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Side chain cholesteric liquid crystalline elastomers: synthesis and phase behaviour

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A series of new side chain cholesteric liquid crystalline elastomers (**P-2–P-6**) containing the nematic crosslinking monomer 4-(10-undecen-1-yloyloxy)benzoyl-4′-allyloxybenzoyl-*p*-benzenediol bisate (**M-1**) and the cholesteric monomer 4-cholesteryl 4-(10-undecen-1-yloyloxy)benzoate (**M-2**) were synthesized. The chemical structures of the monomers and elastomers obtained were confirmed by FTIR and ^{1}H NMR spectroscopy. Their liquid crystalline properties and phase behaviour were investigated by differential scanning calorimetry, polarizing optical microscopy and X-ray diffraction. The effect of the crosslinking units on phase behaviour is discussed. Elastomers containing less than 20 mol% of the crosslinking units showed elasticity, reversible phase transitions and cholesteric Grandjean texture. The experimental results demonstrated that the glass transition and isotropization temperatures of **P-2**–**P-6** increased with the increasing concentration of crosslinking unit **M-1**.

1. Introduction

Present day materials science is directed towards the development of multifunctional and oriented structures. One example of such supramolecular systems is the liquid crystalline networks (LCN) where research into materials with anisotropic properties has recently expanded rapidly [1–10]. Networks with cholesteric structures have attracted special interest, because of their unique properties and potential application in, for example, flat-panel displays, non-linear optics and organic pigment [11–14]. The unique properties of cholesteric liquid crystals are related to the helical supramolecular structure. According to the density of crosslinking, two new classes of cholesteric LCNs emerge: (i) highly crosslinked cholesteric liquid crystalline thermosets (ChLCTs); and (ii) lightly crosslinked cholesteric liquid crystalline elastomers (ChLCEs).

In the last decade, ChLCTs have been extensively investigated because the helical structure of the cholesteric phase is permanently fixed and optical properties become temperature independent [15–20]. In contrast to ChLCTs, ChLCEs combine the basic features of polymer elastomers with the anisotropy of physical properties of cholesteric liquid crystals. Consequently, ChLCEs not only hold the entropic elasticity, but also show reversible phase transitions between liquid crystalline and isotropic phases on heating or cooling. Compared with conventional LCEs, ChLCEs reveal unusual piezoelectricity, besides electro-optical and mechanical properties due to deformation of the helical structure of the cholesteric phase.

Recent theoretical and experimental studies have suggested that piezoelectricity can be obtained from ChLCEs [21–23]. They have the potential to act as a device that transforms a mechanical signal into an optical signal when stress is applied parallel to the cholesteric helix [24]. ChLCEs are therefore considered as a candidate for piezoelectric devices. However, to the best of our knowledge, research on ChLCEs is little reported [21–25]. It has therefore been necessary to synthesize various kinds of side chain ChLCEs in order to explore their potential applications.

In this study, we report the synthesis of side chain ChLCEs derived from 4-(10-undecen-1-yloyloxy)-benzoyl-4′-allyloxybenzoyl-*p*-benzenediol bisate (**M-1**) and 4-cholesteryl 4-(10-undecen-1-yloyloxy)benzoate (**M-2**). Their mesomorphic properties and phase behaviour of the monomers and elastomers were characterized by differential scanning calorimetry (DSC), polarizing optical microscopy (POM) and X-ray diffraction (XRD). The effect of the concentration of crosslinking units on phase behaviour is discussed in detail. The elasticity and piezoelectricity of the ChLCEs obtained are under current investigation.

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2. Experimental

2.1. *Materials*

Polymethylhydrosiloxane (PMHS $M_n = 700–800$) was purchased from Jilin Chemical Industry Co. Undecylenic acid was purchased from Beijing Jinlong Chemical Reagent Co., Ltd, and cholesterol from Henan Xiayi Medical Co. Toluene used in the hydrosilylation reaction was first heated under reflux over sodium and then distilled. All other solvents and reagents were purified by standard methods.

