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Preparation and characterization of side chain cholesteric liquid crystalline polysiloxanes containing two chiral groups

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Preparation and characterization of side chain cholesteric liquid crystalline polysiloxanes containing two chiral groups

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A series of liquid crystalline (LC) polysiloxanes containing diosgeninyl and menthyl groups (from monomers $\mathbf{M}_1$ and $\mathbf{M}_2$, respectively) were synthesized. The chemical structures of the monomers and polymers obtained were confirmed by elemental analysis, Fourier transform infrared spectroscopy, proton NMR and carbon-13 NMR. The LC properties were investigated by differential scanning calorimetry, thermogravimetric analysis, polarizing optical microscopy, and X-ray diffraction. Monomer $\mathbf{M}_1$ showed cholesteric oily-streak and spiral textures. Copolymers $\mathbf{P}_2$–$\mathbf{P}_5$ exhibited cholesteric phases. With increasing concentration of $\mathbf{M}_2$ units, the glass transition and clearing temperatures decreased. Experimental results demonstrated that a flexible polymer backbone and a long flexible spacer tended to favour a lower glass transition temperature, higher thermal stability, and wider mesophase temperature range.

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1. Introduction

In recent years, there has been increasing interest in cholesteric liquid crystalline polymers (LCPs) due to their unique optical properties, including selective reflection of light, thermochromism and circular dichroisms. This has led to advanced applications in, for example, non-linear optical devices, full colour thermal imaging and organic pigments [1–9]. The cholesteric phase is formed by rod-like, chiral molecules responsible for macroscopic alignment of cholesteric domains, depending on chemical structures. Many novel side chain cholesteric LC materials have recently been reported, and work continues in extending the range of these materials and exploring potential applications [10–17].

In comb-like polymers, the mesogenic properties of side chain LCPs depend mainly on the nature of the polymer backbone, the mesogen type, the length of the flexible spacer, and the nature of terminal groups. The polymer backbones of side chain LCPs are primarily polyacrylates, polymethacrylates and polysiloxanes; however, polyacrylates and polymethacrylates, because of the form of their backbones, show higher glass transition temperatures (T_g) and higher viscosity. To obtain higher mobility of the mesophase, and mesogenic properties at moderate temperatures, the polysiloxane backbone with a flexible spacer is usually chosen.

This article, describes the synthesis of a series of LCPs derived from diosgeninyl 4-(10-undecylen-1-yloxy)-benzoate ($\mathbf{M}_1$) and menthyl 4-(10-undecylen-1-yloxy)-benzoate ($\mathbf{M}_2$). The structures and mesogenic properties of the monomers and polymers obtained were characterized by elemental analysis (EA), Fourier transform infrared spectroscopy (FTIR), proton nuclear magnetic resonance (^{1}H NMR), carbon-13 nuclear magnetic resonance (^{13}C NMR), polarizing optical microscopy (POM), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and X-ray diffraction (XRD).

2. Experimental

2.1. *Materials*

Polymethylhydrosiloxane (PMHS, $\bar{M}_n = 700-800$) was purchased from Jilin Chemical Industry Co. Diosgenin was purchased from Wuhan Ruixin Chemical Co, and menthol from Shanghai Kabo Chemical Co. Hexachloroplatinate(IV) catalyst was obtained from Shenyang Chemical Reagent Co. Toluene used in the hydrosilylation reaction was heated under reflux over sodium and then distilled under nitrogen. All other solvents and reagents were purified by standard methods.

2.2. *Characterization*

FTIR spectra were measured on a Perkin-Elmer Spectrum One (B) spectrometer (Perkin-Elmer, Foster

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City, CA). Quantitative elemental analyses were carried out using an Elementar Vario EL III system (Elementar, Germany). ^{1}H NMR (300 MHz) and ^{13}C NMR (75.4 MHz) spectra were obtained with a Varian Gemini 300 spectrometer (Varian Associates, Palo Alto, CA). Optical rotations were obtained on a Perkin-Elmer 341 polarimeter.

