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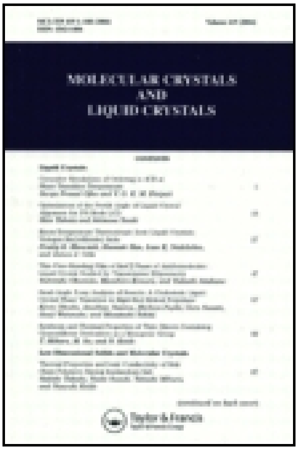

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Substitution Effects on the Liquid Crystalline Properties of Thermotropic Liquid Crystals Containing Schiff Base Chalcone Linkages

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Two homologous series of Schiff base Chalcones, Series-A: 1-{4-[(2,4-dihydroxy benzylidene) amino] phenyl}-3-(4-alkoxy phenyl) prop-2-en-1-one and series-B: 1-(4-{[1-(2,4-dihydroxyphenyl)ethylidene]amino}phenyl)-3-(4-methoxyphenyl)prop-2-en-1-one were synthesized and the physical characterization was carried out along with spectroscopic techniques (FT-IR, ^{1}H NMR, ^{13}C NMR, and MS). The thermotropic properties were investigated by optical polarized light microscopy and differential scanning calorimetry and evaluated as a function of chain length and linking group. In series-A compounds C_3 to C_8 exhibit the nematic mesophase while compounds C_{10}, C_{12}, C_{14}, and C_{16} exhibit a smectic phase as well as the nematic mesophase. Compounds C_1 and C_2 do not exhibit any mesophase. In series-B, all the compounds (C_1 to C_8, C_{10}, C_{12}, C_{14}, and C_{16}) exhibit only the nematic mesophase.

Keywords: chalcone; mesomorphic; nematic mesophase; Schiff base; smectic mesophase

INTRODUCTION

Mesogens with a chalcone central linkage are relatively rare. It has been observed that −CO−CH=CH− linkage is less conducive to mesomorphism compared to −CH=N−(azomethine), −COO− (ester), −N=N−(azo) linkages due to the non linearity and angle strain arising from the keto group [1]. But when −CO−CH=CH− linkage is present

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with other central linkages it becomes condusive to mesomorphism. In the literature there are several reports of mesogenic compounds having chalcone linkage. Many years ago Vora et al. [2] reported a homologous series of polymers containing a chalcone linkage. Soon after words Chudghar et al. [3] reported a homologous series containing on ester-chalcone linkages. Recently Yeap et al. [4] have also synthesized mesomorphic compounds containing on ester-chalcone linkage. In our previous work we have reported the homologous series containing a Schiff base-chalcone linkage [5].

Chalcone is an important class of chemical compound and is being studied extensively because of its significant use or application in various sectors. In the fields of biology and biochemistry, chalcone has been claimed to be one of the compounds that plays a vital role in anti-tumor [6,7], anti-inflammatory [8,9], and anti-malaria [10] activities. It has also been documented that the chalcone possesses a remarkable nonlinear optical (NLO) property, which is an essential element for optical communications devices. The other importance of this compound is its high photosensitivity and thermal stability, which are used in developing various crystalline electro-optical devices [11–13].

In view of the outstanding behavior of chalcone compounds, a systematic study focusing on the synthesis and characterization of this class of compound has been carried out in our present laboratory over past few years [5,14]. In addition, terminal and lateral subtituents also play a vital role in imparting liquid crystallinity to potentially mesogenic compounds [15–19]. The correlation between chemical structure and mesomorphic properties is one of the most important problems in liquid crystals science [20]. An understanding of the influence of different structural elements of the molecules on the physico-chemical characteristics of mesomorphic organic compounds allows chemists to synthesize liquid crystals with required properties.

Laterally substituted mesogens are of considerable interest because these compounds deviate from the classical rod-like shape. However lateral hydroxyl groups are little exploited as it was reported that the phenolic ‘hydroxy’ group may destroy mesomorphism. Gray [21] has proposed that this rarity is associated with intermolecular hydrogen bonding raising the melting point above the mesophase-isotropic liquid transition temperature, and perhaps also encouraging a nonlinear molecular arrangement that is incompatible with mesophase formation. After an initial investigation by Weissflog and Demus [22], a number of homologues series with a trisubstituted benzene nucleus have been reported [23–30]. Schroeder and Schroeder [31] reported a few terminally hydroxy substituted mesogens. Vora and Gupta [32] for the first time reported homologous series with terminal

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and lateral hydroxy groups. Subsequently a few more mesogenic series with a lateral hydroxy group have been reported [33–35]. In general, a rigid lateral substituent disrupts the ordering of the liquid crystalline phase [36–38], causing a significant depression in the clearing point and liquid crystal phase transition.

