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Investigations on Hydrogen-Bonded Liquid Crystals Formed by P-N Alkyl Benzoic Acids and Dodecane Dicarboxylic Acids

R. Rajanandkumar, N. Pongali Sathya Prabu, and M. L. N. Madhu Mohan

Liquid Crystal Research Laboratory (LCRL), Bannari Amman Institute of Technology, Sathyamangalam, India

ABSTRACT

A novel homologous series of hydrogen-bonded liquid crystals (HBLC) have been investigated. These Hydrogen bonds are formed between p-n dodecane dicarboxylic acid (DDC) and various p-n-alkyl benzoic acids (nBA), the present homologous series is referred as DDC + nBA. Seven complexes formed in the present homologous series are characterized by Fourier Transform Infrared spectroscopy (FTIR), Polarizing Optical Microscopy (POM) and Differential Scanning Calorimetry (DSC). Hydrogen bond formation is confirmed by FTIR. Textures observed in DDC + nBA are compared with the standard textures for phase identification. Characteristic phase like nematic, smectic F and smectic G are identified. Odd–even effect is observed in phase transition temperatures and corresponding enthalpy values at isotropic to nematic phase. Phase diagram is constructed from POM and DSC data. The effect of oxygen is discussed with respect to DDC + nBA and DDC + nOBA series. Quantitative treatment for the phase growth is discussed. Light filtering action at various temperatures in nematic phase is also studied.

KEYWORDS

Hydrogen-bonded liquid crystals; light filtering action; phase growth

1. Introduction

Liquid crystalline materials are extensively studied over a century due to the anisotropic nature exhibited by the molecules [1–5]. Owing to tremendous exploitation, these materials have been developed as advanced material for applications in electro optical and display devices. The design and synthesis of hydrogen-bonded liquid crystals (HBLC) with the aid of different chemical structure enhances the utility of these materials in this technical era due to their ease in forming the mesogenic nature due to the complimentary nature exhibited by the chemical constituents involved as the ingredients. Kato and co-workers [6–10] reported a variety of such liquid crystalline materials which paved way for the researchers to study the importance of HBLC. HBLC formed by complementary hydrogen bonds can be synthesized using variety of functional groups [11–13] and by the interaction of weak non covalent bond existing between two dissimilar moieties. These kinds of mesogen are formed by single bond [14–16], double bond [17–19] and even by multiple hydrogen bonds as reported in literature [20, 21]. To obtain mesogen, it is sufficient if one of the ingredients involved in the formation of HBLC exhibits mesogenic properties. Several HBLC, such as molecular liquid crystal [22–26], polymer liquid crystals [27–31], ferroelectric liquid crystals [32, 33] and room temperature liquid crystals [34] have been synthesized and reported.

CONTACT M. L. N. Madhu Mohan ✉ lakshminarayanammohan@bitsathy.ac.in

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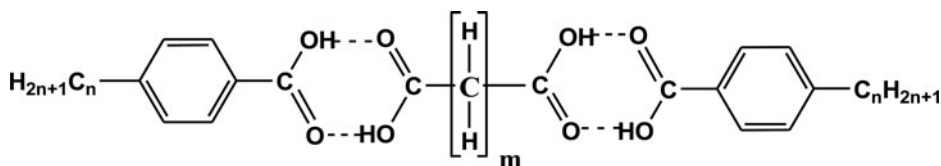


Figure 1. Molecular structure of DDC + nBA, (where $m = 12$).

The present work involves in synthesizing a homologous series of BLC derived from dodecane dicarboxylic acid (DDC) and p-n-alkyl benzoic acids (nBA, n varies from 2 to 8) referred as DDC + nBA resulting in the formation of seven new mesogenic complexes. The presence of carbonyl group stabilizes the hydrogen bond of the mesogens. For application aspects of these synthesized mesogens, thermal stability factor and light filtering action are studied.

2. Experimental

Textural observations are done by Nikon polarizing microscope equipped with Nikon digital CCD camera system having 5 mega pixels and 2560×1920 pixel resolutions. The liquid crystalline textures are processed, analyzed, and stored with the aid of NIS imaging software system. The temperature control of the liquid crystal cell is attained by Instec HCS402-STC 200 temperature controller (Instec, USA) to a temperature resolution of $\pm 0.1^\circ\text{C}$. This unit is interfaced to a computer by IEEE-STC 200 to control and monitor the temperature. Transition temperatures and the corresponding enthalpy values are experimentally determined by Differential Scanning Calorimetry (DSC) (Shimadzu DSC-60). Fourier transform infrared (FTIR) spectra are recorded with KBr pellets (ABB FTIR MB3000) and analyzed by the MB3000 software. The p-n-alkyl benzoic acids (nBA) and dodecane dicarboxylic acid (DDC) are supplied by Sigma Aldrich, Germany and all the solvents used are of HPLC grade.

2.1. Synthesis of hydrogen-bonded liquid crystal homologous series

Intermolecular hydrogen-bonded mesogens are synthesized by adding two moles of p-n-alkyl benzoic acids (nBA) with one mole of dodecane dicarboxylic acid (DDC) in *N,N*-Dimethyl formamide (DMF), respectively. Further, they are subjected to constant stirring for 12 hrs at ambient temperature (30°C) till a white precipitate in a dense solution is formed. The white crystalline crude complexes are obtained by removing excess DMF and are recrystallized. The chemical structure of the present homologous series of p-n-alkyl benzoic acids with dodecane dicarboxylic acid is depicted in Fig. 1. The atomic arrangements of DDC + nBA homologous series with the hydrogen-bonded sites for DDC + 2BA and DDC + 8BA, the first and last homologues, are depicted in Figs. 2(a) and (b), respectively.