2.2. *Monomers synthesis*

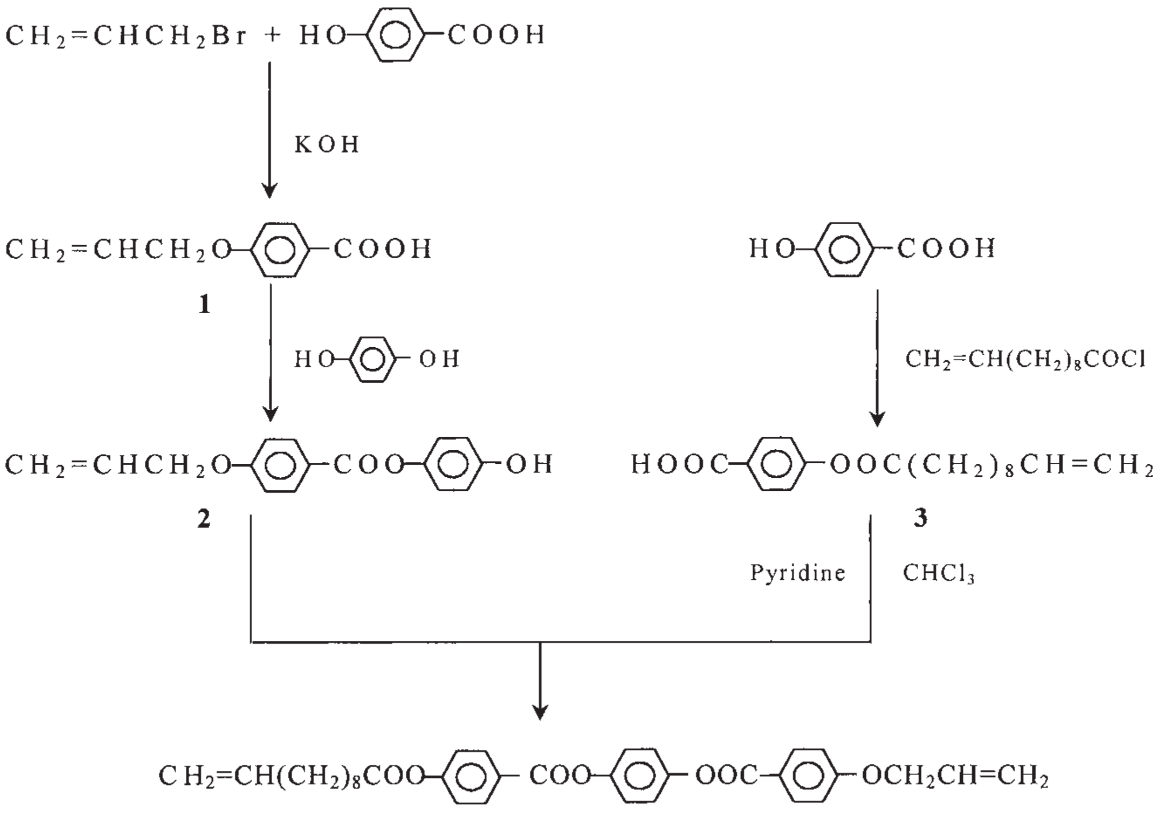
The synthetic route to the olefinic monomers is shown in schemes 1 and 2. The synthesis of 4-hydroxyphenyl 4-allyloxybenzoate **2** and 4-(10-undecen-1-yloyloxy)benzoic acid **3** was carried out according to the procedure reported by Hu et al. [26, 27].

2.2.1. *4-(10-Undecen-1-yloyloxy)benzoyl-4′-allyloxybenzoyl-p-benzenediol bisate (**M-1**)*

4-(10-Undecen-1-yloyloxy)benzoyl chloride (12.9 g, 0.04 mol) was added dropwise to a mixture of 4-hydroxyphenyl 4-allyloxybenzoate **2** (10.8 g, 0.04 mol) and 3.2 ml of pyridine in 50 ml of chloroform at 0–5°C. After stirring at room temperature for 6 h, ethanol was added and the precipitate recrystallized from ethanol; yield 56%, m.p. 98°C. IR (KBr): 1733 (C=O); 1641 (C=C); 1605, 1510 (Ar); 1255, 1162 (ether) cm^{-1}. ^{1}H NMR (CDCl$_3$, TMS) δ ppm: 1.34 [s, 10H, –(C$\underline{H}_2$)$_5$–]; 1.61–2.07 (m, 4H, CH$_2$–CHC$\underline{H}_2$–, –C$\underline{H}_2$CH$_2$COO–); 2.58–2.63 (t, 2H, –C$\underline{H}_2$COO–); 4.63 (t, 2H, –OC$\underline{H}_2$–); 4.93–5.04 [t, 2H, C$\underline{H}_2$–CH(CH$_2$)$_8$–]; 5.33–5.49 [t, 2H, C$\underline{H}_2$–CHCH$_2$O–]; 6.03–6.11 [m, 2H, CH$_2$–C$\underline{H}$(CH$_2$)$_8$–, CH$_2$–C$\underline{H}$CH$_2$O–], 6.99–8.26 (m, 12H, Ar–$\underline{H}$).

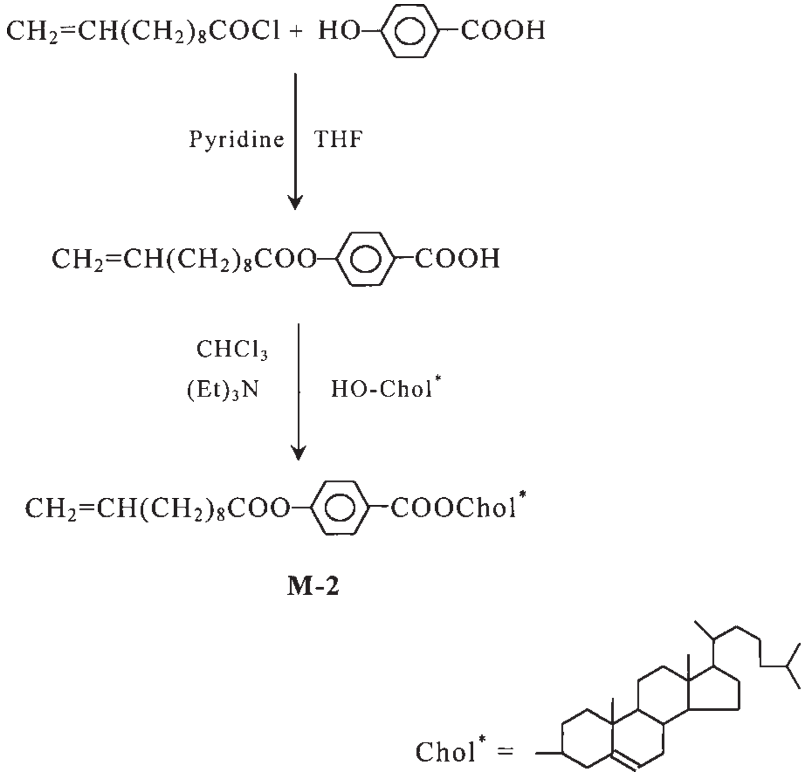
2.2.2. *4-Cholesteryl 4-(10-undecen-1-yloyloxy)benzoate (**M-2**)*

