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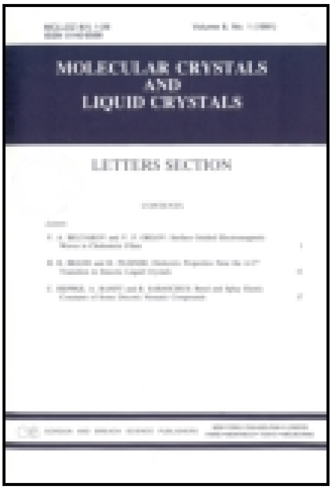

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Synthesis and Mesomorphic Behavior of Calamitic Liquid Crystals with a Biphenyl Moiety

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A new mesogenic homologous series having a biphenyl moiety has been synthesized by condensing 4-hydroxy-4′-nitrobiphenyl- and methoxy-substituted 4-n-alkoxy cinnamoyl chlorides, and their liquid crystalline properties have been studied. All the members of the series are enantiotropic liquid crystals. The methyl to n-pentyl derivatives exhibit both Smectic A (SmA) and Nematic (N) phases; the higher members, starting with the n-hexyl derivative show only a SmA phase. The plot of transition temperatures versus number of carbon atoms in the alkoxy chain exhibits zig-zag pattern for Sm-N and N-Isotropic (Iso) transition temperature curves. The average thermal stability is compared with other related homologous series. The introduction of polar nitro group increases significant intermolecular force of attraction which stabilizes the molecular orientation. This results into the increase in the thermal stability of the system.

Keywords Biphenyl moiety; calamitic liquid crystals; cinnamates; nematic; smectic

1. Introduction

Liquid crystalline materials have been studied for several decades in electrical, optical, and life sciences fields not only for scientific interests but also for their practical applications. Formation of liquid crystalline phases relies on mesogenic groups having either calamitic- or disc-like shapes. These basic molecular structures can be further modified giving rise to controlled structural features of the mesophases. The calamitic liquid crystals can form two main classes of liquid crystal phases, the *nematic* and *smectic* phases. The mesomorphic properties of these materials can largely be influenced by various substituents. This can influence the melting points, the mesophase types, the dielectric properties, *etc*.

Linearity, rigidity, and polarizability are also important factors for a molecule to display liquid crystalline properties [1]. Different aromatic moieties have been used as rigid core in synthesizing liquid crystals. Biphenyl can be seen as rigid core of many mesogenic compounds and liquid crystalline [2] or semi-crystalline aromatic polymers [3]. Moreover, since biphenyl is a molecule interesting and fascinating itself, it has been the object, over many years, of many studies, both experimental [4–6] and theoretical [7, 8]. Such interest is mainly due to its relatively simple structure and to the strong conformational dependence of the molecule on the phase of the material.

Biphenyl derivatives are important in the formation of ferroelectric and anti-ferroelectric liquid crystals [9–13]. Furthermore, biphenyl derivatives are able to undergo

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functionalization reactions, providing access to a variety of compounds [14–25]. Mostly 4, 4′-substituted biphenyl derivatives exhibit one or more smectic phases and extraordinarily high clearing temperatures. Kelly et al. reported 4-arylpyridinium salts using different 4, 4′ substituted biphenyls [26]. Givenchy and co-workers have designed 4, 4′ biphenyl derivatives incorporating a perfluorinated segment and an allylic part as precursors of highly fluorinated polymers [27]. Hentrich et al. have synthesized symmetrical and asymmetrical bolaamphiphiles [28] and concluded that bolaamphiphiles are significantly stabilized by the introduction of a rigid biphenylene unit. Nishiyama and co-workers have investigated chiral trimeric compounds possessing three biphenyl mesogenic cores connected by flexible alkyl spacers [29]. They compared the molecular structure and the mesomorphic properties of these trimers with those of the related dimeric and monomeric systems. Marcelis et al. [30] have reported liquid crystalline dimers consisting of an aromatic unit and a cholesteryl mesogenic unit joined via flexible spacers. To the best of our knowledge, liquid crystalline compounds having a biphenyl moiety with a nitro substituent are very rare. The present investigation was carried out with a view to study the effect of cinnamoyl linkage and nitrobiphenyl moiety on the mesomorphic properties of such molecules.