3. Results and discussion

All the mesogens isolated under the present investigation are insoluble in water and sparingly soluble in common organic solvents such as methanol, ethanol, and benzene and dichloro methane. However, they show a high degree of solubility in coordinating solvents like dimethyl sulfoxide (DMSO) and pyridine. All these mesogens melt at specific temperatures below $\sim 108^\circ\text{C}$ (Table 1). They also show high thermal and chemical stability when

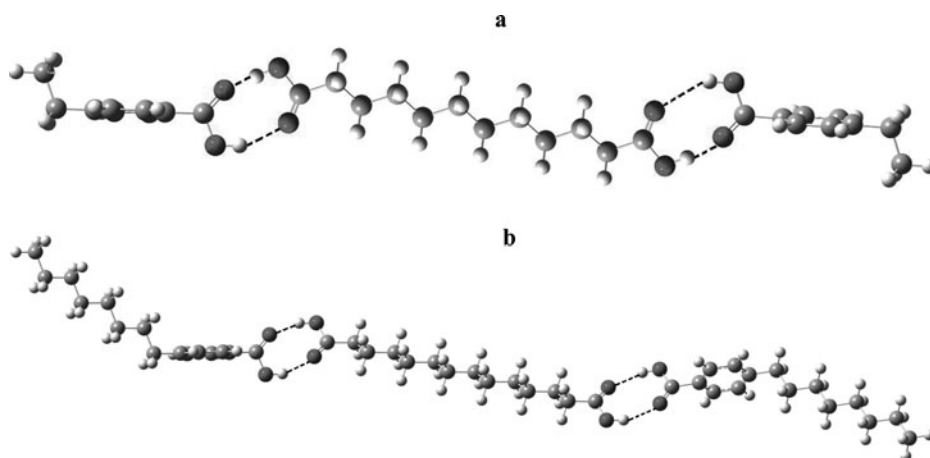


Figure 2. (a) Atomic arrangement of DDC + 2BA depicting formation of H bond. (b) Atomic arrangement of DDC + 8BA depicting formation of H bond.

subjected to repeated thermal scans performed during polarizing optical microscope (POM) and DSC studies.

3.1. Infrared spectroscopy (FTIR)

Infrared spectra for the precursors viz., p-n-alkyl benzoic acids, dodecane dicarboxylic acid, and their seven intermolecular H-bonded complexes (DDC+nBA) are recorded in the solid state (KBr) at room temperature and are presented in Figs. 3(a)–(c). The wavenumber corresponding to the chemical group confirming the intermolecular hydrogen bonding are tabulated in Table 2 for further confirmation. As a representative case, Fig. 3(a) illustrates the FTIR spectra of the mesogen DDC + 8BA in solid state at room temperature (30°C). The doubling

Table 1. Phase transition temperatures of DDC + nBA homologous series obtained by DSC.

Complex	Phase variance	Study	Crystal to Melt	N	F	G	Crystal
DDC + 8BA	N	DSC (h)	92.7 (15.88)	96.6 (0.64)			
		DSC (c)		112.5 (1.08)			83.2 (76.29)
		POM (c)		112.9			83.4
DDC + 7BA	N	DSC (h)	93.4 (21.79)	97 (4.07)			
		DSC (c)		110.2 (2.21)			84.5 (80.9)
		POM (c)		110.6			84.8
DDC + 6BA	N	DSC (h)	89.2 (26.73)	92.8 (2.59)			
		DSC (c)		113.1 (1.17)			79.4 (85.60)
		POM (c)		113.6			79.8
DDC + 5BA	NF	DSC (h)	82.8 (35.30)	97.5 (13.08)			
		DSC (c)		112.4 (1.62)	82.8 (48.42)		74.3 (34.97)
		POM (c)		112.9	83.1		74.4
DDC + 4BA	NF	DSC (h)	90 (46.77)	102.8 (16.75)			
		DSC (c)		114.4 (0.88)	85 (41.53)		77.8 (32.62)
		POM (c)		114.8	85.3		78
DDC + 3BA	G	DSC (h)	107.6 (31.91)				
		DSC (c)				102.6 (21.78)	97.3 (14.85)
		POM (c)				102.9	97.5
DDC + 2BA	G	DSC (h)	93.6 (61.88)				
		DSC (c)				114 (0.59)	84.7 (108.77)
		POM (c)				114.3	84.9

Enthalpy values in J/g are given in parenthesis.

