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Phase characterization of LC (meth)acrylic monomers based on ω -hexyloxy- and ω -undecyloxy-salicylaldehyde groups with different alkoxy tail substitutions

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Phase characterization of LC (meth)acrylic monomers based on ω -hexyloxy- and ω -undecyloxy-salicylalimine groups with different alkoxy tail substitutions

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The synthesis and phase characterization of three homologous series of liquid crystalline acrylic and methacrylic monomers, consisting of 21 new compounds are presented. They are based on ω -hexyloxy- and ω -undecyloxysalicylalimine groups with different alkoxy tail substitutions. The liquid crystalline materials were characterized by polarizing optical microscopy and differential thermal analysis. Smectic A and tilted smectic C phases were observed in the compounds. Near the transition to the isotropic, a narrow nematic phase, coexisting with the smectic A phase, was detected for the pentyloxy and hexyloxy derivatives in the M11 and A11 series. In case of M11R11 and M11R12 only a tilted smectic C phase was detected. The clearing point was comparable for all series, around 100°C.

1. Introduction

The properties and chemical structure dependence of liquid crystal molecules have been extensively studied in recent years, because of their importance in improving materials that will be employed in data handling, display devices, etc. Studies on the liquid crystalline behaviour of different families of compounds have been reported for low molar mass LCs in TN and STN displays [1], polymeric composites [2], lyotropic systems [3], NLO materials [4], bent core molecules [5] and amphiphilic organic materials [6]. These are just some examples of recent activity in liquid crystals research.

Ferroelectrics are materials with a macroscopic electric polarization, switchable between two polarized states. They possess a wide range of possible applications as pyro or piezo sensors. Since 1995 [7] we have been working on a continuous program related to the design of non-chiral polymeric ferro- or antiferroelectric materials. These investigations involve the synthesis of new materials as well as their physical and chemical characterization. Our system comprises monomers dissolved in their own polymeric matrix.

The structure of our materials consists of a salicylalimine rigid core group linked to a methacrylic group via an aliphatic 'spacer' of usually six methylenic groups. In this paper we report the liquid crystalline

behaviour of three new series of compounds based on the ω -hexyloxysalicylalimine group in acrylates (A6 series), and the ω -undecyloxysalicylalimine group in acrylates (A11 series) or methacrylates (M11 series). All of these series involve substitution with different alkoxy tails. Our studies are based on a previous communication [8] that gives details concerning the identification of liquid crystalline phases in compounds involving the ω -butyloxy- (M4 series) and ω -hexyloxy-salicylalimine groups (M6 series). All the monomers possess both smectic A and smectic C phases. For short alkyl chains, a narrow nematic phase is observed, within a I-N-SmA-SmC phase sequence.

The acronyms used, $MnRm$ and $AnRm$, are in accordance with previous work [7–12], where Mn and An indicate the presence of a methacrylate or acrylate group with eleven ($n=11$, M11 and A11 series) or six ($n=6$, A6 series) methylene units acting as spacer, and bonded to the aromatic phenylimine rigid core (R). This group possess, in its *para*-position, an alkoxy flying tail ($k=1$) with m methylene units. In case of alkyl derivatives, ($k=0$), they were differentiated with n ($A6Rmn$), to indicate the absence of the oxygen atom linked to the aromatic core in the terminal chain (see structure in table 1).

A characterization of these series is presented in this paper, based on the previous results. Also some acrylate derivatives with alkyl terminal chains are included to

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Table 1. Elemental analysis and HPLC-mass spectrometry, 70eV, M⁺, 100% (nd=not determined).