4-(10-Undecen-1-yloyloxy)benzoyl chloride (6.45 g, 0.02 mol) was added dropwise to a cold solution of cholesterol (7.74 g, 0.02 mol) and 1.6 ml of pyridine in 30 ml of dichloromethane. The reaction mixture was heated under reflux for 10 h, and, after filtration a crude product precipitated by adding ethanol to the filtrate. This was recrystallized from toluene; yield 46%, m.p.



M-1

Scheme 1. Synthesis of crosslinking monomer M-1.

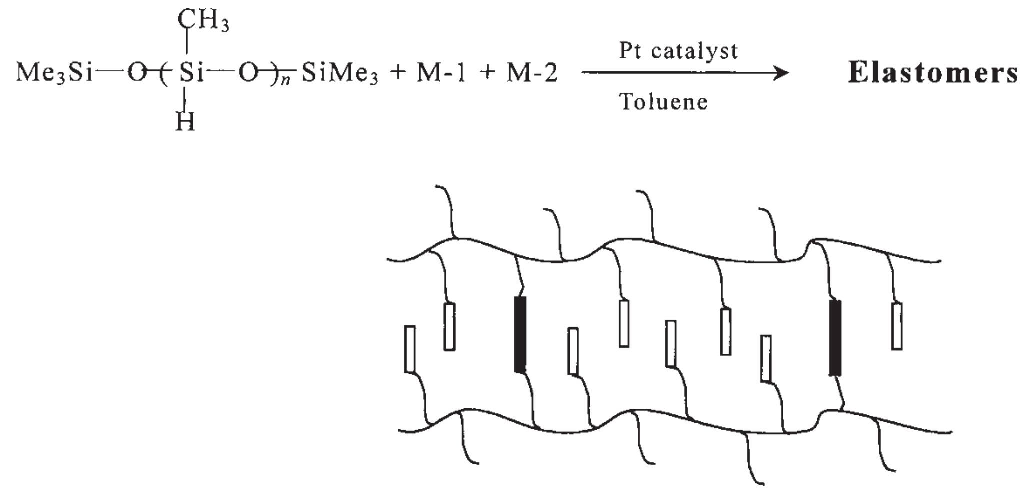


Scheme 2. Synthesis of cholesteric monomer M-2.

107°C. IR (KBr): 1767, 1716 ($C=O$); 1635 ($C=C$); 1603, 1504 (Ar) cm^{-1}. ^{1}H NMR ($CDCl_3$, TMS) δ ppm: 0.61–2.72 (m, 59H, $–(C\underline{H}_2)_8–$, cholesteryl–H); 4.65–5.10 (m, 3H, $C\underline{H}_2–CH$, $–COO–C\underline{H}–$); 5.50 (d, 1H, $–C\underline{H}–$ in cholesteryl), 6.08 (m, 1H, $–C\underline{H}–$), 7.05–8.15 (m, 4H, Ar–$\underline{H}$).

2.3. *Elastomers synthesis*

The synthesis of the polymers was performed as in indicated scheme 3. The same method was adopted for the synthesis of all polymers **P-1**–**P-7**. The synthesis of elastomer **P-4** is given as an example. Monomers **M-1**, **M-2**, and **PMHS** were dissolved in dry toluene. The reaction mixture was heated to 60°C under nitrogen, and then an appropriate amount of a THF solution of hexchloroplatinate hydrate catalyst was injected with a syringe. After holding the reaction mixture at 60°C for 24 h, the polymer was obtained by precipitation with methanol, and then dried under vacuum. IR (KBr):



Scheme 3. Synthesis and schematic representation of elastomers; (▭ mesogenic unit; ▬ crosslinking unit).

1747, 1708 (C=O); 1602, 1501(Ar); 1200–1000 (Si–O–Si) cm^{-1}.

2.4. *Characterization*

Fourier transform infrared (FTIR) spectra were obtained on a Nicolet 510 FTIR spectrometer (Nicolet Instruments, Madison, WI, USA). ^{1}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 using a Perkin-Elmer DSC-7 instrument (Perkin-Elmer, Foster City, CA) equipped with a liquid nitrogen cooling system. Heating and cooling rates were 20°C min^{-1}. Phase transition temperatures were observed during the second heating and first cooling scans. A Leitz Microphot-FX (Leitz, Wetzlar, Germany) polarizing optical microscope equipped with a Mettler FP 82 hot stage and FP 80 central processor was used to observe phase transition temperatures and analyse mesomorphic properties through the observation of optical texture. XRD measurements were performed with a nickel-filtered Cu-K$_\alpha$ radiation with a DMAX-3A Rigaku powder diffractometer.

