/80 LCP/PC blend with that of compression-moulded ternary blends containing 10% PS and 10% 5Zn-SPS. In all three blends, the LCP/ PC ratio was 1:4. The tensile properties are summarized in Table 1; the properties of neat, compression-moulded PC are included for comparison. The addition of LCP to PC increases the tensile modulus and strength and decreases the strain to break. The LCP/PC blend exhibits a yield point prior to failure, while neither ternary blend exhibits macroscopic yielding. PS is immiscible with both the LCP and PC, and the effect of its addition is to lower the ultimate strain and embrittle the blend. The addition of Zn-SPS increases the tensile stress at break by ca. 20%, though the strain to break decreases by 50%. The drop in elongation may be due to embrittlement of the PC matrix by the incorporation of some ionomer into the PC phase, or it may be a consequence of a change in the failure mechanism resulting from improved interphase adhesion between a PC-rich phase and an LCP-rich phase. Both phases may contain Zn-SPS and it is also conceivable that ionomer molecules may bridge the two phases. Although the compositions of the phases in the ternary blends were not determined, the tensile data strongly suggest that the sulfonate ionomer is an effective compatibilizer for this particular blend.

Further evidence for compatibilization of the LCP/PC blend by 5Zn-SPS is provided by scanning electron microscopy (SEM) photomicrographs of the fracture surfaces obtained from the tensile test specimens (*Figure* 5). For the LCP/PC/PS blend (*Figure 5a*), the fracture surface is relatively undeformed, and the spherical holes from where the dispersed LCP phase detached from the matrix indicate poor adhesion between the phases. In contrast, the tensile fracture surface of the LCP/PC/Zn-SPS blend exhibits considerable local drawing and no clear detachment of the phases (*Figure 5b*). Improved interfacial adhesion is consistent with the increased stress required to deform the specimen compared with either the binary LCP/PC blend or the ternary blend containing PS.

The improved adhesion between the LCP and PC that results from the addition of the ionomer was also demonstrated by drawing films above the T_g of the



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Fracture surfaces of (a) 18/72/10 LCP/PC/PS and (b) 18/72/ Figure 6 10 LCP/PC/5Zn-SPS compression-moulded blends after drawing to failure at 0.51 mm min⁻¹ and 110°C

Figure 5 Tensile fracture surfaces of (a) 18/72/10 LCP/PC/PS and (b) 18/72/10 LCP/PC/5Zn-SPS blends. The photomicrographs show the fracture surfaces of the tensile specimens after failure in the experiments analysed in Figure 4

LCP but below that of the PC matrix. Lee and DiBenedetto⁸ recently showed that this is an effective method for achieving a fibrillar LCP dispersed phase in a compatibilized blend. Figure 6 shows SEM micrographs of LCP/PC/PS and LCP/PC/5Zn-SPS blends that were drawn at an extension rate of 0.51 mm min⁻¹ at 110°C until failure occurred. In each case, the LCP/PC ratio was 1:4 and the blend contained 10% of the styrenic polymer based on the total sample mass. Failure occurred at strains of less than 10%. The SEM photomicrograph of the LCP/PC/PS blend (Figure 6a) shows no localized deformation of either the matrix or the dispersed phase. Remnants of a spherical dispersed phase with poor adhesion to the matrix are evident by the undeformed holes present in the specimen. In contrast, the photomicrograph of the blend containing the ionomer exhibits evidence of considerable localized yielding of the matrix and fibrillation of a well-adhered LCP phase (Figure 6b).

LCP/PA66 blends

Previous studies^{30,31} showed that PA66 and HBA/ HNA LCPs are incompatible. Previous work in our laboratory^{18,21} demonstrated that strong, exothermic, intermolecular interactions occur in blends of Zn-SPSs

and aliphatic polyamides. That result and the finding of miscibility, or at least partial miscibility, for 5Zn-SPS and the HBA/HNA LCP used in this study suggest that Zn-SPS ionomers may also be suitable compatibilizers for LCP/PA66 blends. For the LCP/PA66 blends, a more highly functionalized SPS, 9Zn-SPS, was used.

Figure 7 shows SEM photomicrographs of freezefractured surfaces of compression-moulded samples of 20/80 LCP/PA66 and 19/76/5 LCP/PA66/9Zn-SPS. For each blend, the LCP/PA66 ratio was 1:4. The compatibilizing effect of the ionomer is demonstrated by the decrease in the dispersed phase size and better dispersion of the LCP phase when the Zn-SPS is added to the blend.