Compound	C/% (calc.)	H/% (calc.)	N/% (calc.)	M ⁺ <i>m/z</i>	<i>M</i> (calc.)
M11R5	73.69 (73.71)	8.84 (8.81)	2.30 (2.60)	536.5	537.7
M11R6	73.83 (74.01)	8.99 (8.95)	2.23 (2.54)	550.0	551.7
M11R7	73.65 (74.30)	8.81 (9.09)	2.65 (2.48)	564.8	565.8
M11R8	74.52 (74.57)	9.32 (9.21)	1.71 (2.42)	579.8	579.8
M11R9	74.00 (74.83)	9.12 (9.34)	2.19 (2.36)	nd	593.8
M11R10	73.97 (75.08)	9.25 (9.45)	1.92 (2.30)	608.0	607.9
M11R11	75.32 (75.32)	9.65 (9.56)	1.68 (2.25)	621.8	621.9
M11R12	74.70 (75.55)	9.70 (9.67)	1.41 (2.20)	634.9	635.9
A6R7	71.75 (72.35)	8.30 (8.11)	2.69 (2.91)	482.5	481.3
A6R9	71.34 (73.08)	8.46 (8.45)	3.06 (2.75)	nd	509.3
A6R10	71.80 (73.28)	8.53 (8.59)	2.03 (2.68)	524.2	523.3
A6R11	73.64 (73.74)	9.16 (8.75)	2.76 (2.61)	538.6	537.3
A11R5	72.96 (73.79)	8.55 (8.66)	3.00 (2.67)	524.3	523.3
A11R6	73.82 (73.71)	6.80 (8.81)	2.24 (2.60)	537.0	537.7
A11R7	74.49 (74.01)	9.28 (8.95)	1.81 (2.54)	564.8	565.8
A11R8	73.98 (74.30)	9.04 (9.09)	2.12 (2.48)	552.4	551.4
A11R9	73.08 (74.57)	9.54 (9.21)	2.73 (2.42)	579.9	579.4
A11R10	73.53 (74.83)	9.29 (9.34)	2.50 (2.36)	593.7	593.4
A6R7n	74.59 (74.81)	8.45 (8.44)	2.76 (3.01)	466.2	465.3
A11R6n	75.74 (75.97)	9.12 (9.08)	2.12 (2.68)	522.5	521.3
A11R7n	75.52 (76.22)	9.20 (9.22)	2.84 (2.61)	536.4	535.4

contrast their mesomorphism with the investigated series.

2. Experimental

The syntheses of all the monomers was carried out using a convergent synthetic pathway already described [13, 14]. The compounds investigated were characterized by ¹H NMR spectroscopy using a 300 MHz spectrometer (Bruker, WM 300), elemental analysis (Perkin-Elmer, 240 B) and HPLC-mass spectrometry (Fisons Platform).

The phase transition temperatures were determined using a differential thermal analyser (Mettler, FP90 DTA), with an accuracy of ±0.1 K. A polarizing microscope (Leica, DLMP), equipped with a heating stage (HS-1, Instec) was used for temperature dependent investigations of liquid crystal textures. A video camera (Panasonic WVCP414P), installed on the polarizing microscope, coupled with a video capture card (Miro DC-30), allowed real time video capture and image saving. The samples were suspended in a copper plate with a 4 mm diameter hole, and between glass plates.

3. Results

3.1. Chemical characterization

As a representative example, the analytical data for 11-[3-hydroxy-4-(4-pentoxyphenyliminomethyl)phenoxy]undecyl 2-methylacrylate (M11R5) are presented.

¹H-NMR, δ ppm, CDCl₃: 13.86 (s, 1H, OH-Ar); 8.43 (s, 1H, -CH=N-); 7.14 (m, 3H, H-Ar-OH y H-Ar-N=C-); 6.87 (d, 2H, H-Ar); 6.41 (d, 2H, H-Ar-OH); 6.03 (s, 1H, *trans*-CH₂=CR-); 5.48 (s, 1H, *cis*-CH₂=CR-); 4.09 (t, 2H, αCH₂-O-); 3.94 (m, 4H, αCH₂-O-); 1.87 (s, 3H, CH₃-CR=CH₂); 1.75–1.23 (m, 24H, -CH₂-); 0.87 (t, 3H, CH₃-R). MS (70 eV) *m/z* 536.5 (M⁺, 100%). C₃₃H₄₇NO₅, *M*=537.7; calc. C 73.71, H 8.81, N 2.60; found C 73.69, H 8.84, N 2.30%.

