

# Synthesis and Mesomorphic Properties of Side-Chain Liquid Crystalline Ionomers Containing Sulfonic Acid Groups

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**ABSTRACT:** Side-chain liquid crystalline ionomers containing  $M_1$  (4-allyloxybenzoyloxy-4'-amylbenzoyloxy biphenyl) as mesogenic units and  $M_2$  (4-undecylenicoxy-4'-phenylazobenzene sulfonic acid) as ionic units were synthesized by graft copolymerization. Their liquid crystalline properties were characterized by differential scanning calorimetry, polarizing optical microscopy, and X-ray diffraction measurements. The results showed that introduction of nonmesogenic ionic units into polymeric structure could cause additional reduction of the clearing point of the ion-

omers, compared with the corresponding nonion polymers. When the content of ionic units increased to 8.5%, the ionic clusters and mesogens in the ionomers may be dispersed each other to form multiple blocks, but the introduction of ionic groups in LCIs did not change their mesogenic type. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 104: 304–309, 2007

**Key words:** polysiloxanes; sulfonic acid; liquid crystalline ionomers; thermotropic

## INTRODUCTION

Many studies have focused on the synthesis and properties of liquid crystalline polymers (LCPs); recently research has been extended to liquid crystalline ionomers (LCIs). The ionic groups were incorporated either into the main-chain or the side-chain to improve the physical properties of polymers,<sup>1,2</sup> ionic interactions between molecules can improve compatibility between different polymers in polymer blends<sup>3–8</sup> and the ordering of mesogenic fragments can enhance mechanical properties,<sup>9</sup> such a system could combine the characteristic features of both LCPs and ionomers, leading to new and useful materials.<sup>10</sup> In fact, incorporation of small concentration of ions into polymers has greatly influenced the properties of the polymers, not only does the ionic interactions modify structure and properties of LCIs, but also results in the physical crosslink.<sup>11</sup> Some investigation on the synthesis and properties of side chain LCIs have so far been reported, for example, Zental and coworkers<sup>12</sup> prepared copolymers made

from mesogenic and ferrocene-containing monomers and ionomers by oxidation of the ferrocene groups. The methods employed by Gramain and coworkers<sup>13</sup> was based on simultaneous quaternization and polymerization of 4-vinylpyridine in the presence of a mesogenic group, which resulted in LCPs containing ionic charge. As for Vuillaume et al.,<sup>14</sup> they prepared LCPs with side groups containing a pyridine-type unit, which was quaternized to form ionomers; Zhao and coworkers<sup>15,16</sup> have obtained LCIs containing metal ions ( $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ , and  $\text{Mn}^{2+}$ ) by partial neutralization of a copolymer from a mesogenic methacrylate monomer and methacrylic acid comonomer. Meanwhile, other researchers<sup>17</sup> had synthesized a series of new chiral nematic ionomers containing 0.7–9.0 mol % ions of alkali metals Na and transition metals Co(II). However, a little work has been reported on the effect of sulfonic acid ionic groups on the properties of side-chain LCIs.<sup>18,19</sup>

In this paper, we synthesized side-chain LCIs using biphenyl benzoate as mesogenic units and a long flexible nonmesogenic undecylenic compound containing sulfonic acid groups as ionic units and the corresponding side-chain LCPs without ionic units. The mesomorphic properties of the polymers and ionomers were characterized by differential scanning calorimetry (DSC), polarizing optical microscopy (POM), and X-ray diffraction measurements. The influence of the ionic content on the mesogenic properties of side-chain LCIs is discussed.

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## EXPERIMENTAL

### Materials

Polymethylhydrosiloxane (PMHS, MW = 700–800) was obtained from Jilin Chemical Industry Company (China). 4-Amylbenzoic acid, *p*-hydroxybenzoic acid, and antioxidant were purchased from Beijing Chemical. Undecylenic acid was obtained from Shenyang Chemical Co. All solvents and reagents were purified by standard methods.