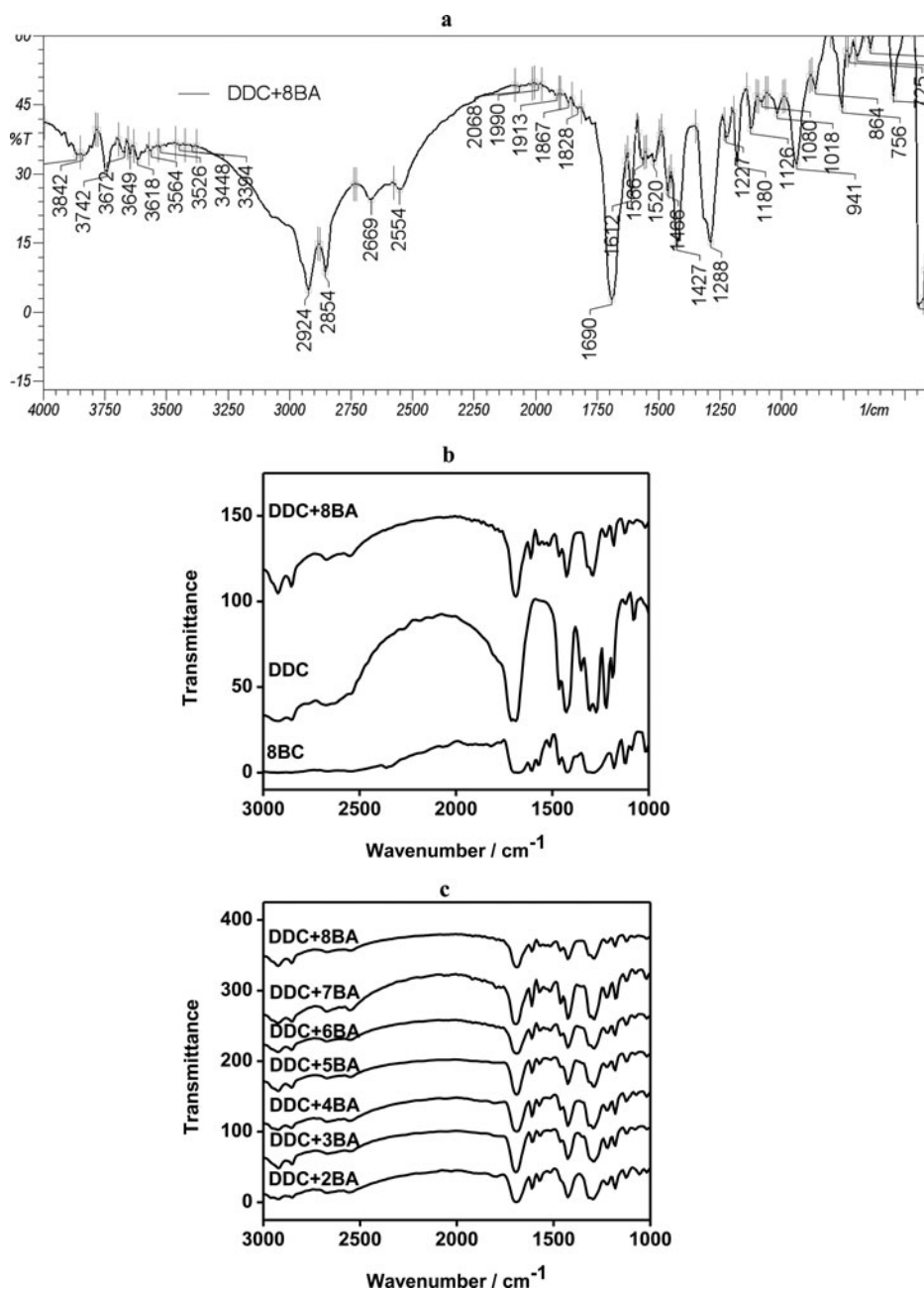


Figure 3. (a) FTIR spectrum of DDC + 8BA complex. (b) FTIR spectra of precursors DDC and 8BA. (c) FTIR spectra of DDC + nBA complexes.

feature of this symmetrical stretching mode confirms the dimeric nature of alkyl benzoic acid at room temperature [35]. A strong intense band noticed at 2924 cm^{-1} in Fig. 3(a) corresponds to the ν (O-H) mode of carboxylic acid group and a sharp peak at 1690 cm^{-1} , clearly suggests the dimer formation of carbonyl group vibration. The FTIR spectra of the precursors viz, DDC and 8BA along with DDC + 8BA is shown in Fig. 3(b). The hypsochromic shift from 1682 cm^{-1} to 1690 cm^{-1} in ν (C=O) of acid ($\sim 8\text{ cm}^{-1}$) and bathochromic shift from 2932 cm^{-1} to 2924 cm^{-1} in the ν (OH) ($\sim 8\text{ cm}^{-1}$) mode of acid in the mesogen suggest

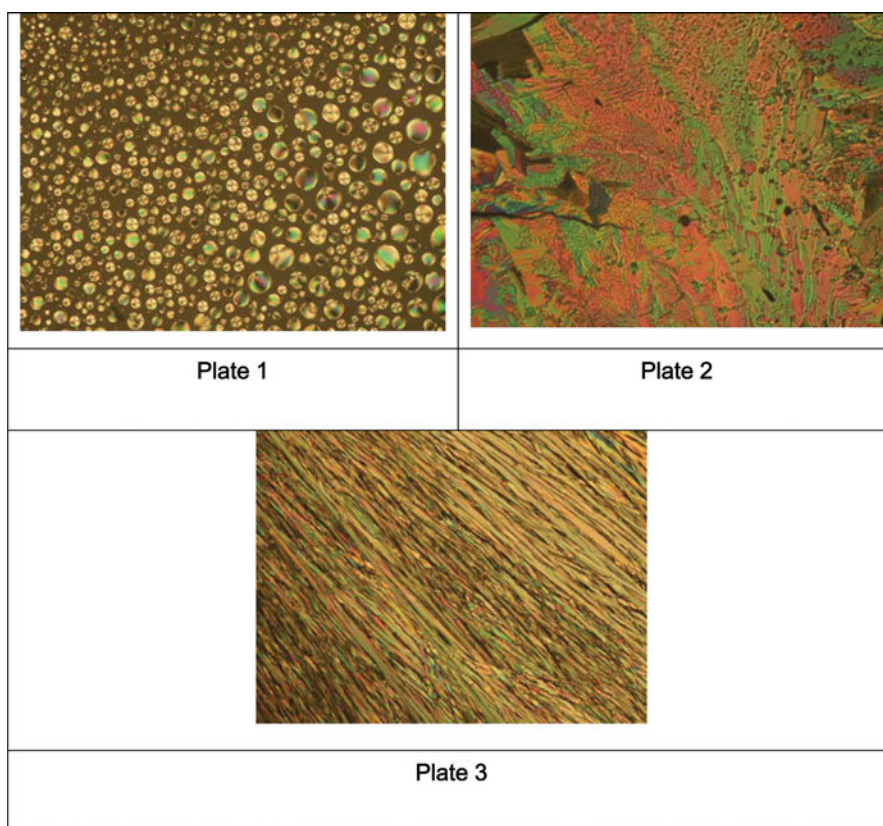
Table 2. FTIR peak assignments for DDC + nBA complexes and their precursors.