3. Results and discussion

3.1. *Synthesis*

The synthetic routes for the target monomers and polymers are shown in schemes 1, 2 and 3.

The liquid crystalline elastomers were prepared by a one-step hydrosilylation reaction between the Si–H groups of PMHS and the olefinic C=C of a cholesteric monomer and difunctional nematic crosslinking agent in toluene, using hexchloroplatinate hydrate as catalyst at 60°C. Yields and polymerization details are summarized in table 1. The network polymers obtained were in the gel form and insoluble in toluene; they took the form of a rubber state. The FTIR spectra of the elastomers showed the complete disappearance of the Si–H stretching band at 2166 cm^{-1} and the olefinic C=C stretching band at about 1641 cm^{-1}. Characteristic absorption bands appeared at 1200–1000cm^{-1}, corresponding to Si–O–Si stretching.

Table 1. Polymerization and yield for P-1–P-7.

Polymer	Feed PMHS mmol	Feed M-1 mmol	Feed M-2 mmol	M-1[a] mol%	Yield %
P-1	1	0.000	7.000	0	93
P-2	1	0.137	6.725	2	88
P-3	1	0.269	6.462	4	91
P-4	1	0.396	6.204	6	89
P-5	1	0.636	5.724	10	90
P-6	1	0.913	5.174	15	87
P-7	1	1.167	4.668	20	91

[a] Molar fraction of monomer M-1 based on M-1+M-2.

3.2. *Thermal analysis*

The phase transition temperatures and corresponding enthalpy changes of the liquid crystalline monomers **M-1**, **M-2** and polymers **P-1**–**P-7** are listed in tables 2 and 3, respectively. Representative DSC traces for **M-1**, **M-2** and **P-1** are presented in figures 1 and 2.

DSC heating thermograms of **M-1** and **M-2**, showed two endothermic peaks representing melting transitions at 98.9 (M-1) and 110.0°C (M2), and liquid crystalline phase to isotropic phase transitions at 208.9 (M-1) and 195.5°C (M2). On the cooling scans, isotropic to liquid crystalline phase transitions appeared at 197.8 and 185.5°C as well as crystallization temperatures at 71.6 and 69.4°C, respectively.

DSC curves obtained for **P-1**–**P-6** revealed a glass transition at low temperature and mesomorphic phase to isotropic transitions at high temperature. As can be seen from the data in table 3, light chemical crosslinking did not remarkably influence the phase behaviour of the liquid crystalline elastomers; reversible phase transitions were observed because of adequate

Table 2. Thermal transitions of the monomers M-1 and M-2. Cr = solid, N = nematic, Ch = cholesteric, I = isotropic.

Monomers	Transition temperature/°C (Corresponding enthalpy changes/J g^{-1}) $\frac{\text{Heating}}{\text{Cooling}}$	Yields %	ΔT^a
M-1	$\frac{\text{Cr98.9(39.46)N208.9(1.98)I}}{\text{I197.8(0.69)N71.6(22.94)Cr}}$	56	110.0
M-2	$\frac{\text{Cr110.0(20.64)Ch195.5(1.82)I}}{\text{I185.5(0.54)Ch69.4(19.16)Cr}}$	46	85.5

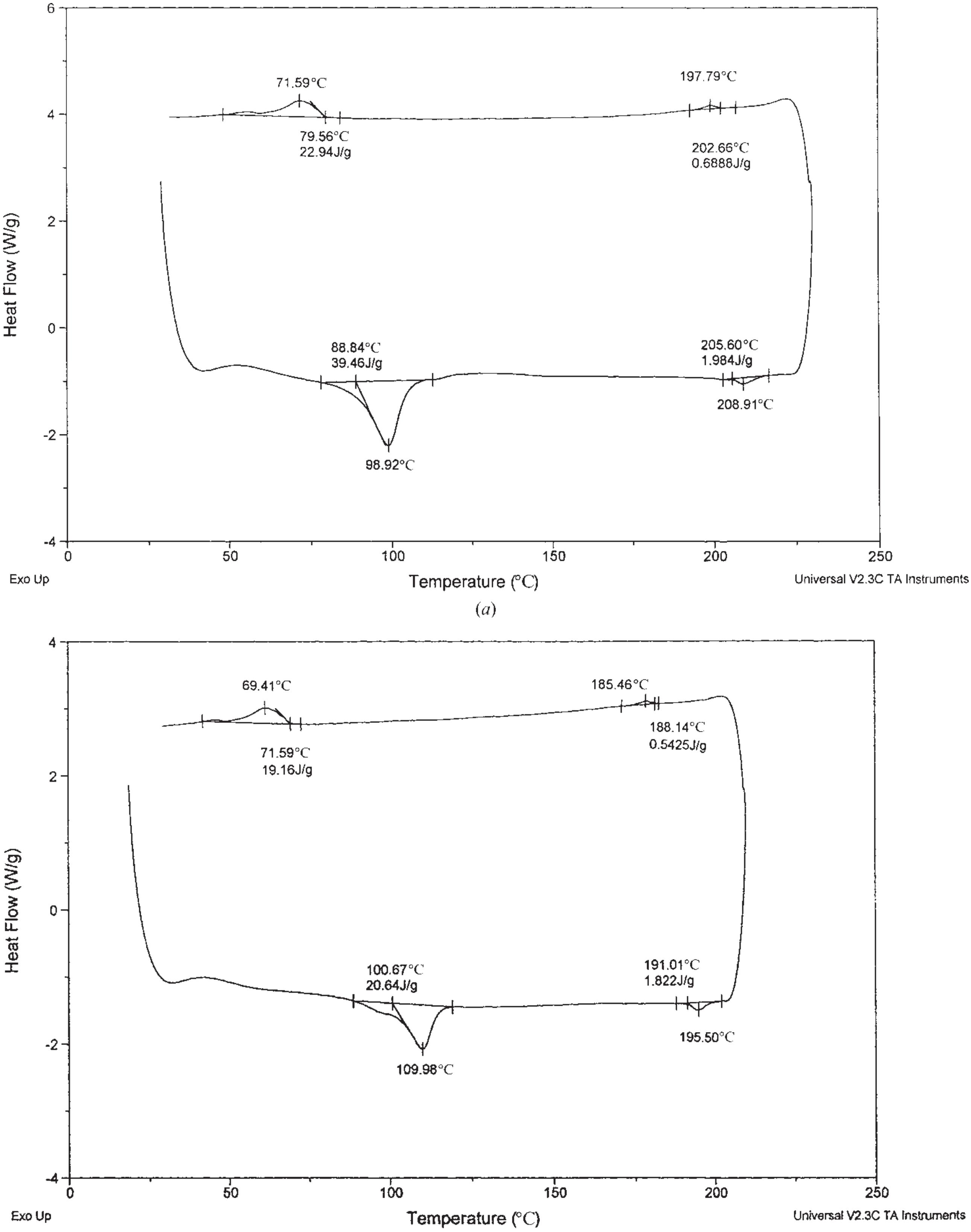
[a] Mesomorphic temperature ranges; peak temperatures were taken as phase transition temperatures.