In our previous work [5] we have synthesized two homologous series containing Schiff base-chalcone linkages having substituted pyrazolone ring as a terminal group. An attempt has been made to synthesize two new homologous series containing the same central linkages, with three aromatic rings in the main core and substituted by a lateral aromatic branch on the terminal ring to study the influence of the terminal and lateral hydroxy group on mesomorphic and thermal stability of these compounds.

EXPERIMENTAL

Reagents and Techniques

For the synthesis of compounds of the homologous series, following materials were used. 4-Hydroxy benzaldehyde, alkyl bromides (Lancaster, England), 2-4 dihydroxy acetophenone, p-amino acetophenone (Lancaster, England) were used without further purification. All the solvents were used after purification using the standard methods described in literature [39].

Elemental analyses (C, H, N) were performed at CDRI, Lucknow. Infrared spectra were recorded with a Perkin-Elmer 2000 FT-IR spectrophotometer in the frequency range 4000–400 cm^{-1} with samples embedded in KBr discs. ^{1}H-NMR spectra of the compound were recorded with JEOL-GSX-400 using $CDCl_3$ as a solvent and TMS as an internal reference at SAIF, IIT Madras, Chennai. ^{13}C NMR spectra of the compound were recorded with BRUKER AVANCE II 400 NMR Spectrometer, SAIF, Chandigarh. Mass spectra (EI) of the compounds were recorded at SAIF, IIT Madras, Chennai. Thin-layer chromatography analyses were performed by using aluminium-backed silica-gel plates (Merck 60 F524) and examined under short-wave UV light.

The phase-transition temperatures were measured using a Shimadzu DSC-50 at heating and cooling rates of 5°C min^{-1}. The DSC data are shown in Table-III. The optical microscopy studies were carried out with a "Leitz Loborlux 12" POL (Wetzler, Germany) polarizing microscope equipped with a Mettler FP52 hot stage and temperature controller. The textures of the compounds were observed using polarized light with crossed polarizers with sample in a thin film between a glass slide and a cover slip.

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SYNTHESIS

Synthesis of n-Alkoxy Benzaldehydes

n-Alkoxy benzaldehydes were prepared by a reported method [40–43].

Synthesis of 1-{4-[(2′,4′-Dihydroxybenzylidene) Amino] Phenyl} Ethanone

A mixture of 2,4-dihydroxy benzaldehyde (1 mmol) and 4-aminoacetophenone(1 mmol) and three drops of acetic acid in absolute ethanol (10 ml) was heated at reflux for 4 hr. The reaction mixture was allowed to cool and stirred at room temperature overnight. The solid was collected and recrystallized from acetone.

Synthesis of 1-(4-{[(1-(2,4-Dihydroxyphenyl)ethylidene]amino} phenyl)ethanone

A mixture of 2,4-dihydroxy acetophenone (1 mmol) and 4-aminoacetophenone(1 mmol) and three drops of acetic acid in absolute ethanol (10 ml) was heated at reflux for 4 hr. The reaction mixture was allowed to cool and stirred at room temperature overnight. The solid was collected and recrystallized from acetone.

Synthesis of 1-{4-[(2,4-Dihydroxybenzylidene) Amino] Phenyl}-3-(4-alkoxyphenyl) Prop-2-en-1-One (Series-A)

A mixture of 1-{4-[(2′,4′-dihydroxybenzylidene) amino] phenyl} ethanone (1 mmol) and n-alkoxy benzaldehyde (1 mmol) was dissolved in the alcoholic sodium hydroxide solution (80 ml ethanol and 10% NaOH solution). The reaction mixture was heated at 80°C for 7 hr. The mixture was left at room temperature overnight. An HCl aqueous solution was added to the mixture, and then yellow precipitate was obtained. The precipitate was washed with water until a neutral aqueous solution was obtained. Then, the solid was washed with methanol and dried under vacuum at 60°C. The obtained yellow solid was purified by recrystallization from acetone. The product was obtained in 45% yield. The physical data of the series A compounds are given in Table 1.