### Characterization

Fourier transform infrared (FTIR) spectra were measured on a Nicolet 510 FTIR spectrometer (Nicolet Instruments, Madison, WI). Proton nuclear magnetic resonance ( $^1\text{H}$  NMR) spectra (300 MHz) were recorded on a Varian WH-90PFT spectrometer (Varian Associates, Palo Alto, CA). Phase transition temperatures and thermodynamic parameters were determined by using a Netzsch DSC 204 (Netzsch, Germany) equipped with a liquid nitrogen cooling system. The thermal stability of the polymers under atmosphere was measured with a Netzsch TGA 209C thermogravimetric analyzer. A Leica DMRX (Leica, Germany) polarizing optical microscope equipped with a Linkam THMSE-600 (Linkam, England) hot stage was used to observe phase transition temperatures and analyze LC properties for the monomers and polymers through observation of optical textures. XRD measurements were performed with a nickel-filtered Cu-K $\alpha$  ( $\lambda = 0.1542$  nm) radiation with a DMAX-3A Rigaku (Rigaku, Japan) powder diffractometer. The scattering vector lies in the horizontal direction and its length is defined as  $q = 4\pi\sin\theta/\lambda$ .

### Synthesis of the monomers

1,4-Allyloxybenzoic acid and 4-hydroxyazobenzene-sulfonic acid were prepared according to previously reported synthetic method.<sup>20</sup>

#### 4-Allyloxybenzoyl-4'-hydroxyl biphenyl (1)

4-Allyloxybenzoic acid (18.4 g, 0.1 mol) and thionyl chloride (25.0 g, 0.21 mol) were added into a round flask equipped with an absorption instrument of hydrogen chloride. The mixture was stirred at room temperature for 2 h, and then heated to 60°C and kept for 3 h in a water bath to obtain chloride. The mixture was stirred at room temperature for 2 h, then heated to 60°C and kept for 3 h in a water bath to ensure that the reaction finished. The excess thionyl chloride was removed under reduced pressure to obtain chloride.

The antioxidant (6.3 g, 0.01 mol) was dissolved in 120 mL pyridine and THF to form a solution. 4-Allyloxy benzoyl chloride (4.1 g, 0.02 mol) was slowly added to the solution and reacted at 50°C for 6 h, cooled, poured in 500 mL of cold water. The precipitate was filtered and was washed by acetone, then acetone was removed and 4-allyloxy benzoyl-4'-hydroxyl biphenyl was obtained: mp: 212.0°C, yield: 56%. IR ( $\text{cm}^{-1}$ ): 3346 (OH), 2963, 2878 ( $\text{CH}_3$ ,  $\text{CH}_2$ ), 1700 (C=O), 1604, 1577, 1510 (Ar).

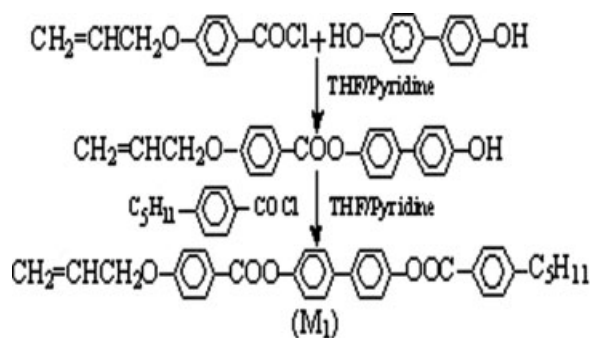
#### 4-Allyloxybenzoyloxy-4'-amylbenzoyloxy-*p*-biphenyl (M<sub>1</sub>)

The 4-amylbenzoyl chloride, prepared by the same procedure of 4-allyloxy benzoyl chloride above, was slowly added to pyridine and THF solution containing (1), then was allowed react for 6 h at 60°C. The mixture was poured into water, precipitated, and filtered; the crude product was washed with ethanol and acetone, respectively, then M<sub>1</sub> was obtained, mp: 159.0°C yield: 80%. IR ( $\text{cm}^{-1}$ ): 3045, 3012(=CH), 2932, 2868 ( $\text{CH}_3$ ,  $\text{CH}_2$ ), 1731(C=O), 1649 (C=C), 1604, 1509 (Ar).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS)  $\delta$  ppm: 0.91 (t, 3H,  $-\text{CH}_3$ ), 1.35(m, 4H,  $-(\text{CH}_2)_2-\text{CH}_3$ ), 1.67(m, 2H,  $-\text{CH}_2-$ ), 2.71(t, 2H, Ar- $\text{CH}_2$ ), 4.64 (d, 2H,  $-\text{CH}_2-\text{O}-$ ), 5.36–5.49(m, 2H,  $\text{CH}_2=$ ), 6.00–6.16(m, 1H, =CH-), 7.00–8.19 (m, 16H, Ar-H).