Sample	OH	COOH
DDC	2924	1690
8BA	2932	1682
DDC + 2BA	2924	1690
DDC + 3BA	2924	1697
DDC + 4BA	2924	1690
DDC + 5BA	2924	1690
DDC + 6BA	2924	1690
DDC + 7BA	2924	1697
DDC + 8BA	2924	1690

the formation of inter molecular hydrogen bonding between the -COOH group of p-n-alkyl benzoic acids and dodecane dicarboxylic acids [36]. This result concurs with the reported data [37, 38].

3.2. Phase variance of DDC + nBA homologous series

The mesogens of homologous series DDC + nBA are found to exhibit three phases with characteristic textures [39], viz., Nematic (droplets, Plate 1), Smectic F (Chequered board, Plate 2) and Smectic G (multicolored domains, Plate 3), respectively. The general phase sequence of



(All plates are recorded under crossed polarizer's with 5x magnification). **Plate 1.** Nematic droplet texture observed in DDC + 6BA. (All plates are recorded under crossed polarizer's with 5 \times magnification). **Plate 2.** Chequered board texture of Smectic F observed in DDC + 4BA. **Plate 3.** Multicolored domains texture of Smectic G observed in DDC + 2BA.

dodecane dicarboxylic acid with alkyl benzoic acids in cooling and heating run are shown as:

Isotropic \rightleftharpoons N \rightleftharpoons Crystal	(DDC + 8BA)
Isotropic \rightleftharpoons N \rightleftharpoons Crystal	(DDC + 7BA)
Isotropic \rightleftharpoons N \rightleftharpoons Crystal	(DDC + 6BA)
Isotropic \rightleftharpoons N \rightarrow Sm F \rightleftharpoons Crystal	(DDC + 5BA)
Isotropic \rightleftharpoons N \rightarrow Sm F \rightleftharpoons Crystal	(DDC + 4BA)
Isotropic \rightarrow G \rightleftharpoons Crystal	(DDC + 3BA)
Isotropic \rightarrow G \rightleftharpoons Crystal	(DDC + 2BA)

Single arrow indicates monotropic transition while double arrow represents the enantiotropic transitions.

3.3. DSC studies

DSC thermograms are studied in heating and cooling cycle of the samples and the thermograms for all the samples in the cooling run are depicted in Fig. 4(a). Initially the formed complexes are heated with a scan rate of 10°C/min in nitrogen atmosphere and held at its isotropic temperature for 1 min to attain thermal stability. The cooling run is performed immediately with a same scan rate of 10°C/min. The respective equilibrium transition temperatures and corresponding enthalpy values of the mesogens of the homologous series are depicted in Table 1. POM studies confirm transition temperatures obtained by DSC data.

Figure 4(b) illustrates the thermogram of DDC + 4BA hydrogen-bonded complex recorded at a scan rate of 10°C/min for the heating and cooling runs. The cooling run of DSC thermogram shows three distinct transitions namely Isotropic to Nematic, Nematic to Smectic F and Smectic F to crystal with transition temperatures 114.4°C, 85°C, 77.8°C and corresponding enthalpy values 0.88 J/g, 41.53 J/g, and 32.62 J/g, respectively. While in the heating cycle two distinct transitions namely crystal to melt, melt to nematic are obtained at 90°C, 102.8°C with corresponding enthalpy values of 46.77 J/g, 16.75 J/g, respectively. Nematic to smectic F transition is observed to be monotropic while isotropic to nematic is observed to be enantiotropic. Further all these transition temperatures of the present homologous series concur with optical polarizing microscopic studies.

3.4. Phase diagram of DDC + nBA homologous series

The phase diagram of dodecane dicarboxylic acid with p-n-alkyl benzoic acid is depicted in Fig. 5(a). A careful observation of Fig. 5(a) reveals the following points:

- The phase diagram comprises of three phase namely Nematic, Smectic F, and Smectic G.
- Nematic phase is present in homologues series starting from DDC + 4BA to DDC + 8BA, while Smectic F and Smectic G phase are induced in two lower homologues of the series viz., DDC + 4BA, DDC + 5BA and DDC + 2BA, DDC + 3BA, respectively.
- Odd–even effect is observed both in the isotropic to nematic phase transition temperatures and corresponding enthalpy values (Figs. 6(a) and (b)).