2. Experimental

2.1. Instrumentation

The synthetic route adopted for the series of mesogens is given in the Fig. 1. All synthesized compounds were purified by column chromatography on silica gel (60–120 mesh). The purity of the samples was checked by thin-layer chromatography. The chemical structures of the compounds were determined by standard spectroscopic methods. IR spectra were determined for KBr pellets, using a Perkin Elmer-RX1 spectrophotometer. ^{1}H NMR spectra was recorded on a Bruker 400 MHz spectrometer using tetramethylsilane (TMS) as internal reference standard. Liquid crystalline properties were investigated using Leica DM 2500P polarizing optical microscope provided with a Linkam heating stage. The transition temperatures were confirmed by Differential Scanning Calorimeter on Mettler Toledo – DSC 822 system which was calibrated using indium as a standard.

2.2. Synthesis and Characterization

2.2.1. Trans-4-n-alkoxy-3-methoxycinnamoyl Chlorides (2). A mixture of 4-hydroxy-3-methoxycinnamic acid [31,32] (4.85 g, 0.025 mol), alkyl iodide, or bromide (0.03 mol), potassium hydroxide (3.5 g, 0.062 mol), and ethanol (50 ml) was refluxed for 4 to 5 hr. After being cooled, the mixture was poured into a stirred mixture of concentrated hydrochloric acid (25 mL) and ice cold water (50 mL). The product was then filtered off, washed with water, dried, and recrystallized from ethanol until constant melting points were obtained. Purity of the compounds are checked by TLC and characterized by spectral data. The melting points are summarized in Table 1.

***n*-butoxy derivative:** IR (KBr): 2594–2956 cm^{-1} –O—H stretch broad band superimposed upon C—H stretch, 2880, 2827 cm^{-1} –C—H stretch, 1683 cm^{-1} >C=O stretch, 1260 cm^{-1} C—O stretch, 979 cm^{-1} strong = C—H bending.

^{1}H NMR (CDCl$_3$, δ, ppm): 0.98–1.02 (t, 3H, CH$_3$), 1.49–1.55 (m, 2H, CH$_2$), 1.83–1.88 (m, 2H, CH$_2$), 3.92 (s, 3H, OCH$_3$), 4.06–4.10 (t, 2H, OCH$_2$), 6.31–6.35 (d, 1H, = CH—), 6.88–6.90 (d, 1H, Ar—CH), 7.09–7.10 (d, 1H, Ar—CH), 7.12–7.14 (dd, 1H, Ar—CH), 7.73–7.77 (d, 1H, —CH=).

The acids were converted into their acid chlorides by treating them with thionyl chloride on water bath till evolution of hydrochloric acid ceased. The excess of thionyl chloride was distilled off. The crude product was used immediately in the next reaction without further purification.

