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Synthesis and characterization of naphthalene-substituted triphenylene discotic liquid crystals

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A series of naphthalene-substituted triphenylene liquid crystals (LCs), viz. triphenylene-2,3,6,7,10,11-hexayl hexakis(6-alkenyloxy-2-naphthoate)s (HTP_nN compounds) were synthesized and characterized. Alkenyloxy groups containing three to eleven carbon atoms were used as peripheral spacers in these liquid crystals. The discotic liquid crystals synthesized trap one to three water molecules depending on the length of their peripheral spacers. Differential scanning calorimetry, polarizing optical microscopy and X-ray diffraction measurements confirm both nematic (N_d) and rectangular disordered columnar (Col_{r,d}) phases for most of the synthesized discotic LCs. The clearing temperatures of these discotic LCs increase with increasing peripheral spacer length. A hybrid aligned HTP₉N sample was investigated to evaluate its optical performance. Retardation values of this hybrid sample decrease with increasing wavelength and increase with increasing measuring angle.

1. Introduction

Discotic liquid crystals were first reported by Chandrasekhar *et al.* in 1977 [1]. Since then, many liquid crystalline materials having disc-like shapes have been synthesized and the field was reviewed by Chandrasekhar in 1993 [2]. Generally, traditional hexa-alkyl substituted triphenylene discotic compounds [3–7] have the tendency to become polycrystalline at room temperature and form a columnar mesophase in which the disc-like mesogens are stacked closely and regularly within columns [8, 9] at elevated temperatures. The situation changed in 1981 when triphenylene-2,3,6,7,10,11-hexayl hexakis(4-*n*-alkoxy benzoate)s were found to exhibit N_d phases [10]. The effects of bulky lateral substituents on the mesomorphism of these triphenylene benzoates were thoroughly investigated by Goodby *et al.* [11, 12].

Recently nematic discotic liquid crystalline materials, which exhibit negative birefringence, have demonstrated their potential for applications as compensation film for wide viewing angle liquid crystal displays (LCDs) [13–15]. Some of the desired properties for an ideal optical compensation film for LCDs are: (1) an appropriate phase retardation value, $d\Delta n$ (where d denotes the film thickness and Δn represents the film birefringence) ranging from $\lambda/8$ to $\lambda/2$, depending on the application;

(2) an ideal phase match with the LC employed so that good compensation results can be achieved over the entire visible region; (3) high optical transparency; and (4) good uniformity [16]. In order to achieve the above requirements, various kinds of discotic LC with different birefringences are needed. Furthermore, according to the literature [13–15], a uniform discotic compensation film can only be prepared by aligning a discotic LC material below its N_d phase. Therefore, an ideal discotic LC material should exhibit a wide N_d temperature range so as to have a wide processing window. So far, there are only a very limited number of discotic LCs that exhibit N_d phases and their N_d phase temperature ranges are rather narrow.

The aim of this study was to synthesize a new series of naphthalene-substituted triphenylene derivatives, the triphenylene-2,3,6,7,10,11-hexayl hexakis(6-alkenyloxy-2-naphthoate)s. The naphthalene groups were introduced into the triphenylene core to alter the birefringence and facilitate the formation of the N_d phase [17]. The alkenyloxy groups were used as peripheral spacers to manipulate the phase transitions and provide the possibility for further oxidation of the terminal vinyl groups to form epoxy derivatives which can then be photopolymerized to yield crosslinked networks. The mesomorphic properties were characterized by differential scanning calorimetry (DSC), polarizing optical microscopy (POM) and X-ray diffraction. The sample of HTP₉N synthesized was aligned as a monodomain and its optical properties were also investigated.

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2. Experimental

2.1. Materials

Allyl bromide, 6-hydroxy-2-naphthoic acid, veratrole and all other reagents were obtained from Aldrich and used as received. 3-Bromo-1-butene, 4-bromo-1-pentene, 5-bromo-1-hexene, 6-bromo-1-heptene, 7-bromo-1-octene, 8-bromo-1-nonene, 9-bromo-1-decene and 10-undecen-1-yl tosylate were prepared according to literature procedures [18, 19]. 6-Alkenyloxy-2-naphthoic acids (*n*NA compounds, $n = 1-9$) were synthesized by esterification of 6-hydroxy-2-naphthoic acid with alkenyl halide according to a literature procedure reported by our laboratories [20]. 2,3,6,7,10,11-Hexahydroxytriphenylene was prepared according to the literature method reported by Frederik *et al.* [21].