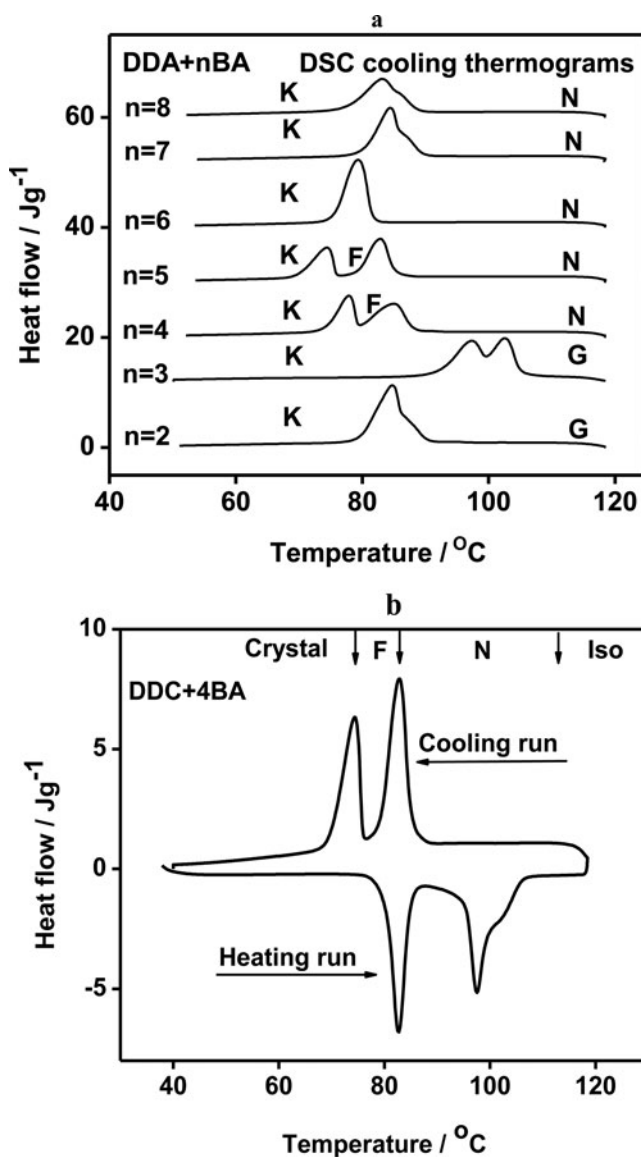


Figure 4. (a) DSC thermogram of DDC + nBA series observed in cooling run. (b) DSC thermogram of DDC + 4BA complex.

- d. In the present homologous series, DDC + 4BA possesses the highest mesogenic thermal range (36.6°C) while DDC + 3BA has the least mesogenic thermal range of 5.3°C
- e. In the higher homologues, DDC + 5BA to DDC + 8BA, the mesogenic thermal range is almost unaltered.

3.5. Influence of oxygen on the mesogenic range

Earlier we have reported [40] the mesogenic nature of dodecane dicarboxylic acid with *p*-n-alkoxy benzoic acid referred as DDC + nOBA (Fig. 5(b)). The present homologous series is designed without oxygen. The following points can be elucidated from the study of DDC + nBA and DDC + nOBA homologues (Figs. 5(a) and (b)).

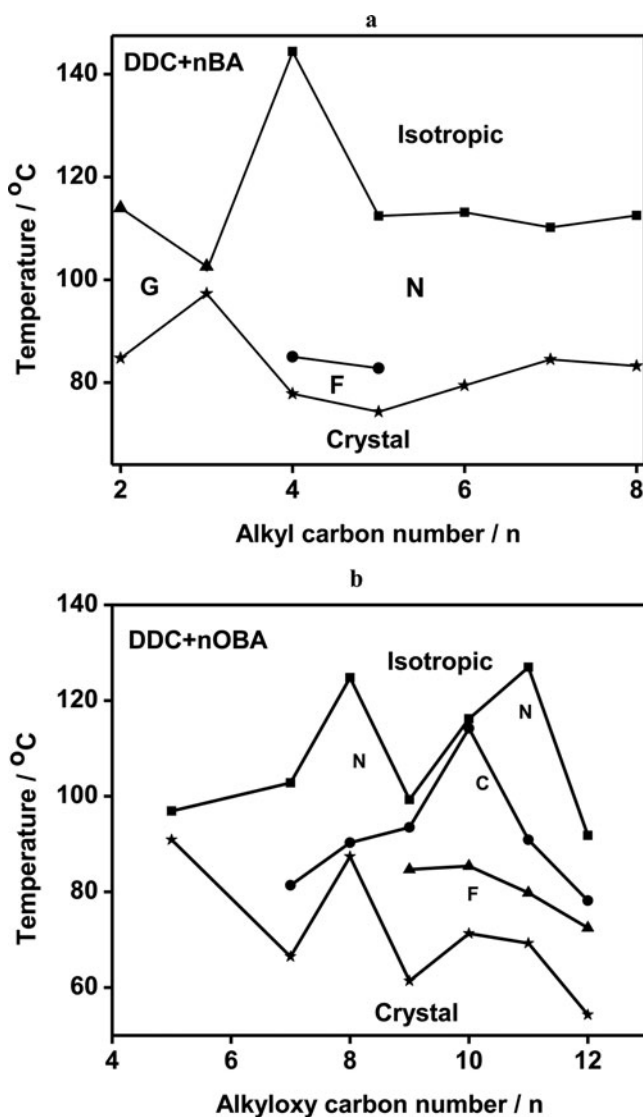


Figure 5. (a) Phase diagram of DDC + nBA homologous series. (b) Phase diagram of DDC + nOBA homologous series.