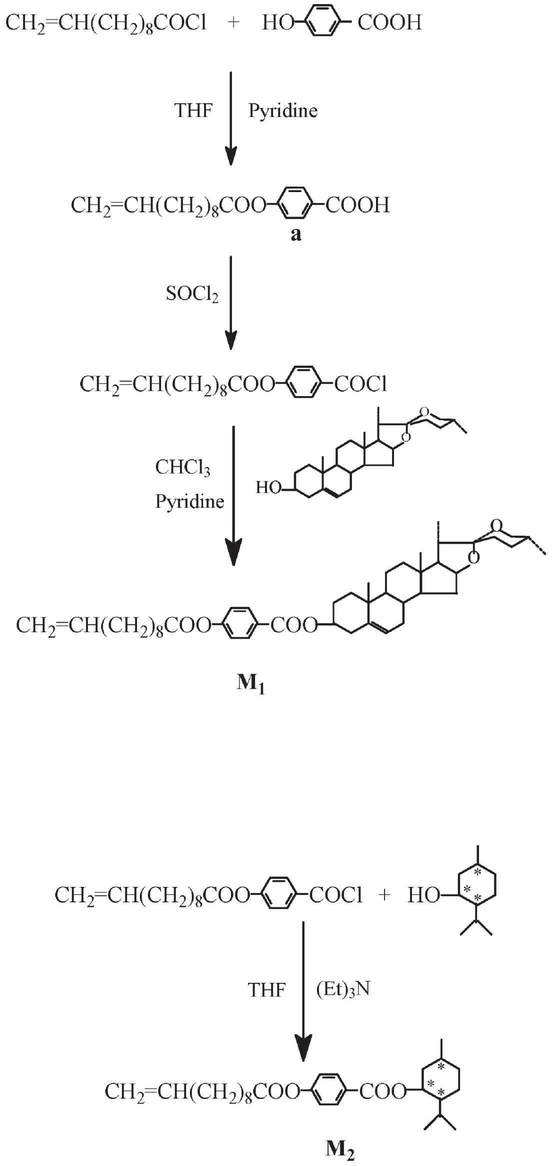
Phase transition temperatures and thermodynamic parameters were determined using a Netzsch DSC 204 (Netzsch, Germany) instrument equipped with a liquid nitrogen cooling system; heating and cooling rates were 10°C min^{-1}. The thermal stability of the polymers in air was measured with a Netzsch TGA 209C thermogravimetric analyser. 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 analyse LC properties for the monomers and polymers through the observation of optical textures. XRD measurements were performed with nickel-filtered Cu-K$_\alpha$ (λ=1.542 Å) radiation using a DMAX-3A Rigaku (Rigaku, Japan) powder diffractometer.

2.3. *Monomer synthesis*

The synthetic route to the olefinic monomers is shown in scheme 1. The starting materials 4-(10-undecylen-1-yloxy)benzoic acid **a** and its acid chloride were prepared according to literature methods [18].