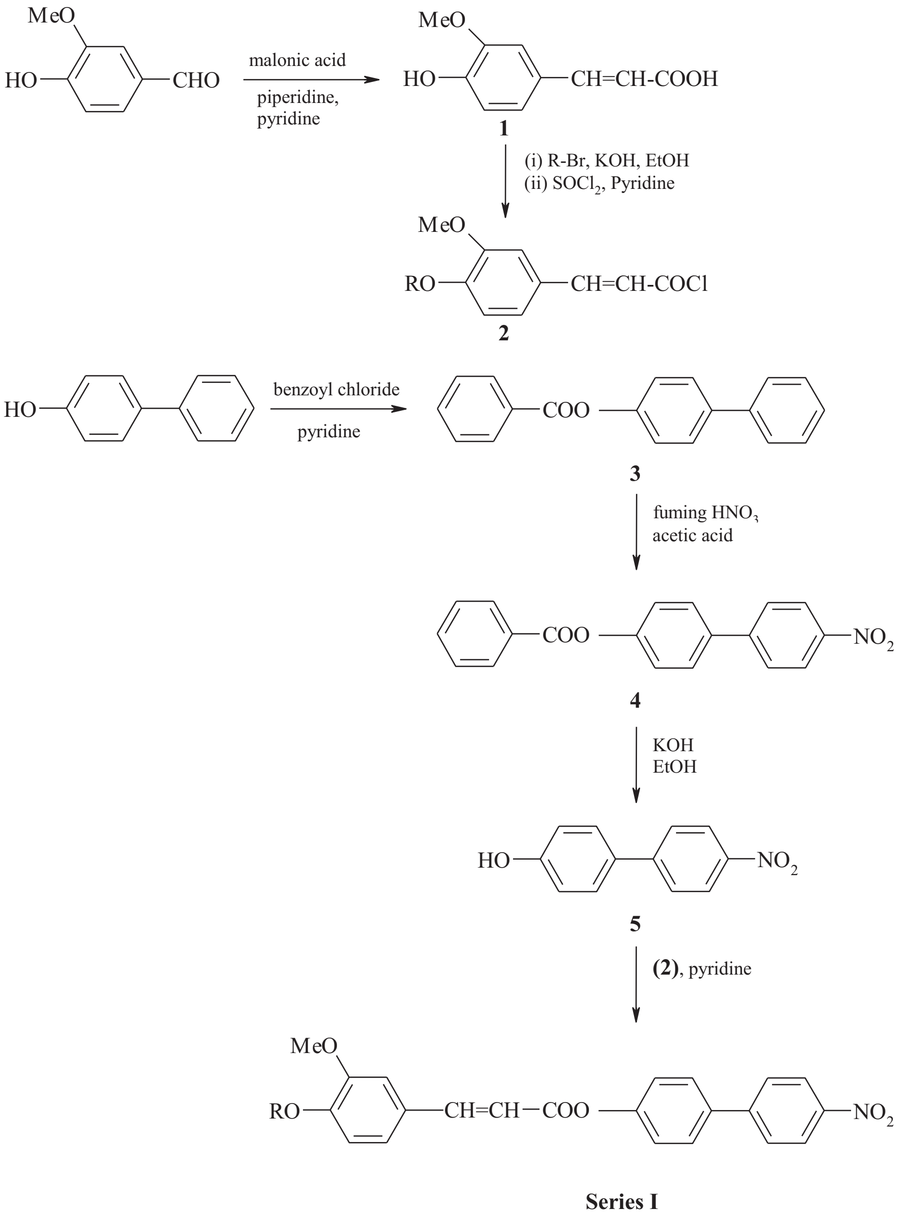

Figure 1. Synthetic route to series I. Note: R = n-C_nH_{2n+1}, n = 1 to 8, 10, 12, 14, 16 and 18.

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Table 1. Melting points (°C) of trans-4′-n-alkoxy-3′-methoxycinnamic acid (5)

Comp. No.	1	2	3	4	5	6	7	8	9	10	11	12	13
R = -C_nH_{2n+1}	1	2	3	4	5	6	7	8	10	12	14	16	18
M.p. (°C)	181	178	163	149	132	125	120	105	103	104	105	107	107

2.2.2. 4-(Benzoyloxy) Biphenyl (3) [33]. 4-Hydroxy biphenyl (0.1 mol) was dissolved in 50 mL of dry pyridine and was added slowly with stirring to cold benzoyl chloride (0.1 mol) contained in a round bottom flask. The mixture was heated on a water-bath for 30 min and was allowed to cool up to room temperature. It was acidified with cold 1:1 hydrochloric acid and precipitates obtained were filtered, washed with water, and dried. The ester was crystallized from ethanol to yield 87% product. Melting Point: 150°C.

2.2.3. 4-(Benzoyloxy)-4′-nitrobiphenyl (4) [33]. 4-(Benzoyloxy) biphenyl (0.145 mol) was taken in round bottom flask, 310 mL of glacial acetic acid was added in the flask and it was heated up to 80 to 90°C. 100 mL fuming nitric acid was added slowly in such a way that the temperature remained between 80 and 90°C. After complete addition, the reaction mixture was cooled to room temperature. The solid separated was filtered and washed with water and methanol. The product was crystallized from glacial acetic acid to yield 51% of product. Melting Point: 210°C.

2.2.4. 4-Hydroxy-4′-nitrobiphenyl (5) [33]. 4-Benzoyloxy-4′-nitrobiphenyl (0.18 mol) was taken in round bottom flask, 300 ml of ethanol was added to it and it was heated to reflux. An aqueous solution of potassium hydroxide (40 g of KOH in 100 mL of water) was then added dropwise at reflux. After complete addition, the mixture was further refluxed for 30 min and cooled overnight. Next day, blue crystals of potassium salt were filtered, washed with tetrahydrofuran until wash was colorless. Pure 4-hydroxy-4′nitrobiphenyl was obtained by dissolving the salt in minimum amount of boiling water and adding 1:1 hydrochloric acid until acidic pH was obtained. The yellow solid was filtered, washed with water, dried, and crystallized from ethanol. Melting Point: 203°C.