- The abundance of nematic thermal range in DDC + nOBA is attributed to the presence of oxygen atoms.
- Further the annihilation of smectic C in DDC + nBA is ascribed to the absence of oxygen atoms.
- Molecular structure, particularly the length of the chains of the terminal groups plays a pivotal role in inducement of smectic C phase. The highest carbon number for DDC + nBA is eight, whereas for DDC + nOBA, it is twelve. Hence, the annihilation of smectic C phase in DDC + nBA can be attributed to the length of the terminal molecules.
- The absence of oxygen atoms leads to the elevation of the isotropic temperatures.
- In all the higher order homologous of DDC + nOBA, distribution of thermal ranges corresponding to four different phases is uniform and is attributed to the presence of oxygen.

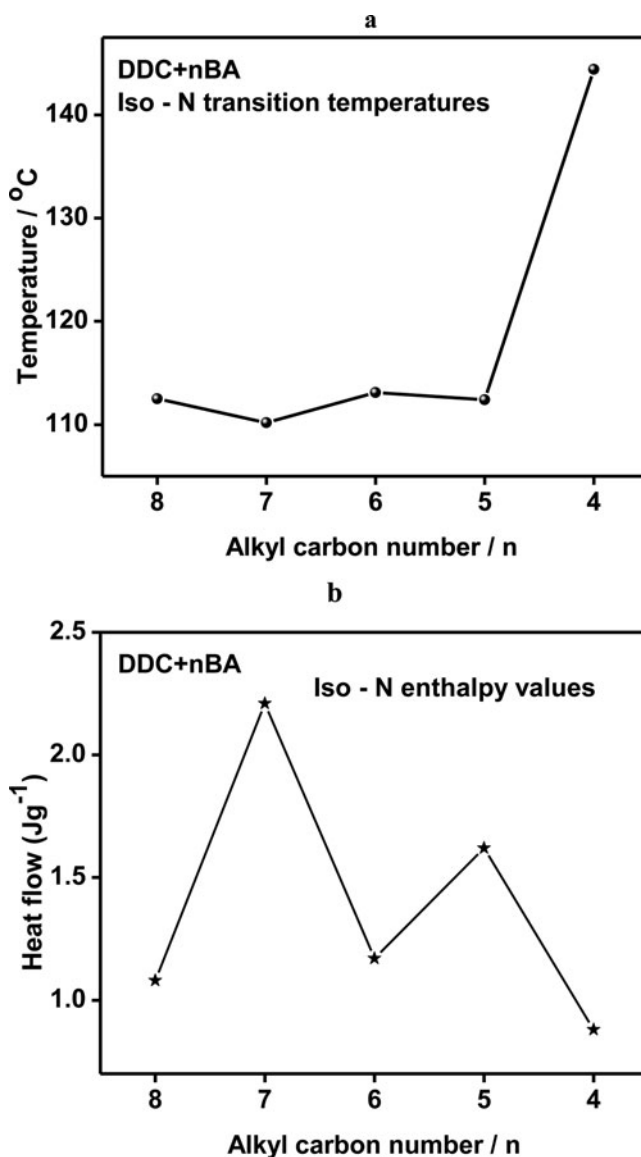


Figure 6. (a) Odd–even effect of DDC + nBA series observed in transition temperatures. (b) Odd–even effect of DDC + nBA series observed in enthalpy values.

3.6. Quantitative treatment of phase growth

Usually from the phase diagrams the occurrence and thermal ranges of various phases are considered. For the first time in the history of Liquid Crystals an attempt is made for the quantitative treatment of phase growth. Areas of individual phases are calculated from the phase diagram and discussed. An interesting and informative observations regarding the area of the particular mesogenic phase spreaded over the entire mesogenic thermal range are made from the phase diagrams of the homologous series DDC + nBA (Fig. 5(a)) and DDC + nOBA (Fig. 5(b)). The two phase diagrams are constructed on an identical area of 270 cm². The areas occupied by the individual phases are calculated from the two phase diagrams and the results are discussed later:

