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Blends of side-chain liquid crystalline polymers: towards self-assembled interpenetrating networks

Yue Zhao*, Guoxiong Yuan, Philippe Roche

Département de chimie, Université de Sherbrooke, Sherbrooke, Québec, Canada J1K 2R1

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Abstract

The possibility of using various types of non-covalent cross-linking, to promote the structure of interpenetrating networks in blends based on side-chain liquid crystalline polymers, was investigated. The polymers used included: (1) random copolymers of liquid crystalline polyacrylates containing benzoic acid units for hydrogen-bond cross-linking through the formation of intermolecular dimers; (2) liquid crystalline polyacrylate-ionomers bearing sodium salt groups for cross-linking through ionic aggregates; and (3) a styrene–butadiene–styrene triblock copolymer having a network structure with polystyrene microdomains as cross-links. The preparation method consists of making a dilute solution of two polymers, then evaporating the solvent and drying the mixture. No evidence of network interpenetrating was observed. The results suggest, however, that blends composed of two components having a network structure, for both H-bond/ionic-aggregate and H-bond/triblock-copolymer combinations, have a greater miscibility, compared with blends containing only one component capable of forming a network structure through non-covalent cross-linking. The improved miscibility is characterized by reduced domain sizes and, for blends of two liquid crystalline polymers, changes in the phase transition behavior. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Interpenetrating networks; Side-chain liquid crystalline polymers; Liquid crystal polyacrylate-ionomers

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1. Introduction

Increasingly, effort is being made to explore new liquid crystalline materials based on side-chain liquid crystalline polymers (SCLCP). These include block and graft copolymers [1,2] and elastomers [3,4]. Relatively little attention has been paid to blends of SCLCP. Generally, these blends are immiscible [5], and the phase separation results in grossly heterogeneous mixtures with no useful properties. Interpenetrating two cross-linked polymers is known as an effective means to restrict the phase separation process by cross-links, which can often result in microphases, or even the miscibility with one single phase for the otherwise immiscible components [6]. In a previous study, we have investigated the possibility of extending the concept of interpenetrating networks to SCLCP, and prepared some polyacrylate-based sequential LC interpenetrating polymer networks (LC-IPN) [7]. The mesophase transitions in these LC-IPN samples are modified as compared with individual cross-linked SCLCP, indicating an increased miscibility between the components. LC-IPN has the potential to bring together intimately different SCLCP and, in principle, to offer structural possibilities with varying degrees of miscibility and interfaces. This means that distinct properties or different features of two SCLCP, could be exhibited by a single system.

The purpose of the present study was to investigate the use of non-covalent cross-linking to build LC-IPN. Such self-assembled LC-IPN may have the advantage over covalent LC-IPN to be thermoplastic. As a matter of fact, the preparation of thermoplastic IPN was demonstrated, involving thermoplastic elastomers blended with semi-crystalline polyamides [8] and ionomers [9]. Three kinds of non-covalent cross-links were used in the present work on SCLCP. As schematically depicted in Fig. 1, these are intermolecular hydrogen-bonds, ionic aggregates, and microdomains in triblock copolymers. The idea is to blend two modified SCLCP which contain a number of functional groups capable of forming non-covalent cross-links and, thus, promoting network structures. The basic preparation method consists in solution-mixing two SCLCP, each of which is ready to convert into its own network form upon solvent evaporation. It should also be mentioned that the preparation of single, self-assembled LC network, through the use of H-bonding, was reported [10,11]. Those studies focused on promoting or inducing the liquid crystallinity in polymers by an exclusive action of H-bonds, which either attach mesogenic molecules to a chain backbone or build up the pendent mesogenic groups.

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* Corresponding author. Tel: +1-819-821-7090; Fax: +1-819-821-8017.