2.2. Techniques

^1H NMR spectra of all the compounds synthesized were recorded on a Varian VXR-300 NMR spectrometer (300 MHz) using CDCl_3 as solvent and tetramethylsilane

(TMS) as internal standard. A Seiko/SSC/5200 differential scanning calorimeter was used for determining thermal transitions. Heating and cooling rates were $10^\circ\text{C min}^{-1}$. A Carl-Zeiss Axiophot polarizing microscope equipped with a Mettler FP 82 hot stage and a Mettler FP 80 central processor was used to analyse the thermal transitions and observe the mesomorph textures. X-ray diffraction measurements were made with a Rigaku powder diffractometer using nickel-filtered CuK_α radiation. Optical properties were studied using an Otsuka Multichannel retardation measuring system, RETS-2000 (Osaka, Japan).

2.3. Synthesis of triphenylene-2,3,6,7,10,11-hexayl hexakis (6-alkenyloxy-2-naphthoate)s (HTP*n*N compounds, $n = 1-9$)

All HTP*n*N compounds were synthesized by the esterification of 2,3,6,7,10,11-hexahydroxytriphenylene with the corresponding 6-alkenyloxy-2-naphthoic acids. An example for the synthesis of HTP9N is given below.

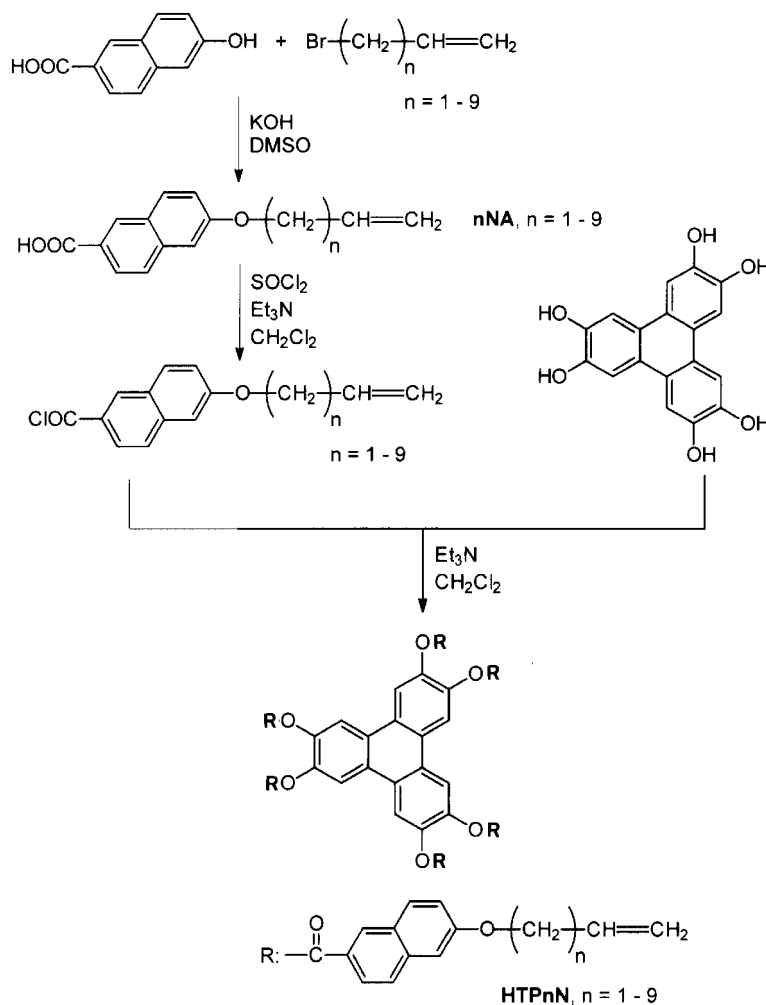


Figure 1. Synthetic routes to the naphthalene-substituted discotic liquid crystals HTP*n*N.

6-10-Undecenyloxy-2-naphthoic acid (5 g, 0.015 mol) was allowed to react with a two-fold excess of thionyl chloride containing 5 drops of *N,N*-dimethylformamide in 50 ml of methylene chloride at room temperature for 2 h. The solvent and excess of thionyl chloride were removed under reduced pressure to furnish the crude acid chloride. The crude product was then dissolved in 50 ml of methylene chloride and added slowly to an ice-cold solution of 2,3,6,7,10,11-hexahydroxytriphenylene (0.661 g, 0.025 mol) and triethylamine (2.23 g, 0.022 mol) in 50 ml of methylene chloride. The mixture was stirred for 12 h and then the solvent was removed by rotary evaporation. The solid obtained was dissolved in ethyl acetate and washed with 5% aqueous K_2CO_3 and water and dried with anhydrous $MgSO_4$. Ethyl acetate was removed under reduced pressure and the crude product was purified by column chromatography (silica gel, CH_2Cl_2 as eluent) to yield 4.0 g (85%) of yellowish solid. 1H NMR ($CDCl_3$, 300 MHz) δ (ppm) = 1.32 (m, 72H, $(-CH_2)_6-$); 1.76 (m, 12H, $O-CH_2-CH_2-$); 2.03 (m, 12H, $CH_2=CH-CH_2-$); 3.89 (t, $J = 6.3$ Hz, 12H, $O-CH_2-$); 4.94 (m, 12H, $CH_2=CH-$); 5.80 (m, 6H, $CH_2=CH-$); 6.59 (s, 6H, H_{arom}); 6.76 (d, $J = 8.9$ Hz, 16H, H_{arom}); 7.01 (d, $J = 8.8$ Hz, 6H, H_{arom}); 7.15 (d, $J = 9.0$ Hz, 6H, H_{arom}); 7.76 (d, $J = 9.2$ Hz, 6H, H_{arom}); 8.24 (s, 6H, core); 8.54 (s, 6H, H_{arom}).

