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Synthesis and characterization of a series of side chain chiral smectic liquid crystalline elastomers

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A series of new chiral smectic liquid crystalline elastomers was prepared by graft polymerization of a nematic monomer with a chiral and non-mesogenic crosslinking agent, using polymethylhydrosiloxane as backbone. The chemical structures of the monomers and polymers obtained were confirmed by FTIR and ^1H NMR. The mesomorphic properties were investigated by differential scanning calorimetry, thermogravimetric analysis, polarizing optical microscopy and X-ray diffraction. Monomer M_1 showed a nematic phase during heating and cooling. Polymer P_0 exhibited a smectic B phase; elastomers P_1 – P_3 showed the smectic A phase, P_4 – P_6 showed a chiral smectic C (SmC^*), and P_7 displayed stress-induced birefringence. Elastomers containing less than 15 mol % M_2 displayed elasticity, reversible phase transitions with wide mesophase temperature ranges, and high thermal stability. With increasing content of the crosslinking unit, glass transition temperatures first increased, then fell, then increased again; isotropization temperatures and mesophase temperature ranges steadily decreased.

1. Introduction

Liquid crystalline elastomers (LCEs) have been paid much attention since their synthesis was reported in 1981, due to the combination of LC properties with rubber-like elasticity [1–7]. Chiral LCEs form an attractive research fields because of their potential piezoelectric, ferroelectric and pyroelectric properties [8–10]. More recently, smectic C (SmC) elastomers composed of chiral materials have attracted both industrial and scientific interest. Chiral smectic C (SmC^*) elastomers can be obtained by the combination of a chiral component and a mesogenic crosslinking unit [11–13]. Consequently, cholesteric liquid crystalline elastomers (cholesteric LCEs) have been considered as candidates for the fabrication of piezoelectric devices [14–20]. Cholesteric LCEs can be obtained by three main methods: (i) by directly combining cholesteric materials with a crosslinking agent, (ii) by photocrosslinking, (iii) by combining chiral materials with a nematic LC crosslinking agent.

Much research has been carried out in this laboratory in the field of cholesteric LCEs [21–23]. In the past, they have been obtained by combining a chiral non-mesogenic crosslinking agent with a nematic LC monomer; we now wish to know whether SmC^* elastomers can be obtained by combining a chiral

crosslinking agent with a nematic monomer. In general side chain SmC^* elastomers can be obtained by combining chiral mesogenic or non-mesogenic units with a non-chiral crosslinking agent. The present paper deals with a new preparation of side chain smectic LCEs, in which the nematic monomer 4'-allyloxybiphenyl-4-yl 4-heptyloxybenzoate (M_1) and chiral and non-mesogenic crosslinking agent isosorbide-undecanoyloxy bisate (M_2), are linked directly to a polymethylhydrosiloxane backbone. We expected to obtain SmC^* or cholesteric LCEs by this method. Isosorbide was chosen because it possesses a high twisting power and can better introduce chirality into LCEs [24]. However, to the best of our knowledge, few SmC^* elastomers bearing a chiral and non-mesogenic crosslinking agent have so far been described; it is necessary to synthesize and examine various kinds of side chain SmC^* LCEs with a chiral crosslinking agent to explore their potential application.

2. Experimental

2.1. Materials

Polymethylhydrosiloxane (PMHS, $M_n=700$ – 800) was purchased from Jilin Chemical Industry Co. 4,4'-Biphenol was purchased from Beijing Chemical Industry Co., undecylenic acid from Beijing Jinlong Chemical Reagent Co. Ltd, and isosorbide from Yangzhou Shenzhou New Material Co. Ltd.

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1-Bromopentane was obtained from Yancheng Longsheng Fine Chemical Industry Co., and heptyl 4-hydroxybenzoate from Shenyang Xinxi Chemical Reagent Company. Toluene used in the hydrosilylation reaction was first heated under reflux over sodium, then distilled under nitrogen. All other solvents and reagents were purified by standard methods.

2.2. Characterization

IR spectra were measured on a Perkin Elmer Spectrum One FTIR spectrometer (Perkin-Elmer Instruments, USA). ^1H NMR spectra (300 MHz) were recorded on a Varian WH-90PFT spectrometer. Specific rotation was performed with a Perkin Elmer 341 polarimeter. Phase transition temperatures and thermodynamic parameters were determined using a Netzsch DSC 204 instrument (Netzsch, Germany) with a liquid nitrogen cooling system. The thermal stability of the polymers was measured with a Netzsch TGA 209C thermogravimetric analyser, with heating and cooling rates of $10^\circ\text{C min}^{-1}$. A Leica DMRX (Leica, Germany) polarizing optical microscope (POM) equipped with a Linkam THMSE-600 (Linkam, England) hot stage was used to observe phase transition temperatures and analyse LC properties for the monomers and polymers through observation of optical textures. X-ray diffraction (XRD) measurements were performed with nickel-filtered $\text{Cu-K}\alpha$ ($\lambda=0.1542\text{ nm}$) radiation using a DMAX-3A (Rigaku, Japan) powder diffractometer. The scattering

vector lies in the horizontal direction and its length is defined as $q=4\pi \sin \theta / \lambda$.