2.2.5. 4′-nitro Biphenyl-4-yl-(2E)-3-(4-butoxy-3-methoxyphenyl)-prop-2-enoate (Series I). The respective 4-n-alkoxy-3-methoxycinnamoyl chloride (0.01 mol) was dissolved in about 5 mL of dry pyridine. The solution of compound **5** (0.01 mol) in 10 mL of dry pyridine was added slowly to a cold solution of the above acid chloride. The mixture was heated on a water bath for about half an hour and was allowed to stand overnight. It was acidified with cold 1:1 hydrochloric acid. Precipitates were filtered and washed with water followed by diluted sodium hydroxide solution and water. The residue was purified by column chromatography on silica gel with mixture of ethyl acetate and petroleum ether (10:90) as an eluent.

***n*-butoxy derivative:** IR (KBr): 2958, 2872 cm^{-1} —C—H stretch, 1697 cm^{-1} >C=O stretch, 1602 cm^{-1} olefinic —C=C-stretch, 1295 cm^{-1} C—O stretch, 1504 cm^{-1} NO_2 asymmetric stretch, 1348 cm^{-1} NO_2 symmetric stretch, 1209 cm^{-1} C—N stretch, 1654, 1414, 1395 cm^{-1} ring breathing vibrations of C—H bending.

^{1}H NMR ($CDCl_3$, δ, ppm): 1.01–1.03 (t, 3H, CH_3), 1.52–1.58 (m, 2H, CH_2), 1.90–1.98 (m, 2H, CH_2), 4.02 (s, 3H, OCH_3), 4.13–4.17 (t, 2H, OCH_2), 6.33–6.37 (d, 1H, = CH—), 6.69–6.91 (d, 1H, Ar—CH), 7.25–7.27 (dd, 1H, Ar—CH), 7.34–7.36 (d, 1H, Ar—CH),

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Table 2. Phase transition temperatures of series I compounds

		Transition temperatures (°C)						
Sr. no.	R = *n*-alkyl group	Cr		SmA		N		I
1	Methyl	·	169.0	·	187.0	·	199.0	·
2	Ethyl	·	171.0	·	194.5	·	210.0	·
3	Propyl	·	174.0	·	195.0	·	205.0	·
4	Butyl	·	185.0	·	211.0	·	222.3	·
5	Pentyl	·	163.9	·	196.5	·	210.0	·
6	Hexyl	·	174.1	·	—	—	213.2	·
7	Heptyl	·	185.0	·	—	—	227.0	·
8	Octyl	·	182.2	·	—	—	229.5	·
9	Decyl	·	181.1	·	—	—	231.0	·
10	Dodecyl	·	181.0	·	—	—	229.3	·
11	Tetradecyl	·	175.9	·	—	—	221.8	·
12	Hexadecyl	·	177.4	·	—	—	216.0	·
13	Octadecyl	·	171.0	·	—	—	198.2	·

7.42–7.44 (d, 2H, Ar—CH), 7.57–7.61 (d, 1H, —CH=), 7.70–7.72 (d, 2H, Ar—CH), 7.76–7.78 (d, 2H, Ar—CH), 8.33–8.35 (d, 2H, Ar—CH).

3. Results and Discussion

The number of homologous series [34, 35] with biphenyl moiety is reported in literature. Most of these series have terminal a cyano group in the moiety. Low melting mesogenic cyano biphenyl compounds gave impetus for applications [1]. Gray [34] mentioned that terminal nitro group normally imparts yellow color hence they did not pursue it further. However, terminal nitro group has provided some interesting new mesogens. The effect of

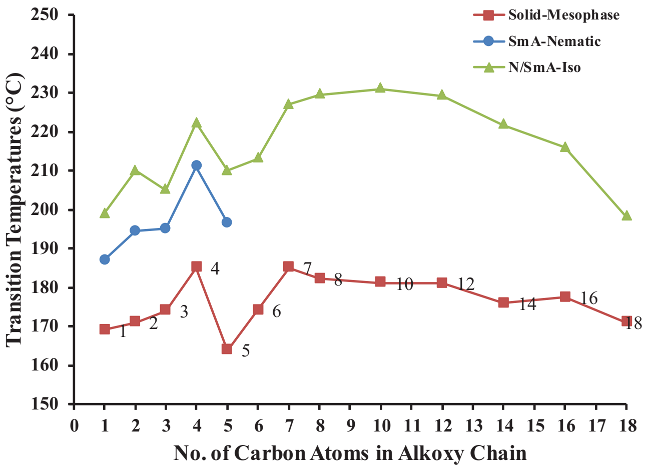

Figure 2. Plot of transition temperatures (°C) versus number of carbon atoms in the alkoxy chain.