3. Results and discussion

3.1. Mesomorphic behaviour of 6-alkenyloxy-2-naphthoic acids (*nNA* compounds, $n = 1-9$)

Phase transition temperatures and phase transition enthalpies of the *nNA* compounds are summarized in table 1. All the 6-(alkenyloxy)-2-naphthoic acids synthesized have hydrogen bonded dimeric structures. Compounds with short spacer lengths (1NA and 2NA) exhibit only a monotropic nematic phase. The five compounds with medium spacer lengths (3NA–7NA) give enantiotropic nematic phases, and the other two compounds with longer spacers (8NA and 9NA) give a smectic A phase besides a nematic phase. Figure 2 depicts the DSC thermograms of 8NA. As can be seen, compound 8NA displays enantiotropic nematic and smectic phases. The clearing temperature decreases as the number of carbon atoms increases as shown in figure 3, and a typical odd–even effect is also evident.

3.2. Characterization and thermal behaviour of triphenylene-2,3,6,7,10,11-hexayl hexakis (6-alkenyloxy-2-naphthoate)s (*HTPnN* compounds, $n = 1-9$)

The 1H NMR spectrum of HTP8N was representative and showed a peak at 1.6 ppm which belongs to the protons of the included water molecules. This peak did not disappear when distilled, dry $CDCl_3$ was used as

Table 1. Phase transitions and transition enthalpy changes for *nNA* compounds Cr = crystalline, SmA = smectic A, N = nematic, I = isotropic.

Compound	Transition temp./°C (enthalpy change/kcal mol ⁻¹)	2nd heating 1st cooling
1NA	Cr 206.6 (24.8) I I 206.4 (– 0.05) N 152.4 (– 28.5) Cr	
2NA	Cr 199.9 (33.1) I I 187.5 (– 1.8) N 179.9 (– 28.5) Cr	
3NA	Cr 151.5 (20.5) N 197.7 (3.3) I I 194.9 (– 2.7) N 134.2 (– 20.4) Cr	
4NA	Cr ₁ 30.0 (0.7) Cr ₂ 148.0 (12.1) N 191.1 (2.0) I I 184.7 (– 2.3) N 133.7 (– 16.9) Cr ₂ 122.4 ^a Cr ₁	
5NA	Cr ₁ 34.0 (4.4) Cr ₂ 148.0 (12.1) N 191.2 (2.0) I I 188.0 (– 2.0) N 133.5 (– 11.2) Cr ₂ 3.8 (– 4.3) Cr ₁	
6NA	Cr ₁ 57.9 (6.5) Cr ₂ 105.7 (0.2) Cr ₃ 147.6 (15.3) N 180.9 (1.9) I I 178.0 (– 2.2) N 133.9 (– 14.9) Cr ₃ 94.2 (– 0.1) Cr ₂ 43.7 (– 7.3) Cr ₁	
7NA	Cr ₁ 34.0 (1.4) Cr ₂ 102.4 (13.7) Cr ₃ 139.1 (13.0) N 181.2 (2.2) I I 178.0 (– 2.2) N 128.8 (– 12.7) Cr ₃ 26.4 (– 13.6) Cr ₂ – 0.2 (– 0.3) Cr ₁	
8NA	Cr 129.5 (12.0) SmA 133.9 (0.8) N 174.2 (2.1) I I 172.2 (– 1.8) N 131.3 (– 0.8) SmA 115.7 (– 3.9) Cr	
9NA	Cr ₁ 120.6 (30.2) Cr ₂ 126.5 ^a SmA 137.2 (0.4) N 171.0 (1.9) I I 168.3 (– 2.0) N 133.4 (– 0.6) SmA 104.9 (– 10.3) Cr ₃ 101.7 ^a Cr ₂ 59.3 (– 3.4) Cr ₁	

^a Overlapped transitions.