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Table 1
Characteristics of the individual polymers

Polymer	Acid (mol.%)	T_g (°C)	T_{sn} (°C)	T_{ni} (°C)	M_w (g mol^{-1})	M_w/M_n
P6CN	0	31		130	27 000	1.7
P6CN-0.07COOH	7	26		115	6400	1.2
P6CN-0.26COOH	26	29	126	131	6100	1.2
P6OCH$_3$	0	25	94	119	16 500	1.3
P6OCH$_3$-0.08COOH	8	16	85	92	12 800	1.8
P6OCH$_3$-0.15COOH	15	25	76	105	7700	1.3
P6OCH$_3$-0.24COOH	24	28	72	106	6500	1.2
BiP5CN	0	39	113	117	79 000	1.8
BiP5CN-0.025Na	2.5	38	113	117		
BiP5CN-0.05Na	5	38	93	97		

The modified SCLCP used in this study include: (1) LC random copolymers containing benzoic acid units for H-bond cross-linking through the formation of acid dimers; (2) LC ionomers bearing COO$^-$Na$^+$ groups for cross-linking through ionic aggregates; and (3) a styrene–butadiene–styrene triblock copolymer (SBS) having a network structure with polystyrene microdomains as cross-links. Since no LC triblock copolymers were available for this work, SBS was utilized in order to assess the feasibility of the approach. SCLCP carrying no functional units for non-covalent cross-linking, as well as a styrene–butadiene diblock copolymer (SB), were also used for comparison. It will be shown that, although no evidence of network interpenetrating can be claimed, a combination of two network-forming polymers may result in an improved miscibility, as compared with any combination with only one polymer capable of forming non-covalent cross-links in the blend.

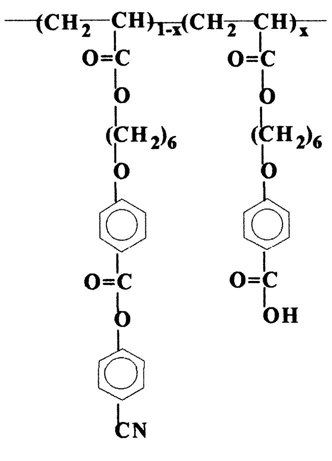
2. Experimental

2.1. Samples

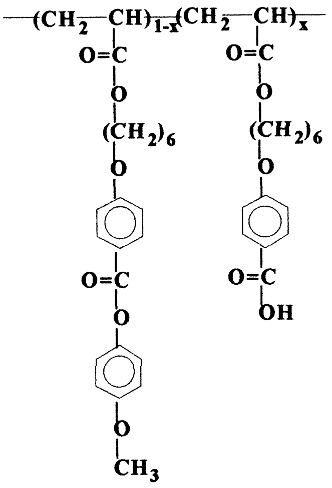
The chemical structures of the used SCLCP are shown below

P6CN-*x*COOH and P6OCH$_3$-*x*COOH are two series of polyacrylate-based random copolymers; the *x* in the acronyms of the samples designates the mole fraction of the benzoic acid units. Various copolymers were prepared by free radical copolymerizations at 60°C–70°C using 1% of AIBN as the initiator; the content of the benzoic acid comonomer units was determined by ^{1}H NMR and titration. All monomers, including 4-(6-(acryloyloxy)hexyloxy)benzoic acid, were synthesized through well-documented methods [12]. BiP5CN-*x*Na refers to LC polyacrylate-ionomers bearing the sodium salt of acrylic acid, *x* being the mole fraction of the ionic groups. BiP5CN-*x*Na was obtained through partial hydrolysis of the SCLCP by sodium hydroxide [13].

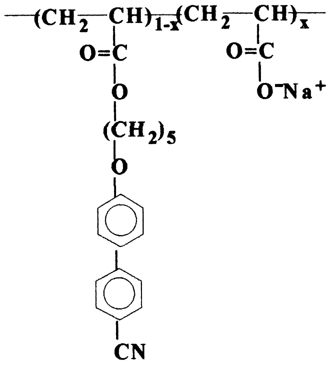
Summarized in Table 1 are the characteristics of all the individual SCLCP, including the phase transition temperatures of smectic-to-nematic T_{sn}, nematic-to-isotropic T_{ni}, and the glass transition temperature T_g. The incorporation of the 4-(6-(acryloyloxy)hexyloxy)benzoic acid unit in P6CN and P6OCH$_3$ does not have a simple dilution effect which lowers the transition temperatures. For instance, P6CN-0.07COOH has a lower T_{ni} than P6CN, but in the case of P6CN-0.26COOH, its T_{ni} raises and there is a smectic phase which is absent for the nematic-only P6CN. This observation can be explained by the fact that H-bonded dimers of the benzoic acid units can be mesogenic [14].