Table 3. Average thermal stabilities (°C) of series I, A and B compounds

Series	I	A	B
N/SmA – Iso (C_1-C_{18})	216.3	180.9	243.0
SmA - N	196.8(C_1–C_5)	148.1(C_7–C_{14})	195.6(C_4–C_8)
Commencement of SmA mesophase	C_1	C_7	C_4

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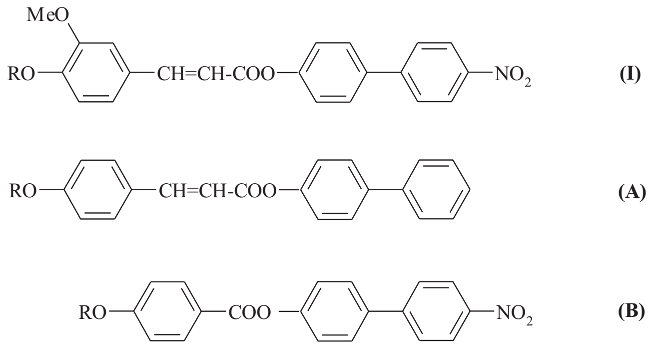

Figure 3. Comparative geometry of series I, A, and B compounds.

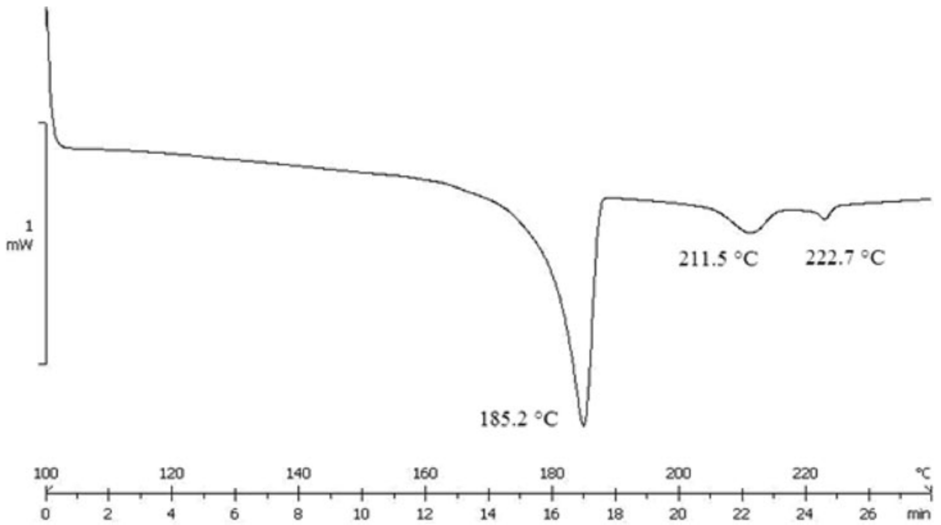

Figure 4. Representative DSC thermograms of n-butyloxy derivative at heating rate of 5°C min^{-1}.

Table 4. Calorimetric data

Sr. No.	R-n-alkyl group	Transition state	Peak temps. (microscopic reading) (°C)	ΔH kJ/mole
1	Butyl	K-SmA	185.2 (185.0)	10.45
		SmA-N	211.5 (211.0)	5.12
		N-Iso	222.7 (222.3)	1.83
2	Pentyl	K-SmA	164.5 (163.9)	10.68
		SmA-N	197.1 (196.5)	4.71
		N-Iso	210.0 (210.0)	1.84
3	Hexyl	K-SmA	174.8 (174.1)	19.27
		SmA-Iso	212.5 (213.2)	9.07
4	Heptyl	K-SmA	185.7 (185.0)	18.58
		SmA-Iso	227.7 (227.0)	7.22

terminal nitro group on the commencement of smectic mesophase is quite interesting. It was thought quite interesting to synthesize and study the mesogenic properties of biphenyl derivatives having terminal nitro group.

