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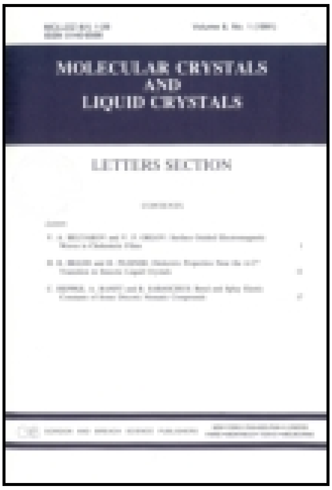

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

Design, Synthesis and Analysis of Chlorohydroquinone Derivatives—Liquid Crystalline Complexes

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Published online: 27 May 2014.

To cite this article: V. Hariharan, N. Pongali Sathya Prabu & M. L. N. Madhu Mohan (2014) Design, Synthesis and Analysis of Chlorohydroquinone Derivatives—Liquid Crystalline Complexes, Molecular Crystals and Liquid Crystals, 593:1, 78-92, DOI: 10.1080/15421406.2013.867225

To link to this article: http://dx.doi.org/10.1080/15421406.2013.867225

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Mol. Cryst. Liq. Cryst., Vol. 593: pp. 78–92, 2014
Copyright © Taylor & Francis Group, LLC
ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2013.867225

Design, Synthesis and Analysis of Chlorohydroquinone Derivatives—Liquid Crystalline Complexes

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A novel linear double hydrogen-bonded liquid crystalline material is designed and isolated. The ingredients of the mesogens synthesized are chloro hydroquinone (ClHQ) and p-n alkyloxy benzoic acid (nBAO) where n varied from pentyl to dodecyl alkyloxy carbon numbers. The series is abbreviated as ClHQ + nBAO, and seven of eight synthesized complexes are found to exhibit liquid crystalline phases. These liquid crystalline complexes are characterized by Fourier Transformation Infrared spectroscopy (FTIR) in order to confirm the formation of hydrogen bond. Mesophases and their corresponding transition temperatures are studied by polarizing optical microscopy (POM) and their respective enthalpy values along with the order of the transitions are determined by Differential Scanning Calorimetry (DSC). The experimental measurement of tilt angle is carried out by optical extinction method and fitted to the theoretically predicted mean field exponent which is found to be in good agreement. Dielectric relaxations at different temperatures in nematic phase for the complexes have been carried out and their corresponding activation energy values are calculated from Cole–Davidson plots. Light filtering action in the liquid crystalline complexes is done to validate the commercial applications of these mesogens.

Keywords Benzoic acid; dielectric relaxations; DSC; hydrogen bond; liquid crystals

1. Introduction

Liquid crystals (LC) are well established in basic research as well as in development for applications and commercial uses due to its intermediate state between ordinary liquids and three dimensional solids [1]. Depending on the transition temperature and thermal span, hydrogen-bonded liquid crystals (HBLC) can form single [2–4]-, double [5–8]-, and multiple-bonded HBLC [9,10]. On the other hand, the application of supramolecular chemistry in the field of liquid crystalline materials yields a proper combination of molecular interactions and the shape of the molecules [11–15]. HBLC mesogens exhibits various new phases and lower the electrical and thermal threshold values required for device applications, hence interest in HBLCs grew for the past few decades [16–19]. From the materials point of view, HBLC hold promising issues relating to optical, thermal, and electrical properties

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[20–25]. We have earlier reported [5] that the light modulation and optical shuttering action was achieved through HBLC and also the wide thermal range of nematic and smectic C phases. Some of the HBLC are found to exhibit ambient temperature phases which facilitates for device applications. Dielectric studies will support to understand different mesophases through various relaxation modes. This information is critical for obtaining the threshold values, activation energies, and dielectrical responses of a given mesogenic phases.

In view of the above material requirement, a novel series of HBLC are designed, isolated, and characterized. Thermal, electrical properties of these mesogens are also discussed.