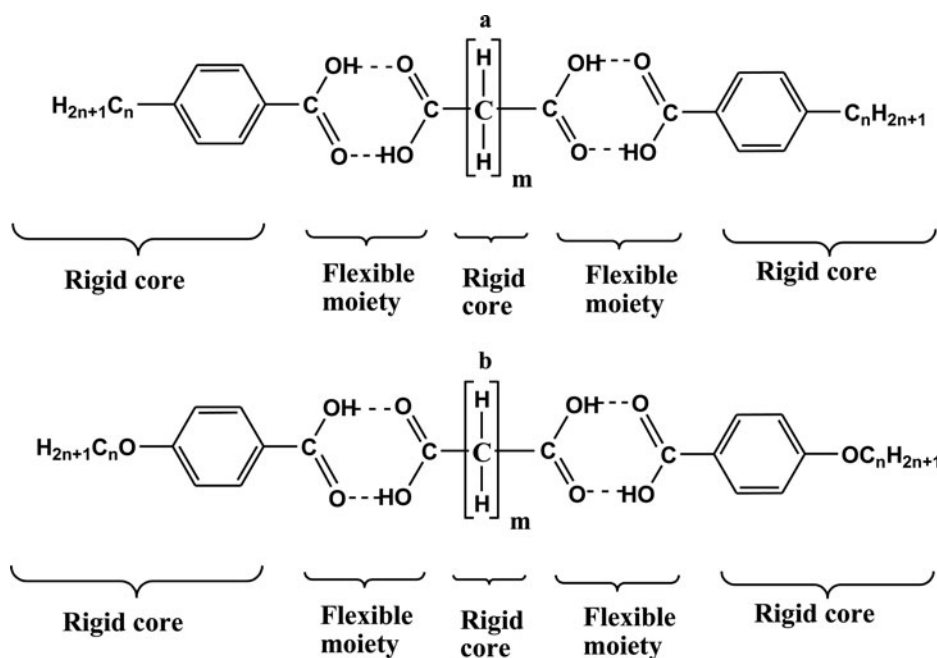


Figure 7. (a) Molecular modeling indicating various types of cores in DDC + nBA series. (b) Molecular modeling indicating various types of cores in DDC + nOBA series.

- i. In DDC + nOBA series, the nematic mesogenic area spreads over 43 cm^2 , where as in the case of DDC + nBA series the area gets doubled viz., the area spreads to 88.2 cm^2 . The area of nematic in DDC + nBA is exactly 2.05 times more than that of the area occupied by the nematic phase in DDC + nOBA. Hence the absence of oxygen increased the presence of nematic phase.
- ii. Area occupied by the long range ordered smectic F phase in DDC + nBA homologous series (4 cm^2) is drastically reduced viz., 3.63 times lesser when compared to the DDC + nOBA homologous series (14.52 cm^2).
- iii. Smectic G phase growth is completely arrested in DDC + nOBA while it showed its presence in DDC + nBA (8.32 cm^2).
- iv. Even though, the magnitude of the mesogenic phases in DDC + nBA is quite less when compared to DDC + nOBA, the area of the total mesogenic range for DDC + nBA (100.52 cm^2) is 1.25 times greater than the DDC + nOBA (80.13 cm^2) series.

3.7. Odd–even effect in DDC + nBA homologous series

The odd–even effect for present homologous series is studied by the plot constructed with alkyl carbon number along x axis and corresponding transition temperatures, enthalpy values on y axis. Figures 6(a) and (b) depict the variation of transition temperatures, enthalpy values with carbon numbers in DDC + nBA homologous series, respectively. The even complexes, namely DDC + 8BA, DDC + 6BA and DDC + 4BA exhibits one type of behavior while the odd complexes DDC + 7BA, DDC + 5BA follow another type of behavior. In the literature [41], such behavior is referred as odd–even effect. This is understood by molecular structure as

Table 3. Thermal stability factor obtained for various phases in DCC + nBA complexes.

Complex	N	F	G
DDC + 8BA	2867	—	—
DDC + 7BA	2502	—	—
DDC + 6BA	3244	—	—
DDC + 5BA	2889	668	—
DDC + 4BA	2931	586	—
DDC + 3BA	—	—	530
DDC + 2BA	—	—	2911

shown in Figs. (7(a) and (b)). The rigid core is the alkyl benzoic acid moiety while the flexible moiety is the hydrogen-bonded structure. The length of rigid core varies with increment in the alkyl benzoic acid carbon number. The rich liquid crystalline phase polymorphism and the associated enthalpy values with increment of alkyl carbon number are thus attributed to this part of the chemical structure. Hence, the rigid core plays a vital role in establishing the pronounced odd–even effect as evinced in the present homologous series (DDC + nBA).

3.8. Thermal stability factor

It is reported [42,43] that when the liquid crystal molecules have two symmetric end chains the phase transition temperatures are higher for the systems. In a symmetric system, the end chains affect the phase transition temperatures as well as the temperature ranges of various phases. The molecular weights of terminal chain could be considered as the measure of balancing and if they are nearly equal, the system is balanced. In other words the system is symmetric about its molecular short axis.

Phase stability is one of the important parameters that govern the utility of the mesogen. In the present case phase stability of nematic is discussed. The term nematic phase stability can be attributed to isotropic to nematic transition temperature as well to the temperature range of nematic phase. It is reasonable to consider both the above factors and define a parameter called stability factor (S). The stability factor for nematic, S_N , is given by

$$S_N = T_{\text{mid}} * \Delta T_N$$

T_{mid} is the mid nematic temperature and ΔT_N , the nematic thermal range. In this manner, the thermal stability of nematic, smectic F and smectic G exhibited by different homologues are calculated and tabulated in Table 3. It can be noticed that even homologues viz., DDC + 4BA and DDC + 6BA exhibit higher thermal stability compared to their odd counterparts.

3.9. Light filtering action

From the report [44, 45] the nematic liquid crystals are capable of transmitting light at all wavelength. These nematic liquid crystals can be used as filter [46] with wide range of wave length from ultraviolet to infrared region. Figures 8(a)–(e) shows the filtering action carried out at different nematic temperatures. DDC + nBA complexes exhibits trends of high pass, low pass band pass and notch filter configurations. DDA + 4BA acts as high pass filter at 88.3°C, as band pass at 87.3°C and as notch filter at 89.3°C. Two high pass and notch filters are observed in DDC + 5BA complex at 86.3°C, 92.2°C, and 90.3°C, 94.2°C, respectively. DDC + 6BA follows the trend of notch filter at 89.3°C, 93.2°C and the trend of low pass filter at 90.3°C. Similarly for other two complexes DDC + 7BA and DDC + 8BA low pass

filter configuration is observed at 93.7°C, 92.2°C, and 93.2°C, high pass filter trend at 92.8°C and 94.2°C, notch filter trend at 91.8°C. The transmission of polarized light indicates that the filtering is stopped at ultraviolet region compared to visible region. Hence these liquid crystal complexes can be used as effective filters for specific region.