Thirteen homologues were synthesized by condensing 4-n-alkoxy-3-methoxy cinnamoyl chlorides with 4-hydroxy-4′-nitrobiphenyl. All the homologues have enantiotropic mesophases. The methyl to n-pentyl derivatives exhibit both SmA and N phases; the higher members, starting with the n-hexyl derivative show only SmA phase. Series I was synthesized by the route shown in Fig. 1. The transition temperatures are summarized in Table 2. Gray [36] and Major and Baumgartner [37] have suggested that the addition of each methylene unit simultaneously increase the overall polarizability of the molecules and

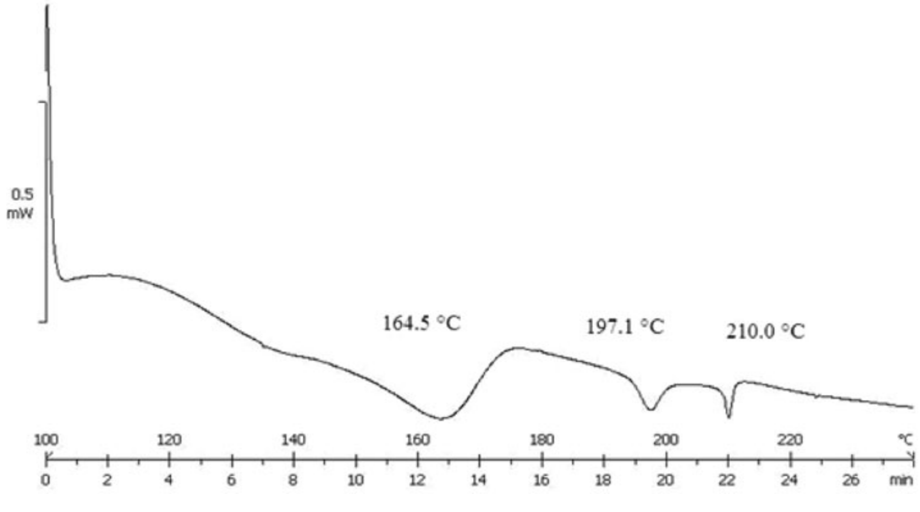

Figure 5. Representative DSC thermograms of n-pentyloxy derivative at heating rate of 5°C min^{-1}.

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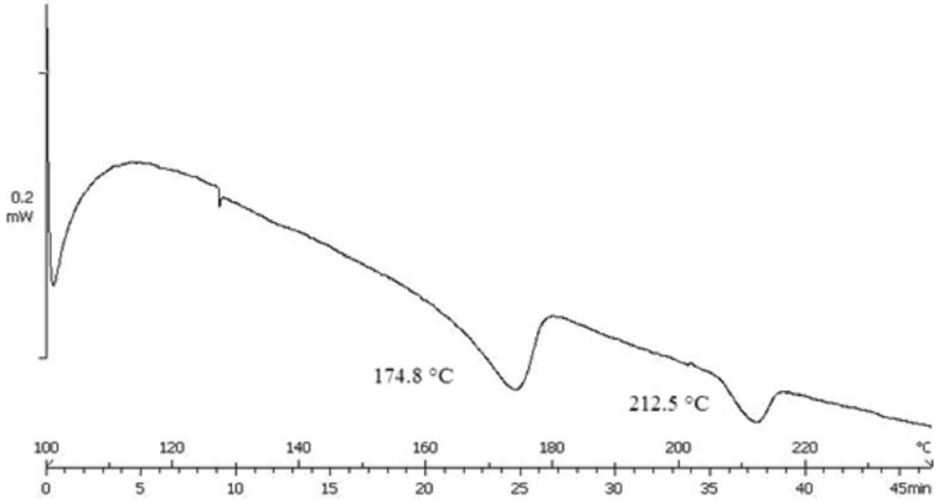

Figure 6. Representative DSC thermograms of n-hexyloxy derivative at heating rate of 3°C min^{-1}.

the lateral intermolecular attraction. The lower homologues are SmA and N, the separation of aromatic nuclei is at minimum and the terminal cohesions are strongest. As we ascend the series, only SmA phase commences because as the alkyl chain length increases, the lateral cohesive forces are also increase and the molecules align themselves in the layer structure. Hence with increase in the alkyl chain length, the Smectogenic character should predominate at the expense of nematic mesophase. Therefore in series I, at certain chain length of alkyl group, nematic mesophase would be observed and system would exhibit pure SmA phase. At this stage, SmA mesophase will directly pass into isotropic liquid stage presumably because the terminal intermolecular attractions are inadequate to maintain the