2. Experimental

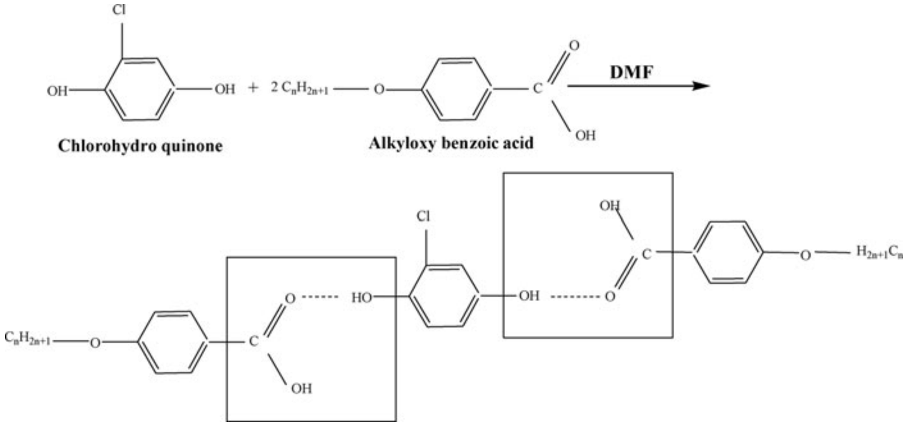
Optical textural observations were recorded using Nikon polarizing microscope equipped with Nikon digital Charge-Coupled Device (CCD) camera system with 5 mega pixels and 2560×1920 pixel resolutions. Liquid crystalline textures were stored, retrieved, and analyzed using NIS imaging software system. Temperature control of the liquid crystal cell was equipped by instec HCS 402–STC 200 temperature controller (Instec, USA) to a temperature resolution of ± 0.1 °C. This unit was further interfaced to computer by IEEE –STC 200 to control and monitor the temperature by using Wintemp software. Liquid crystal sample was filled by capillary action into a commercially available (Instec, USA) polyamide-buffed cell with 10 μm spacer. Transition temperatures and corresponding enthalpy values were experimentally deduced by differential scanning calorimetry (DSC) (Shimadzu DSC-60, Kyoto, Japan). Fourier Transformation Infrared spectroscopy (FTIR) was recorded (ABB FTIR MB3000, Quebec, Canada) and analyzed with MB3000 software. Dielectric studies were performed with Agilent impedance analyzer (4192A, USA). The p-n-alkyloxy benzoic acid (nBAO) and chloro hydroquinone were supplied by Sigma Aldrich, (Steinheim, Germany) and all the solvents were of High-Performance Liquid Chromatography (HPLC) grade.

3. Synthesis of ClHQ + nBAO

The liquid crystalline complexes of Chloro hydroquinone and alkyloxy benzoic acids (ClHQ + nBAO; $n = 5$–12) are synthesized by mixing one mole of chloro hydroquinone with two moles of alkyloxy benzoic acids (pentyl to dodecyl) to obtain intermolecular HBLC abbreviated as ClHQ + nBAO. The molecular structure and scheme of ClHQ + nBAO are represented as Scheme 1.

4. Results and Discussion

ClHQ + nBAO complexes synthesized are white solid powder in nature and are found to be stable at room temperature (∼30°C). The solubility test indicated that they are insoluble in water and sparingly soluble in common organic solvents such as methanol, ethanol, benzene, and dichloro methane. They show a high degree of solubility in coordinating solvents like dimethyl sulfoxide (DMSO), dimethyl formamide (DMF), and pyridine. Moreover, the prepared complexes have melting points between 74.5°C to 116.5°C (Table 1) and exhibited high thermal and chemical stability when subjected to repeated thermal scans performed during polarizing optical microscopy (POM) and DSC studies.



Scheme 1. Scheme representing the formation of ClHQ + nBAO hydrogen-bonded series.

4.1. Phase Identification

The occurrence of various phases and observed phase transition temperatures with corresponding enthalpy values obtained both the cooling and heating cycles of DSC for the ClHQ + nBAO complexes along with the phase variance of the precursors (nBAO) is tabulated as Tables 1 and 2, respectively. The observed transition temperatures obtained by DSC are in reasonable agreement with POM analysis (Table 1). Further from this data, the phase transitions are classified as monotropic and enantiotropic.

4.2. Mesogenic Nature of ClHQ + nBAO

ClHQ + nBAO homologous series, found to exhibit various LC phases such as nematic (N) (schlieren four brushes, Plate 1), smectic C (broken focal conic texture, Plate 2), and smectic G (multicolored smooth mosaic like texture, Plate 3, i.e., mosaic platelets separated by very fine lines containing schlieren-like brushes, but does not show actual intersections in the form of crosses), respectively. Phase sequence of ClHQ + nBAO series in heating and cooling run of temperature may be represented as:

$$\text{Isotropic} \rightarrow \text{N} \rightarrow \text{Sm C} \rightarrow \text{Sm G} \rightleftharpoons \text{Crystal(ClHQ + 12BAO)}$$

$$\text{Isotropic} \rightarrow \text{N} \rightarrow \text{Sm C} \rightleftharpoons \text{Sm G} \rightleftharpoons \text{Crystal(ClHQ + 11BAO)}$$

$$\text{Isotropic} \rightarrow \text{N} \rightleftharpoons \text{Sm C} \rightleftharpoons \text{Sm G} \rightleftharpoons \text{Crystal(ClHQ + 10BAO)}$$

$$\text{Isotropic} \rightarrow \text{N} \rightleftharpoons \text{Sm C} \rightarrow \text{Sm G} \rightleftharpoons \text{Crystal(ClHQ + 9BAO)}$$

$$\text{Isotropic} \rightarrow \text{N} \rightarrow \text{Sm C} \rightleftharpoons \text{Sm G} \rightleftharpoons \text{Crystal(ClHQ + 8BAO)}$$

$$\text{Isotropic} \rightarrow \text{N} \rightarrow \text{Sm G} \rightleftharpoons \text{Crystal(ClHQ + 7BAO)}$$

$$\text{Isotropic} \rightleftharpoons \text{N} \rightarrow \text{Sm G} \rightleftharpoons \text{Crystal(ClHQ + 6BAO)}$$

In the above phase variance, monotropic and enantiotropic transitions are depicted as single and double arrows, respectively. All synthesized complexes exhibit mesogenic in nature except ClHQ + 5BAO.