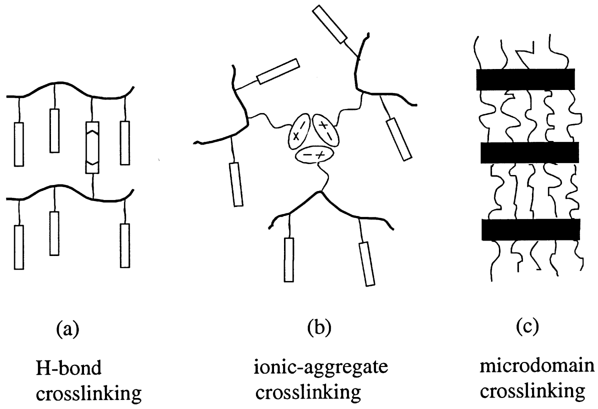
P6CN-xCOOH



P6OCH$_3$-xCOOH



BiP5CN-xNa



(a) H-bond crosslinking

(b) ionic-aggregate crosslinking

(c) microdomain crosslinking

Fig. 1. Schematic representation of the three types of non-covalent crosslinking used to build interpenetrating networks.

Samples of SBS triblock and styrene–butadiene (SB) diblock copolymers,[1] both containing approximately 30% of styrene, were purchased from Aldrich. The average M_w of SBS is approximately 140 000, and that of SB is approximately 70 000.

2.2. Preparation of LC-IPN

Two series of blends of non-covalent network combinations were investigated for the preparation of LC-IPNs. The first was the H-bond/ionic-aggregate system which was made up from two SCLCP having, respectively, H-bonding and ionic aggregates. For this system, samples of P6CN-*x*COOH and BiP5CN-*x*Na were utilized. The second system was prepared from P6OCH$_3$-*x*COOH and SBS, combining the network of the H-bonded SCLCP and the network arising from the triblock copolymer. All blends have a weight ratio of 50/50, corresponding approximately to a mole ratio of 50/50 for P6CN-*x*COOH/BiP5CN-*x*Na, and 20/80 for P6OCH$_3$-*x*COOH/SBS.

For the preparation of the H-bond/ionic-aggregate system, the two samples, one P6CN-*x*COOH and one BiP5CN-*x*Na, were dissolved in THF with a total polymer concentration less than 2%; the solution was clear, indicating no phase separation. Thin films were obtained by solution-casting. The rate of the solvent evaporation was controlled to be the same for all samples: a slow evaporation of 2 h at ambient, followed by drying in vacuum at 60°C for 2 days. Fast solvent evaporation leads to films whose surfaces are not smooth enough for polarizing microscopy observations. In the case of the P6OCH$_3$-*x*COOH/SBS systems, the preparation procedure was the same, but pyridine was used as the solvent to make a clear solution of the two components.

2.3. Characterization

Phase transition temperatures were measured using a Perkin–Elmer DSC-7 (heating rate: 10°C min^{-1}). A Leitz DMR-P polarizing microscope equipped with an Instec hot stage was used to observe the liquid crystalline textures and the morphology of the samples, and to take the photomicrographs under crossed polarizers. All samples, both mixtures and individual polymers, were treated in their isotropic state for 10 min for equilibrium before characterization. Transmission electronic microscopy observations were conducted on a JEOL JEM 100S at 60 kV. Infrared spectra were recorded on a Bomem MB-102 FTIR spectrometer at a resolution of 4 cm^{-1}, and the molecular weights were measured on a Waters-GPC system using polystyrene standards.

3. Results and discussion

3.1. Systems involving hydrogen bonding and ionic aggregates

As the solvent evaporation progresses from an initially clear solution of a P6CN-*x*COOH with a BiP5CN-*x*Na, phase separation is going to take place. However, if the two SCLCP are able to form their network structures before or even during the phase separation process, the non-covalent cross-links, i.e. H-bonds for P6CN-*x*COOH and ionic aggregates for BiP5CN-*x*Na, may restrict the phase separation as the covalent cross-links do. This is conceivable, since interpenetrated network structures can emerge in the solution becoming more and more concentrate during the solvent evaporation. Indeed, in solvents like THF, the intermolecular H-bonded dimers of the COOH groups can be formed and, on the other hand, ionic aggregates (or clusters) responsible for inter-chain associations, were observed even in dilute ionomer solutions [15]. Should this happen, the resulting LC-IPN must prevent gross phase separation from occurring, and exhibit some changes in the mesophase transition behavior as a result of more interactions between the mesogens of the two SCLCP. These aspects can be revealed by comparing LC-IPN with corresponding blends which have only one network-forming component and, consequently, have no possibility for interpenetrating of two networks. The non-modified SCLCP, P6CN, P6OCH$_3$ and BiP5CN, can serve this purpose.