Data

A_{12}, Molecular Formula: $C_{34}H_{41}O_4N$

Elemental analysis, calculated for C 71.70%; H 7.20% and N 9.84%, found: C 71.78%; H 7.12% and N 9.93%, **FT-IR (in KBr)**, 3432 cm^{-1}

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TABLE 1 Transition Temperature Data of the Series (Series-A)

		Transition temperature in °C		
Code No.	R = n-alkyl	S	N	I
A_2	Ethyl	–	–	183
A_3	Propyl	–	–	167
A_4	Butyl	–	146	159
A_5	Pentyl	–	131	154
A_6	Hexyl	–	129	146
A_7	Heptyl	–	112	141
A_8	Octyl	–	106	135
A_{10}	Decyl	–	103	121
A_{12}	Dodecyl	81	99	123
A_{14}	Tetradecyl	85	94	121
A_{16}	Hexadecyl	66	79	109
A_{18}	Octadecyl	69	82	98

(OH-phenolic); 2879, 2968 cm^{-1} (C-H aliophatic) 1573 cm^{-1} (C-H phenyl ring); 1681 cm^{-1} (C=O, chalcone group); 1641 cm^{-1} (-C=N of azomethine); 1597 cm^{-1} (-C=C-, vinyl group of chalcone); 1120–1029 cm^{-1} (C-O-C); **^{1}H-N MR ($CDCl_3$, δ, ppm):** 0.96–1.33 (3 H, t, CH_3, alkyl chain), 1.29–1.71 (m, -$(CH_2)n$, alkyl chain), 3.94–4.03 (2 H, t, -CH_2O, alkoxy chain), 7.56 & 7.90 (2 H, d, olefinic H), 6.32–7.80 (m, phenyl protons), 11.09 (s, phenolic OH), **Mass (FAB):** Molecular ion peak: A_{12} {m/z −528 $(M+1)^+$}.

1-(4-{[1-(2,4-Dihydroxyphenyl)ethylidene]amino}phenyl) -3-(4- methoxyphenyl) Prop-2-en-1-one (Series-B)

A mixture of 1-(4-{[1-(2,4-dihydroxyphenyl)ethylidene]amino}phenyl) ethanone (1 mmol) and n-alkoxy benzaldehyde (1 mmol) was dissolved in the alcoholic sodium hydroxide solution (80 ml ethanol and 10% NaOH solution). The reaction mixture was heated at 80°C for 7 hr. The mixture was left at room temperature overnight. An HCl aqueous solution was added to the mixture, and then yellow precipitate was obtained. The precipitate was washed with swater until a neutral aqueous solution was obtained. Then, the solid was washed with methanol and dried under vacuum at 60°C. The obtained yellow solid was purified by recrystallization from acetone. The product was obtained in 45% yield. The physical data of the series-B compounds are given in Table 2.

TABLE 2 Transition Temperature Data of the Series (Series-B)

		Transition temperature in °C		
Code No.	R = n-alkyl	S	N	I
B_2	Ethyl	–	124	132
B_3	Propyl	–	111	120
B_4	Butyl	–	116	126
B_5	Pentyl	–	110	118
B_6	Hexyl	–	113	124
B_7	Heptyl	–	97	115
B_8	Octyl	–	102	121
B_{10}	Decyl	–	95	108
B_{12}	Dodecyl	–	98	112
B_{14}	Tetradecyl	–	99	110
B_{16}	Hexadecyl	–	112	120
B_{18}	Octadecyl	–	78	119

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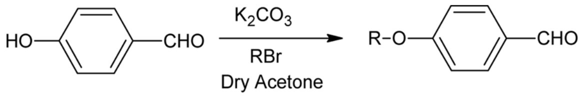
Data

