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Optical, Thermal Studies on Binary and Ternary Hydrogen-Bonded Liquid Crystal Complexes

T. Mahalingam¹ · T. Venkatachalam² · R. Jayaprakasam³ · V. N. Vijayakumar⁴

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Abstract Hydrogen-bonded ferroelectric liquid crystalline (HBFLC) complexes are synthesized from binary mixtures of L-(+)-tartaric acid with 4-dodecyloxybenzoic acid and cholesteryl acetate. A ternary complex has been obtained from L-(+)-tartaric acid, 4-dodecyloxybenzoic acid, and cholesteryl acetate. Fourier transform infrared spectroscopy (FTIR) studies confirm the formation of an intermolecular hydrogen bond in the binary as well as the ternary complex. The L-(+)-tartaric acid does not show any mesomorphic behavior, but the hydrogen-bonded binary and ternary complexes are exhibiting the nematic phase along with tilted smectic phases. Phase transition properties of HBFLC mixtures have been investigated by means of differential scanning calorimetry (DSC) and polarizing optical microscope (POM). The DSC and POM clearly reveal the existence of nematic and smectic phases in the HBFLC mixtures. The optical tilt angle of binary and ternary mixtures for smectic C* phase and thermal stability factors of the mesogenic phases have been discussed. The noteworthy observation

is that there is a significant reduction of phase transition temperatures with enhanced phase width, lowering melting temperature, and clearing point in the HBFLC ternary complex.

Keywords HBFLC · Binary mixture · Ternary mixture · FTIR · DSC · POM · Smectic · Nematic

1 Introduction

In recent years, there has been significant quantity of research conducted with respect to the properties of binary liquid crystalline materials [1, 2]. Liquid crystals (LCs) are truly attractive materials in terms of their properties which are important for the fundamental understanding of molecular self-assembly and their remarkable success in commercial applications [3]. LCs are prepared by self-assembly process through the formation of intermolecular interactions such as hydrogen bonding which are promising materials for the production of new functional materials [4]. The formation of a hydrogen bond (H-bond) through the non-covalent interactions of molecules is a powerful tool for self-assembling the molecules to form the liquid crystal complexes [5]. Cholesteric liquid crystals, a supramolecular helical assembly of nematics, cover a wide range of potential applications in displays, polarizers, organic pigments, thermography, and photonic devices [6–10]. The distinguished characteristic features of liquid crystals paved the way for their implementation in the field of applied science and engineering. It is further classified into many sectors such as ferroelectric liquid crystals, achiral liquid crystals, and hydrogen-bonded liquid crystals [11–13]. The characterization and application aspects of mesogenic materials

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made scientists turn their attention upon the soft condensed matter and work for further development [14–17].

The field of hydrogen-bonded ferroelectric liquid crystal (HBFLC) is wide open for the design, synthesis, and characterization of various new combinations of mesogens and nonmesogens. HBFLCs and their complexes formed by mixtures possess interesting properties due to their intermolecular hydrogen bonds [18–20]. H-bonds induce a rich phase polymorphism between mesogenic and nonmesogenic mixtures [21]. H-bonds form ideal non-covalent interactions in the design of supramolecular architectures since they are highly selective and directional [22].

The strength of the H-bond depends on the solvent and temperature which makes the H-bonding unit an attractive molecular trigger responding to humidity and heat [23]. In the case of some binary mixture liquid crystalline molecules, polymerization may fix the liquid crystalline phases, resulting in a wide variety of highly ordered supramolecular materials [24]. In addition, a multicomponent liquid crystalline mixture can be used to derive applicational perspectives of LC materials [23]. Due to the presence of a heteroatom in the ring structures, it possesses high dipole moment [25] which results in greater optical anisotropy and birefringence of the material. The chiral center molecule exhibits ferroelectric behavior due to its configuration for transverse dipole moment. The tilted smectic C phases of LC materials are a potential candidate in designing the electro-optic devices [26, 27]. The mesomorphic thermal stability of ferroelectric LC compounds is discussed in the domain of symmetry and flexibility of the alkyloxy end chain length attached to the chiral center [28]. Thus, the binary mixture contains L-(+)-tartaric acid with a chiral center exhibiting ferroelectric properties. In order to exploit these materials in device applications, it should possess low melting point and exhibit a wide range LC phases. The thermal and mesophase behavior of ternary complex formed from hydrogen-bonded nematic liquid crystals reveal that the nematic ranges of the produced mixtures are affected in a way that is dependent on both the mixture ratio and the difference in the alkyloxy chain lengths [29]. The asymmetrically substituted benzene derivative with biphenyl moiety shows a low melting point [30]. With our previous experience in synthesis and characterization of various types of hydrogen bonded liquid crystal (HBLC) [31–34], we made an attempt to design, synthesize, and characterize the chiral-centered binary and ternary mixture complexes. The authors probed such a possibility by mixing different mesogenic acids which enabled them to understand the correlation between the physical and the chemical structure of the HBLC moieties.