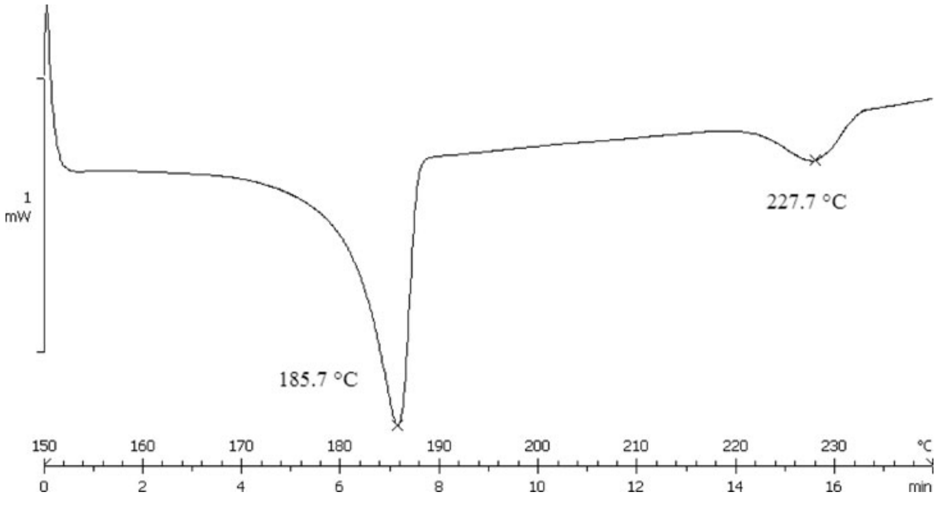

Figure 7. Representative DSC thermograms of n-heptyloxy derivative at heating rate of 5°C min^{-1}.

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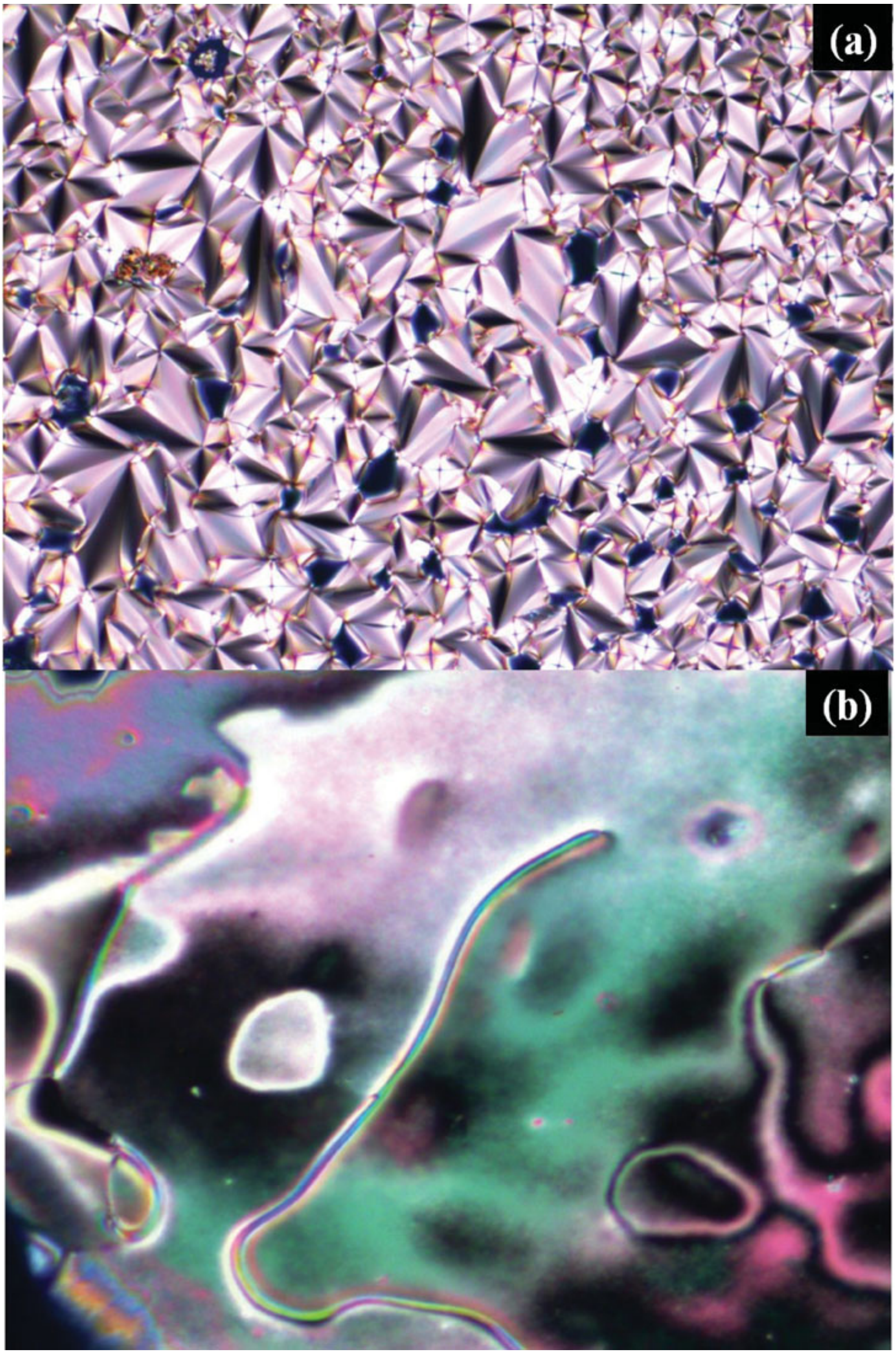