Table 3. DSC and POM result for polymers P-1–P-7.

Polymer	DSC T_g °C	T_i °C	ΔH/J g^{-1}	ΔT^a	POM T_{cl}^b/°C	T_{lc}^c/°C
P-1	38.6	233.6	1.73	195.0	241	232
P-2	30.0	229.2	3.89	199.2	234	233
P-3	30.4	228.3	1.72	197.9	236	230
P-4	32.6	232.4	1.54	199.8	239	232
P-5	39.2	240.6	1.42	201.4	244	240
P-6	40.7	245.8	0.71	205.1	255	247
P-7	46.5	—	—	—	—	—

[a] Mesomorphic temperature ranges (T_i-T_g).

[b] Temperature at which the birefringence disappeared completely.



(*a*)

(*b*)

Figure 1. DSC thermograms of monomers: (*a*) M-1, (*b*) M-2.

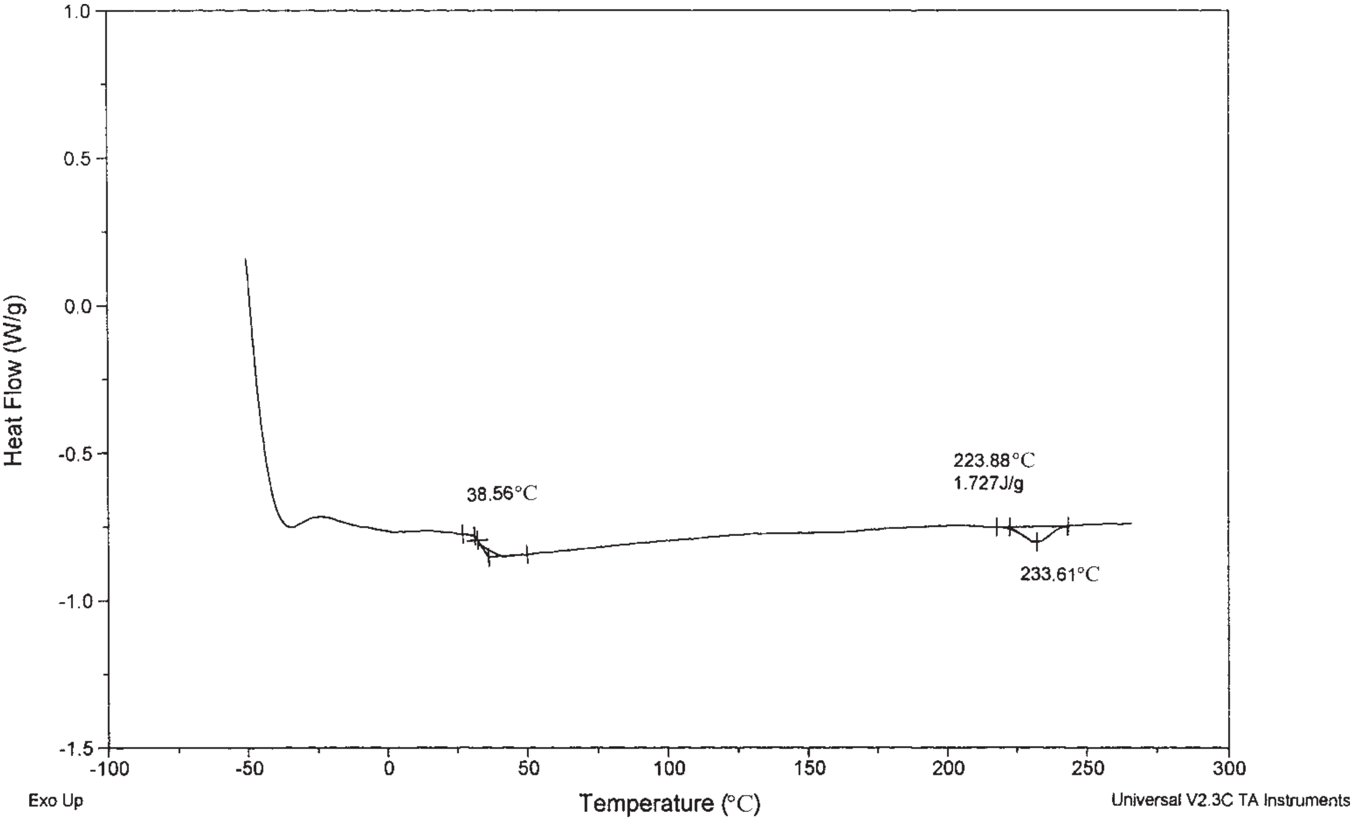


Figure 2. DSC thermograms of polymer P-1.

molecular motion and ready mesogenic orientation. In contrast, higher chemical crosslinking had a strong effect on phase behaviour; it could cause the mesomorphic phase to disappear due to the depression of liquid crystalline order. Consequently, the DSC curve of P-7 shows only a glass transition; no liquid crystalline to isotropic phase transition is seen. Above all, the phase transitions are reversible and do not change on repeated heating and cooling cycles; transition temperatures obtained by DSC agree with those observed by POM.