2 Experimental

2.1 Materials and Methods

L-(+)-tartaric acid (LTA), 4-dodecyloxybenzoic acid (12BAO) and cholesteryl acetate (CHA) were supplied by Sigma-Aldrich, Germany, and all the solvents used were E-Merck grade. The LC sample is filled in its isotropic state to a homogenous alignment liquid crystal cell of $5 \times 5 \text{ mm}^2$ indium tin oxide coated area with a $4 \text{ }\mu\text{m}$ spacer by capillary action.

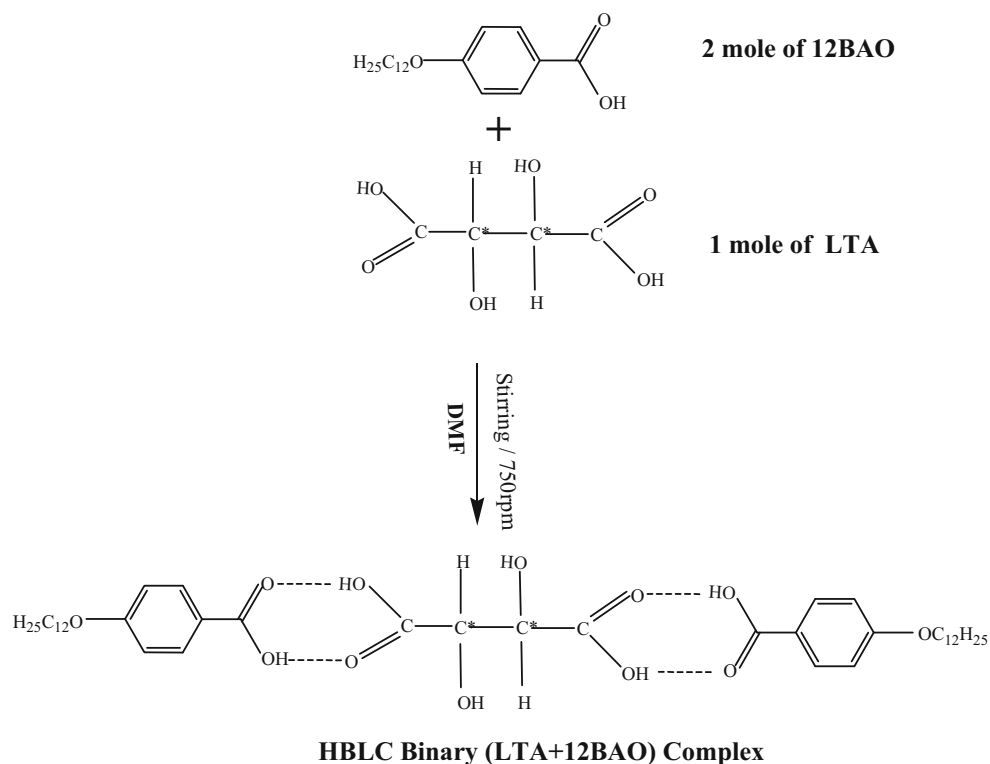
This LC cell is placed on a hot stage MHCS400 (MicroOptik) where the temperature is monitored by a MTDC600 temperature controller (MicroOptik) which is interfaced with a computer and controlled by a software program to a temperature resolution of $\pm 0.1 \text{ }^\circ\text{C}$. The hot stage is placed under the crossed polarizer of a Ningbo polarizing microscope for optical textural studies equipped with a digital unit (ToupTek). Optical images of mesogens are captured using a Sony CCD.17.UH310 USB CCD 3.1 MP camera. Heating and cooling runs at the rate of $1 \text{ }^\circ\text{C min}^{-1}$ and the corresponding textures are analyzed by software.