2.3. Synthesis of the monomers

The synthesis of the olefinic monomers is shown in scheme 1. 4-Heptyloxybenzoic acid and 4-allyloxy-4'-hydroxybiphenyl were prepared in this laboratory.

2.3.1. 4'-Allyloxybiphenyl-4-yl 4-heptyloxybenzoate (M_1). Monomer M_1 was prepared by procedures similar to those previously reported [25]. IR(KBr): 3078, 3038(=C-H), 2980–2850(– CH_3 , – CH_2), 1731(C=O), 1647(C=C), 1605–1450(Ar–), 1258 cm^{-1} (C–O–C). ^1H NMR(CDCl_3) δ (ppm): 0.88–0.93(t, 3H, – CH_3), 1.33–1.86(m, 10H, – CH_2 –), 4.03–4.08(d, 2H, – OCH_2 –), 4.59–4.61(d, 2H, $\text{CH}_2=\text{CHCH}_2\text{O}$), 5.30–5.49(m, 2H, $\text{CH}_2=$), 6.00–6.20(m, 1H, $\text{CH}_2=\text{CH}$ –), 6.98–8.18(m, 12H, ArH). In figure 1 the ^1H NMR spectra of M_1 is shown.

2.3.2. Isosorbide-undecanoyloxybisate (M_2). A few drops of DMF were added to a suspension of undecenoic acid (0.1 mol) in freshly distilled thionyl chloride (40 ml) and the reaction mixture was heated under reflux for 10 h. Excess thionyl chloride was then removed under reduced pressure to give the corresponding undecylenic acid chloride. Isosorbide (0.03 mol) was dissolved in a mixture of dry triethylamine (10 ml) and dry THF (100 ml); the

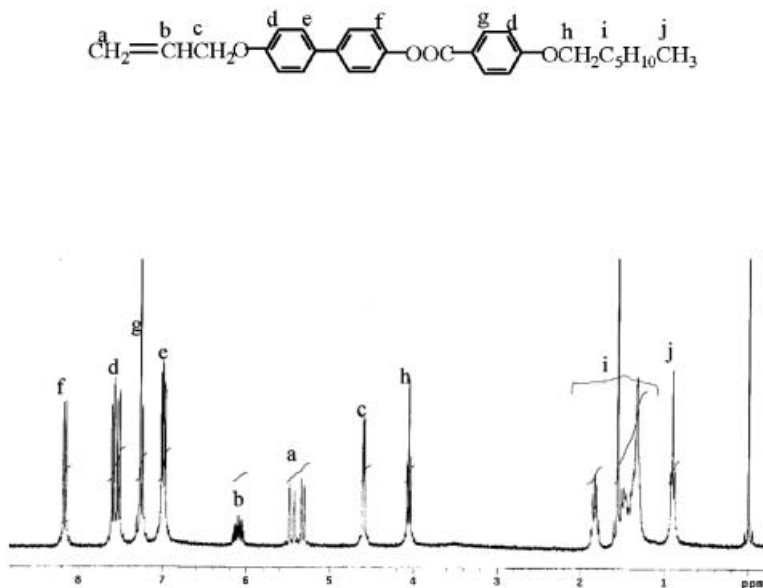


Figure 1. ^1H NMR spectra of M_1 .

undecylenic acid chloride was then added quickly at ice bath temperature and the reaction mixture was held under a condenser reflux for 72 h at room temperature. The cold reaction mixture was poured into water and the crude product obtained by separating the organic// layer, extracting the aqueous layer with toluene, and combining the organic layers, which were then washed with water, dilute alkali, water again and then dried with magnesium sulphate. The product was obtained by complete evaporation of the toluene under reduced pressure; yield 65%, m.p.22°C, $[\alpha]_{589}^{18.0} = +63.2^\circ$. IR(KBr): 3057(=CH), 2975, 2855(-CH₃, -CH₂), 1742(C=O), 1640(C=C), 1605–1450 (Ar-), 1235 cm⁻¹ (C–O–C). ¹H NMR (CHCl₃) δ (ppm): 1.29–1.66(m, 12H, (-CH₂)₆), 2.00–2.07(d, 2H, CH₂=CHCH₂), 2.28–2.39(m, 2H, -CH₂CH₂COO), 3.77–4.48 (m, 8H, isosorbide), 4.81–5.19 (m, 2H, CH₂=CH), 5.74–5.85(m, 1H, CH₂=CH).