Shown in Fig. 2 are differential scanning calorimetry (DSC) curves which allow for such a comparison. First, the curve of the P6CN-0.26COOH/BiP5CN blend, Fig. 2(b), looks like mainly the addition of the curves of the two individual polymers Fig. 2(a) and (c), showing all the transition peaks. The slight changes in peak position and intensity are comparable with those observed for phase-separated blends containing a SCLCP and a non-liquid crystalline polymer [16]. Also, despite the small difference in the T_gs of P6CN-0.26COOH and BiP5CN, two T_gs can be noticed for their blend. These results indicating gross phase separation are expected, since only P6CN-0.26COOH can form a network structure through H-bonding, while BiP5CN, bearing no ionic groups, cannot. By contrast, the

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[1] The SB sample actually contains 80% of diblock copolymer.

curve of P6CN-0.26COOH/BiP5CN-0.05Na, Fig. 2(d), displays greater changes as compared with the two individual polymers Fig. 2(c) and (e). The mesophase transition temperatures of P6CN-0.26COOH are lowered by approximately 10°C, and its smectic-to-nematic and nematic-to-isotropic transition peaks can no longer be distinguished. These results suggest an improved miscibility in the expected LC-IPN sample.

The polarizing microscopy observations support the above analysis. Fig. 3 shows the photomicrographs of P6CN-0.26COOH/BiP5CN and P6CN-0.26COOH/BiP5CN-0.05Na taken at two temperatures for each system. In the case of P6CN-0.26COOH/BiP5CN, the gross phase separation is evident from the photomicrograph at 120°C. At this temperature, BiP5CN appears as the continuous dark region, as it is still in the isotropic state, while the dispersed birefringent regions come from P6CN-0.26COOH in its smectic phase (see Fig. 2); the texture within the P6CN-0.26COOH phase is the same as the individual polymer. The sizes of the dispersed domains reach to over 130 μm. When the blend is cooled to 110°C, BiP5CN also enters into the liquid crystalline phase, and the whole sample becomes birefringent, showing a simple mixture of the textures arising from the two components. As for the P6CN-0.26COOH/BiP5CN-0.05Na system, the result is totally different. At 115°C, BiP5CN-0.05Na is isotropic and P6CN-0.26COOH is liquid crystalline, but no grossly separated phases can be observed. The liquid crystalline P6CN-0.26COOH component is only reflected by bright spots uniformly distributed throughout the sample, and no liquid crystalline texture like that of the individual P6CN-0.26COOH can be noticed. This is a strong indication of very small phases or some kind of microphases of P6CN-0.26COOH in this system. Cooling the sample to 90°C, BiP5CN-0.05Na also becomes liquid crystalline; the resulting apparent texture is different from that of the P6CN-0.26COOH/BiP5CN blend.

Similar results were obtained when comparing P6CN/BiP5CN-0.05Na with P6CN-0.07COOH/BiP5CN-0.05Na. Here, the reference system having no possibility for network interpenetrating is different. The component able to form a network structure in the P6CN/BiP5CN-0.05Na blend is BiP5CN-0.05Na, which has ionic aggregates as non-covalent cross-links instead of H-bonds, as in the P6CN-0.26COOH/BiP5CN blend discussed earlier. Only the DSC results are shown in Fig. 4. Again, the changes are slight for the P6CN/BiP5CN-0.05Na blend, showing a decrease in the sharpness of the transition peaks and a slight decrease of the transition temperatures for BiP5CN-0.05Na. Two T_gs can still be noticed for the blend. By contrast, for the P6CN-0.07COOH/BiP5CN-0.05Na system, the results suggest a great extent of miscibility induced between the two components. It is seen that the mesophase transition peak of P6CN-0.07COOH is superimposed with the peaks of BiP5CN-0.05Na, and that a single T_g appears at 29°C, which is intermediate between the T_gs of the two individual polymers (Table 1).