The glass transition temperature (T_g) is an important parameter in connection with structures and properties. For side chain ChLCEs, T_g is influenced by the nature of the polymer backbone, the rigidity of the mesogenic group, the length of the flexible spacer and crosslinking density. In general, chemical crosslinking imposes additional constraints on the motion of chain segments and increases T_g, but the effect may be small for lightly crosslinked polymers. T_g can also be affected by flexible crosslinking chains similar to the plasticization. Compared with the precursor polymer **P-1**, the T_g of lightly crosslinked polymers may fall. Figure 3 shows the effect of the concentration of the crosslinking unit **M-1** on the phase transition temperatures of **P-1**–**P-7**. It can be seen that the T_g of **P-2**–**P-4** was lower than that of **P-1**; however, the general tendency is toward increased T_g with increasing crosslinking density. For **P-2**–**P-7**, when

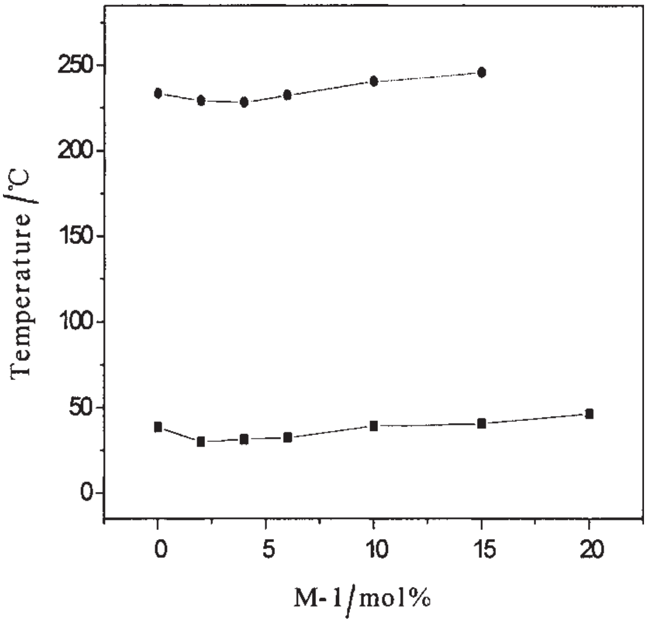


Figure 3. Effect of M-1 concentration on phase transition temperatures of elastomers: ■ = T_g; ● = T_I.

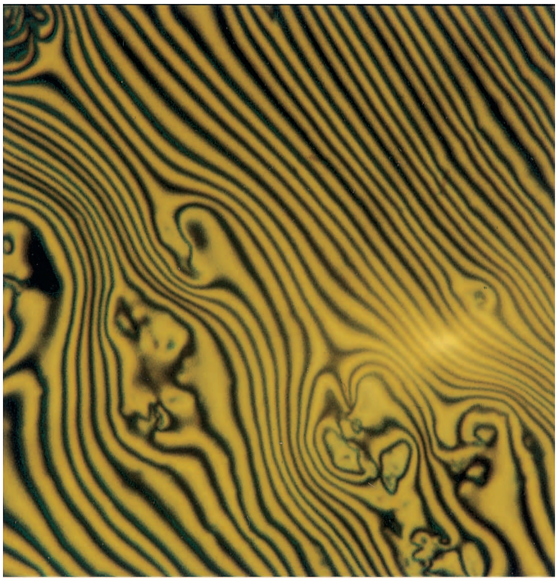
the concentration of the crosslinking unit **M-1** increased from 2 to 20 mol%, T_g increased from 30.0 to 46.5°C.