2.4. Synthesis of the polymers

The synthetic methods for preparing fun uncrosslinked and crosslinked polymers (elastomers) are similar. The synthetic routes and schematic representation of the elastomers are outlined in scheme 2. The mesogenic monomers and varying content of chiral crosslinking agent reacted with the Si–H of PMHS to form elastomers in the presence of a Pt catalyst. The monomers **M**₁, **M**₂ and PMHS were dissolved in dried, freshly distilled toluene. The mixture was heated to 65°C under nitrogen and anhydrous conditions, and then a suitable amount of THF solution of hexachloroplatinate hydrate catalyst was injected with a syringe. The reaction mixture was held at 60–70°C for three days, and then treated with methanol; the precipitated products were dried in vacuum at room temperature. The elastomers obtained were insoluble in toluene, xylene, DMF and chloroform, but could swell in these solvents. Polymer details are listed in table 1. IR(KBr):

2928–2856(-CH₃, -CH₂), 1733(C=O), 1605–1450(Ar-), 1200–1000 cm⁻¹ (Si–O–Si).

3. Results and discussion

3.1. Synthesis

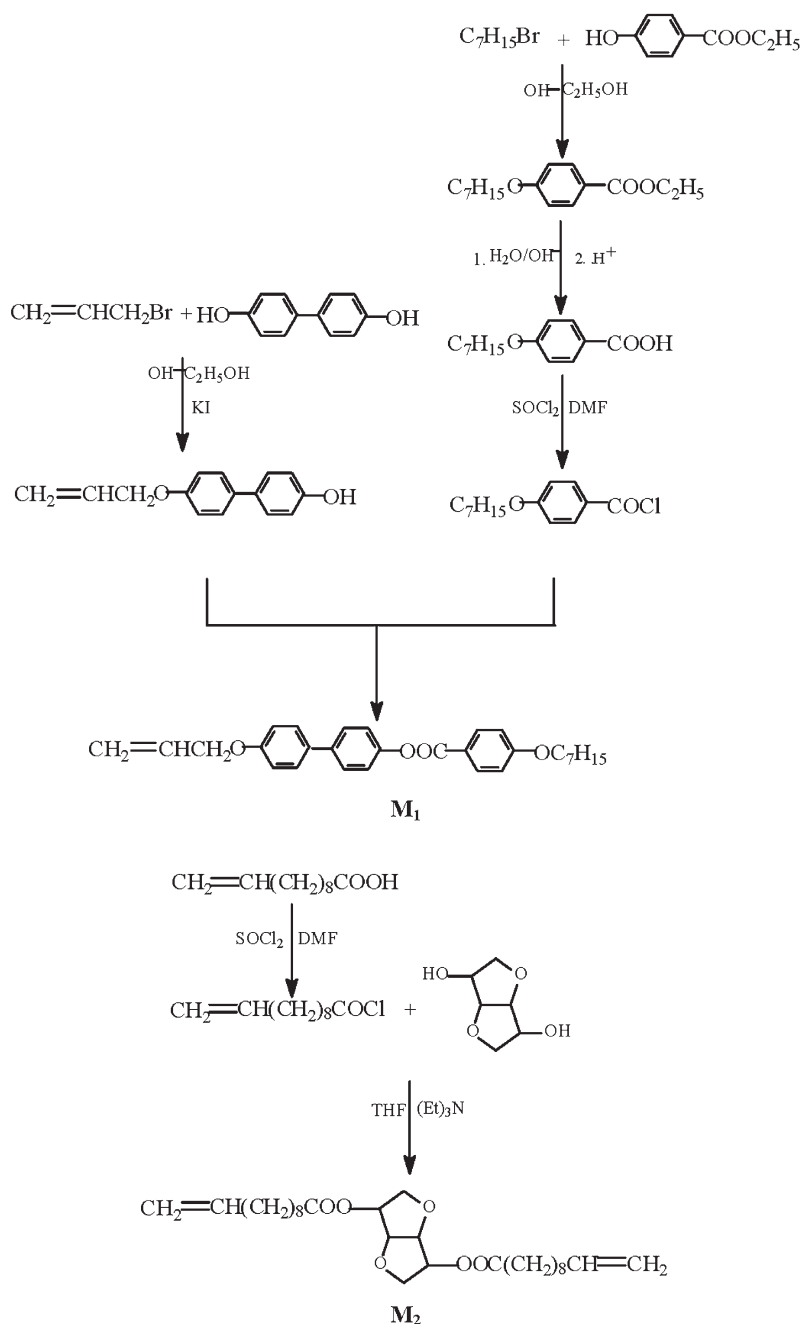
The novel nematic liquid crystalline monomer **M**₁ and a chiral crosslinking agent **M**₂ have been synthesized using reaction pathways shown in scheme 1. **M**₁ was prepared by (1) mono-etherification of 4,4'-hydroxybiphenyl with 3-bromopropene to give 4-allyloxy-4'-hydroxybiphenyl; (2) esterification of 4-heptyloxybenzoic acid chloride with 4-allyloxy-4'-hydroxybiphenyl. **M**₂ was prepared by esterification of undecenoic acid chloride with isosorbide. The chemical structures of the two monomers were characterized by FTIR and ¹H NMR spectroscopy. The FTIR spectra of **M**₁ and **M**₂ showed characteristic bands at 1742–1731 cm⁻¹ originating from ester C=O stretching, 1647–1640 cm⁻¹ due to olefinic C=C stretching and 1605–1450 cm⁻¹ corresponding to aromatic C=C stretching. The ¹H NMR spectra of **M**₁ and **M**₂ showed multiplets at 6.98–8.18, 5.74–6.20, 4.81–5.49, 4.03–4.61 and 0.88–2.39 ppm corresponding to aromatic, vinyl, methyleneoxy, isosorbide and methyl, methylene protons.