2.3.1. Diosgeninyl 4-(10-undecylen-1-yloxy)benzoate (M$_1$).

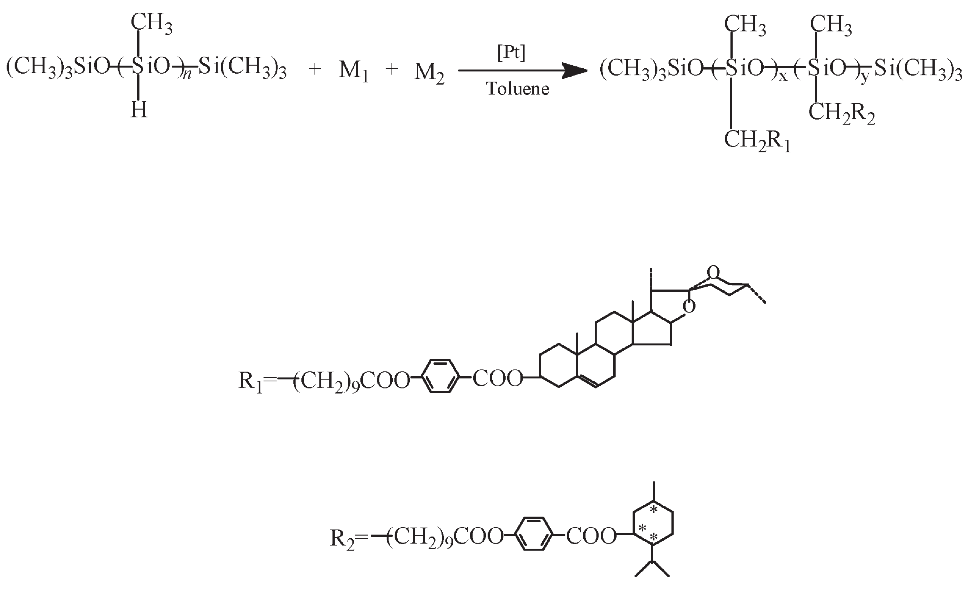
4-(10-Undecylen-1-yloxy)benzoyl chloride (16.2 g, 0.05 mol) was added dropwise to a cold solution of diosgenin (20.7 g, 0.05 mol) in chloroform (100 ml) and pyridine (4 ml). The reaction mixture was heated under reflux for 13 h. Excess solvent and the precipitate were removed, the crude product was then obtained by adding ethanol to the filtrate. It was recrystallized from ethanol to give a white solid; yield 59%, m.p. 116°C, $[\alpha]_D^{28}$ −33.9° (c=0.769, toluene). IR (KBr, cm^{-1}): 3073 (=C−H); 2927, 2854 (–CH$_3$, –CH$_2$–); 1763, 1712 (C=O); 1641 (C=C); 1604, 1457 (Ar–). Elemental analysis: calcd for $C_{45}H_{64}O_6$, C 77.10, H 9.20; found, C 76.91, H 9.28%. ^{1}H NMR (CDCl$_3$, TMS) δ ppm: 0.78–2.56 (m, 52H, –(CH_2)$_8$– and diosgeninyl–H); 3.33–3.51 (m, 3H, $>$CHO– and –OCH_2– in diosgeninyl); 4.40 (m, 1H, –ArCOOC$H$$<$); 4.87 (d, 2H, C$H_2$=CH–); 5.42 (d, 1H, =C$H$– in diosgeninyl), 5.82 (m, 1H, =CH–); 7.13–8.08 (m, 4H, Ar–H). ^{13}C NMR (CDCl$_3$, TMS, δ, ppm): 6.6, 15.6, 20.1, 20.4 (methyl–C); 21.3, 23.8, 25.3, 27.5, 30.1, 30.3, 30.6, 30.9, 31.7, 32.5, 32.9, 33.1, 33.2, 33.5, 34.1, 38.9, 68.1 (methylene–C); 28.8, 30.2, 35.5, 41.1, 43.5, 47.4, 67.2, 76.8 (tertiary C in diosgeninyl); 120.8, 129.6 (aromatic tertiary C); 35.3, 39.7, 112.3 (quaternary C in diosgeninyl); 127.7, 158.1 (aromatic quaternary C); 113.2 (CH$_2$=); 141.5 (=CH–); 122.4 (=CH– in diosgeninyl); 148.1 ($>$C= in diosgeninyl); 166.3, 170.4 (C=O).



Scheme 1. Synthesis of the LC monomers.

2.3.2. Menthyl 4-(10-undecylen-1-yloxy)benzoate (M$_2$).

4-(10-Undecylen-1-yloxy)benzoyl chloride (13.0 g, 0.03 mol) was added dropwise to a cold solution of menthol (6.3 g, 0.04 mol) in tetrahydrofuran (50 ml) and triethylamine (4.2 ml). After heating under reflux for 10 h, the reaction mixture was poured into excess water. The crude product was obtained by filtration, and



Scheme 2. Synthesis of the polysiloxanes.