B_{12}, Molecular Formula: $C_{35}H_{43}O_4N$

Elemental analysis, calculated for C 77.63%; H 7.94% and N 2.58%, found: C 77.58%; H 7.87% and N 2.54%, **FT-IR (in KBr)**, 3439 cm^{-1} (OH-phenolic); 2000–1667 cm^{-1} (C-H phenyl ring, out of plane bending); 1684 cm^{-1} (C=O, chalcone group); 1632 cm^{-1} (-C=N); 1606 cm^{-1} (-C=C-, vinyl group of chalcone); 1120–1030 cm^{-1} (C-O-C); **^{1}H-NMR ($CDCl_3$, δ, ppm):** 0.89–0.93 (3H, t, CH_3, alkyl chain), 0.94 (3H, s, CH_3 of acetophenone) 1.44–1.86 (m, -(CH_2)n, alkyl chain), 4.03–4.06 (2H, t, -CH_2O, alkoxy chain), 6.68 and 7.73 (2 H, d, olefinic H), 7.0–7.97 (m, phenyl protons), 9.90 (s, phenolic OH), **Mass (FAB)**: Molecular ion peak: B_{12} {m/z – 540 $(M-1)^+$}.

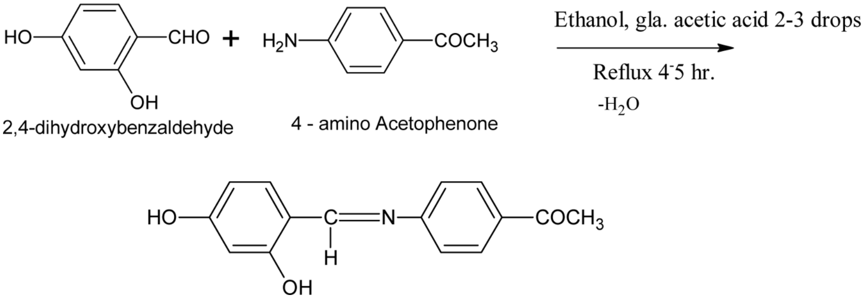
Scheme: Synthetic Route for Series-A & Series-B

Synthesis of -n-alkoxy benzaldehydes

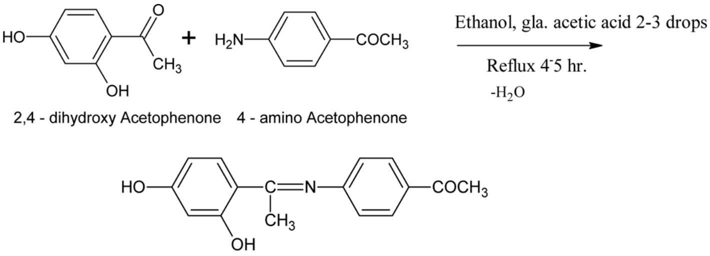


R= C_nH_{2n+1}, n=1 to 8,10,12,14,16,

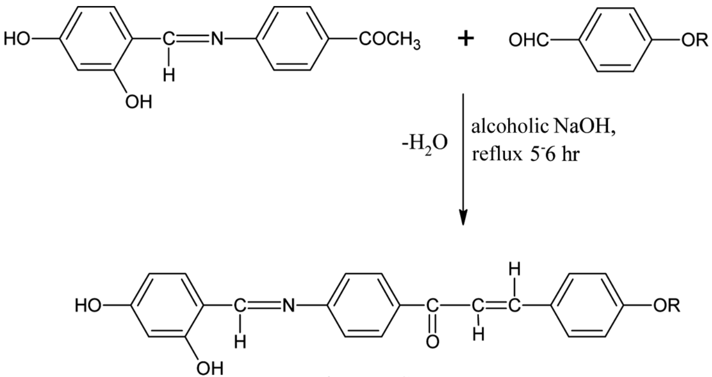
Synthesis of 1-{4-[(2′,4′-dihydroxybenzylidene) amino] phenyl} ethanone



Synthesis of 1-(4-{[(1-(2,4-dihydroxyphenyl)ethylidene]amino}phenyl) ethanone



Synthesis of 1-{4-[(2,4-dihydroxybenzylidene) amino] phenyl}-3-(4-alkoxyphenyl) prop-2-en-1- one (Series-A)



Where R=C_nH_{2n+1} , n= 1 to 8,10,12,14,16.