The new series of elastomers **P**₁–**P**₇ were prepared by a one-step hydrosilylation reaction. The yields and properties of **P**₀–**P**₇ are summarized in table 1. The elastomers **P**₁–**P**₇ were insoluble in toluene, xylene, DMF and chloroform, but could swell in these solvents. The disappearance of the Si–H stretching band at 2166 cm⁻¹ showed the successful incorporation of monomers into the polysiloxane chains. Characteristic absorption bands appeared at 1732, 1605–1450 and 1200–1000 cm⁻¹ representing ester C=O stretching aromatic and Si–O–Si, respectively. It can be concluded that the chemical structures of the monomers and polymers are consistent with expectation.

Table 1. Polymerization of **P**₀–**P**₇.

Polymer	Feed/mmol			Yield/%	Solubility ^b		
	M ₁	M ₂	M ₂ ^a /mol %		toluene	xylene	DMF
P ₀	3.500	0.000	0	83.2	+	+	+
P ₁	3.360	0.070	2	76.5	–	–	–
P ₂	3.220	0.140	4	73.8	–	–	–
P ₃	3.080	0.210	6	70.2	–	–	–
P ₄	2.940	0.280	8	68.5	–	–	–
P ₅	2.800	0.350	10	60.2	–	–	–
P ₆	2.450	0.525	15	65.2	–	–	–
P ₇	2.100	0.700	20	55.3	–	--	–

^aMolar fraction of **M**₂ based on **M**₁+**M**₂. ^b+soluble; – insolubility or swelling.



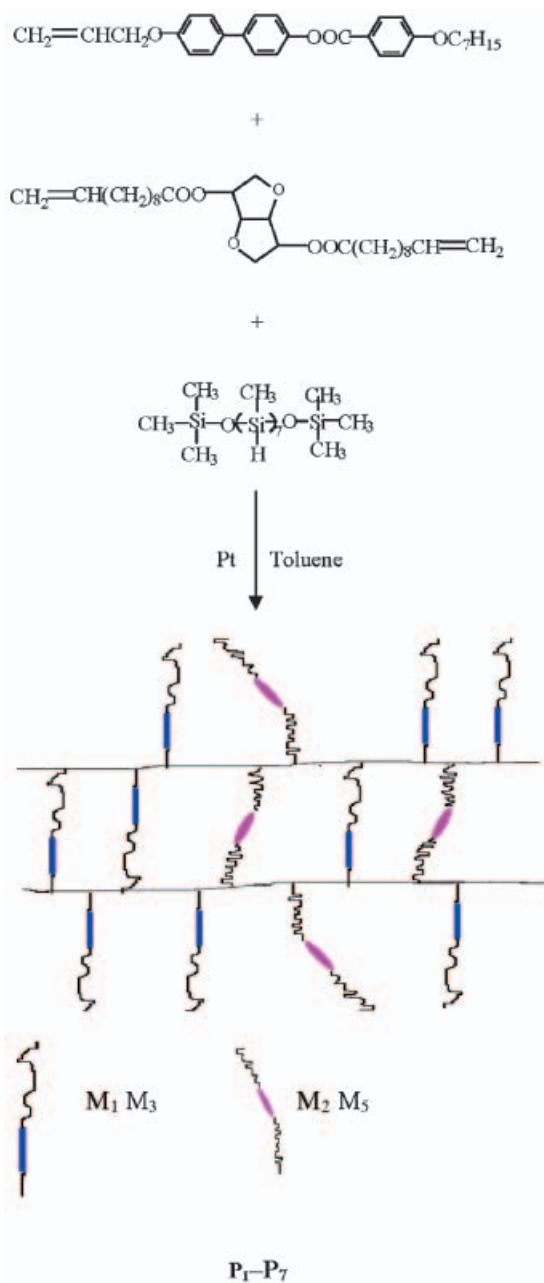
Scheme 1. Synthetic route to the monomers.