Using the solvent evaporation method, the above results clearly show the consequences of a simultaneous formation of two non-covalent networks in the blends, that is, an improved blend miscibility with reduced domain sizes. This improved miscibility appears only when the two SCLCP components can form network structures through H-bonding and ionic aggregates; grossly phase-separated blends are formed when only one SCLCP component has H-bonding or ionic aggregates. The amount of ionic or benzoic acid groups in the SCLCP components should determine, in principle, the number of the non-covalent cross-links, and higher cross-linking densities generally are more efficient for network interpenetrating. However, the complete effects of the network density may be

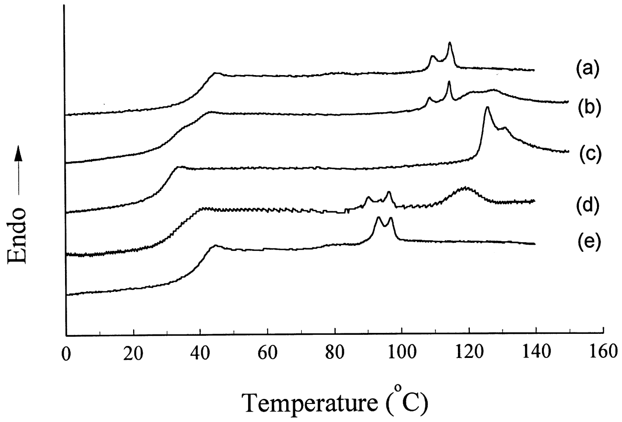

Fig. 2. DSC heating curves for: (a) BiP5CN; (b) P6CN-0.26COOH/BiP5CN; (c) P6CN-0.26COOH; (d) P6CN-0.26COOH/BiP5CN-0.05Na; and (e) BiP5CN-0.05Na.

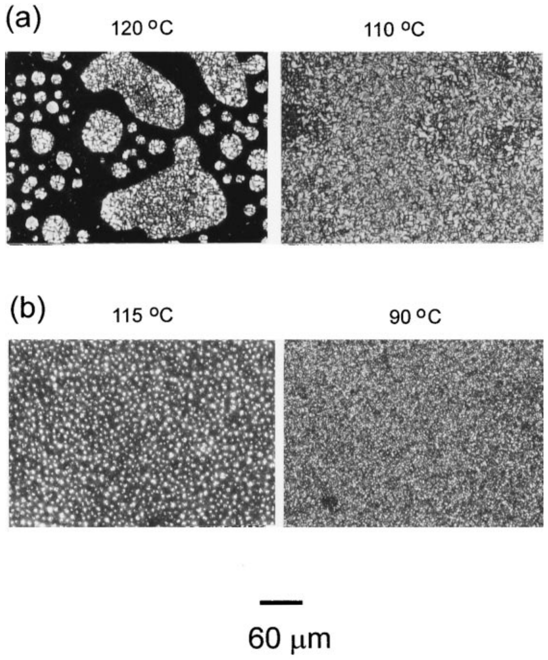

Fig. 3. Polarizing photomicrographs for: (a) P6CN-0.26COOH/BiP5CN at 120°C then cooled to 110°C; and (b) P6CN-0.26COOH/BiP5CN-0.05Na at 115°C then cooled to 90°C.

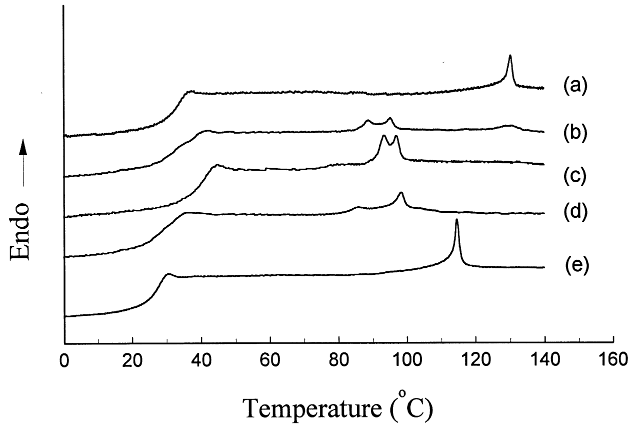

Fig. 4. DSC heating curves for: (a) P6CN; (b) P6CN/BiP5CN-0.05Na; (c) BiP5CN-0.05Na; (d) P6CN-0.07COOH/BiP5CN-0.05Na; and (e) P6CN-0.07COOH.