A Shimadzu DSC60 Plus with Ta60 software (version 2.21) and TAC-60i mechanical auto-cooling system is used for obtaining transition temperatures and enthalpy values of mesogens. A 4 g mass of the mesogens and ternary mixture (LTA + 12BAO + CHA) were accurately weighed by using a Shimadzu microbalance. The weighed mesogens and HBLC complex (LTA + 12BAO + CHA) are sealed in aluminum pans and are heated up to a temperature above the estimated clearing temperature and held at their isotropic temperature for 2 min so as to attain thermal stability. Once a stable heat flow is sustained, the samples are cooled at a rate of $5 \text{ }^\circ\text{C min}^{-1}$ to $30 \text{ }^\circ\text{C}$ followed by a heating scan at the rate of $5 \text{ }^\circ\text{C min}^{-1}$ to a temperature above the expected clearing temperature. All differential scanning calorimetry (DSC) curves are corrected using baselines recorded under identical conditions. Fourier transform infrared (FTIR) spectra are recorded (ABB FTIR MB3000) by making pellets of the (LTA + 12BAO + CHA) complex and KBr, and then, they are analyzed to identify the formation of the H-bond in the (LTA + 12BAO + CHA) complex using the MB3000 software.

2.2 Synthesis

2.2.1 Synthesis of (LTA + 12BAO) HBLC Binary Complex

The binary complex (Fig. 1) is designed and synthesized by adding 1 mol of LTA and 2 mol of 12BAO in *N,N*-dimethyl formamide (DMF). This mixture is subjected to

Fig. 1 Molecular structure of the LTA + 12BAO binary complex

constant stirring (magnetic stirrer) for 12 h with 750 rpm at an ambient temperature of 30 °C till white precipitate is formed. The white crystalline crude complex is obtained after removal of excess DMF. This same is recrystallized with dimethyl sulfoxide (DMSO). The designed molecular structure of the (LTA + 12BAO) HBLC binary complex is depicted in Fig. 1.

2.2.2 Synthesis of (LTA + CHA) HBLC Binary Complex

The (LTA + CHA) binary complex is synthesized by adding 1 mol of LTA in DMF and 1 mol of CHA in ethyl acetate as shown in Fig. 2. Further, the mixture is subjected to constant stirring (magnetic stirrer) for 12 h with 750 rpm at ambient temperature of 30 °C till white precipitate is formed. The white crystalline crude complex is obtained after removal of excess DMF. This same is recrystallized with DMSO. The designed molecular structure of the (LTA + CHA) binary complex is depicted in Fig. 2.

2.2.3 Synthesis of (LTA + 12BAO + CHA) Ternary Mixture

The ternary mixture liquid crystal (Fig. 3) is synthesized by the addition of 1 mol of cholesteryl acetate in ethyl acetate and 1 mol of synthesized binary mixture (LTA + 12BAO) in DMF. It is subjected to constant stirring for 12 h at an ambient temperature of 30 °C till a white

precipitate in a dense solution is formed. The white crystalline crude complex so obtained after removing excess DMF is then recrystallized with DMSO. The designed molecular structure of the (LTA + 12BAO + CHA) ternary mixture is depicted in Fig. 3.