3.2. Optical analysis

The optical textures of the monomers and polymers were characterized by POM using a hot stage under a nitrogen atmosphere. M_1 exhibited an enantiotropic nematic phase. When M_1 was heated to 134.2°C, the sample began to melt and flow and a threaded texture gradually appeared, characteristic of the nematic phase. A droplet texture appeared close to the melting point.

During cooling, a droplet texture was briefly seen, immediately turning into a threaded texture; photomicrographs are shown in figures 2(a) and 2(b), respectively.

The uncrosslinked polymer P_0 showed bright colours, but no clear textures. On heating to the clearing point, an interesting strapped texture appeared at 246°C, and flashed particles at 256.7°C. This may be because the



Scheme 2. Synthesis and schematic representation of the elastomers.

molecules have enough energy to overcome steric hindrance and move freely. Photomicrographs of the homopolymer are shown in figures 2(c) and 2(d), respectively. Elastomers $\text{P}_1\text{-P}_3$ exhibited smectic step particle textures, characteristic of SmA or SmB phases, $\text{P}_4\text{-P}_6$ exhibited lined texture which is the special texture of the SmC* or cholesteric phase, while P_7 displayed stress-induced birefringence. Photomicrographs of these elastomers are shown in figures 3(a-d), respectively.

The polymer LC phase type was further characterized by X-ray diffraction.

3.3. X-ray diffraction

XRD analysis provided a complementary assessment of the nature of the phases observed by DSC and POM, giving additional information about their structural parameters. Representative XRD curves of quenched samples are shown in figure 4; XRD data for quenched polymers are shown in table 2. The XRD patterns of the quenched polymer $\text{P}_0\text{-P}_6$ films showed sharp reflection peaks at 2θ of $5.11^\circ\text{-}5.40^\circ$ ($d=18.64\text{-}18.16\text{ \AA}$, $q=0.346\text{-}0.337\text{ \AA}^{-1}$) corresponding to the smectic layer spacing. The d -spacings of $\text{P}_0\text{-P}_3$ were close to the molecular lengths of the fully stretched mesomorphic units, calculated as 19.09 \AA , while d -spacings of $\text{P}_4\text{-P}_6$ were shorter. It may be concluded that the $\text{P}_4\text{-P}_6$ molecules are more inclined to slantwise arrangement [26]. The $\text{P}_0\text{-P}_6$ films showed another peak (first sharp, and then broad) at 2θ of $20.44\text{-}20.75^\circ$ ($d=4.75\text{-}4.81\text{ \AA}$, $q=1.304\text{-}1.323\text{ \AA}^{-1}$) due to, first, high order (P_0) and then low order ($\text{P}_1\text{-P}_6$) corresponding to the distance between the mesogenic side groups in the smectic layer [27]. As the level of crosslinking agent increased, these broad peaks became weaker; this is simply related to the difference between the intrachain scattering of the mesogenic repeat and the crosslinking unit. Combining POM with XRD shown that polymer P_0 was in a smectic B phase, elastomers $\text{P}_1\text{-P}_3$ were in the smectic A phase, while $\text{P}_4\text{-P}_6$ were chiral smectic C elastomers and P_7 displayed stress-induced birefringence [26, 27]. It may be concluded that the chirality content of the chiral crosslinking agent has a strong effect on the polymer phase, and that increased content of crosslinker and chiral crosslinker with a long flexible chain favours formation of the chiral smectic C phase.

3.4. Thermal analysis

The DSC curves and phase transition temperatures of monomer M_1 during heating and cooling are shown in figure 5. The heating thermogram shows two endotherms, at 134.3 and 218.6°C . On subsequent cooling, there are three endotherms, at 187.9 , 92.6 and 86.0°C . The DSC measurements reveal a melting to nematic point at 134.3°C and a clearing point at 218.6°C . On cooling, the isotropic phase becomes nematic at 187.9°C , crystallizing at 92.6 and 86.0°C due to different rates of crystallization. Monomer M_1 is an enantiotropic liquid crystal. The results of optical and thermal analysis are consistent.

Representative DSC curves of that elastomers, obtained during second heating, are presented in