The flexible mesogenic crosslinking units influenced the isotropization temperature (T_i) in two regards, for lightly crosslinked polymers. On one hand, the flexible crosslinking chains acted as a diluent, and led to a decrease in T_i. On the other hand, the rigid mesogenic core of crosslinking unit **M-1** promoted the formation of a mesophase in the network polymers, and led to an increase in T_i. According to table 3, T_i of **P-2** and **P-3** decreased by 4.4 and 5.3°C compared with **P-1**. For **P-3**–**P-6**, when the concentration of crosslinking unit **M-1** increased from 4 to 15 mol%, T_i increased from 228.3 to 245.8°C. The enthalpy changes (ΔH) decreased from 3.89 J g^{-1} for **P-2** to 0.71 J g^{-1} for **P-6**. When the concentration of crosslinking unit **M-1** was greater than 15 mol%, the liquid crystalline properties of the network polymers disappeared. In addition, as the concentration of crosslinking unit **M-1** increased, the mesomorphic temperature ranges (ΔT) of the liquid crystalline elastomers also increased.

3.3. *Optical micrograph analysis*

POM observation of the monomers **M-1** and **M-2** revealed enantiotropic nematic and cholesteric phases. When the crosslinking agent **M-1** was heated to about 94°C, typical nematic schlieren texture appeared, and birefringence disappeared at 216°C. When the isotropic phase was cooled to 215°C, a nematic droplet texture appeared and the liquid crystalline phase began to crystallize at 87°C. Photomicrographs of **M-1** are shown in figures 4 (*a*) and 4 (*b*). When mesogenic monomer **M-2** was heated to 104°C, a typical cholesteric oily-streak texture and reflection colour appeared at 157°C. Selective reflection of light shifted to shorter wavelengths and the reflection colour changed through yellow, green, blue to violet with increasing temperature. Cholesteric texture and reflection colour disappeared at 202°C. On cooling the sample from the isotropic melt, a focal-conic texture was formed, which was easily transformed to an oily-steak texture by shearing. The reflection colour disappeared and a broken focal-conic texture appeared at 152°C; the sample crystallized at 82°C. Photomicrographs of **M-2** are shown in figures 5 (*a*) and 5 (*b*).

During heating and cooling cycles, the precursor polymer **P-1** exhibited cholesteric Grandjean and oily-streak textures; elastomers **P-2**–**P-6** exhibited cholesteric Grandjean texture. A photomicrographs of **P-3** is shown in figure 6 as an example. **P-7** showed only elasticity with no other texture, this is consistent with the results obtained by DSC. Thus, liquid crystalline phase type and texture did not change when the concentration of crosslinking unit was less than 15 mol% for **P-1**–**P-6**.


(*a*)

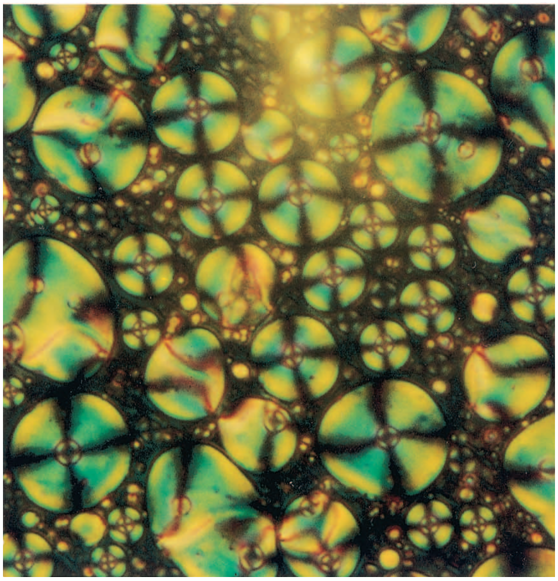

(*b*)

Figure 4. Optical polarizing micrographs of monomer M-1 (200×): (*a*) schlieren texture at 188°C; (*b*) droplet texture at 213°C.

3.4. *X-ray diffraction analysis*

X-ray diffraction studies were carried out to obtained more detailed information on the liquid crystalline phase structures. In general, a sharp and strong peak at low angle ($1° < 2\theta < 4°$) appears in small angle X-ray scattering (SAXS) curves only for the smectic liquid