#### 4-Undecylenicoxy-4'-phenylazo benzene sulfonic acid (M<sub>2</sub>)

10-Undecylenic acids (18.4 g, 0.1 mol) and thionyl chloride (25.0 g, 0.21 mol) were added into a round flask equipped with an absorption instrument of hydrogen chloride. The mixture was stirred at 60°C until the reaction finished. The mixture was distilled under reduced pressure to obtain 12.4 g 10-undecylenoyl chloride at 160–170°C/20 mmHg in the yield of 61%.

4-Hydroxyazobenzene-sulfonic acid (6.3 g, 0.01 mol) was dissolved in 120 mL pyridine to form a solution. 11-undecylenoyl chloride (4.1 g, 0.02 mol) was added to the solution and was allowed react at 60°C for 6 h, poured into water, and acidified with 6N  $\text{H}_2\text{SO}_4$ . The precipitated crude product was filtered and recrystallized from ethanol and dried overnight at 85°C under vacuum to obtain orange powdered product with a yield of 76%. No melting point. IR (KBr,  $\text{cm}^{-1}$ ): 3080(=C-H); 2921 ~ 374; 2850 ( $\text{CH}_3-$  and  $-\text{CH}_2-$ ); 1757(C=O); 1188, 1053 (S-O in  $-\text{SO}_3\text{H}$ ).  $^1\text{H}$  NMR (DMSO, TMS)  $\delta$  ppm: 1.33–2.06 (m, 14H,  $-(\text{CH}_2)_7-$ ); 2.58(t, 2H,  $-\text{CH}_2-\text{O}-$ ), 4.92–5.02(m, 2H,  $\text{CH}_2=$ ), 5.72–5.84(m, 1H, =CH-), 7.28–8.13(m, 8H, Ar-H).

Scheme 1 Synthetic route of  $M_1$ .

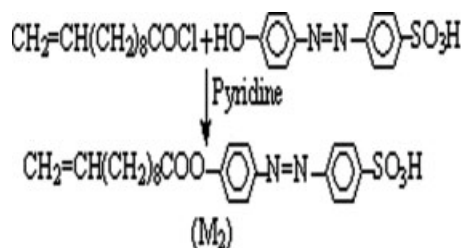
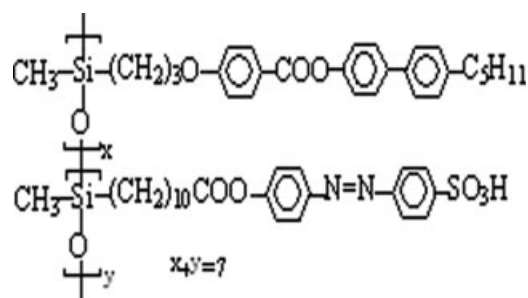
### Synthesis of polymers and ionomers

The monomers  $M_1$ ,  $M_2$ , and PMHS were dissolved in freshly distilled chloroform. The mixture was heated to  $65^\circ\text{C}$  under nitrogen and anhydrous conditions, and then 1 mL THF solution of hexachloroplatinate (IV) catalyst (5 mg/mL) was injected with a syringe. The progress of the reaction was monitored by IR spectra until the Si-H absorption peak of PMHS at  $2160\text{ cm}^{-1}$  disappeared. The crude polymer was purified by precipitation in chloroform with ethanol, and dried under vacuum. IR (KBr,  $\text{cm}^{-1}$ ): 3440 ( $-\text{OH}$ ); 2960–2850 ( $\text{CH}_3-$  and  $-\text{CH}_2-$ ); 1731 ( $\text{C}=\text{O}$ ); 1603, 1508 (Ar); 1260 ( $\text{Si}-\text{C}$ ); 1190 ( $\text{C}-\text{O}-\text{C}$ ); 1100–1000 ( $\text{Si}-\text{O}-\text{Si}$ ).

## RESULTS AND DISCUSSION

### Syntheses

The synthetic routes of the monomers  $M_1$  and  $M_2$  were outlined in Scheme 1 and 2 respectively. The polymer and ionomers were synthesized according to procedure in the literature,<sup>21</sup> and their structure was outlined in Scheme 3. The ionomers and the corresponding polymer were prepared by hydrosilyzation from polymethylhydrosiloxane with the synthesized monomers  $M_1$  and  $M_2$ . Hydrosilyzation is a known method for obtaining conventional LC polysiloxanes; this reaction is hardly quantitative, and however, when the reaction mixture included ionic monomer  $M_2$ , the polymer yields decrease. One probable reason is the ionization of the ion-pair sup-

Scheme 2 Synthetic route of  $M_2$ .