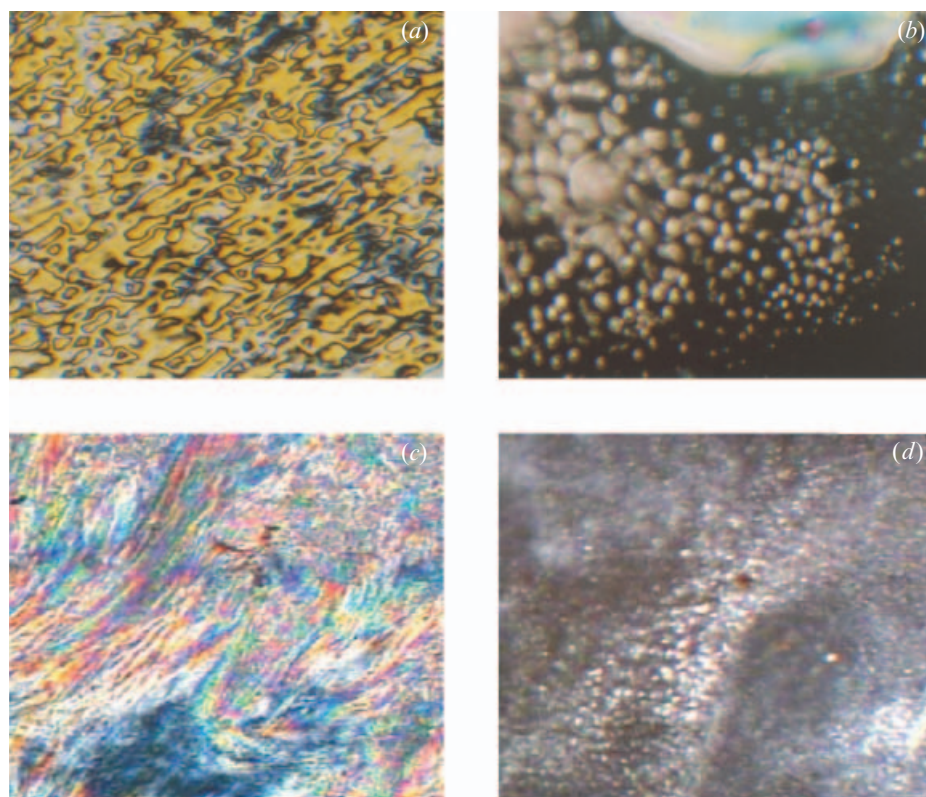


Figure 2. Optical textures of monomer M_1 and homopolymer $P_0(200X)$: (a) thread-like texture of M_1 on heating to 182.6°C ; (b) droplet texture of M_1 on heating to 221.0°C ; (c) texture of P_0 on heating to 249.5°C ; (d) texture of P_0 on heating to 256.7°C .

figure 6; phase transition temperatures and the results of thermal analysis are shown in table 3; and the effect of the content of crosslinking units on the phase transition is shown in figure 7. The glass transition temperature (T_g) and the temperature of mesophase to isotropic phase transition (T_i) can be seen. All transitions are reversible and do not change on repeated heating and cooling; the phase transition temperatures noted in DSC thermograms are consistent with the mesomorphic transition temperatures observed by POM. As seen from the data in the table 3 and figure 7, T_g values increase, decrease, and then increase again with the increasing content of the chiral crosslinking agent. The factors influencing T_g may be summarized as the properties of the polymer backbone, the rigidity of the mesogenic unit, the flexible spacer length and the content of crosslinking agent.

In elastomers, the crosslinking agent usually plays the main role, having two opposing actions: crosslinking and plasticization. Crosslinking imposes additional constraints on the segment motion of the polymer chains, and might be expected to raise T_g ; plasticization tends to reduce the T_g value, and when the two effects balance, only small T_g changes might be expected. At first, the introduction of the crosslinker places a

constraint on the segment motion, so the T_g values of P_0 – P_2 increased from 90.0 to 94.7°C ; the T_g value of P_3 then decreased abruptly to 85.5°C , due to the influence of plasticization. The T_g values of P_2 – P_4 fell from 94.7 to 85.3°C , and the T_g values of P_3 and P_4 were 85.5 and 85.3°C , only a small change due to the balance of crosslinking and plasticization. Finally, the crosslinking effect gradually becomes predominant again after the crosslinking agent content reaches a critical value, so T_g of P_4 – P_7 increased from 85.3 to 92.3°C .

Similarly, crosslinking influences the clearing point T_i in two ways. Firstly, crosslinking units may act as a non-mesogenic diluent and lead to a downward shift of the clearing point with increasing content in the LC polymer. Secondly, for a crosslinked sample, heating to the isotropic state requires additional energy to distort the polymer backbone from the anisotropic state at crosslinking; this leads to an increase in the clearing point with increasing content of chiral crosslinking agent. In this study, a non-LC crosslinking agent is introduced into the polymer backbone, so the first factor is predominant. It can be seen that the T_i value fell with increasing content of crosslinking agent. As the content increased from 2 to 15%, the T_i values of P_1 – P_6 decreased from 251.4 to 204.0°C ; at the same time,