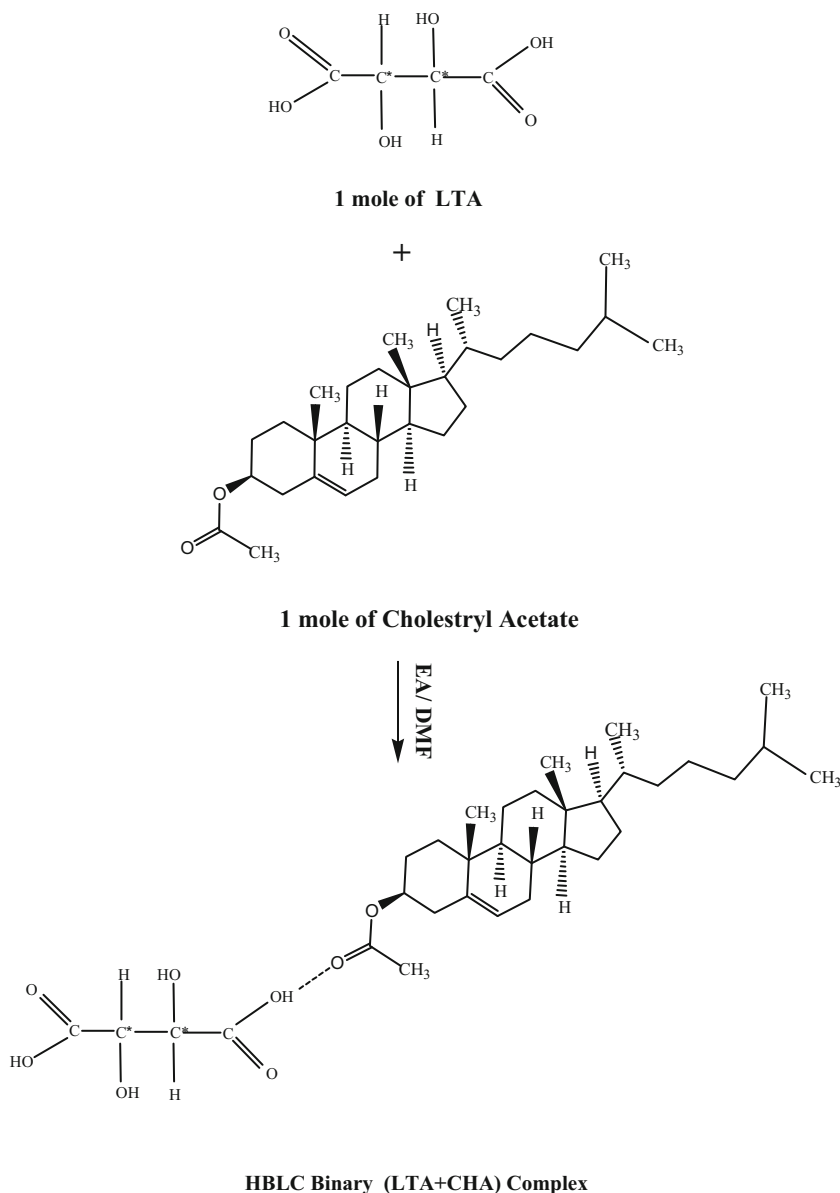
3 Results and Discussion

3.1 FTIR Studies

Intermolecular hydrogen bonding between the chemical moieties could be resolved using the FTIR spectra. A covalent bond is identified due to the absorption of various frequencies of electromagnetic radiation in the infrared region. Thus, the absorption of infrared gives rise to various energy transactions, and so, the bond formation and the types of stretching are inferred. Only the bonds having a dipole moment that change as a function of time are capable of absorption of infrared radiation. So the FTIR spectrum can be used to identify the molecular environment chemical moieties.

The FTIR spectra (Fig. 4) of intermolecular H-bonded binary complexes (LTA + 12BAO, LTA + CHA) and ternary complex (LTA + 12BAO + CHA) are recorded in the solid state (KBr) at room temperature. It is reported [35–37] that in the alkyl benzoic acids, carboxylic acid exists in monomeric form and the stretching vibration of

Fig. 2 Molecular structure of the LTA + CHA binary complex



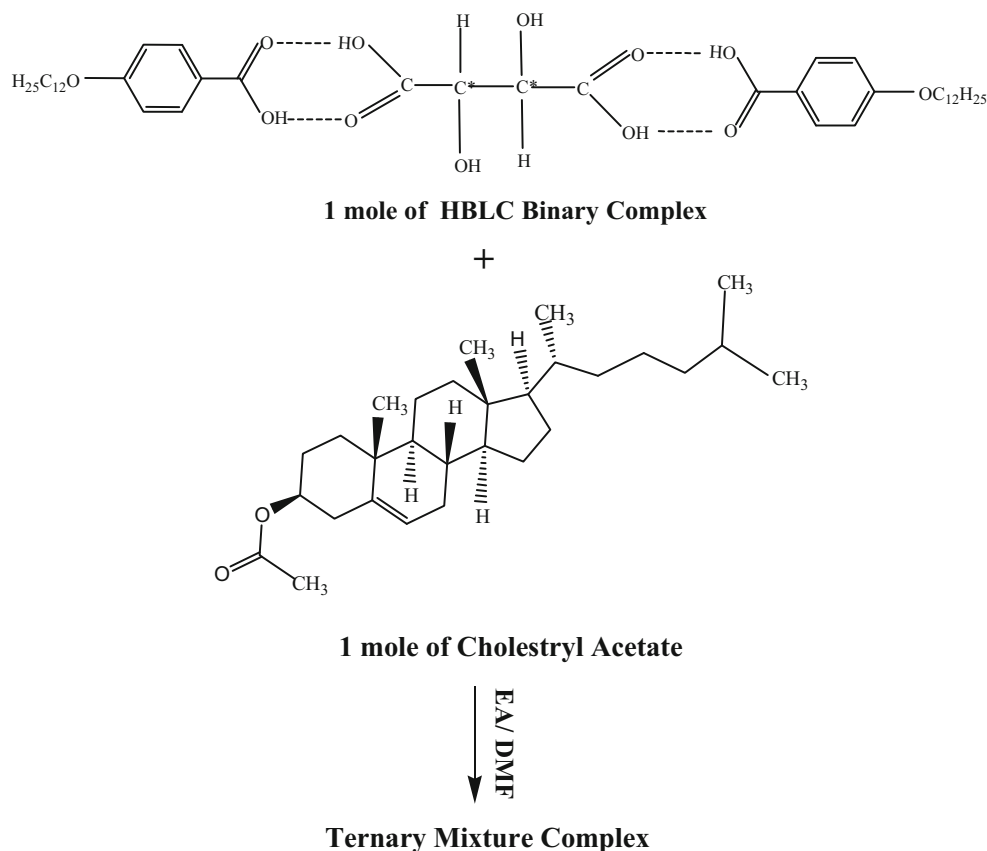
C=O is observed at 1728 cm^{-1} . In the FTIR spectrum of LTA + 12BAO (Fig. 4), there is a sharp peak at 1682 cm^{-1} which clearly suggests the dimer formation, in particular the carbonyl group vibration. The presence of the H-bond in the present complex is further inferred by the appearance of a strong band which shows the carboxylic acid group ν (O–H) at 2924 cm^{-1} . The spectrum of the LTA + CHA + 12BAO shows a peak at 1689 cm^{-1} which is due to carbonyl group vibration. The presence of the H-bond in the present complex is further inferred by the appearance of a new band which confirms the carboxylic acid group ν (O–H) at 2854 cm^{-1} , and the same is not observed in the pure CHA and 12BAO mesogens.