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washed with water to give white crystals; yield 61%, m.p. 62°C, $[\alpha]_D^{28}$ $-1.2°$ (c=0.667, toluene). IR (KBr, cm^{-1}): 3070 (=C–H); 2952, 2855 (–CH_3, –CH_2–); 1764, 1715 (C=O); 1640(C=C); 1603, 1510 (Ar–). Elemental analysis: calcd for $C_{28}H_{42}O_4$, C 75.98, H 9.56; found, C 75.25, H 9.48%. ^1H NMR ($CDCl_3$, TMS) δ ppm: 0.93–2.26 (m, 34H, –$(CH_2)_8$– and menthyl –*H*); 4.09 (m, 1H, –ArCOOC*H*<); 4.98 (d, 2H, CH_2=CH–); 5.82 (m, 1H, =C*H*–); 7.15–7.99 (m, 4H, Ar–*H*). ^{13}C NMR ($CDCl_3$, TMS, δ, ppm): 18.5, 19.3 (methyl–C); 24.8, 29.5, 30.0, 30.5, 33.5, 34.1 (aliphatic methylene –C); 19.8, 32.8, 37.1 (methylene–C in menthyl); 22.4, 24.9, 42.5, 68.2 (tertiary C in menthyl); 120.9, 130.4 (aromatic tertiary C); 127.4, 158.1 (aromatic quaternary C); 112.8 (CH_2=); 141.5 (=CH–); 166.3, 170.4 (C=O).

2.4. *Polymer synthesis*

The synthesis of the polysiloxanes $\mathbf{P}_1-\mathbf{P}_7$ is shown in scheme 2. All were made by similar methods; the synthesis of $\mathbf{P}_2$ may be quoted as typical. Monomers $\mathbf{M}_1$, $\mathbf{M}_2$, and PMHS, (in feed quantities indicated in table 1) were dissolved in toluene. The reaction mixture was heated to 65°C under nitrogen, then 2 ml of a THF solution of hexachloroplatinate(IV) catalyst (5 mg ml^{-1}) was injected with a syringe. The hydrosilylation reaction was completed within 30 h as indicated by IR. The polymers were obtained by precipitation with methanol and dried in vacuum. IR (KBr): 2928–2854 (–CH_3, –CH_2–); 1766, 1717 (C=O); 1605, 1508 (Ar–); 1300–1000 cm^{-1} (Si–O–Si, C–Si and C–O–C).

3. Results and discussion

3.1. *Synthesis*

The monomers and polymers obtained were synthesized according to schemes 1 and 2. $\mathbf{M}_1$ and $\mathbf{M}_2$ were prepared by the reaction of 4-(10-undecylen-1-yloxy)-benzoyl chloride with diosgenin and menthol, respectively. The structures of $\mathbf{M}_1$ and $\mathbf{M}_2$ were characterized by IR, ^1H and ^{13}C NMR; a ^1H NMR spectrum of $\mathbf{M}_1$ is shown in figure 1. The spectra of $\mathbf{M}_1$ and $\mathbf{M}_2$ indicate high purity, which was confirmed by EA.

The polymers were prepared by a hydrosilylation reaction. IR spectra of the polymers showed complete disappearance of the Si−H stretching band at 2166 cm^{-1}. Characteristic Si–O–Si stretching bands appeared at 1300–1000 cm^{-1}. The polymerization, yields and solubility of the polymers are summarized in table 1. All polymers were in powder form, and were soluble in toluene and xylene; they were sparingly soluble in tetrahydrofuran.

Table 1. Polymerization and solubility of polymers $\mathbf{P}_1$–$\mathbf{P}_7$.