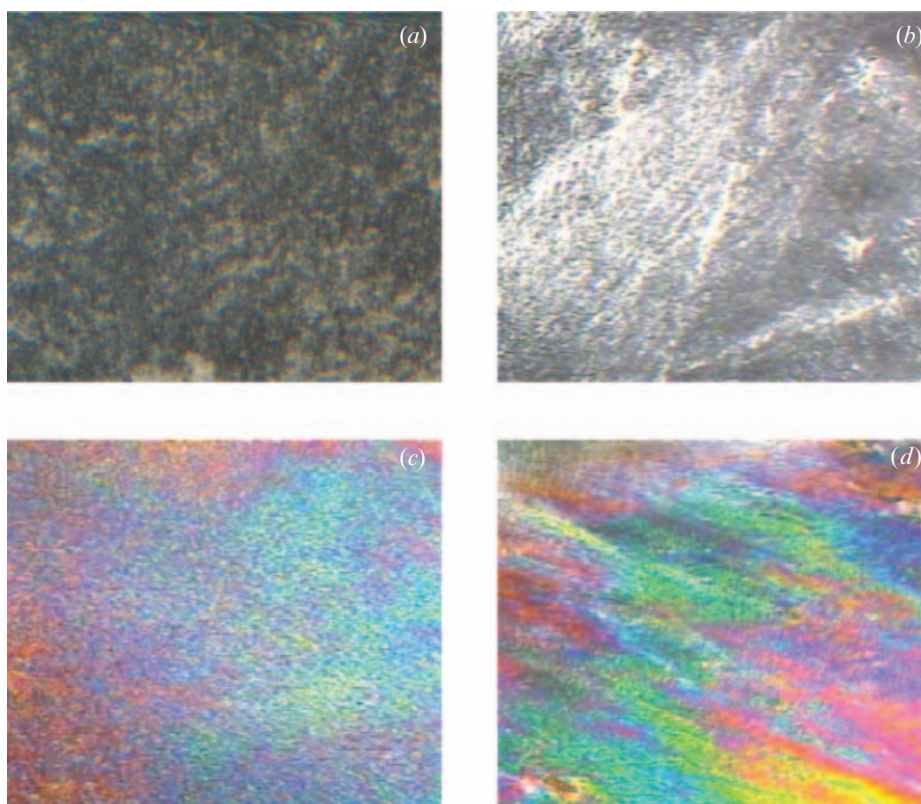


Figure 3. Optical textures of elastomers (200 \times): (a) step particletexture of P_3 on heating to 117.6 $^{\circ}\text{C}$; (b) lined texture of P_4 on heating to 196.0 $^{\circ}\text{C}$; (c) lined texture of P_5 on heating to 163.3 $^{\circ}\text{C}$; (d) lined texture of P_6 on heating to 157.3 $^{\circ}\text{C}$.

P_1 – P_6 displayed wide mesophase temperature ranges (ΔT) from 158.5 to 116.9 $^{\circ}\text{C}$, i.e. ΔT values of the elastomers decreased with increasing content of cross-linking agent. The DSC curve of P_7 showed only a glass transition and no mesophase to isotropic transition due

to the high levels of crosslinking which disturbed the LC order.

TGA of the elastomers showed that the temperatures at which 2% weight loss occurred (T_d) were greater than 340 $^{\circ}\text{C}$ for P_0 – P_7 ; the synthesized elastomers therefore have a high thermal stability.

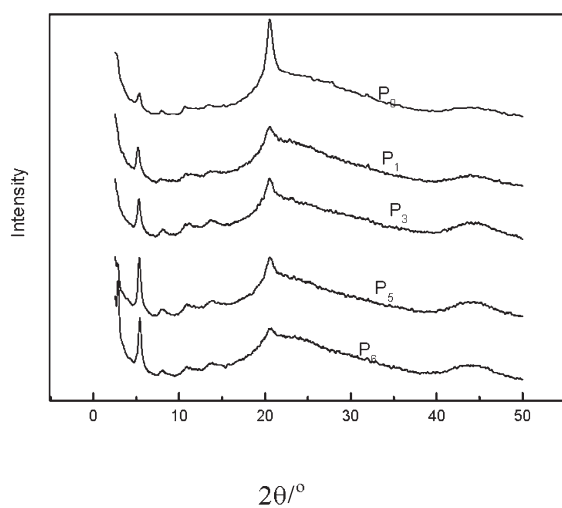


Figure 4. X-ray diffraction patterns of quenched samples.

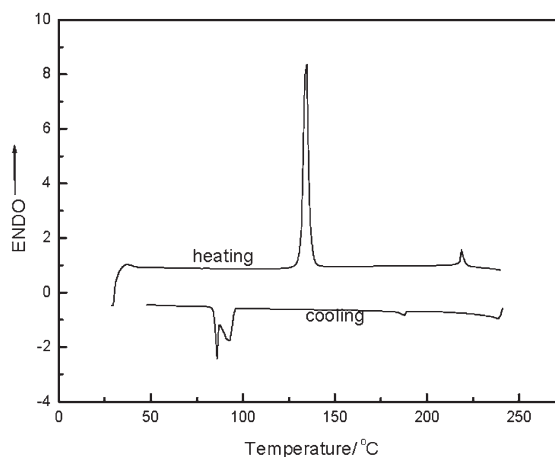


Figure 5. DSC thermographs of liquid crystal monomer M_1 .