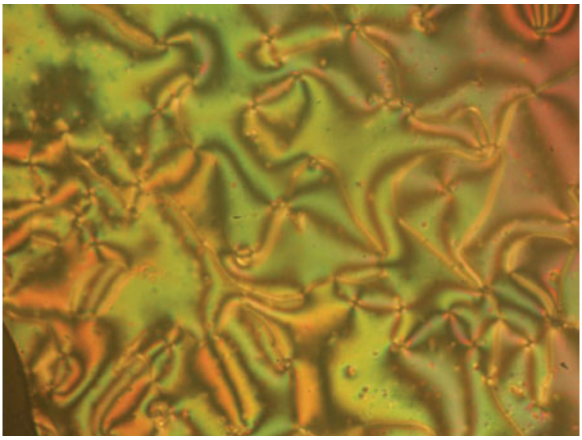


Plate 1. Nematic threaded four brush texture observed in ClHQ + 10BAO complex.

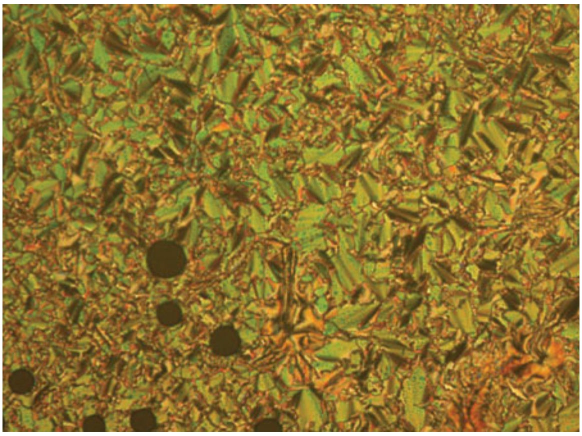


Plate 2. Broken focal conic Smectic C texture observed in ClHQ + 11BAO complex.

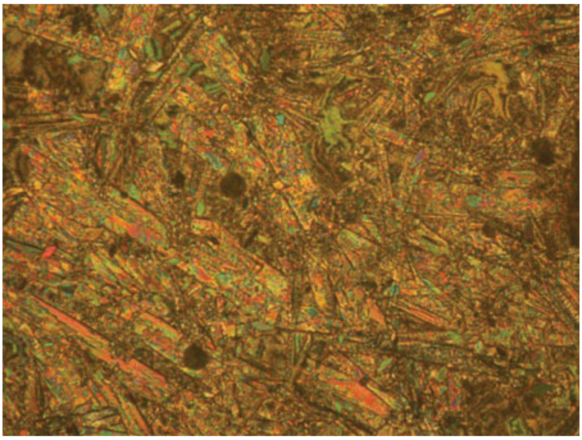


Plate 3. Multicolored smooth mosaic like schlieren texture of smectic G observed in ClHQ + 7BAO complex.

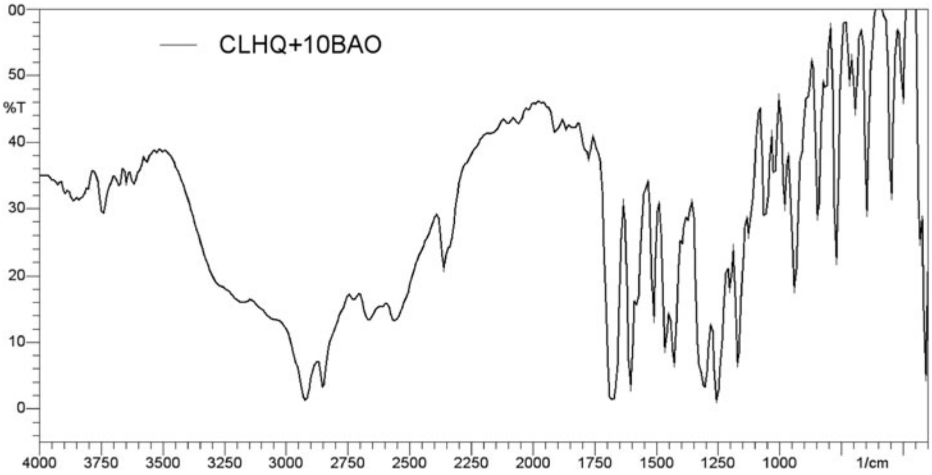


Figure 1. FTIR spectrum of ClHQ + 10BAO complex.