	Feed/mmol				Solubility	
Polymer	$\mathbf{M}_1$	$\mathbf{M}_2$	$\mathbf{M}_2^{a}$ /mol%	Yield /%	toluene	THF
$\mathbf{P}_1$	7.00	0.00	0	92	+	−
$\mathbf{P}_2$	6.65	0.35	5	89	+	−
$\mathbf{P}_3$	6.30	0.70	10	88	+	−
$\mathbf{P}_4$	5.60	1.40	20	90	+	−
$\mathbf{P}_5$	3.00	3.50	50	88	+	−
$\mathbf{P}_6$	1.40	5.60	80	91	+	−
$\mathbf{P}_7$	0.00	7.00	100	90	+	−

[a] Molar fraction of $\mathbf{M}_2$ based on ($\mathbf{M}_1$+$\mathbf{M}_2$).

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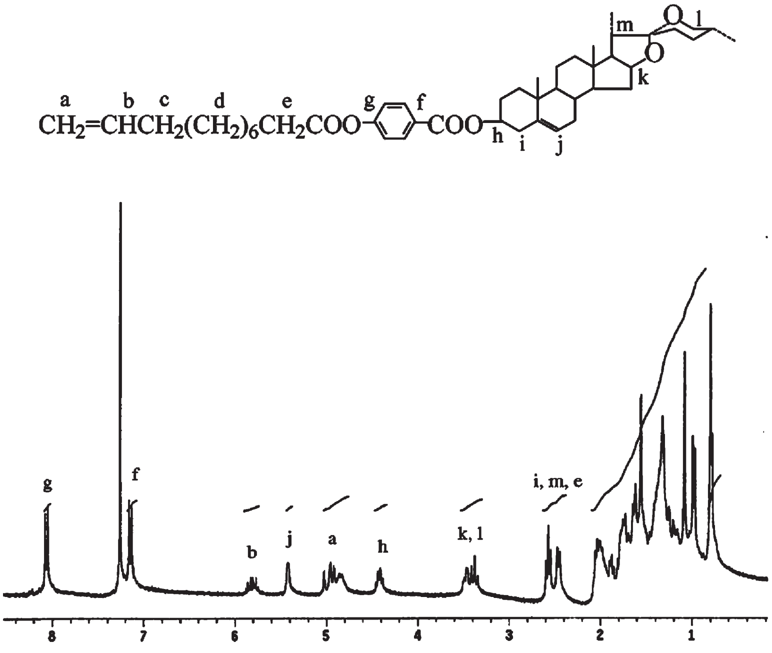

Figure 1. ^{1}H NMR spectra of monomer $\mathbf{M}_1$.

3.2. *Phase behaviour*

The phase transition temperatures and corresponding enthalpy changes of the monomers and polymers, obtained on the second heating and first cooling scans, are summarized in tables 2 and 3, respectively. Representative DSC traces of monomer $\mathbf{M}_1$ and polymers $\mathbf{P}_3$ and $\mathbf{P}_4$ are presented in figures 2 and 3, respectively. All phase transitions were reversible and did not change on repeated heating and cooling cycles. The phase transition temperatures determined by DSC were consistent with POM results.

The DSC heating thermogram of monomer $\mathbf{M}_1$ contained two endothermic peaks, representing a melting transition at 116.3°C and a cholesteric–isotropic phase transition at 156.9°C. On cooling scans of $\mathbf{M}_1$, the isotropic–cholesteric phase transition and crystallization temperatures appeared at 152.1 and 68.2°C, respectively. However, the DSC thermogram of $\mathbf{M}_2$ showed only a melting transition or crystallization transition, which is consistent with POM results. DSC thermograms of polymers $\mathbf{P}_2$–$\mathbf{P}_5$ showed a glass transition at low temperature and a LC–isotropic phase transition at high temperature. However, DSC curves for $\mathbf{P}_1$, $\mathbf{P}_6$ and $\mathbf{P}_7$ showed only glass transitions and no LC-isotropic phase transition. On the other hand, POM results did show LC properties for $\mathbf{P}_1$, but not for $\mathbf{P}_6$ and $\mathbf{P}_7$.