Table 2. X-ray diffraction data for the polymers.

Polymer	Small angle				Wide angle			
	$2\theta/^\circ$	$d/\text{\AA}$	$q/\text{\AA}^{-1}$	Intensity ^a	$2\theta/^\circ$	$d/\text{\AA}$	$q/\text{\AA}^{-1}$	Intensity ^a
P ₀	5.11	19.19	0.327	wS	20.59	4.78	1.313	sS
P ₁	5.11	19.19	0.327	mS	20.59	4.78	1.313	sB
P ₂	5.27	18.60	0.337	mS	20.44	4.81	1.304	sB
P ₃	5.26	18.64	0.336	vsS	20.44	4.81	1.304	sB
P ₄	5.40	18.16	0.346	vsS	20.75	4.75	1.323	sB
P ₅	5.40	18.16	0.346	vsS	20.75	4.75	1.323	sB
P ₆	5.40	18.16	0.346	vsS	20.59	4.78	1.313	sB

^aKey: w, weak; m, medium; s, strong; vs, very strong; B, broad; S, sharp.

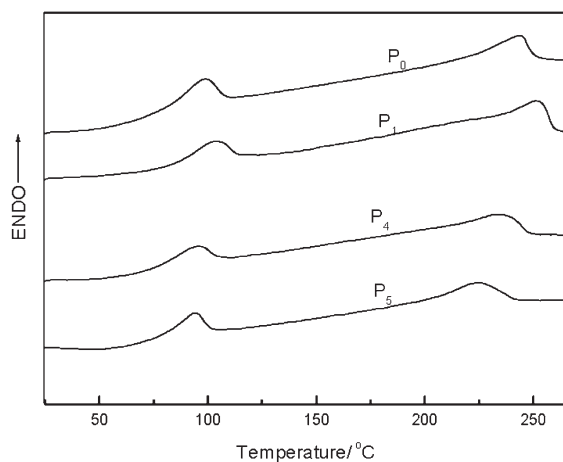
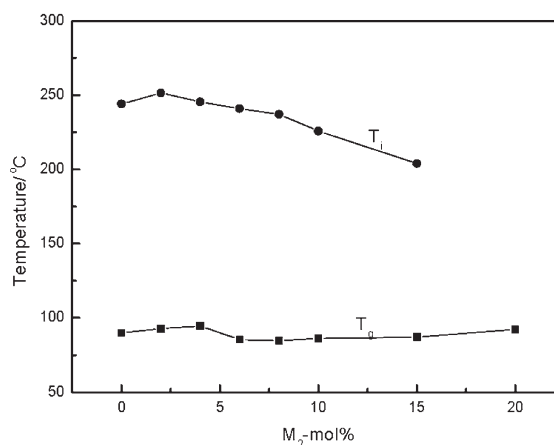


Figure 6. DSC thermographs of liquid crystal elastomers.

Figure 7. Effect of M₂ content on phase transition temperature of the elastomers.

4. Conclusion

In this study, a series of new side chain smectic LCEs containing nematic monomer groups (M₁: 4'-allyloxybiphenyl-4-yl 4-heptyloxybenzoate) and chiral crosslinking agent (M₂: isosorbide-undecanoyloxybisate) were synthesized and characterized. All of the polymers obtained showed very wide mesophase

temperature ranges and high thermal stability. Elastomers containing less than 15% of crosslinking units showed elasticity and reversible phase transitions. The liquid crystalline phase of the homopolymer and elastomers changed from SmB to SmA and then to SmC* with increasing content of the chiral crosslinker. For polymers P₀–P₇, the glass transition temperatures first increased, then decreased and

Table 3. Thermal properties of polymers P₀–P₇.

Polymer	$T_g/^\circ\text{C}$	$T_i/^\circ\text{C}$	$\Delta H/\text{J g}^{-1}$	ΔT^a	$T_d^b/^\circ\text{C}$
P ₀	90.0	244.0	3.20	154.0	359.6
P ₁	92.9	251.4	3.96	158.5	363.8
P ₂	94.7	245.5	3.88	150.8	356.7
P ₃	85.5	240.9	2.38	155.4	349.7
P ₄	85.3	237.1	2.75	152.4	357.9
P ₅	86.4	225.8	2.42	139.4	353.9
P ₆	87.1	204.0	2.55	116.9	354.0
P ₇	92.3	—	—	—	347.1

^aMesophase temperature ranges ($T_i - T_g$). ^bTemperature at which a 2% weight loss occurred.

finally increased again with increasing content of crosslinking agent; meanwhile the isotropization temperatures and mesophase temperature ranges decreased.

Acknowledgements

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