4.3. Fourier Transform Infrared Spectroscopy (FTIR)

To illustrate the formation of hydrogen bond between Chloro hydroquinone and alkyloxy benzoic acid, FTIR spectroscopic technique is performed for all the complexes of ClHQ + nBAO in the solid state (KBr) at room temperature. As a representative case, the FTIR spectra of ClHQ + 10BAO in solid state at room temperature depicted as Fig. 1 is discussed briefly. It has been already reported that [26] the alkyloxy benzoic acids exhibits the stretching vibration of C=O around 1760 cm^{-1} which is present in its monomeric form. Similarly in the case of chloro hydroquinone OH stretching, C–H scissoring and C–Cl stretching vibration occurs around 3325 cm^{-1}, 1470 cm^{-1}, and 743 cm^{-1}, respectively [27]. Literature report [28] suggested that formation of hydrogen bonds due to alkyloxy benzoic acid results in lowering the frequency of the carbonyl complexes which has been evidenced in the present hydrogen-bonded complexes. It is interesting to note that the appearance of a sharp peak around 1670 cm^{-1} for ClHQ + 10BAO complex which clearly suggests the dimer formation [29]. It is known that this dimerization weakens the C=O bond and lowers the stretching force constant K, resulting in a lowering of the carbonyl frequency of saturated acid to $\sim$1670 cm^{-1}. Hence, in the present complexes, the formation of H bonding is evidenced by FTIR analysis. A similar pattern of results are observed in all the synthesized hydrogen-bonded complexes.

4.4. Differential Scanning Calorimetry

The DSC is calibrated for transition temperatures and enthalpy values by a standard reference material (Indium). The set procedure to record DSC thermograms in heating and cooling cycle for all the complexes are as follows: The empty aluminum pan is weighed initially and the desired mesogen is filled to the predetermined weight before crimping it. The crimped aluminum pan along with the sample is placed in the heating chamber of DSC. Another empty crimped aluminum pan is taken as the reference. In heating chamber nitrogen gas is purged at a constant rate. DSC thermograms are recorded, stored, and analyzed by TA60 data software. The mesogen is heated with scan rate of 5°C/min and held at its isotropic temperature for 2 min so as to attain thermal stability. A computer program

Table 1. Transition temperatures and enthalpy values obtained for ClHQ + nBAO HBLC series by various techniques

Complex	Phase variance	Techniques	Crystal to Melt	N	C	G	Crystal (K)
ClHQ + 12BAO	NCG	DSC(h)	96.8 (76.26)	@	@	@	—
		DSC(c)	—	125.7 (8.00)	117.0 (11.41)	83.9 (19.19)	79.3 (49.65)
		POM(c)		126.5	117.5	84.2	79.5
ClHQ + 11BAO	NCG	DSC(h)	97.5 (61.20)	@	119.3 (0.98)	@	—
		DSC(c)	—	128.5 (2.61)	118.2 (1.90)	82.5 (12.73)	69.9 (9.09)
		POM(c)		130.0	118.6	82.7	70.0
ClHQ + 10BAO	NCG	DSC(h)	87.1 (18.44)	@	113.4 (0.75)	93.3 (2.14)	—
		DSC(c)	—	126.9 (2.54)	113.1 (1.27)	90.4 (9.78)	69.8 (7.87)
		POM(c)		127.8	113.6	90.7	70.1
ClHQ + 9BAO	NCG	DSC(h)	94.1 (63.22)	@	107.1 (1.40)	@	—
		DSC(c)	—	122.1 (2.53)	105.3 (2.27)	87.7 (14.78)	62.6(33.59)
		POM(c)		122.9	105.9	88.1	62.7
ClHQ + 8BAO	NCG	DSC(h)	74.5 (31.60)	@	@	100.1 (19.98)	—
		DSC(c)	—	134.4 (5.0)	99.4 (2.12)	94.0 (22.72)	53.0 (30.34)
		POM(c)		135.0	99.9	94.4	53.2
ClHQ + 7BAO	NG	DSC(h)	92.8 (59.84)	@	—	@	—
		DSC(c)	—	131.9 (3.47)	—	85.8 (19.68)	61.5 (24.22)
		POM(c)		132.6		86.3	61.7
ClHQ + 6BAO	NG	DSC(h)	105.1 (29.76)	120.4 (0.63)	—	@	—
		DSC(c)	—	118.1 (6.57)	—	106.6 (3.63)	91.2 (27.69)
		POM(c)		118.9		107.1	91.4
ClHQ + 5BAO	—	DSC(h)	116.5 (49.28)	—	—	—	—
		DSC(c)	—	—	—	—	109.9 (53.93)
		POM(c)					110.3

Temperatures in °C, Enthalpy (J/g) is given in parenthesis, @ monotropic transition, (h) heating run, (c) cooling run