Figure 4 shows the effect of the copolymer composition on phase behaviour of the LCPs. With increasing concentration of $\mathbf{M}_2$ units, T_g and the clearing temperature (T_i) of the polymers fell. For side chain LCPs, T_g is affected by the nature of the polymer backbone, the rigidity of the mesogenic groups, the length of the flexible spacer, and the copolymer

Table 2. Phase transition temperatures of the monomers. Cr=solid, Ch=cholesteric, I=isotropic: peak temperatures were taken as phase transition temperatures.

Monomer	Transition temperature/°C (Corresponding enthalpy changes/J g^{-1}) $\frac{\text{Heating}}{\text{Cooling}}$	ΔT^{a}
$\mathbf{M}_1$	$\frac{\text{Cr116.3(25.3)Ch156.9(0.5)I}}{\text{I152.1(0.5)Ch68.2(22.9)Cr}}$	40.6
$\mathbf{M}_2$	$\frac{\text{Cr62.4(23.7)I}}{\text{I50.1(20.7)Cr}}$	–

[a]Mesophase temperature ranges on heating.

Table 3. Thermal properties of the polymers.

Polymer	T_g/°C	T_i/°C	ΔH_i/J g^{-1}	ΔT^{b}/°C	T_d^{c}/°C
$\mathbf{P}_1$	66.5	294^a	—	227.5	326.1
$\mathbf{P}_2$	54.2	288.9	1.9	234.7	305.8
$\mathbf{P}_3$	50.2	286.0	1.6	235.8	304.3
$\mathbf{P}_4$	45.1	283.5	1.2	238.4	302.2
$\mathbf{P}_5$	33.4	272.6	0.9	239.2	292.2
$\mathbf{P}_6$	22.7	—	—	—	284.9
$\mathbf{P}_7$	9.7	—	—	—	295.0

[a]Detection with POM at 10°C min^{-1}. [b]Mesophase temperature ranges ($T_i - T_g$). [c]Temperature at which 5% weight loss occurred.

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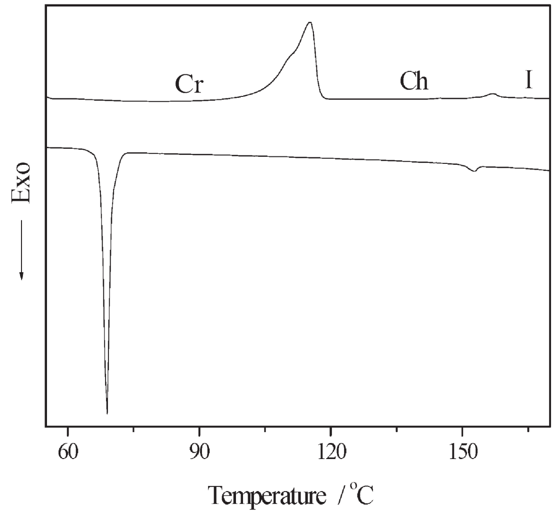

Figure 2. DSC thermograms of monomer $\mathbf{M}_1$.

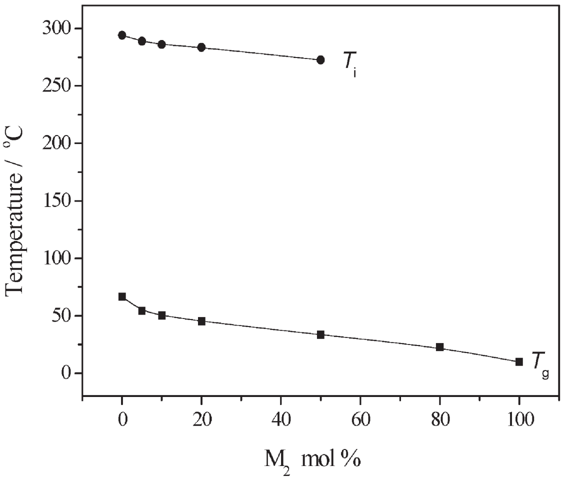

Figure 4. Effect of $\mathbf{M}_2$ concentration on the phase transition temperatures of the polymers.

composition. As shown in table 3, T_g decreased from 66.5°C for $\mathbf{P}_1$ to 9.7°C for $\mathbf{P}_7$ as the concentration of $\mathbf{M}_2$ increased from 0 to 100 mol%, due to the increase in flexibility of the side groups (similar to the plasticization effect).