Scheme 3 Structures of ionomers.

pressed the growth of the chain; another reason is a reduction of the catalyst by the presence of the sulfonic acid. The catalysts used in the hydrosilyzation are THF solution of hydrogen hexchloroplatinate (IV) hydrate. The Si-H stretching at  $2160\text{ cm}^{-1}$  disappeared and their characteristic absorption bands were mentioned earlier. For organic sulfonic acid, the IR absorption range of  $\text{O}=\text{S}=\text{O}$ , the asymmetric and symmetric stretching mode lied in  $1150\text{--}1260$  and  $1010\text{--}1080\text{ cm}^{-1}$ , respectively, and that of the S-O stretching mode lied in  $600\text{--}700\text{ cm}^{-1}$ . Because of the overlap found for stretching bands of  $\text{O}=\text{S}=\text{O}$  with ester C-O stretching in the monomer  $M_1$ , we chose the S-O stretching mode ( $600\text{--}700\text{ cm}^{-1}$ ) to identify sulfur groups in the ionomers, and Figure 1 showed the FTIR spectrum in the range of  $400\text{--}1000\text{ cm}^{-1}$  for  $P_1$ ,  $P_2$ , and  $P_4$ . Comparing the FTIR spectra of polymer and ionomers synthesized, there was no S-O stretching mode found in  $P_1$ , however a weak band ( $636\text{ cm}^{-1}$ ) was found for the sample of  $P_2$ , and this absorption band became stronger as ionic content of ionomers increases; this result indicated successful incorporation of monomers into the polysiloxanes chains.<sup>20</sup>

### LC behavior of the monomers

The thermal property of  $M_1$  and  $M_2$  were characterized by DSC and their textures were observed by polarized optical microscope (POM).

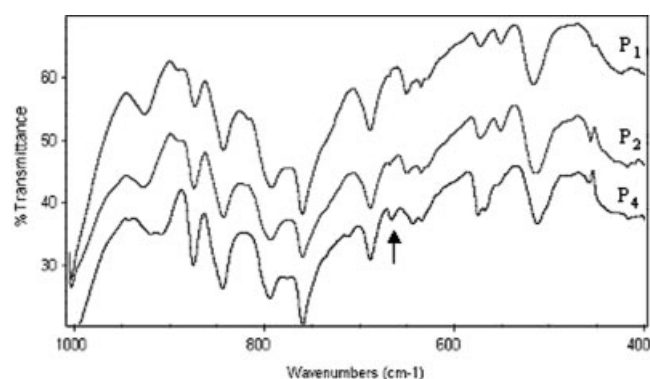
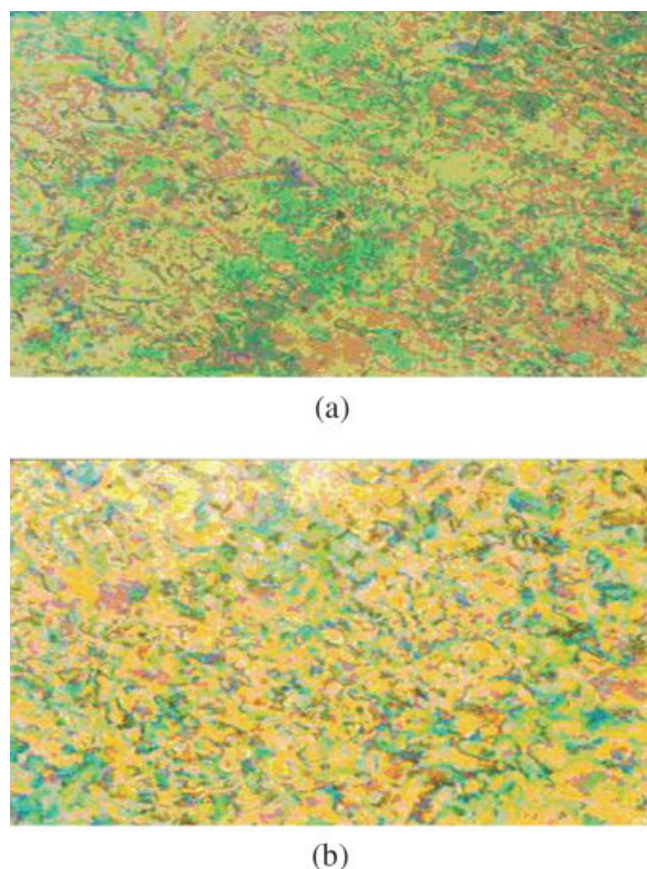


Figure 1 FTIR spectrum of polymer and ionomers.