Tables 1 and 2 summarize the elemental analysis, mass spectrometry and ¹H NMR, of the new compounds.

3.2. Texture identification

By using polarizing optical microscopy (POM) on samples of M11R5, M11R6, A11R5 and A11R6, the presence of a nematic phase on cooling from the isotropic state in a narrow temperature range can be seen, immediately disappearing to give a smectic A phase. The presence of this nematic phase was detected as droplets forming on cooling from the isotropic state. No POM evidence for the nematic phase, at the transition to the isotropic state, was obtained for other compounds in the series.

Typically, in free standing films the orthogonal smectic A phase develops as a homeotropically aligned phase where a texture is seen the resembling radial lines crossing a cavity, see figure 1(a). This behaviour is indicative of a low viscosity medium and therefore a

Table 2. ^1H NMR spectroscopic characterization of the investigated compounds, δ ppm, CDCl_3 .

Compound															
$R_1 = \text{CH}_3, k = 1$															
$n = 9, m = 3-10$	R_1 s	H_1 s	H_2 s	H_3 t	H_5, H_{13} m	H_6 s	H_7 d	H_8 d	H_9, H_{12} m	H_{10} s	H_{11} d	H_4, H_{14} m	H_{15} t		
M11R5	1.87	5.48	6.03	4.09	3.94	13.86	7.14	6.39	6.87	8.43	6.41	1.23-1.75	0.87		
M11R6	1.87	5.47	6.02	4.10	3.94	13.85	7.16	6.38	6.86	8.43	6.41	1.23-1.74	0.86		
M11R7	1.87	5.48	6.03	4.09	3.94	13.87	7.16	6.38	6.87	8.43	6.41	1.24-1.77	0.85		
M11R8	1.94	5.54	6.09	4.15	3.98	13.95	7.24	6.44	6.89	8.49	6.47	1.29-1.83	0.88		
M11R9	1.87	5.48	6.03	4.09	3.94	13.86	7.16	6.39	6.87	8.43	6.41	1.24-1.75	0.85		
M11R10	1.87	5.48	6.03	4.09	3.94	13.86	7.16	6.39	6.86	8.43	6.41	1.23-1.77	0.84		
M11R11	1.94	5.53	6.09	4.10	3.96	13.94	7.20	6.44	6.89	8.49	6.47	1.30-1.83	0.87		
M11R12	1.87	5.48	6.03	4.09	3.94	13.88	7.17	6.38	6.87	8.43	6.41	1.20-1.77	0.83		
$R_1 = \text{H}, k = 1$															
$n = 4, m = 5, 7-9$	R_1 m	H_1 m	H_2 m	H_3 t	H_5, H_{13} m	H_6 s	H_7 d	H_8 d	H_9, H_{12} m	H_{10} s	H_{11} d	H_4, H_{14} m	H_{15} t		
A6R7	6.15	6.33	5.75	4.08	3.90	13.95	7.24	6.30	6.82	8.44	6.42	1.18-1.72	0.88		
A6R9	6.10	6.32	5.73	4.12	3.95	13.93	7.20	6.30	6.86	8.42	6.42	1.15-1.77	0.85		
A6R10	6.08	6.39	5.75	4.11	3.93	13.95	7.19	6.29	6.85	8.41	6.44	1.12-1.75	0.82		
A6R11	6.10	6.40	5.82	4.17	3.99	13.95	7.24	6.39	6.93	8.51	6.45	1.26-1.86	0.89		
$n = 9, m = 3-8$	R_1 m	H_1 m	H_2 m	H_3 t	H_5, H_{13} m	H_6 s	H_7 d	H_8 d	H_9, H_{12} m	H_{10} s	H_{11} d	H_4, H_{14} m	H_{15} t		
A11R5	6.17	6.46	5.84	4.18	4.02	13.94	7.24	6.38	6.94	8.51	6.43	1.31-1.82	0.97		
A11R6	6.09	6.37	5.76	4.10	3.93	13.86	7.18	6.30	6.86	8.43	6.41	1.23-1.74	0.84		
A11R7	6.10	6.35	5.76	4.10	3.94	13.86	7.16	6.30	6.86	8.43	6.41	1.22-1.76	0.84		
A11R8	6.09	6.37	5.75	4.10	3.93	13.86	7.16	6.30	6.86	8.42	6.40	1.22-1.74	0.83		
A11R9	6.09	6.37	5.75	4.10	3.93	13.89	7.16	6.29	6.86	8.42	6.40	1.23-1.71	0.84		
A11R10	6.09	6.30	5.75	4.10	3.93	13.80	7.16	6.30	6.86	8.42	6.40	1.19-1.76	0.83		
$R_1 = \text{H}, k = 0$															
$n = 4, m = 5$	R_1 m	H_1 m	H_2 m	H_3 t	H_5 t	H_{13} t	H_6 s	H_7, H_9, H_{12} m	H_8 s	H_{10} s	H_{11} d	H_4, H_{14} m	H_{15} t		
A6R7n	6.10	6.36	5.73	4.10	3.93	2.57	13.72	7.16	6.30	8.45	6.40	1.21-1.79	0.83		
$n = 9, m = 4, 5$	R_1 m	H_1 m	H_2 m	H_3 t	H_5 t	H_{13} t	H_6 s	H_7, H_9, H_{12} m	H_8 s	H_{10} s	H_{11} d	H_4, H_{14} m	H_{15} t		
A11R6n	6.10	6.38	5.75	4.10	3.94	2.57	13.83	7.16	6.30	8.45	6.41	1.23-1.74	0.84		
A11R7n	6.06	6.31	5.73	4.10	3.93	2.57	13.72	7.12	6.31	8.45	6.38	1.21-1.80	0.81		