Along with T_g, T_i is an important parameter of the LC. The copolymer composition affected the T_i of LCPs in two ways: (1) the flexible $\mathbf{M}_2$ units acted as a diluent in the polymer, causing a decrease in T_i; (2) the introduction of the non-mesogenic $\mathbf{M}_2$ units into the polymer discouraged the orientation and formation of mesogenic molecules. Therefore, T_i and ΔH_i of $\mathbf{P}_2$–$\mathbf{P}_5$ decreased from 288.9 to 272.6°C and 1.9 to 0.9 J g^{-1} as the concentration of $\mathbf{M}_2$ units increased from 5 to 50 mol%. This indicates that the mesomorphism reduces on moving from $\mathbf{P}_2$ to $\mathbf{P}_5$. The mesomorphism of the polymers disappeared when the concentration of non-mesogenic $\mathbf{M}_2$ units became greater than 50%. In addition, $\mathbf{P}_1$–$\mathbf{P}_6$ displayed wider mesophase temperature ranges (ΔT), which increased from 227.5 to 239.2°C with increasing concentration of $\mathbf{M}_2$ because T_i decreased less than T_g. In comparing the phase transition temperatures of the polymers, a flexible polymer backbone and a long flexible spacer tended to lead to a low glass transition temperature and a wide mesophase temperature range.

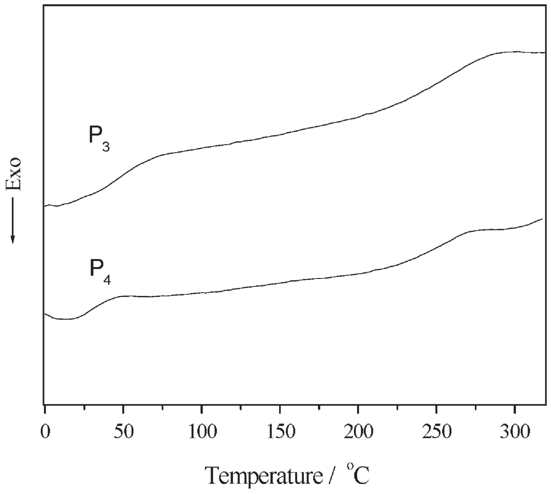

Figure 3. DSC thermograms of polymers.

In TGA results, the temperatures at which 5% weight loss occurred (T_d) were greater than 290°C for $\mathbf{P}_1$−$\mathbf{P}_5$, showing that the synthesized side chain LC polysiloxanes have a high thermal stability. Moreover, thermal stability decreased with increasing concentration of $\mathbf{M}_2$, see table 3.

The optical textures of the monomers and polymers were observed by POM with a stage under nitrogen. $\mathbf{M}_1$ exhibited an enantiotropic cholesteric texture, while $\mathbf{M}_2$ showed no texture on either heating or cooling. When $\mathbf{M}_1$ was heated to 116°C, the sample began to melt; on heating to 138°C, a cholesteric oily-streak texture appeared and the reflection colour changed from red to green to blue with increasing temperature to 149°C. The texture disappeared at 160°C. On cooling from the isotropic to LC state, focal-conic and spiral textures appeared. The optical textures of $\mathbf{M}_1$ are shown in figure 5.