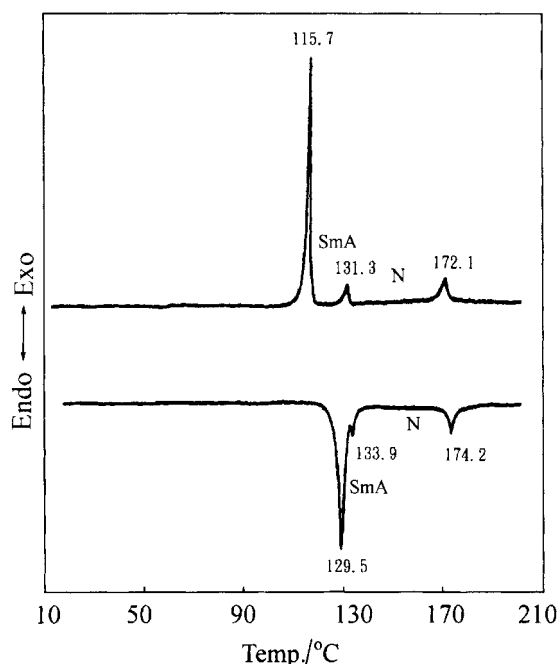


Figure 2. DSC traces for 8NA (from top to bottom: first cooling, second heating runs).

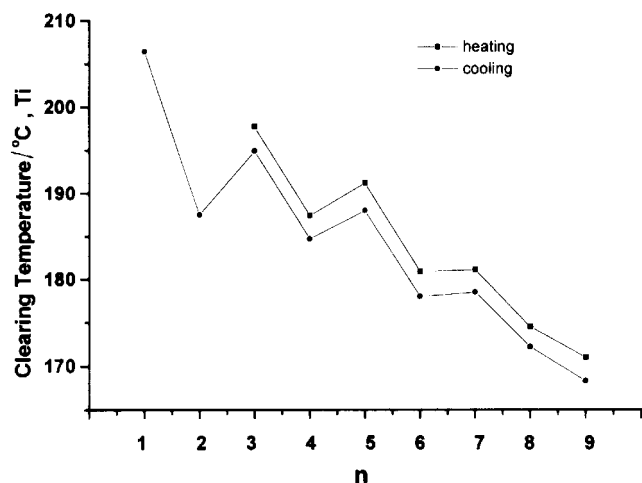


Figure 3. Clearing temperatures of n NAs as a function of n .

solvent. The elemental analysis results for the HTP n N compounds listed in table 2 confirmed the inclusion of the water molecules. It is clear that the HTP n N compounds include one to three water molecules depending on their spacer length. As a general trend, the number of included water molecules first increases and then decreases as the carbon number of the peripheral alkenyl chain increases. Frederik *et al.* [21] also reported this water-inclusion phenomenon.

Table 3 summarizes the phase transition temperatures and phase transition enthalpies for the HTP n N compounds. All these discotic compounds exhibit N_d and

Table 2. Elemental analysis data for HTP n N compounds

Compound	Formula	Calculated/%	Found/%
HTP1N	$C_{102}H_{72}O_{18}H_2O$	C: 76.49 H: 4.66	^a
HTP2N	$C_{108}H_{84}O_{18}H_2O$	C: 76.85 H: 5.14	C: 76.50 H: 5.04
HTP3N	$C_{114}H_{96}O_{18}2H_2O$	C: 77.27 H: 5.57	C: 77.10 H: 5.77
HTP4N	$C_{120}H_{108}O_{18}2H_2O$	C: 76.90 H: 6.02	C: 77.82 H: 5.91
HTP5N	$C_{126}H_{120}O_{18}2H_2O$	C: 77.28 H: 6.38	C: 77.46 H: 6.40
HTP6N	$C_{132}H_{132}O_{18}3H_2O$	C: 77.62 H: 6.71	C: 77.81 H: 6.80
HTP7N	$C_{138}H_{144}O_{18}3H_2O$	C: 77.28 H: 7.05	C: 77.21 H: 7.01
HTP8N	$C_{144}H_{156}O_{18}3H_2O$	C: 77.60 H: 7.33	C: 77.70 H: 7.24
HTP9N	$C_{150}H_{168}O_{18}2H_2O$	C: 78.50 H: 7.55	C: 78.38 H: 7.53

^a No satisfactory data are available.

Col_{rd} mesophases except for HTN1N, which shows no mesophase on melting with decomposition. It is found that the clearing temperatures generally decrease with increasing number of carbon atoms in the peripheral alkenyl chains.