complex. The amount of acid or ionic groups may influence the initial miscibility between the two SCLCP components in the solution, and the phase separation kinetics upon the solvent evaporation, while the interplay between those factors and the rate of gelation through non-covalent cross-links determine the efficiency of the network interpenetrating. Nevertheless, the requirement of a minimum cross-link density for each network for the formation of an LC-IPN is conceivable. Fig. 5 shows the photomicrograph of the P6CN-0.26COOH/BiP5CN-0.025Na system under crossed polarizers at 120°C. The isotropic BiP5CN-0.025Na, having only 2.5 mol.% of the ionic groups, appears dark and forms a continuous phase which coexists with smaller spherical domains dispersed inside the birefringent regions of the liquid crystalline P6CN-0.26COOH. Combining the results shown in Fig. 3 for P6CN-0.26COOH with BiP5CN and with BiP5CN-0.05Na, this observation illustrates the change in morphology as a function of the ionic-aggregate cross-link density. It is interesting to note that this kind of morphology change is known for IPN of conventional polymers [6].

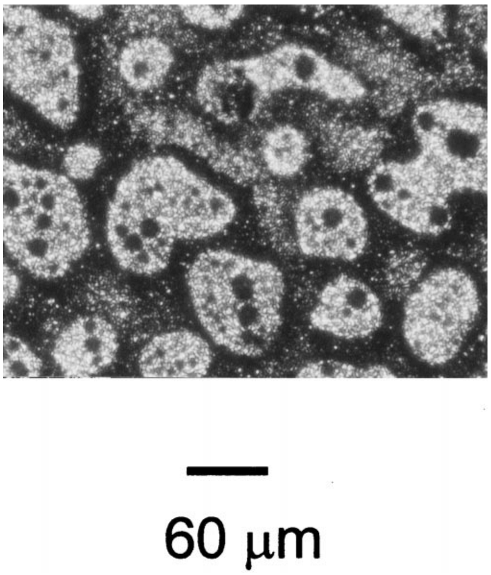

Fig. 5. Polarizing photomicrograph for P6CN-0.26COOH/BiP5CN-0.025Na at 120°C.

The crucial question is how to ensure a non-interfering route to the interpenetrating of two networks involving H-bonding and ionic aggregates. Are the observed features really due to a network interpenetrating, or are these the results of a miscibility-enhancement induced by specific intermolecular interactions between the two components? A comparison between P6CN/BiP5CN-0.05Na and P6CN-0.07COOH/BiP5CN-0.05Na helps us to obtain some answers. Looking at the chemical structures of the components involved for the two systems, the difference is between P6CN and P6CN-0.07COOH; and the difference is that 7 mol.% of the COO-phenyl groups in P6CN are replaced by the COOH groups in P6CN-0.07COOH. Therefore, it is reasonable to assume that possible ion–dipole or dipole–dipole interactions in P6CN/BiP5CN-0.05Na must be essentially the same as in P6CN-0.07COOH/BiP5CN-0.05Na. In other words, the difference, if there is any, in the intermolecular interactions inside the two systems is unlikely to be responsible for the results shown in Figs 2–4. If the hypothesis is pushed further, one can suspect that in the P6CN-0.07COOH/BiP5CN-0.05Na system, the 7 mol.% of the acid groups build up some kind of interaction with the 5 mol.% of the ionic groups. If this is the case, this particular type of interaction, probably ion–dipole, must be very strong and predominant over any other kinds of specific interactions which could exist in P6CN/BiP5CN-0.05Na. As a result, the acid dimers should be severely altered. Fig. 6 shows the infrared spectra, in the range of 1000–2300 cm^{-1}, for P6CN-0.07COOH/BiP5CN-0.05Na compared with P6CN-0.07COOH/BiP5CN, and also P6CN-0.26COOH/BiP5CN-0.05Na for comparison with P6CN-0.26COOH/BiP5CN. The band characteristic of the intermolecular acid dimers at 1700 cm^{-1} is present in the two blends containing BiP5CN-0.05Na, showing no sign of severe alternation on the dimers, due to any strong interaction between the acid and the ionic units. The slight change