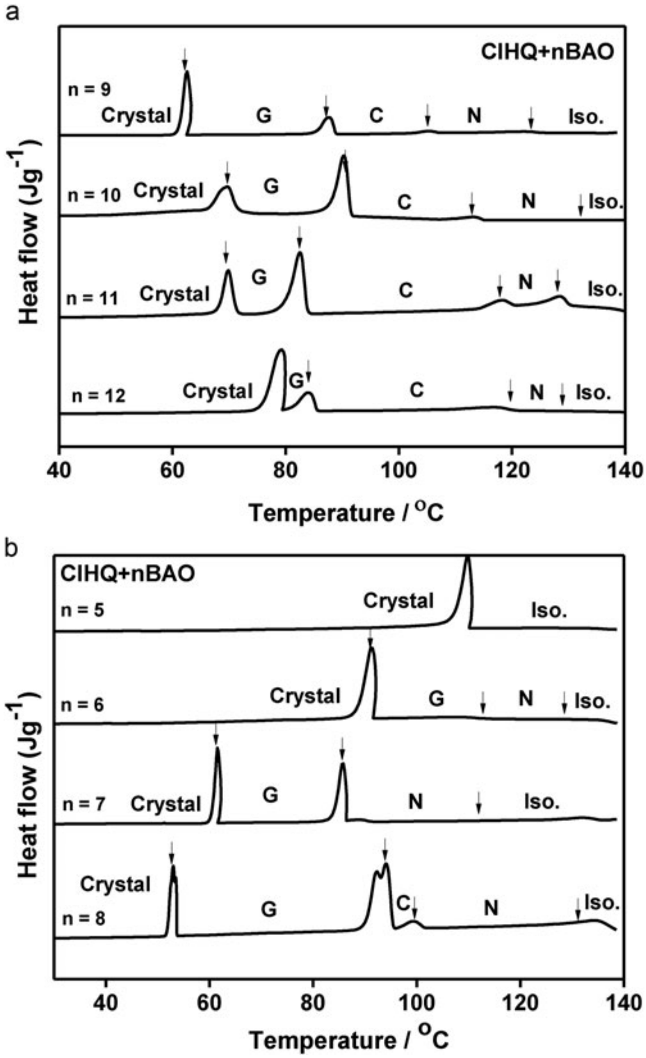


Figure 2. **(a) & (b)** DSC exothermic thermograms of ClHQ + nBAO series.

is used for heating and cooling the sample. The cooling run is performed with the same scan rate. Experimentally obtained equilibrium transition temperatures and corresponding enthalpy values of the mesogens are listed separately in Table 1 which is in agreement with the POM analysis. The eight DSC exothermic thermograms of ClHQ + nBAO complexes are shown in Figs. 2a and b.

4.5. Phase Diagram

The phase diagram of the present homologous series is constructed by POM and DSC data. It is shown as Fig. 3 and is discussed below:

Table 2. Phase variance exhibited by ClHQ + nBAO series

S. NO	ClHQ + nBAO series	Phase variance	Precursor (nBAO)	Phase variance
1	ClHQ + 12BAO	NCG	12BAO	NC
2	ClHQ + 11BAO	NCG	11BAO	NC
3	ClHQ + 10BAO	NCG	10BAO	NC
4	ClHQ + 9BAO	NCG	9BAO	NC
5	ClHQ + 8BAO	NCG	8BAO	NC
6	ClHQ + 7BAO	NG	7BAO	NC
7	ClHQ + 6BAO	NG	6BAO	N
8	ClHQ + 5BAO	*	5BAO	N

*Represents nonmesogenic nature.

Table 3. Values of relaxation frequency and corresponding temperature along with the activation energies for ClHQ + 9BAO and ClHQ + 10BAO complexes

Complex (nematic phase)	T (°C)	f_r (KHz)	ε'' max	α (rad)	Activation energy (eV)
ClHQ + 9BAO	116.2	2.1	1.7943	0.8004	4.095
	114.2	2.2	1.5716	0.7308	
	112.2	2.7	1.3438	0.7134	
ClHQ + 10BAO	121.7	0.8	0.0782	0.3654	0.6339
	117.7	0.8	0.0791	0.3480	
	113.7	0.9	0.0770	0.3306	
	111.7	0.85	0.0775	0.2436	

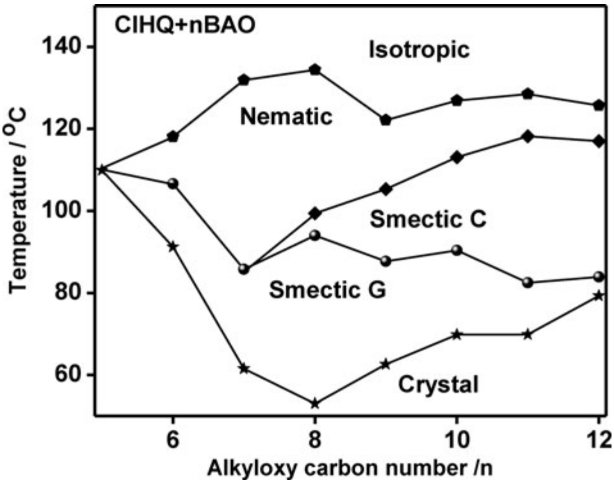


Figure 3. Phase diagram of ClHQ + nBAO homologous series.

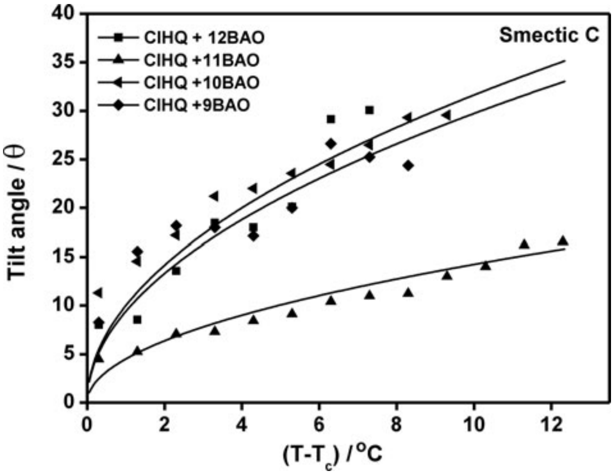


Figure 4. Temperature variation of tilt angle in smectic C phase of ClHQ + nBAO series.