Typical tensile stress-strain curves of melt-drawn fibres (draw ratio of 25) of the LCP/PA66 and LCP/ PA66/9Zn-SPS blends are shown in Figure 8, and the tensile properties are summarized in *Table 1*. The tensile properties of the neat, compression-moulded PA66 are also included in Table 1 for comparison. As with the PC, the addition of the LCP to PA66 increases the modulus and stress at break, but decreases the strain to break. The addition of ca. 5 wt% 9Zn-SPS increases the stress at break by 35% and may have slightly increased the modulus and the strain to break. The yield stress was unaffected by the addition of the ionomer, but the yield strain decreased by 35%. The increased modulus is probably a consequence of better fibrillation of the LCP phase and better alignment of the LCP molecules within



Figure 7 Freeze-fractured surfaces of compression-moulded blends of (a) 20/80 LCP/PA66 and (b) 19/76/5 LCP/PA66/9Zn-SPS. In each blend, the LCP/PA66 ratio was 1:4



Figure 9 Freeze-fractured surfaces of extruded fibres (draw ratio of 25) of (a) 20/80 LCP/PA66 and (b) 19/76/5 LCP/PA66/9Zn-SPS blends. In each blend, the LCP/PA66 ratio was 1:4

the phase during processing of the blend fibres. Both of these are expected to result from improved stress transfer from the continuous polyamide phase to the dispersed LCP phase in the melt if better interphase adhesion occurs between the phases. Similarly, the lower yield strain and higher ultimate stress are the result of improved interphase adhesion that resulted from the addition of the ionomer.

SEM photomicrographs of fracture surfaces of the extruded blend fibres are shown in *Figure 9*. The photomicrograph of the LCP/PA66 blend (*Figure 9a*) exhibits little evidence of deformation of the spherical dispersed LCP phase, which is probably a result of the higher viscosity of the LCP phase, as has been reported by other investigators^{30,31} for the same system. However, the photomicrograph of the blend containing the ionomer (*Figure 9b*) clearly shows evidence of distortion or fibrillation of the dispersed phase, which is a direct consequence of improved interphase adhesion.

CONCLUSIONS

Zn-SPS ionomers have been shown to be effective



Figure 8 Representative tensile stress-strain curves for extruded fibres (draw ratio of 25) of (a) 20/80 LCP/PA66 and (b) 19/76/5 LCP/PA66/ 9Zn-SPS blends. The LCP/PA66 ratio in each blend was 1:4. Stress and strain are defined in the text

compatibilizers for blends of an HBA/HNA LCP with either PC or PA66. The ionomer is fully or partially miscible with all three polymers, though the origins of miscibility with the polyesters and with the polyamide are different. Zn-SPS complexes strongly with aliphatic polyamides, primarily by either a transition metal complex with the amide nitrogen or by an ion-dipole interaction with the amide oxygen^{18,21}. No associative interactions between the ionomer and the PC or the LCP were found, and in those cases mixing of the polymers was thought to occur by a copolymer effect^{32,33} that involves intramolecular repulsion within the ionomer.

For the compatibilized LCP/PC/ionomer blends, no specific interaction occurs between any of the component polymers. However, the binary pairs PC/Zn-SPS and LCP/Zn-SPS are miscible or nearly miscible. Compatibilization of the LCP/PC blend by the ionomer is thought to result from sufficient partitioning of the ionomer into each phase so that the interphase adhesion is substantially improved.

Compatibilization of the LCP/PA66 blend by Zn-SPS is somewhat surprising in that the ionomer complexes strongly with the polyamide. One might expect a priori that all of the ionomer would partition into the polyamide phase, which would provide no improvement in the interphase adhesion with the LCP. However, that was clearly not the case for the system studied and Zn-SPS proved to be an effective compatibilizer even though its interactions with the two other polymers were quite different.

A third case, that where the ionomer may complex with both polymers, was not discussed above. Preliminary studies, however, of blends of PA66 and a poly(ester amide) LCP (Vectra X from Hoechst-Celanese) indicate that Zn-SPS ionomers are also effective at compatibilizing that system.

In addition to the demonstration of SPS ionomers as effective compatibilizers for LCP blends, a significant, though unexpected, result was the miscibility of the ionomer and the LCP. Although rigid and flexible polymer chains are normally immiscible, it is expected that miscibility may be accomplished by promoting strong associative interactions between the two polymers. To our knowledge, however, the current study represents the first report of miscibility occurring between a relatively rod-like polymer and a flexible chain polymer as a result of intramolecular repulsive interactions.

The unique aspect of using SPS ionomers as compatibilizing agents in LCP-containing thermoplastic blends is that compatibilization is accomplished solely through physical effects with no formation of intermolecular covalent bonds. This fact distinguishes it from most other approaches for compatibilization, which generally involve reactive processing. Although reactive processing can be very effective, the formation of intermolecular covalent bonds is not reversible and separation of the constituent polymers is not possible after reaction. The physical approach has advantages with regard to the control of properties and possible recycling of the blend and/or its components.

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