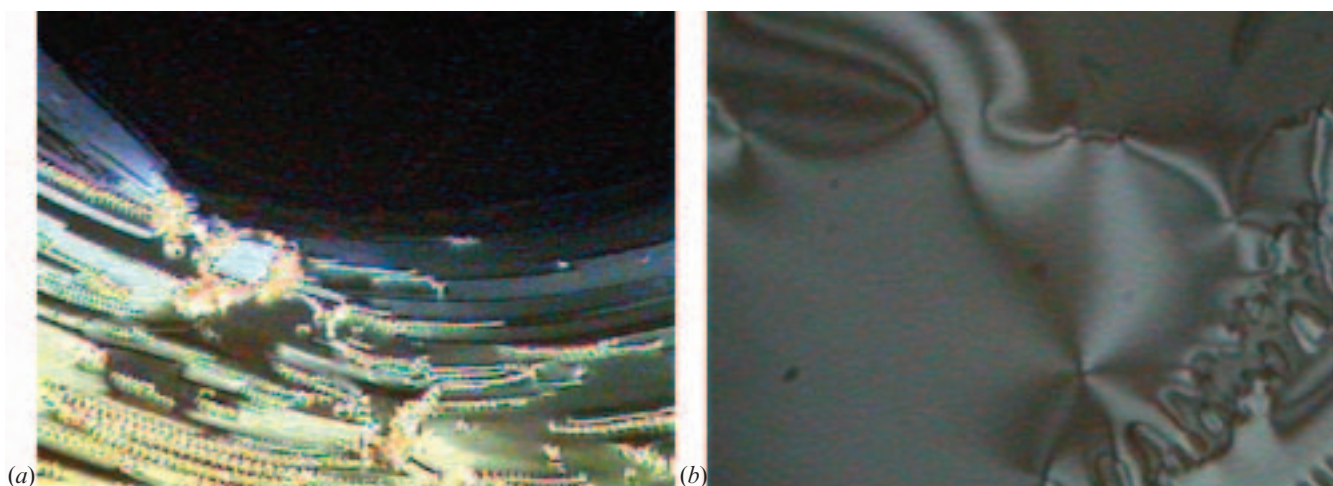
Figure 1. Observed texture of (a) smectic A phase (A6R11, $T = 96.0^\circ\text{C}$) homeotropically oriented; (b) smectic C phase (A6R11, $T = 96.0^\circ\text{C}$).