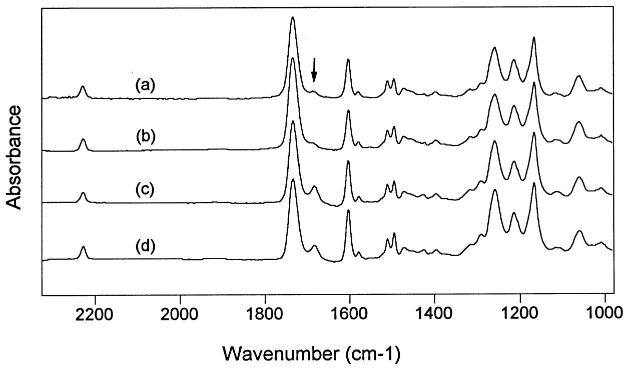

Fig. 6. Infrared spectra in the 1000–2300 cm^{-1} range for: (a) P6CN-0.07COOH/BiP5CN; (b) P6CN-0.07COOH/BiP5CN-0.05Na; (c) P6CN-0.26COOH/BiP5CN; and (d) P6CN-0.26COOH/BiP5CN-0.05Na.

in the intensity is more likely to arise from a 'dilution' effect as a result of the induced miscibility between the two components, while the two blends containing BiP5CN are grossly phase-separated, and the band comes from phases of essentially pure P6CN-0.07COOH and P6CN-0.26COOH. Also, the infrared spectra show no evidence of ion exchange between Na^+ and H^+ of the two polymers; no characteristic bands at 770 and 1103 cm^{-1} of the sodium salt of benzoic acid can be noticed [13]. The choice of solvent is important. The use of a non-ionizing solvent like THF, prevents the ionization of the salt groups of the LC ionomer from occurring in the initial solution. When pyridine is used, these bands appear, indicating ion exchange in the resulting blends. This analysis supports the suggestion that some kind of interpenetrating of the two self-associated networks is at the origin of the improved miscibility between the two SCLCP components. In some comparisons, differences in the molecular weights of the samples can be noticed, but overall, the usual molecular weight effects cannot explain the results.

3.2. Systems involving hydrogen bonding and triblock copolymers

Triblock copolymers like styrene–butadiene–styrene (SBS) represents another type of self-associated network structure. In the case of SBS having a styrene content of 30 wt.%, the polystyrene (PS) microdomains are known to form cylinders dispersed in the polybutadiene (PB) matrix. The PS microdomains interconnect the PB chains and play the role of cross-links. To investigate the use of this type of network in the preparation of self-assembled LC-IPNs, the combination of SBS with $P6OCH_3$-*x*COOH, being able to form a H-bonded network, was studied. In order to reveal the features of interpenetrating networks, we made the same comparisons as those discussed above, using systems containing only one component which can form a network structure upon solvent evaporation. Those reference systems were either SBS/$P6OCH_3$ ($P6OCH_3$ has no acid groups) or, more interestingly, $P6OCH_3$-*x*COOH with a diblock styrene-butadiene (SB) copolymer, which also has a styrene content of 30 wt.% but cannot form a network like SBS.

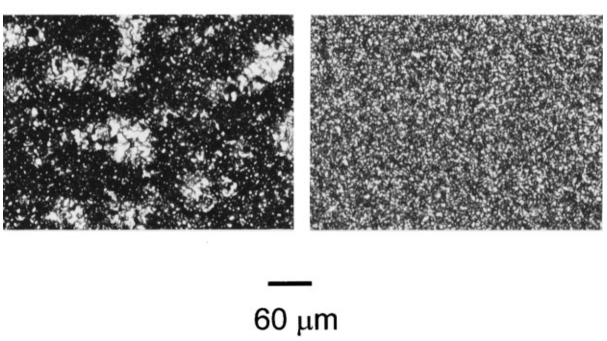

Fig. 7. Polarizing photomicrographs, at 90°C, for: (a) $P6OCH_3$-0.08COOH/SB; and (b) $P6OCH_3$-0.08COOH/SBS.

Indeed, the systems of H-bonded SCLCP with SBS display morphologies different from those reference blends having no possibility to form interpenetrating networks. This can be seen from the examples of polarizing photomicrographs presented in Fig. 7. The image in Fig. 7(a) is for the $P6OCH_3$-0.08COOH/SB blend. The gross phase separation is clear with the amorphous diblock copolymer appearing dark under crossed polarizers, and the liquid crystalline $P6OCH_3$ at 90°C showing a smectic texture within its dispersed regions of 60 μm in size. The image in Fig. 7(b) is taken from the $P6OCH_3$-0.08COOH/SBS system at the same temperature. A much more homogeneous morphology emerges with no large dark regions for the amorphous triblock copolymer. Similar results of comparison were obtained with other $P6OCH_3$-*x*COOH samples containing more than 8 mol.% of the acid groups. In the case of $P6OCH_3$/SBS, where only the triblock copolymer can form a network structure, gross phase separation occurred. Some measurements on samples prepared from THF solutions were also made, and similar results were obtained.