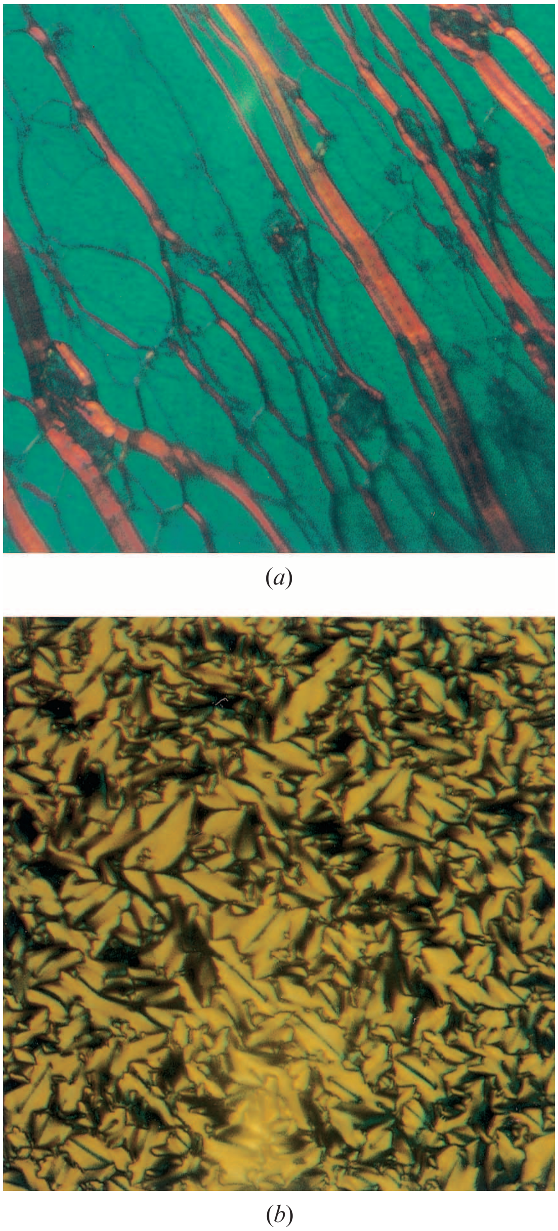


Figure 5. Optical polarizing micrographs of monomer M-2 (200×): (*a*) oily-streak texture at 172°C; (*b*) focal-conic texture at 180°C.

crystalline phase; a broad peak associated with lateral packing at $2\theta=18$–$21°$ is observed in wide angle (WAXD) curves for nematic, smectic and cholesteric liquid crystalline phases. Figure 7 shows XRD curves of quenched samples. A sharp peak associated with the smectic layers at low angle did not appear in the SAXS curves; a broad peak associated with lateral packing at $2\theta\approx20°$ occurred in WAXD curves. Thus, the cholesteric phase structure of **P-1**–**P-6** was confirmed by DSC, POM and XRD results.

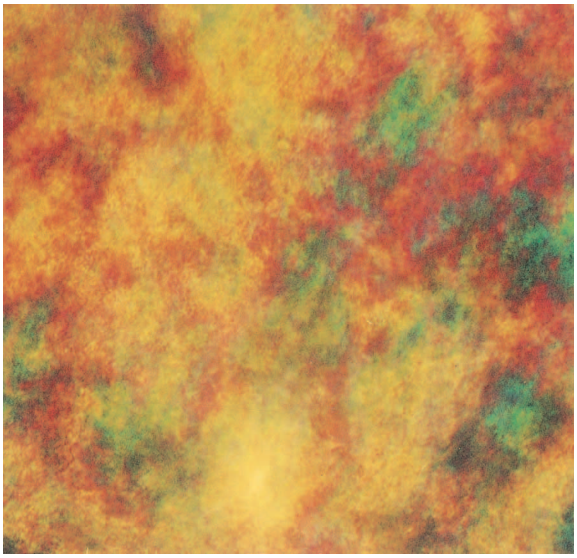


Figure 6. Optical texture of elastomer P-3 at 194°C (200×).

4. Conclusions

In this paper, the preparation of side chain cholesteric liquid crystalline elastomers containing both 4-(10-undecen-1-yloyloxybenzoyl-4′-allyloxybenzoyl-*p*-benzenediol bisate and 4-cholesteryl 4-(10-undecen-1-yloyloxy)benzoate is described. The monomer **M-1** exhibited nematic schlieren and droplet textures, and M-2 displayed cholesteric oily-streak and focal-conic textures. Compared with uncrosslinked polymer, light chemical crosslinking did not markedly influence the

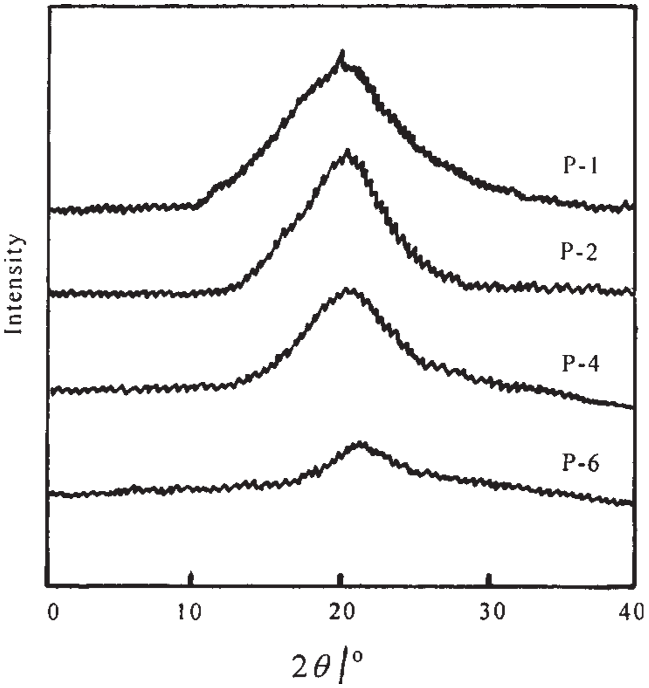


Figure 7. X-ray diffractograms of quenched samples.

phase behaviour of the liquid crystalline elastomers. Elastomers containing less than 20 mol% of crosslinking units revealed elasticity, reversible phase transitions and cholesteric texture on heating or cooling. The use of a flexible nematic divinyl monomer as crosslinking agent can promote the formation of mesophases in lightly crosslinked polymers. For elastomers **P-2–P-6**, glass transition and isotropization temperatures increased with increasing concentration of crosslinking units.

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