(i) The phase diagram is composed of an orthogonal and two smectic phases. The observed orthogonal phase is nematic and smectic phases that are C and G, respectively.
(ii) Nematic C and smectic C are observed in the entire series, whereas smectic C originates from octyl carbon number quenching nematic.
(iii) The total mesogenic range gradually increases with increase in the carbon number and saturates.
(iv) Heptyl carbon number possesses largest nematic thermal range, whereas dodecyl carbon number possesses largest thermal range for smectic C phase as the carbon number is increased.
(v) Smectic C phase grows quenching smectic G and nematic thermal ranges.

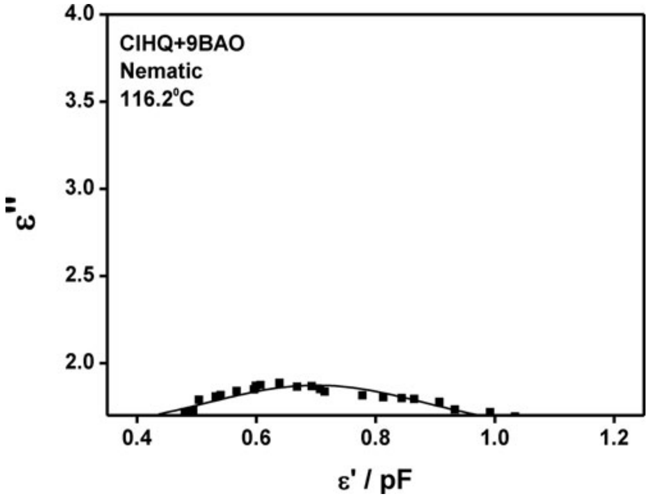


Figure 5. Cole–Davidson plot in smectic C phase of ClHQ + 9BAO complex.

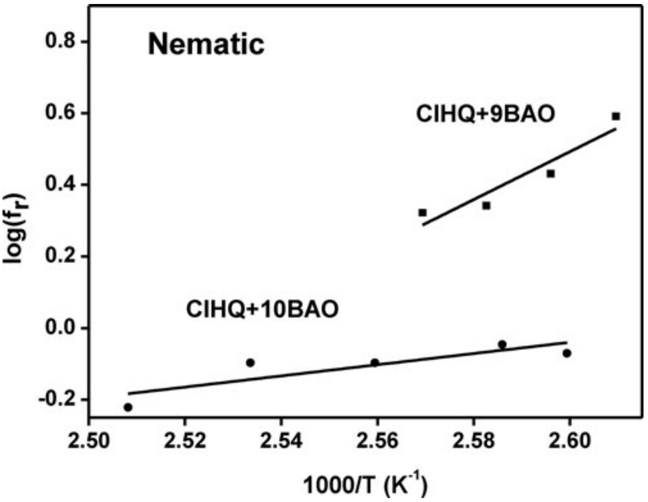


Figure 6. Arrhenius plots for ClHQ + 9BAO and ClHQ + 10BAO complexes.

(vi) Interestingly, odd–even effect [30] is observed in the envelope of smectic C to smectic G phase transitions temperatures.

4.6. Optical Tilt Angle Studies in Smectic C

Optical tilt angle has been experimentally measured by optical extinction method [31]. Figure 4 shows the temperature dependence on the optical tilt angle of ClHQ + 12BAO, ClHQ + 11BAO, ClHQ + 10BAO, and ClHQ + 9BAO complexes, respectively. The theoretical fit obtained from the mean field theory [32] is represented by solid lines. From all the complexes, it is observed that the tilt angle increases with decrement in temperature. The magnitudes of maximum tilt angle in the case of ClHQ + 12BAO, ClHQ + 11BAO,

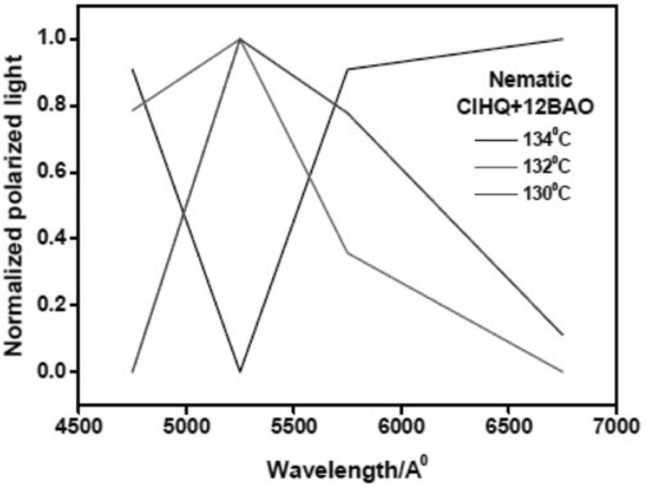


Figure 7. Filtering action in nematic phase of ClHQ + 12BAO complex.