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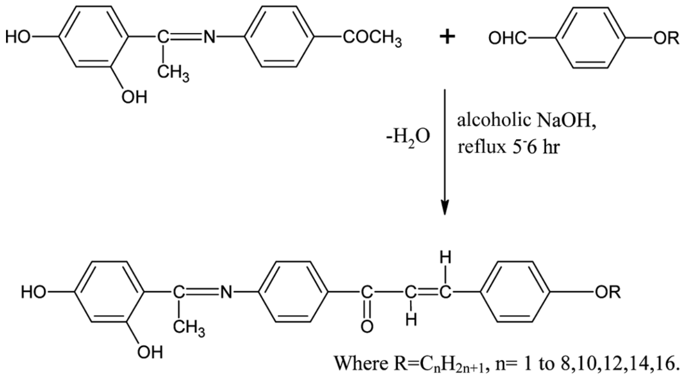
Synthesis of 1-(4-{[1-(2,4-dihydroxyphenyl)ethylidene]amino}-phenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (Series-B)



Where R=C_nH_{2n+1}, n= 1 to 8,10,12,14,16.

RESULT AND DISCUSSION

In the present study, 12 homologues from each of the two series, 1-{4-[(2,4-dihydroxybenzylidene) amino] phenyl}-3-(4-alkoxyphenyl) prop-2-en-1-one (Series-A) and 1-(4-{[1-(2,4-dihydroxyphenyl)ethylidene] amino}phenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (Series-B) were synthesized and their mesomorphic properties studied. The mesomorphic properties of all the synthesized compounds have been characterized by differential scanning calorimetry (DSC) and polarizing optical microscope (POM) attached with Mettler hot stage. Phase identification was based on the optical textures, and the magnitude of the isotropization on enthalpies is consistent with the assignment of each mesophase type, using the classification systems reported by Sack Mann and Demus [44], and Gray and Goodby [45].

In series-A, out of twelve compounds, only ten compounds exhibit the enantiotropic nematic mesophase along with the Smectic-C phase. In series-A, the first two compounds C_1 & C_2 do not exhibiting mesomorphism. The terminal benzene ring is disubstituted by –OH group. One –OH group is ortho to the imine linkage and meta to another –OH group which does not show linearity in the molecule. The two ends of the molecule possess –OH and either –OCH_3 or –OC_2H_5 groups which do not increase polarity and polarizability of the molecule, and hence the first two compounds do not show mesomorphism. As the alkoxy chain length increases, the linearity and polarizability of the molecules increase which increases the cohesive forces, resulting in showing nematic mesomorphism.

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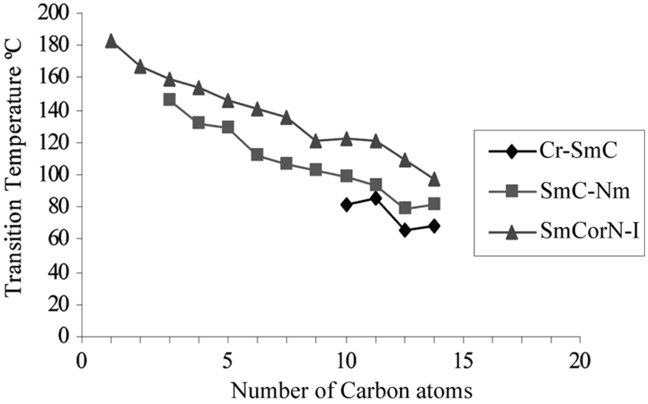

FIGURE 1 Mesomorphic behavior as a function of the number of carbon atoms (n) in the terminal alkoxy chain for series-A.

The transition temperature ranges Δt (S – N) and Δt (N – I) are between 9–18°C and 13–30°C respectively. No odd-even effect is observed for N-I transition temperature in series-A. The smectic mesophase appears for the n-decyloxy to n-hexadecyloxy derivatives. The transition temperature are recorded in Table 1. The plot of transition temperature against the number of carbon atoms in the alkoxy chain (Figure 1) shows a smooth falling tendency for the nematic-isotropic transition temperature throughout the series. Series-A also exhibits a falling tendency of the S_C-N transition temperature for higher homologous.

By incorporating on electron donating group (–CH_3) on the imine linkage in series-B, leading to a disruption of S_C phase and it's only appearance of the nematic phase. The transition temperature are recorded in Table 2. All phase transition temperatures for the methyl substituted compounds are significantly lower compared with the non-substituted series-A. The plot of transition temperature against the no. of carbon atoms in the alkoxy chain shown in Figure 2. This effect is well known and can be explained in terms of steric influence of the methyl group on molecular packing [46].

Comparison of series-A and B indicates that the smectic phase was disappeared in series-B, which was observed in series-A. The methyl lateral group on the imine linkage decreases the S_C phase stability and this also affects the stability of nematic phase, which results in a reduced N–I range compared to series-B [47].