**Figure 2** Polarized optical micrographs of  $M_1$  (200 $\times$ ) (a) 165 $^{\circ}\text{C}$  (b) 217 $^{\circ}\text{C}$ . [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

The POM observations showed that  $M_1$  exhibited colorful texture during the heating and cooling cycles. When  $M_1$  was heated to the melting temperature (159 $^{\circ}\text{C}$ ), the obvious threadlike texture appeared and the texture became more colorful when the temperature was increased. Meanwhile the texture did not change until anisotropic-to-isotropic transition temperature ( $T_i$ ) 283 $^{\circ}\text{C}$ . When it was cooled to 254 $^{\circ}\text{C}$ , droplets texture was observed at first, and then it turned to threadlike texture and kept this texture until crystallizing temperature reached 120 $^{\circ}\text{C}$ . Moreover, the quenched sample of  $M_1$  was studied by small angle X-ray scattering (SAXS), and no sharp peak in the lower Bragg angle region was observed. This result

suggested that monomer  $M_1$  was a nematic thermotropic LC monomer, and the photomicrographs of  $M_1$  were shown in Figure 2;  $M_2$  was a nonmesogenic monomer. From the structure of  $M_2$ , it showed that the orientation of rod-like azobenzene unit was influenced because of the end-capped sulfonic acid group, so  $M_2$  did not have mesogenic property.

### LC behavior of polymer and ionomers

The phase behavior of  $P_1 \sim P_4$  evaluated by DSC and POM was summarized in Table I. The DSC curves of all the polymer and ionomers synthesized were shown in Figure 3.

For polymer  $P_1$  with no ionic group, the glass transition ( $T_g$ ) was 107.2 $^{\circ}\text{C}$ , but  $T_i$  was not observed, so it was determined by POM observations. When  $P_1$  was heated to  $T_g$ , the colorful texture appeared and did not change until  $T_i$ .

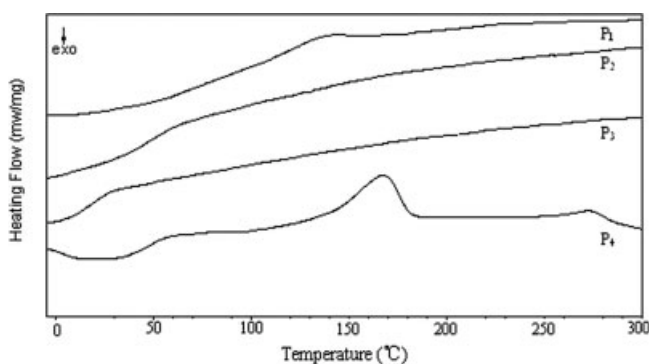
It is well known that the incorporation between the ions generally constrains the segmental motion of polymer chains on amorphous, flexible polymers.<sup>19</sup> When the ionic groups were linked into main-chain or side-chain polymers, ionic groups may increase the interaction between the molecules and disturb the orientation of mesogenic units, therefore, LC behavior of LCIs may be influenced by the introduction of ionic groups.

The experimental results showed that glass-transition temperatures  $T_g$  of the side-chain LCIs decreased first, and then increased in the case of a relatively low proportion of ionic monomer, compared with the corresponding polymer  $P_1$  without ionic units. Generally,  $T_g$  increased with ionic content because ionic groups increased the interaction between the molecules. This implied that another role of the flexible space, which makes  $T_g$  of polymer decrease, had been played simultaneously, that was undecyl group between sulfonic groups and backbone. Besides  $T_i$  tended to reduce from 318.0 to 283.0 $^{\circ}\text{C}$ . This result was similar to that of experiments in the literature.<sup>16</sup> Nevertheless, there was another peak of DSC endothermic curve of  $P_4$  and the temperature of this endotherm was 168.7 $^{\circ}\text{C}$ . To study the phase behavior, liquid crystalline property of  $P_4$  were observed by POM and the observations showed that there was