Compounds HTP2N–HTP6N present a similar thermal behaviour. Figure 4 depicts a representative DSC thermogram for compound HTP6N. The first heating curve shows a melting transition at 170.3°C, a Col_{rd} to N_d transition at 201.0°C and a N_d to isotropic transition at 273.3°C. The first cooling scan reveals an isotropic to N_d transition at 270.1°C, and an N_d to Col_{rd} transition at 131.2°C, but no crystallization transition is shown. When the sample was further cooled to room temperature, the Col_{rd} texture was frozen in and did not change for more than one week. The second heating scan shows only a Col_{rd} to N_d transition at 198.2°C and an N_d to isotropic transition at 273.1°C. Figure 5 depicts the N_d and Col_{rd} textures shown by HTP6N.

HTP7N–HTP9N reveal similar thermal behaviours. Figure 6 presents the representative DSC thermogram for HTP8N. The first heating scan shows a recrystallization transition at 155.2°C, a Col_{rd} to N_d phase transition at 174.2°C and an N_d to isotropic phase transition of low enthalpy at 224.9°C, whereas the first cooling scan shows only an isotropic to N_d phase transition at 223.6°C. The second heating scan looks very similar to the first heating scan except for its broader recrystallization peak. Figure 7(a) shows the typical nematic schlieren texture

Table 3. Phase transitions and transition enthalpy changes for HTP n N compounds Col $_{r,d}$ = rectangular disorder and columnar mesophase, N $_d$ = discotic nematic mesophase, I = isotropic phase.

Compound	Transition temp./°C (enthalpy change/kcal mol ⁻¹)	2nd heating 1st cooling
HTP1N	No transition was observed before decomposition	
HTP2N	Col $_{r,d}$ 249.9 (1.67) N $_d$ 330.1 ^a I I 325.1 ^a N $_d$ 214.8 (-0.1) Col $_{r,d}$	
HTP3N	Col $_{r,d}$ 227.4 (1.67) N $_d$ 327.5 ^a I I 324.2 ^a N $_d$ 178.6 (-2.00) Col $_{r,d}$	
HTP4N	Col $_{r,d}$ 227.4 (0.86) N $_d$ 288.8 ^a I I 285.7 ^a N $_d$ 146.8 (-1.08) Col $_{r,d}$	
HTP5N	Col $_{r,d}$ 222.0 (2.15) N $_d$ 286.5 (0.04) I I 280.1 (-0.04) N $_d$ 164.2 (-2.37) Col $_{r,d}$	
HTP6N	Col $_{r,d}$ 198.2 (2.24) N $_d$ 273.1 (0.05) I I 270.1 (-0.04) N $_d$ 131.2 (-2.89) Col $_{r,d}$	
HTP7N	Col $_{r,d}$ 174.2 (1.84) N $_d$ 235.7 (0.05) I I 233.8 ^a N $_d$	
HTP8N	Col $_{r,d}$ 174.2 (1.84) N $_d$ 224.9 (0.05) I 233.6 ^a N $_d$	
HTP9N	Col $_{r,d}$ 155.5 (0.45) N $_d$ 217.6 (0.05) I I 214.2 ^a N $_d$	

^a Transition observed via POM.

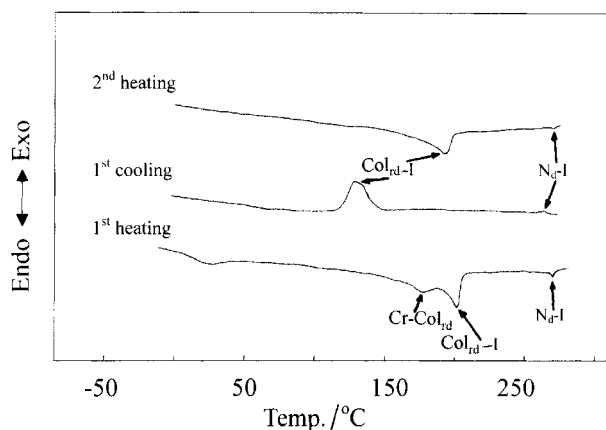


Figure 4. DSC traces for HTP6N.

exhibited by HTP8N. Upon cooling to room temperature, the nematic texture was frozen and recrystallized during the second heating scan, see figure 7 (b). The high molecular mass, which makes these discotic compounds turn into glassy states, may be responsible for the inhibition of columnar mesophase formation during the cooling processes.