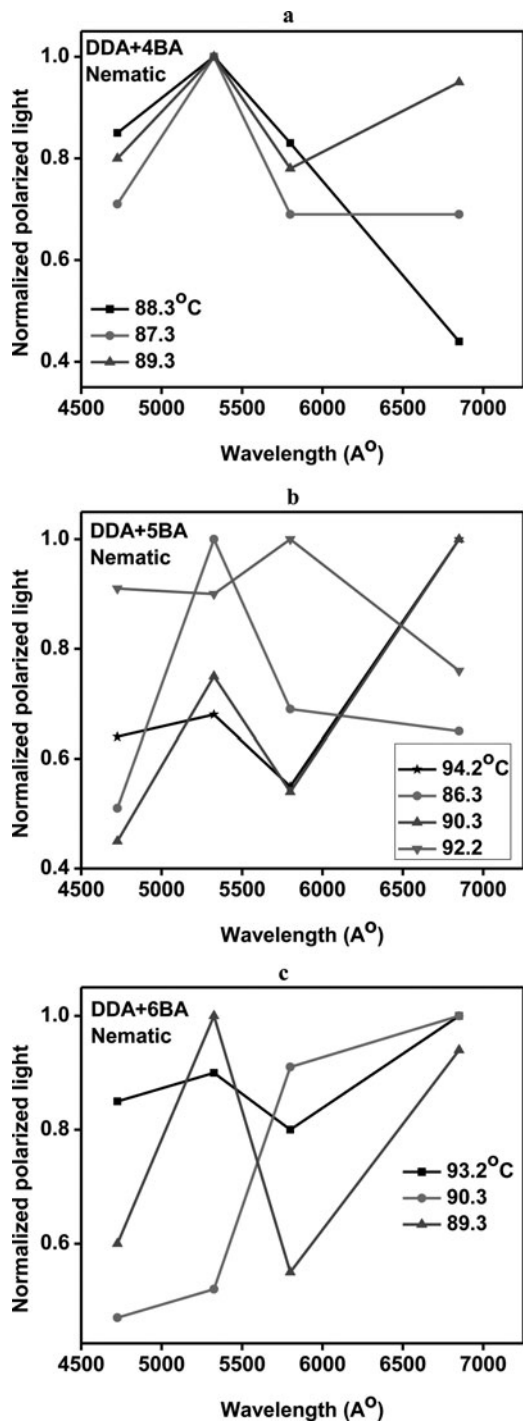


Figure 8. (Continued)

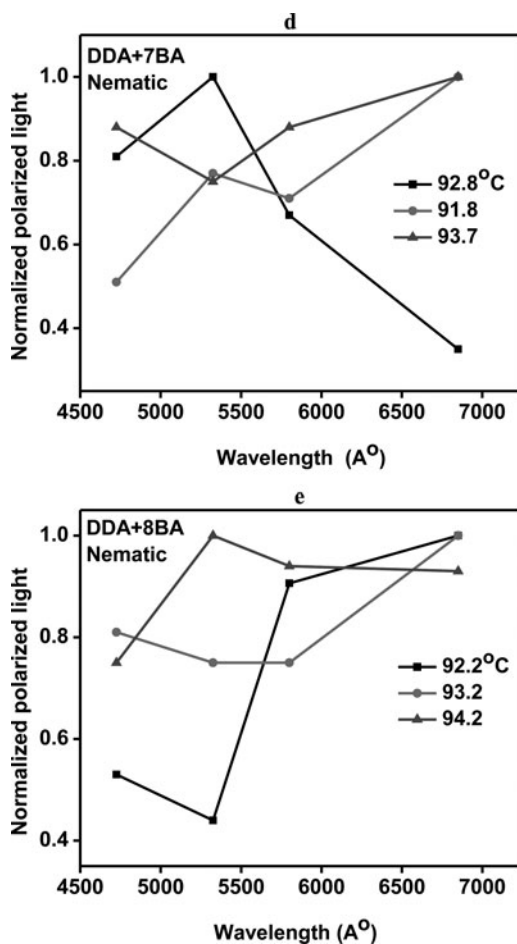


Figure 8. (a) Filtering action observed in DDC + 4BA homologous series. (b) Filtering action observed in DDC + 5BA homologous series. (c) Filtering action observed in DDC + 6BA homologous series. (d) Filtering action observed in DDC + 7BA homologous series. (e) Filtering action observed in DDC + 8BA homologous series.

4. Conclusion

- A successful attempt is made in designing, synthesizing and characterizing seven inter-molecular HBLCs formed between p-n alkyl benzoic acids (nBA) and dodecane dicarboxylic acid (DDC) is made.
- The results obtained for the present homologous series (DDC + nBA) are compared with DDC + nOBA homologous series.
- The influence of the oxygen atoms to the rigid core of the chemical moieties with respect to the phase abundance, their thermal range and thermal stability are discussed.

Acknowledgments

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