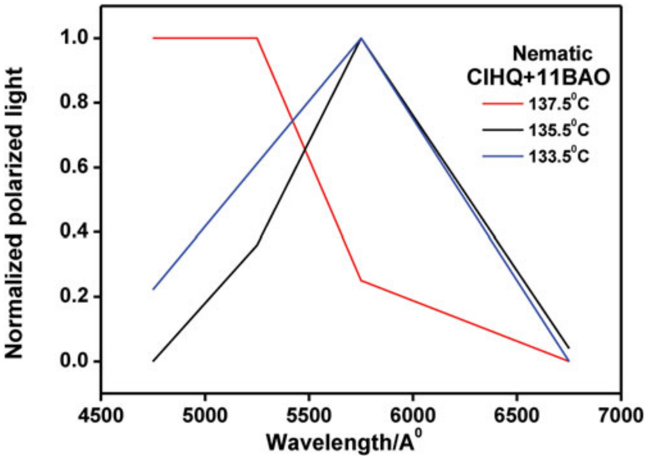


Figure 8. Filtering action in nematic phase of ClHQ + 11BAO complex.

ClHQ + 10BAO, and ClHQ + 9BAO are observed to be 27°, 16.54°, 21°, and 24.36° respectively. These large magnitudes of the tilt angle are due to the direction of soft covalent hydrogen bond interaction which spreads along molecular long axis with finite inclination.

Since the tilt angle is a primary order parameter [33] and the temperature variation is estimated by fitting the observed data of θ (T) to the relation

$$\theta\ (T)\ \alpha\ (T_C - T)^{\beta}$$

The critical exponent of β value estimated by fitting the data of θ (T) to the above equation is found to be 0.50 to agree with the Mean Field prediction value. Further, the

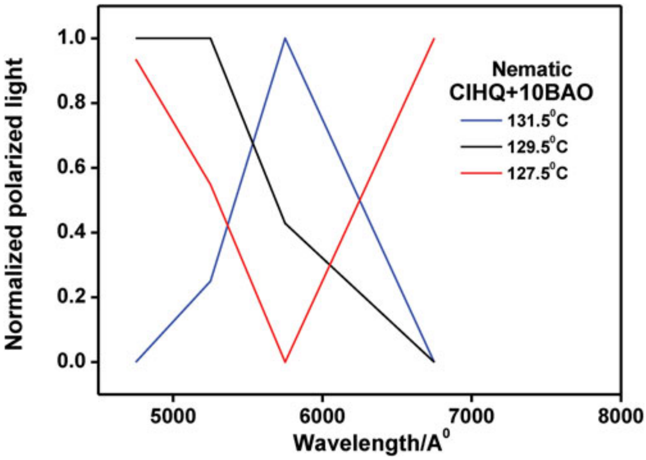


Figure 9. Filtering action in nematic phase of ClHQ + 10BAO complex.

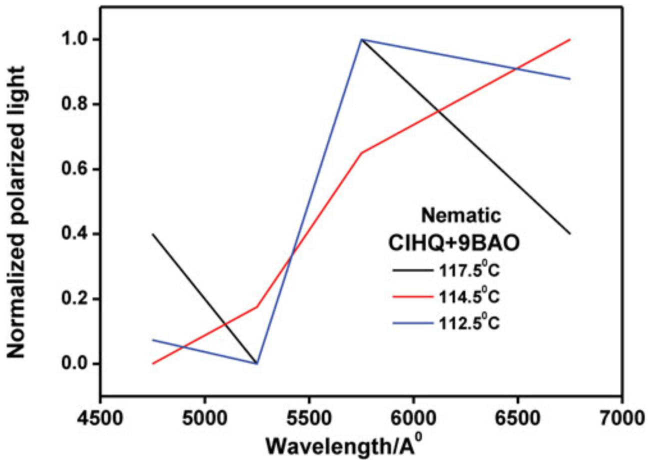


Figure 10. Filtering action in nematic phase of ClHQ + 9BAO complex.

agreement of magnitude of β (0.5) with Mean Field predicted value (0.5) infers the long-range interaction of transverse dipole moment for the stabilization of tilted smectic C phase.

4.7. *Dielectric Relaxations in Nematic Phase of ClHQ + nBAO*

To perform dielectric relaxation studies in ClHQ + 9BAO and ClHQ + 10BAO complexes, empty conducting cell of 10 micron spacing (Instec, USA) is calibrated with temperature (30°C to 150°C) and frequency (5 Hz to 13 MHz) by a known substance (benzene) to estimate the lead capacitance.