The phase assignments for the HTP n N compounds were confirmed by X-ray diffraction. Figure 8 shows the temperature dependent X-ray diffraction patterns obtained from a powder sample of HTP8N. Curve A, which was obtained at 210°C, shows a very broad diffraction peak in the wide angle region and a very strong diffraction

peak in the small angle region. The wide angle diffraction peak relates to the lateral distance between triphenylene cores, while the small angle diffraction peak is attributed to the diameter of the triphenylene core. This confirms the formation of an N $_d$ phase. By lowering the temperature from 210 to 170°C, the small angle diffraction peak splits into two peaks at 29.1 and 35.4 Å as shown in curve B. In addition, the large angle region shows only a very broad diffusion peak. This provides evidence for the formation of a rectangular disordered columnar phase [21].

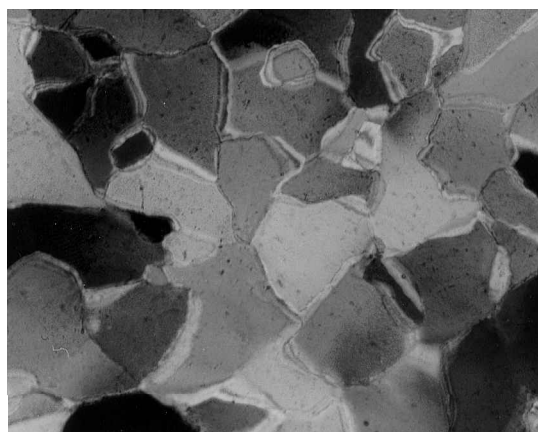
In comparison with triphenylene-2,3,6,7,10,11-hexayl hexakis(4-*n*-alkoxybenzoate)s, it is obvious that the incorporation of six naphthalene moieties facilitates the formation of the discotic nematic mesophase. Tinh *et al.* [10] reported that triphenylene-2,3,6,7,10,11-hexayl hexakis(4-decyloxybenzoate) gave an N $_d$ phase for a range of only 21.0°C. However, the corresponding hexanaphthalene substituted triphenylene discotic material HTP8N gives a much broader N $_d$ range of over 250.0°C as shown by the cooling scan process.

3.3. Optical anisotropy measurement

Some optically anisotropic properties of HTP9N were next investigated. A small amount (0.1 g) of HTP9N was dissolved in 0.5 ml of methyl ethyl ketone and spin-coated onto a rubbed polyimide surface. The sample

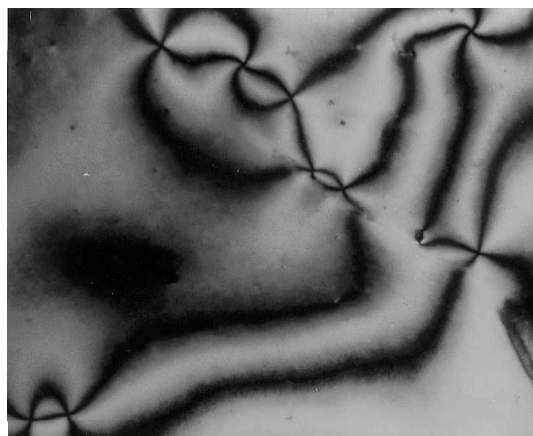


(a)

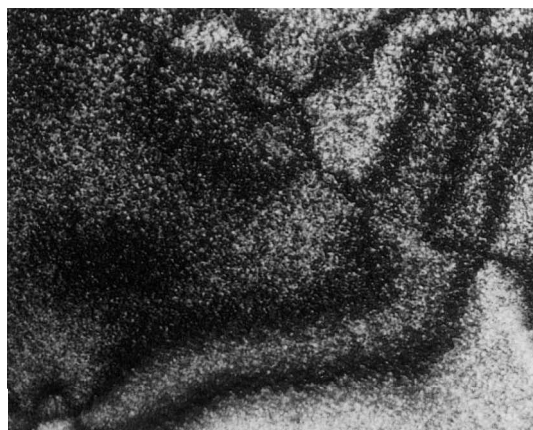


(b)

Figure 5. Optical textures of HTP6N at (a) 257°C, N_d ; (b) 160°C, Col_d (crossed polarizers, magnification $\times 200$).



(a)



(b)

Figure 7. Optical textures of HTP8N at (a) 187°C, N_d ; (b) 160°C, frozen N_d (crossed polarizers, magnification $\times 200$).