The homopolymer $\mathbf{P}_1$ exhibited a smectic fan-shaped texture. The expected cholesteric texture did not appear because the polymeric chains hinder the formation of a helical supramolecular structure of the mesogens, and the mesogenic moieties are ordered in a smectic orientation with their centres of gravity in planes. The copolymers $\mathbf{P}_2$−$\mathbf{P}_5$ exhibited cholesteric Grandjean

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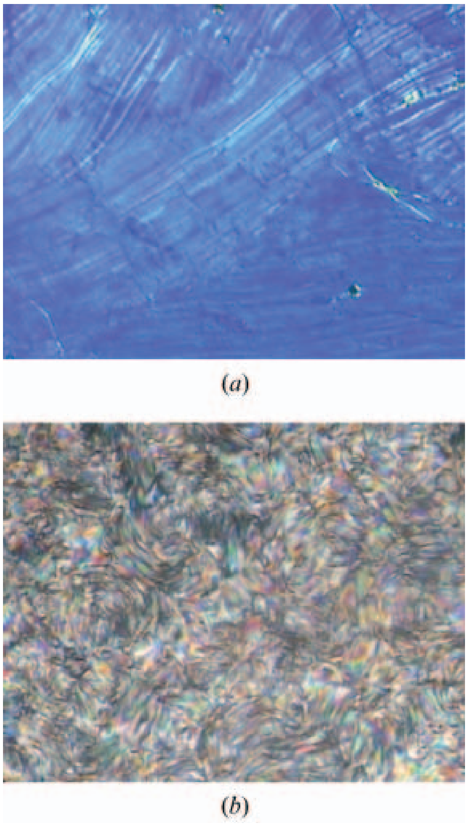

Figure 5. Optical textures of monomer $\mathbf{M}_1$ (200 ×): (*a*) oily-streak texture at 147.8°C; (*b*) spiral texture at 69.6°C.

textures, but no selective reflection properties were observed. $\mathbf{P}_6$ and $\mathbf{P}_7$ showed neither texture nor mesogeic properties.

X-ray studies were carried out to obtain more detailed information on LC phase structure. In general, for a cholesteric structure no peak appears in small angle X-ray scattering curves and a broad peak occurs at about $2\theta=16°$. Figure 6 shows representative XRD curves of quenched samples. For polymer $\mathbf{P}_1$ a strong small angle reflection associated with smectic layers was observed at about $2\theta=3.2°$ and a broad peak associated with lateral packing at about $2\theta=20.4°$, corresponding to *d*-spacings of 27.6 and 4.4 Å. However, for $\mathbf{P}_2$–$\mathbf{P}_5$, no strong small angle reflection was observed, but a broad peak appeared at about $2\theta=16°$, corresponding to a *d*-spacing of 5.5 Å.

4. Conclusions

In this study, new cholesteric LCPs containing side chains of diosgeninyl 4-(10-undecylen-1-yloxy)benzoate and menthyl 4-(10-undecylen-1-yloxy)benzoate were synthesized and characterized. Monomer $\mathbf{M}_1$ showed cholesteric oily-streak and spiral textures. Polymer $\mathbf{P}_1$ showed a smectic phase, while $\mathbf{P}_2-\mathbf{P}_5$ showed cholesteric Grandjean textures. All of the polymers obtained exhibited wide mesophase temperature ranges and high thermal stability. T_g and T_i of the polymers decreased with increasing concentration of $\mathbf{M}_2$ units. The mesogenic properties of the polymers disappeared when the concentration of $\mathbf{M}_2$ units became greater than 50%.

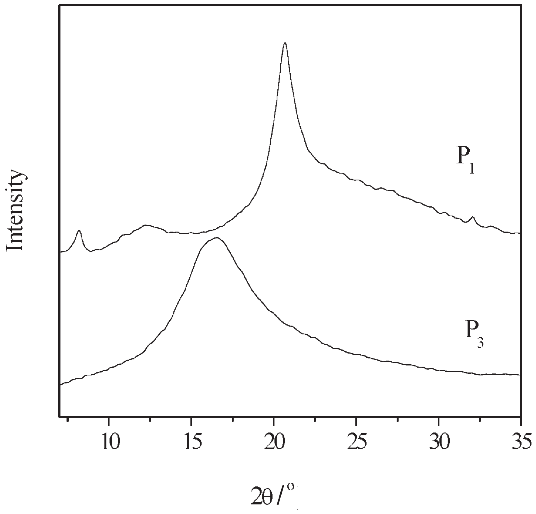

Figure 6. X-ray diffraction patterns of quenched samples.

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