Table 3. Phase transition temperatures and enthalpy changes obtained on cooling (nd=not determined).

Compound	Transition temperatures/ $^{\circ}\text{C}$	Enthalpy/ kJ mol^{-1}
M11R5	Cr-59.7-SmC-87.4-SmA-87.7-I	49.52-2.20-3.29
M11R6	Cr-55.0-SmC-93.1-SmA-93.7-I	48.50-3.86-3.86
M11R7	Cr-52.3-SmC-79.0 ^a -SmA-91.9-I	36.77-nd-7.02
M11R8	Cr-47.6-SmC-87.0 ^a -SmA-91.8-I	35.13-nd-7.30
M11R9	Cr-51.5-SmC-82.3 ^a -SmA-92.8-I	38.90-nd-7.24
M11R10	Cr-51.2-SmC-90.0 ^a -SmA-95.6-I	43.77-nd-8.87
M11R11	Cr-59.0-SmC-97.7-I	73.40-11.44
M11R12	Cr-56.9-SmC-95.3-I	49.22-9.73
A6R7	Cr-43.0-SmC-78.8 ^a -SmA-101.8-I	48.61-nd-6.60
A6R9	Cr-43.8-SmC-74.7 ^a -SmA-94.6-I	17.77-nd-3.84
A6R10	Cr-48.4-SmC-65.0 ^a -SmA-100.8-I	41.29-nd-6.43
A6R11	Cr-57.8-SmC-86.3 ^a -SmA-101.1-I	51.74-nd-6.93
A11R5	Cr-67.6-SmC-86.6-SmA-90.9-I	64.89-1.32-1.70
A11R6	Cr-63.6-SmC-94.2-SmA-95.7-I	68.28-3.35-2.24
A11R7	Cr-39.1-SmC-80.0 ^a -SmA-92.8-I	32.17-nd-6.78
A11R8	Cr-36.8-SmC-83.0 ^a -SmA-100.0-I	nd-nd-8.37
A11R9	Cr-55.3-SmC-85.0 ^a -SmA-94.7-I	56.32-nd-5.91
A11R10	Cr-70.6-SmC-96.5 ^a -SmA-97.6-I	84.85-nd-10.03
A6R7n	Cr-22.4 ^a -SmA-61.3-I	nd-2.56
A11R6n	Cr-26.9 ^a -SmA-67.9-I	nd-5.42
A11R7n	Cr-25.7 ^a -SmA-73.1-I	nd-7.49

By POM.

less ordered smectic phase. This smectic A phase was observed in most cases, the only exceptions being the longer molecules M11R11 and M11R12.

Apart from the A6 Series, where only the orthogonal smectic A phase was observed, all the compounds show other low viscosity phases on cooling. In case of M11R11 and M11R12 derivatives, this is the only

liquid crystalline phase detected. In this phase, schlieren textures were developed, with disclination points showing four-brush singularities ($s \pm 1$). An example of this texture is shown in figure 1 (b), being similar to textures prepared under homeotropic boundary conditions. Therefore, this birefringence must be associated with a biaxial smectic C phase.