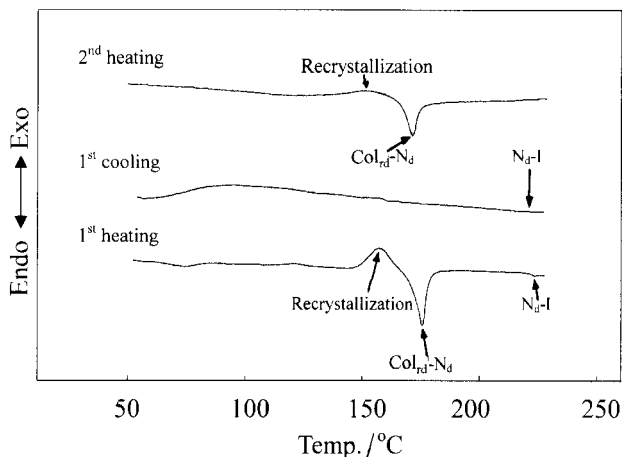


Figure 6. DSC traces for HTP8N.

was annealed at 180°C for 5 min to achieve a monodomain hybrid arrangement as shown in figure 9. This hybrid structure was confirmed by optical measurements (see figure 10) at several different viewing angles (10–60°)

and azimuthal angles (0– $\pm 50^\circ$). This monodomain hybrid arrangement remained unchanged at room temperature (about 25°C) for over two months even though it was not a crosslinked network.

Figures 11 and 12 depict, respectively, the angle-dependent and wavelength-dependent retardations measured for the hybrid aligned HTP9N sample (with azimuthal angle = 0°). Its retardation values are in the range 20–90 nm. The retardation values increase with increasing measuring angle and decrease with increasing wavelength.

This mono-domain aligned HTP9N sample shows different retardation values at a fixed angle from different directions. Figure 13 shows that the retardation value decreases gradually from 0 to $\pm 50^\circ$ azimuthal angle (with measuring angle = 50°). This indicates that the HTP9N molecules are arranged in a biaxial way in which the discotic molecule has its plane inclined from the plane of polyimide substrate at an angle varying along the direction of the depth of the anisotropic layer.

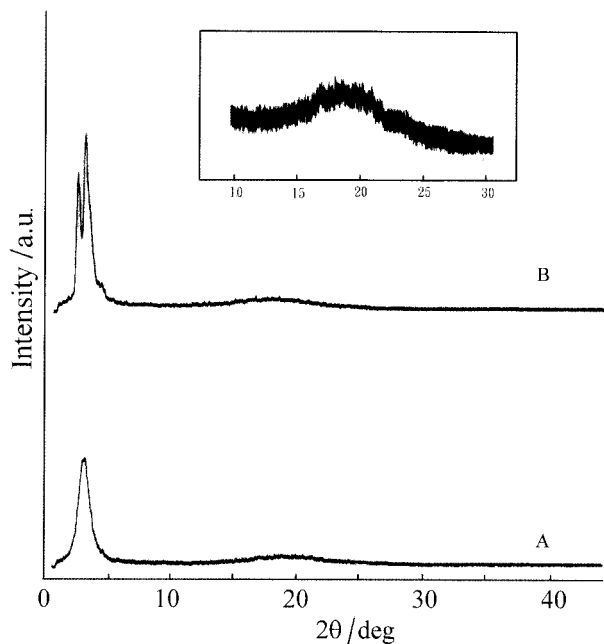


Figure 8. X-ray power diffractograms of HTP8N at (A) 210°C, N_d ; (B) 170°C, $Col_{r,d}$.

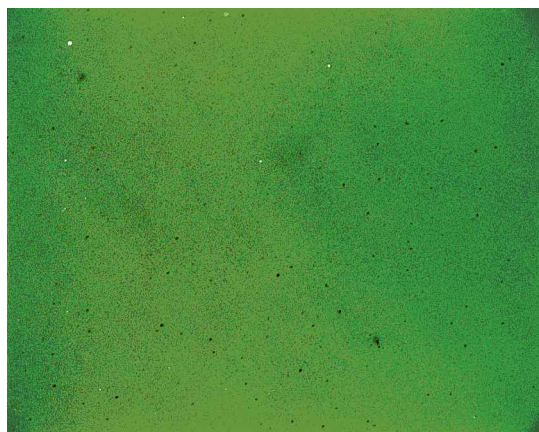


Figure 9. Optical texture of monodomain prepared from HTP9N (crossed polarizers, magnification $\times 200$).

From the above results, it is clear that these naphthalene-containing discotic liquid crystals can be used in preparing optical retardation films for improving the viewing angle of LCDs.

In order to form a cross-linked network, the HTP n N compounds were oxidized with *m*-chloroperoxybenzoic acid (MCPBA) to yield the epoxy derivatives which might then be photopolymerized to form a crosslinked network. Unfortunately, we only obtained mixtures of the epoxy derivatives, and pure materials could not be obtained from these epoxide mixtures.