The increase in the S_C-N transition temperature with increasing the alkoxy chain length in compounds of series-A ($n = 10, 12, \ldots.$ etc.), can be explained by the increasing polarisability of the molecule.

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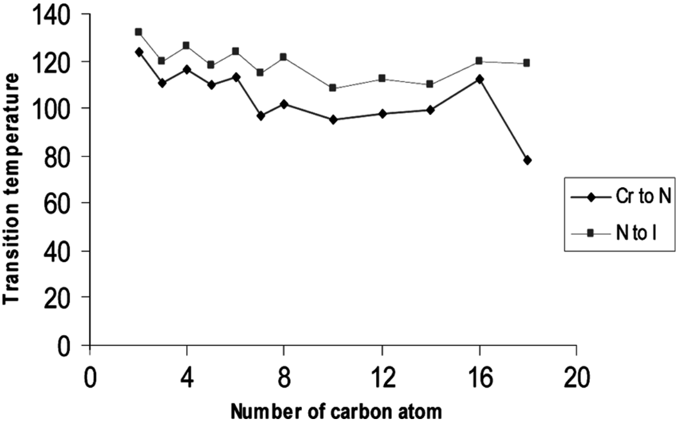

FIGURE 2 Mesomorphic behavior as a function of the number of carbon atoms (n) in the terminal alkoxy chain for series-B.

This will increase the cohesive forces, acting between the sides and plane of the molecules, increasing the tendency for forming the smectic layers. An increase in the nematic and smectic mesophase range is observed with increasing number of methylene units in the terminal group [36]. The enthalpies of the n-tetradecyloxy derivatives of each of the series were measured by DSC. The data is recorded in Table 3.

Both the series are structurally similar, consisting of the three aromatic cores, imine and chalcone linkages, and n-alkoxy as one of the terminal group. Molecules of series-A & B differ only in the lateral group on imine linkage. Series-A has a simple imine linkage and series-B has a lateral methyl group on imine central linkage. It should be remembered that the one central group in both the series is chalconyl group and its effect has been taken to be similar for both series [48,49].

TABLE 3 Transition Temperature and DSC Data of the Series-A & B

Compound	Transition	Peak Temp. (Microscopic temp.) °C	ΔH (Jg^{-1})	ΔS ($jg\text{-}lk^{-1}$)
A_{12}	Cr–S	81 (81)	16.07	0.0453
	S–N	99 (99)	18.01	0.0484
	N–I	123 (124)	20.67	0.0520
A_{14}	Cr–S	85 (86)	11.09	0.0308
	S–N	94 (95)	6.63	0.0180
	N–I	121 (121)	13.06	0.0331
B_{12}	Cr–N	97.89 (98)	7.90	0.0213
	N–I	111.34 (112)	10.78	0.0280
B_{14}	Cr–N	98.79 (99)	9.89	0.0265
	N–I	109.69 (110)	14.56	0.1324

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There is a decrease in thermal stabilities of the liquid crystal phases in the series-B compounds, this may be attributed to the broadening effect caused by the lateral methyl group in an otherwise relatively linear molecule [50]. An increase in the molecular breadth forces the long axes of the molecules apart, as a result of which the interactions are decreased and consequently the liquid crystal temperatures are lowered. A change in the degree of conjugation between the alkene and the carbonyl group in the chalcone linkage will alter the polarisability. This effect of the change in resultant moments is subtle; however, consequentially, the decrease in the polarisability will cause a decrease in the thermal stability of liquid crystal phase in series-B. Variation of $-CH_3$ group in series-B made it possible to observe the effects of structural changes on mesomorphic behavior in a system which had not been studied previously.

CONCLUSION

Systematic studies on a homologous series of compounds have allowed for better understanding of the relationship between the molecular structures and mesomorphic properties. Mesomorphic data obtained in this work suggests that the smectic mesophases might be thermodynamically disrupted due to the presence of a lateral methyl group on imine central linkage. Strong molecular interactions in the mesophase could be partially overcome with surrounding by a more or a less polarisable terminal group and mesophase stabilities of chalcone linkage is less compare to ester linkage liquid crystals. The present study completes our objective of analyzing and establishing the effect of different structural modifications on mesomorphism.

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