The above comparison is significant and straightforward. In addition to the fact that both SBS and SB are essentially non-polar polymers, the two samples have the same styrene content and SBS has a higher molecular weight than SB. To explain the obtained results, the possibility of having any kind of specific intermolecular interactions which exist between the SCLCP and SBS but are absent between the SCLCP and SB, can be ruled out. The only difference is that in the presence of the SCLCP which can be H-bonded, the triblock SBS copolymer can be converted into a network form, while the diblock SB copolymer cannot. These observations are consistent with those for the systems involving H-bonding and ionic aggregates, and indicate the condition for obtaining LC-IPNs, or at least inducing improved miscibility.

The condition is that the two components must be able to self-associate into their own network form through a non-interfering route. A simultaneous formation of the two networks during the solvent evaporation may be necessary for an effective network interpenetrating and restriction of the phase separation.

All the samples were characterized by other techniques. The intermolecular H-bonded dimers of the COOH groups were always confirmed by the characteristic infrared band at 1700 cm^{-1} (spectra not shown). Unlike the systems made up from two SCLCP components, DSC measurements show little changes in the mesophase transition temperatures and T_gs for the systems using SBS and SB. This suggests that the main effect for the LC-IPN systems like $P6OCH_3$-0.08COOH/SBS and $P6OCH_3$-0.24COOH/SBS is on the sizes of the separated phases, and that there is no significant interaction between the polymer chains of the two components. In other words, there is mainly a phase interpenetrating, which is a situation often observed for IPNs [6]. The

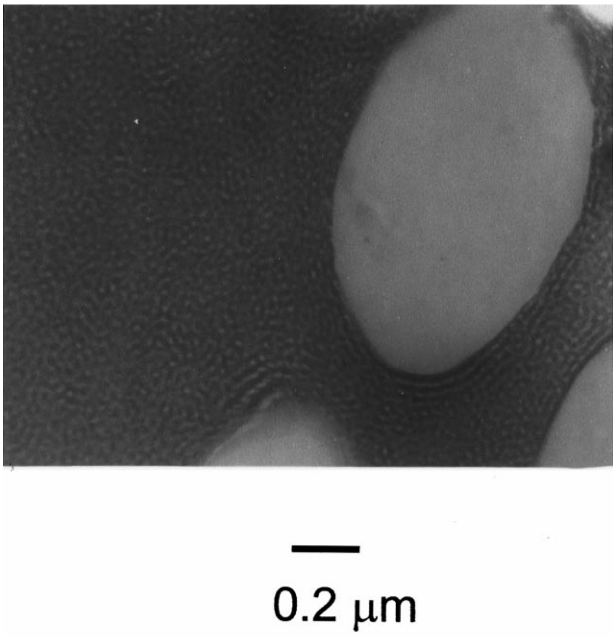

Fig. 8. TEM micrograph for P6OCH$_3$-0.24COOH/SBS.

transmission electronic microscopy seems to confirm this feature. Fig. 8 shows the morphology of a film of P6OCH$_3$-0.24COOH/SBS. The SBS phase is clearly distinguishable with the PB blocks preferentially stained by OsO$_4$ appearing dark. The small SCLCP domains of 1 μm in size are dispersed in the continuous SBS phase, with a sharp interface between the two components.

4. Conclusion

Non-covalent cross-linking such as hydrogen bonding, ionic aggregates and microdomains in triblock copolymers, can be used to prepare blends of SCLCPs which behave like self-assembled LC-IPNs, characterized by reduced domain sizes and an improved miscibility. The basic method consists in making a clear solution of two polymers, then evaporating the solvent and drying the mixture. The condition is that the two components must be able to self-associate through non-covalent cross-linking to form their own network structure; combinations of two polymers with only one component capable of forming non-covalent cross-links, lead to gross phase separation. The use of two SCLCPs carrying acid and ionic groups to form LC-IPNs through a non-interfering route is shown to be possible using non-ionizing solvents.

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