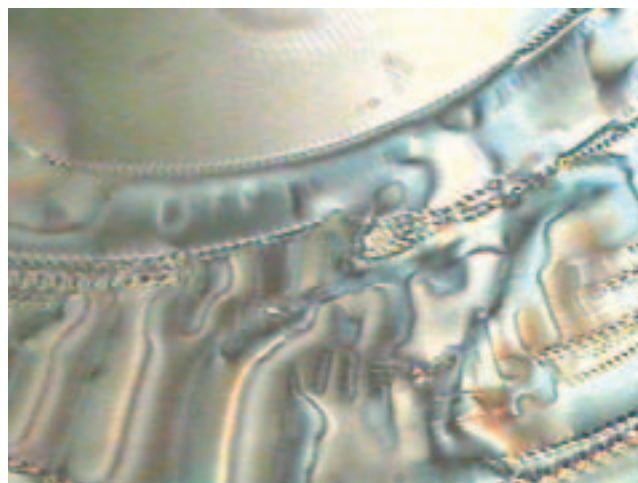
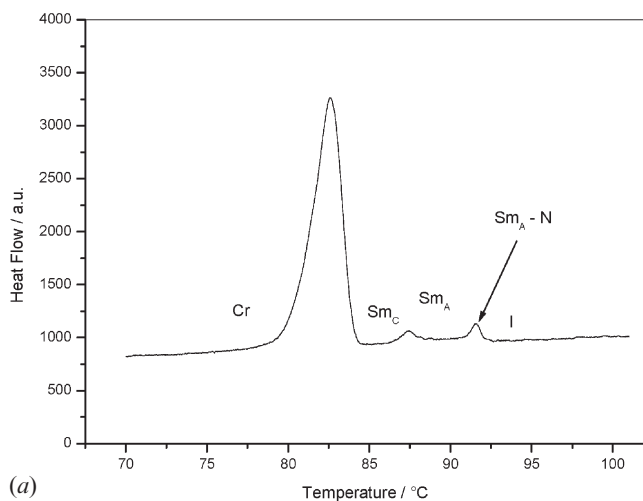


Figure 2. (a) DTA trace for A11R5 at a heating rate of $1^{\circ}\text{C min}^{-1}$ on heating; (b) observed texture of the smectic A-smectic C phase transition (A11R8, $T=83.0^{\circ}\text{C}$).

3.3. Thermodynamic results

The phase transition temperatures and enthalpies obtained by DTA while cooling, and confirmed by POM, are summarized in table 3. In monomers with a narrow nematic range, no evidence of this phase was detected by DTA. The reported enthalpy values correspond sequentially to the transitions Cr–SmC and SmA–I. In the case of the shorter monomers M11R5, M11R6, A11R5 and A11R6, the enthalpy of the SmC–SmA transitions is also reported. Contrasting with the other samples, these small molecules developed a first order phase transition from the SmA to SmC phase, as shown in the thermogram of figure 2(a) for A11R5. For the other molecules, a continuous second order phase transition was observed.

Apart from M11R12, a pronounced first order phase transition at the melting point was observed in the M11 series. In acrylate compounds, only the A6R7 and A6R11 monomers show this behaviour.

In figure 3 the corresponding phase diagrams for the three investigated series (a) M11, (b) A11 and (c) A6 under cooling are displayed. Transition temperatures detected by POM are reported as open circle. The reported temperatures are more or less constant at the transition from the isotropic to the liquid crystalline state in the series. The smectic A phase is more stable in the A6 series. An odd-even effect could just be observed in the A11 series at the transition to the isotropic state. In other cases there is no appreciable variation of the phase transition temperatures when within the series.

4. Discussion

For all the monomer families the existence of layered mesophases has been observed. The phase smectic A to smectic C transition could just be recognized as a first order phase transition, figure 2(b), in thermograms of compounds that also possess a short nematic phase at the isotropic phase transition. This nematic phase appears to be due to a destabilization of the orthogonal smectic A phase, associated with the chain flexibility of longer spacers.