The relaxation frequencies for ClHQ + 9BAO complex are investigated at three temperatures namely 116.2°C, 114.2°C, and 112.2°C, respectively, which cover the entire nematic thermal range. The points are fitted to a semi-circle and as the center of the circle

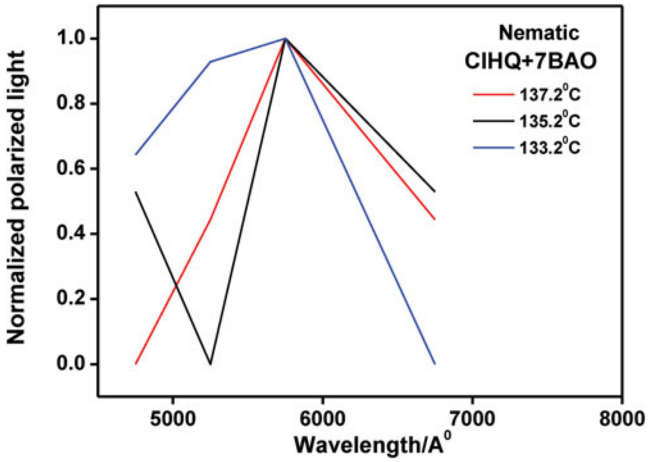


Figure 11. Filtering action in nematic phase of ClHQ + 7BAO complex.

lies outside the x-axis, these relaxations mechanisms are referred as Cole–Davidson [34]. As a representative case, the Cole–Davidson plot for ClHQ + 9BAO complex at 116.2°C is shown in Fig. 5. The relaxation frequency varied between 2100 Hz and 2700 Hz for the temperatures 116.2°C, 114.2°C, and 112.2°C, respectively. The shift of the relaxation frequency to higher side with decrease in temperature can be noticed from Fig. 5. The relaxation frequency at 116.2°C is 2100 Hz with a ε'' value of 1.7943, it has increased to 2200 Hz as the temperature has decreased to 114.2°C with a ε'' value of 1.5716. Thus in this complex, the relaxation frequency in nematic phase is inversely proportional to the temperature and is termed as anomalous dielectric relaxation behavior.

Similarly in the case of ClHQ + 10BAO complex, the relaxation frequency varied between 800 Hz and 900 Hz for the temperatures 121.7°C, 117.7°C, 113.7°C, and 111.7°C. The same above said procedure is followed in the case of ClHQ + 10BAO complex to determine the relaxation frequencies. For the temperature at 117.7°C, the relaxation frequency is observed at 800 HZ with a ε'' value of 0.0791, which is increased to 900 Hz linearly with temperature decreased to 113.7°C with ε'' value of 0.0770. In this complex, the relaxation frequency in nematic phase is directly proportional to the temperature.

From the above analyses, Arrhenius plots are drawn (Fig. 6) and the corresponding activation energy is estimated from the slope of the graph plotted between logarithm of the relaxation frequency (f_r) versus the inverse of the absolute temperature (1000/T) in the ClHQ + 9BAO and ClHQ + 10BAO cases and the corresponding activation energies that are found to be 4.095 eV and 0.6339 eV, respectively (Table 3).

4.8. Light Filtering Action

It is reported that [35] nematic liquid crystal is capable of transmitting light substantially at all wavelengths while reflecting light over a single, generally narrow, wavelength band and it is suitable for the application for optical-filtering applications. From the literature of nematic liquid crystals [36], it can be inferred that the unique optical properties of liquid crystal elements can be exploited to provide a wide variety of narrow band filtering functions extending over a wide wavelength range from the near ultraviolet to the far infrared. Figures 7–11 represents the suitability of ClHQ + nBAO (n = 12, 11, 10, 9 & 7) complexes as different kinds of filters with respect to the desired wavelength. It is to be noticed in all the cases that the complexes from $n = 12$ to 7 can be used as notch filter at certain temperature. Individual observations starting from Figs. 7–11 suggest that the complexes ClHQ + 12BAO, ClHQ + 11BAO, and ClHQ + 10BAO can be used as low-pass filters, whereas Fig. 11 indicates the application of ClHQ + 7BAO complex as high pass filter at desired temperature which ranges throughout the entire thermal range of nematic phase. It is common in all cases that transmission of polarized light which clearly indicates that the visible region is allowed to pass, while the ultraviolet radiation is blocked. Thus these liquid crystal complexes can be used as an effective filter for ultraviolet region of the spectrum.

5. Conclusions

Chlorohydrquinone derivatives of hydrogen-bonded liquid crystalline complexes (ClHQ + nBAO) are successfully synthesized for their electrical and optical applications. Elaborate studies of POM and DSC for all the complexes revealed that the carbon chain plays a major role in determining the transition temperatures for phase formation. Phase diagram drawn from the analysis of DSC showed an interesting odd–even effect at smectic C to smectic G phase transition. Dielectric relaxation analysis on ClHQ + 9BAO and ClHQ + 10BAO complexes and their corresponding activation energies are found to be 4.095 and 0.6339 eV, respectively. This result clearly indicates that higher molecular carbon chain complex showed more chemical activity than that of lower molecular carbon chain complexes. Optical tilt angle measurement also showed the same result that ClHQ + 12BAO had more tilt angle when compared to the rest of the complexes. Light filtering action was carried out in nematic liquid crystalline phase of the complexes and the observations suggest that all the complexes can be used as low-pass, high-pass, and notch filters at certain wavelength region.

Acknowledgments

The divine and graceful blessings of almighty Bannari Amman, the infrastructural support rendered by Bannari Amman Institute of Technology, and the financial support rendered by the Board of Research in Nuclear Sciences (BRNS) of Department of Atomic Energy (DAE), India (Sanction No. 2012/34/35/BRNS), are gratefully acknowledged by the authors.

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