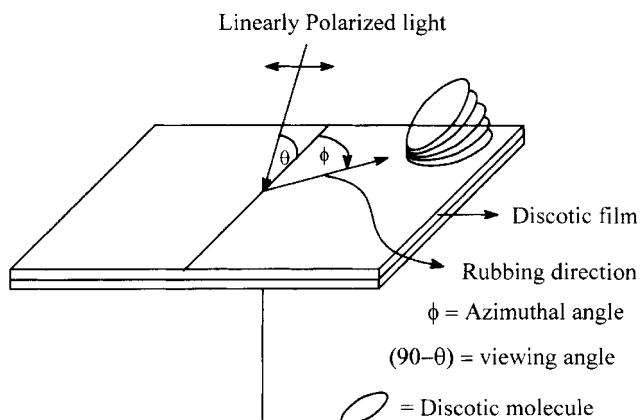


Figure 10. Experimental set-up for measurement of optical properties.

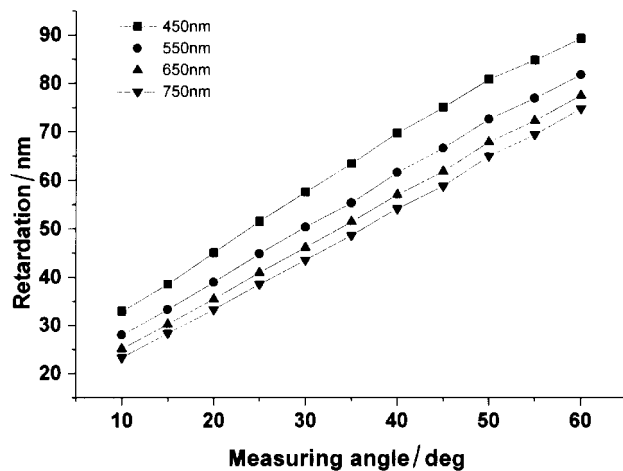


Figure 11. Retardation values of HTP9N for different wavelengths as a function of measuring angle.

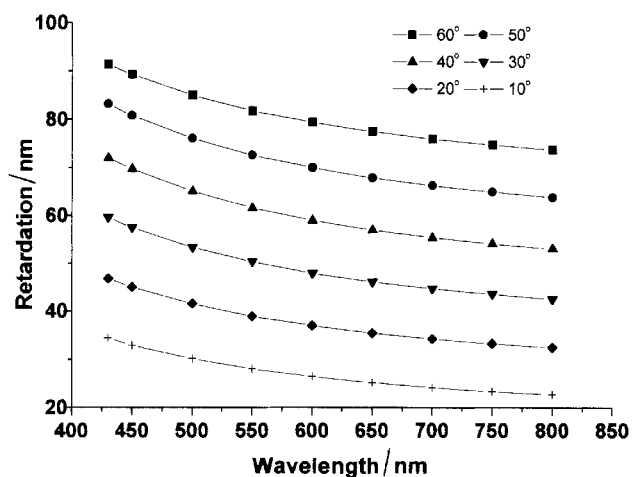


Figure 12. Retardation values of HTP9N for different measuring angles as a function of wavelength.

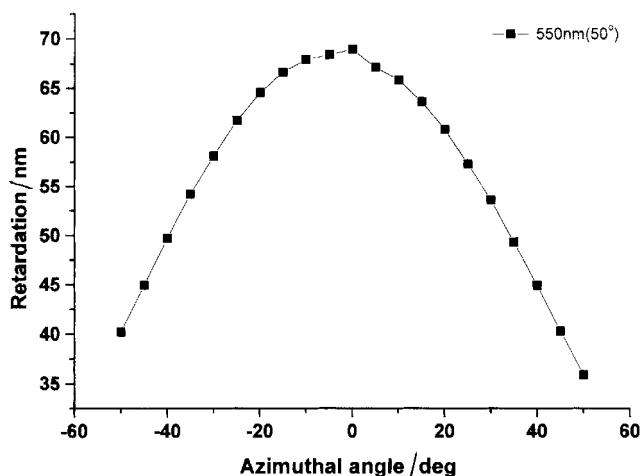


Figure 13. Retardation values of HTP9N measured from different azimuthal angles (measuring angle: 50° , wavelength: 550 nm).

4. Conclusion

A new series of naphthalene-substituted LCs, viz. triphenylene-2,3,6,7,10,11-hexayl hexakis(6-alkenyloxy-2-naphthoate)s were synthesized and characterized. All the HTP n N compounds prepared, except HTP1N, gave N_d and Col_{rd} phases. The results demonstrate that introducing six naphthalene groups into the triphenylene core widens the mesomorphic temperature range. Most of the HTP n N compounds obtained have very wide temperature range N_d phases. They could be easily aligned on glass substrates which were coated with unidirectionally rubbed polyimide; a mono-domain hybrid arrangement was then obtained. These HTP n N compounds can be mixed with other UV-curable discotic monomers to form mixtures for the fabrication of compensation films with desired retardation values.

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