POM gave better experimental evidences for recognition of the second order phase transition from SmA to SmC observed in the other compounds. As was described for the M6 Series [8], the difference between the SmC phase and the smectic A, was clearly observed in the phase transition, due to changes of the homeotropic orientations of the orthogonal smectic A phase to birefringent zones. In figure 2(b) the transition from SmA to SmC is shown, where the homeotropically oriented phase at the upper part of the picture begins to

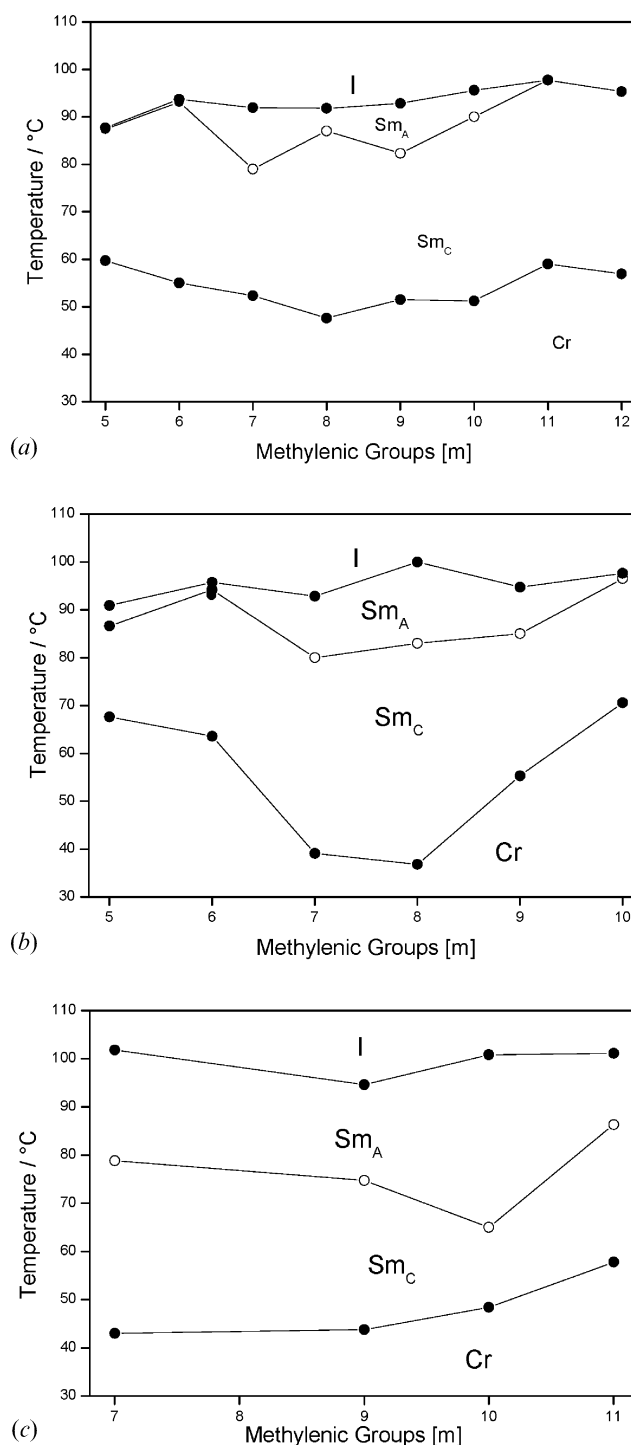


Figure 3. Phase diagram obtained under cooling. Full circles from DTA thermograms and empty circles by POM: (a) series M11; (b) series A11; (c) series A6.

tilt, forming the birefringent zones of the SmC phase, at the bottom. Characteristic corrugated schlieren textures of the smectic C phase were observed in the odd