Figure 8. Polarized optical microphotographs of homologue 05 (a) Smectic A (focal conic texture) liquid crystalline phase at 189.3°C on heating; (b) Nematic (thread like texture) liquid crystalline phase at 204.6°C on heating.

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parallel molecular orientation which is required for the normal nematogenic homologous series.

The plot of transition temperatures versus number of carbon atoms in the alkoxy chain is given in Fig. 2. The SmA-N and N-Iso transition temperature curves exhibit zig-zag pattern in a lower derivatives (up to C_5) of series. The N-Iso transition temperature curve raises steeply up to C_{10} derivative and then fall down up to the last member of the series.

There is a close relationship between liquid crystalline properties and molecular constitution of organic compounds, hence the thermal stability; a measure of liquid crystalline properties can be correlated with the molecular constitution of the compounds.

The average thermal stabilities of different mesogenic homologous series are compared and recorded in Table 3.

Comparison of molecular structure of present series I with reported Series:

1. Biphenyl-4-Trans-p-n-Alkoxycinnamates; Series: A [38]
2. 4(4″-n-Alkoxy benzoyloxy)-4′-nitrobiphenyls; Series: B [39]

The geometry of these series is given in Fig. 3. The average isotropic thermal stabilities of series I is higher than those of series A. Series I and series A have the same molecular geometry except the terminal and lateral substituents. Series I have lateral methoxy and terminal nitro substituent. Introduction of lateral methoxy is expected to be deterrent to the thermal stability of the system but the presence of polar nitro group increases significant intermolecular force of attraction, which stabilizes the molecular orientation. It also increases the length and polarizability of the molecules. This results into the increase in the thermal stability of the system [36]. Hence thermal stability of the series I is higher compare to series A. The difference between the thermal stabilities of series I and A is of the order of 35°C. This shows potential effect of a terminal nitro group on the mesomorphic property of homologous series.

The Nematic thermal stabilities of series I and of series B are almost the same. They differ only in central linkage and lateral substituent at one end of the molecule. Methoxy group joined to phenyl ring at ortho position would have steric effect compared to the molecules of series B. However, the ortho methoxy group would not have fullest breadth increasing effect. Series B has a biphenyl nucleus and an ester linkage which could contribute appreciably to the polarizability of the molecule in the direction of molecular axis, thereby enhancing thermal cohesions. This would give higher nematic–isotropic transition temperatures than series I.

The mesogens were screened by DSC method and calorimetric data are recorded in Table 4. Figures 4–7 shows DSC thermograms for n-butoxy to n-heptyloxy derivatives, respectively. n-Hexyloxy derivative was scanned at the heating rate of 3°C min^{-1}, rest of the derivatives were scanned at the heating rate of 5°C min^{-1}. In the present study, SmA and nematic mesophases are inferred from the textures observed under Leica DM 2500P polarizing optical microscope provided with a Linkam heating stage (Fig. 8).

4. Conclusions

All the compounds of the series show mesomorphic properties. The methyl to n-pentyl derivatives exhibit both SmA and N phases; the higher members, starting with the n-hexyl derivative show only SmA phases. The plot of transition temperatures versus number of carbon atoms in the alkoxy chain exhibits zig-zag pattern for SmA-N and N-Isotropic transition temperature curves. Lateral methoxy group is deterrent to mesomorphic properties while polar nitro group increases significant intermolecular force of attraction, which stabilizes the molecular orientation. This results into the increase in the thermal stability of the system.

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