**TABLE I**  
Polymerization and Thermal Analysis Results of the Polymer and Ionomers

| Polymer | PHMS/ $M_1$ / $M_2$ | $\chi$ (mol%) | $T_{g1}$ ( $^{\circ}\text{C}$ ) | $T_{g2}$ ( $^{\circ}\text{C}$ ) | $T_i$ ( $^{\circ}\text{C}$ ) | $T_d$ ( $^{\circ}\text{C}$ ) | Mesophase |
|---------|---------------------|---------------|---------------------------------|---------------------------------|------------------------------|------------------------------|-----------|
| $P_1$   | 0.5/3.50/0.00       | 0.00          | 107.2                           | –                               | 318.0                        | 415.0                        | N         |
| $P_2$   | 0.5/3.43/0.07       | 2.00          | 87.7                            | –                               | 310.0                        | 372.0                        | N         |
| $P_3$   | 0.5/3.32/0.18       | 4.50          | 53.0                            | –                               | 302.0                        | 370.5                        | N         |
| $P_4$   | 0.5/3.16/0.34       | 8.50          | 72.6                            | 168.7                           | 283.0                        | 365.6                        | N         |

$T_d$ , temperature at which 5% weight loss occurred.  
 $\chi$  (mol%): mole percent of  $M_2$  based on  $M_1 + M_2$ .



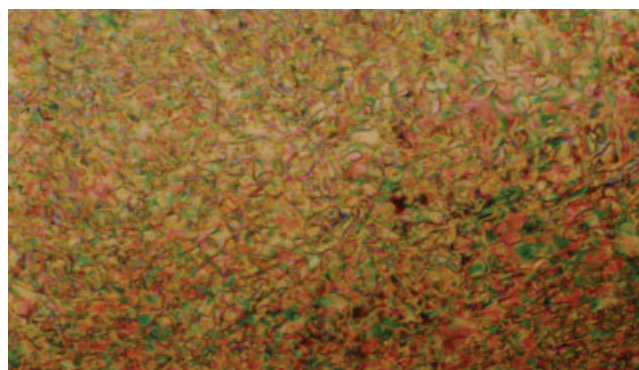
**Figure 3** DSC thermogram (second heating) of polymer and ionomers.

no change of texture in the range of 100–200°C, that was to say, this was not behavior of phase transition. It was attributed to the formation of two-phase behavior due to ionic aggregation, which was generally accepted that ionic groups formed aggregates, called multiplet. In the ionomer literature, the features of two glass transitions were characteristic of a biphasic material and may readily be explained by the Eisenberg–Hird–Moore (EHM) “multiplet-cluster” concept developed for classical ionomers.<sup>22</sup> At very low ion content, only multiplet were present. As the number density of the multiplet increased with ion content, the regions of restricted mobility started to overlap and formed large contiguous regions of reduced mobility.<sup>23</sup> In this state, the ionic clusters and LC phase were coexistent. The ionic clusters and the LC segments may be dispersed each other to form multiple blocks. The ionomers exhibited two glass transitions: one for the matrix region was found at lower temperatures and another for the cluster region was located at higher temperatures.

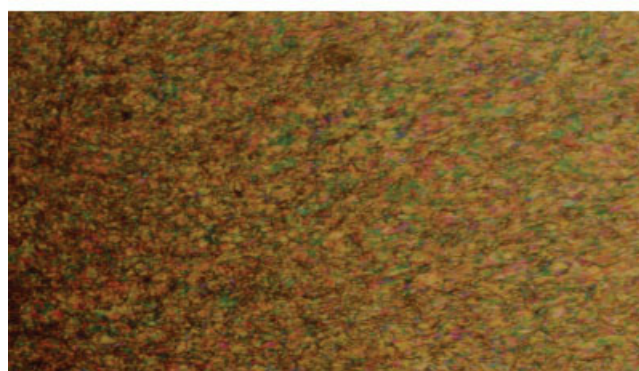
In our previous study, we also have prepared several kinds of side-chain liquid crystalline polysiloxanes containing sulfonic acid groups.<sup>19–21</sup> The distance between sulfonic acid and the main-chain backbone was short, because of the orienting effect of LC units, the formation of ionic cluster phases was not easy. In our experiments, polymethylsiloxane had low glass transition temperature, and the phase separation process should be fast because the siloxane matrix has high mobility. On the other hand, in the structure of LCI that we had prepared, there was a long flexible place between the sulfonic acid groups and the main-chain backbone, so the sulfonic acid groups should be easy to move and phase behavior of SLCI depended on the competition between the formation of ionic cluster phases and LC phase. Therefore, P<sub>4</sub> showed thermotropic LC behavior and ionomer property. Photomicrographs of P<sub>1</sub>, P<sub>2</sub>, and P<sub>4</sub> were shown in Figure 4(a–c). It indicated that introduction of ionic monomer M<sub>2</sub> did not remarkably affect their texture.