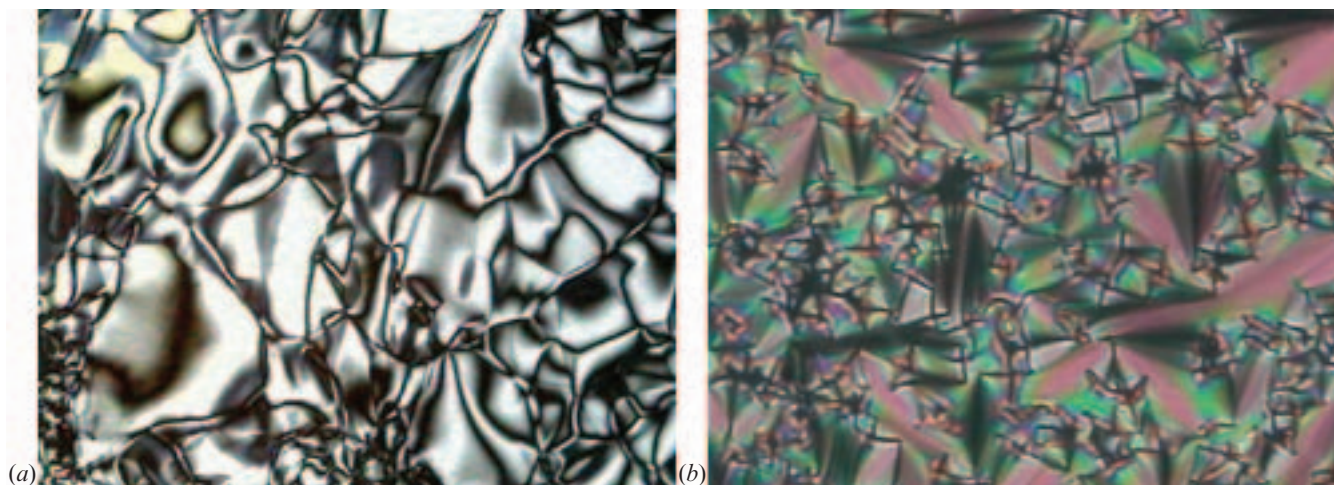


Figure 4. Observed textures of (a) schlieren of a SmC phase (A6R11, $T=70^{\circ}\text{C}$); (b) focal-conic of a SmA phase (A11R6n, $T=54,5^{\circ}\text{C}$).

acrylates of the A6 series. Figure 4(a) shows a representative texture for A6R11 at 70.0°C .

This polymorphism was also compared with the monomers A6R7n, A11R6n and A11R7n, without oxygen in the aliphatic terminal group. They show only the occurrence of a smectic A phase, developing characteristic textures between glass plates for this well studied phase. Figure 4(b) illustrates the common observation of this molecular arrangement, where a fan-shaped texture is growing by cooling from a well defined focal-conic texture.

The presence of the oxygen atoms in the aliphatic tails provokes a drastic fall in the transition temperatures. We reported this effect in a previous communication [15], where M6R8n, compared with M6R8, possesses phase transition temperatures about 30°C lower. This effect was also observed in other series of Schiff's bases [16, 17] and ester compounds [18]. We believe that this decrease is related to the flexibility of the alkyl chain as compared with the alkoxy chain, and the polar effect of the alkoxy chain due to the presence of the electro-negative oxygen atom.

5. Conclusion

The synthesis and phase characterization of 21 new liquid crystalline monomers have been carried out. All the homologues exhibit thermotropic liquid crystalline behaviour. The existence of disordered lamellar phases was determined by the use of polarizing optical microscopy. A nematic phase observed in M11R5, M11R6 A11R5 and A11R6 was detected only by POM in a

narrow temperature range at the phase transition to the isotropic.

An odd-even effect related to the alkoxy substitution length was observed in the A11 series for the phase transition from liquid crystalline to isotropic. The other series do not show this effect.

For series with longer spacers (M11 and A11), the tendency is to show a shorter smectic A region, stabilizing the smectic C phase. Smectic C phases were absent in compounds with alkyl instead of alkoxy chains, while smectic A phases were absent in monomers with long alkoxy tails. The presence of the oxygen in the aliphatic tails of the monomers promotes the appearance of tilted lamellar phases.

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