Meanwhile, TGA results of the polymer and ionomers were shown in Table I also. TGA results showed that the temperatures at which 5% weight loss occurred ( $T_d$ ) were greater than 350°C for P<sub>1</sub> ~ P<sub>4</sub>. This displayed that the synthesized polymers had a high thermal stability.

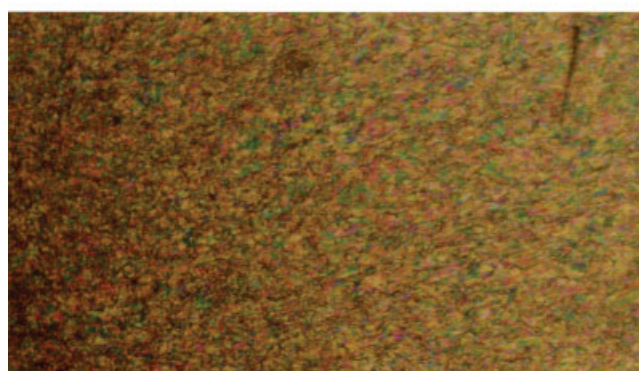
X-ray diffraction studies were carried out to obtain more detailed information on the LC phase and ionic cluster structure. Figure 5 reveals representative X-ray diffraction curves of P<sub>1</sub>, P<sub>2</sub>, and P<sub>4</sub>. No scattering



(a)



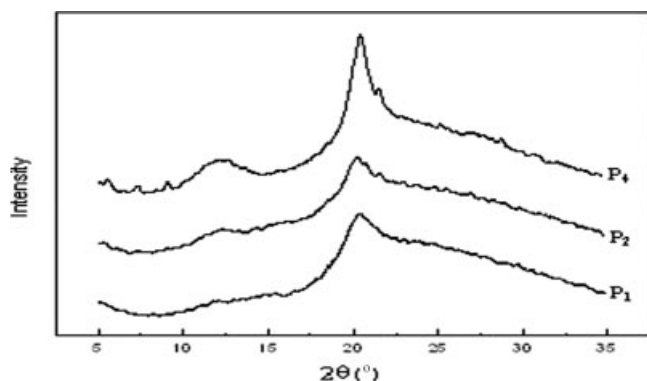
(b)



(c)

**Figure 4** Polarized optical micrographs of polymers (200×) (a) P<sub>1</sub> at 180°C (b) P<sub>2</sub> at 180°C (c) P<sub>4</sub> at 200°C. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]





**Figure 5** X-ray diffraction patterns of quenched samples  $P_1$ ,  $P_2$ , and  $P_4$ .

peak appeared at the lower Bragg angle region and a broad amorphous diffuse peak at about  $20^\circ$  occurred in wide-angle X-ray diffraction curves for all the quenched samples, and corresponding to the  $P_1$ , its intensity obviously increased with increase in the content of ionic groups. It suggested that the crystallinity of ionomers increased with increase of the content of ionic groups, and this explained that the structure of ionic monomer  $M_2$  contains rod-like azobenzene groups; it maybe incorporated into the arrangement of mesogenic groups of monomer  $M_1$ , which increases the orientation of LCP. Meanwhile, a diffuse peak at about  $12^\circ$  at wide scattering angle appeared. It was resulted by the interaction between Si-O and the sulfonate ionic groups. Therefore, nematic phase structure of the nonionic LCP  $P_1$  and LCIs  $P_2 \sim P_4$  were confirmed by X-ray diffraction, which coincides with POM results described. It also indicated that the formation of ionic structure in the ionomers did not change their mesogenic type.

### CONCLUSIONS

A series of side-chain LCIs containing sulfonic acid groups were prepared by graft copolymerization, and their LC properties showed that the ionic

groups lead to additional reduction of the clearing point of the ionomers compared with the corresponding polymer without ionic groups. A little content of the sulfonic acid remarkably affected phase behavior of LCIs, resulting in a decrease in the extent of mesogenic phase. When the content of sulfonic acid was 8.5%, the ionic aggregates appeared, but the introduction of ionic groups in LCIs did not change their mesogenic type.

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