3.2 Phase Characterization

The synthesized HBLC complex is highly stable at ambient temperature ($\sim 30\text{ }^{\circ}\text{C}$). HBLC complex melts at a specific temperature below $\sim 106\text{ }^{\circ}\text{C}$. It shows high thermal and chemical stability when subjected to repeated thermal scans performed during polarizing optical microscope (POM) and DSC studies. The observed textural changes through POM under crossed polarizer and corresponding transition temperatures are simultaneously recorded. The characteristic textures are compared with reported [38] textures.

The phase observed by heating and cooling cycles is called enantiotropic while that which is observed in the cooling cycle

Fig. 3 Molecular structure of the LTA + 12BAO + CHA ternary complex



only is termed as monotropic. When examined by POM, the binary and ternary mixtures show nematic phase (N^*). On further cooling, mixture results transition into the lower temperature phases. The phase sequence of different binary and ternary mixtures is given below:

Isotropic	$\rightarrow N^*$	\rightleftharpoons	Sm	C^*	\rightleftharpoons	Sm	G^*	\leftarrow	Crystal	(LTA + 12BAO)
Isotropic	$\rightarrow C$	\rightleftharpoons	h	\leftarrow	Crystal					(LTA + CHA)
Isotropic	$\rightarrow N^*$	\leftarrow	Sm	C^*	\rightleftharpoons	Sm	G^*	\leftarrow	Crystal	(LTA + 12BAO + CHA)

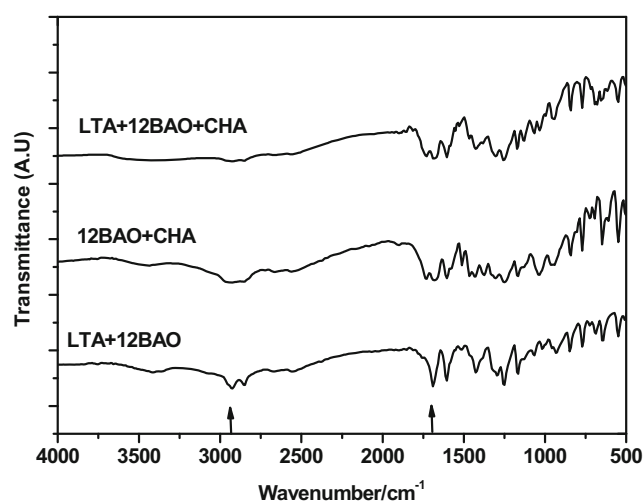


Fig. 4 FTIR spectra of binary and ternary mixture complexes

The LTA + 12BAO HBLC binary complex exhibits enantiotropic phase-like nematic (N^*) droplets and smectic (C^*) schlieren texture (Fig. 5a, b) in the heating and cooling cycles. The monotropic-phase smectic G^* multicolored mosaic texture (Fig. 5c) is observed only in the cooling cycle. Similarly, on the LTA + CHA HBLC binary complex, a brush-like cholesteric (Ch) texture (Fig. 6a) is observed in the cooling cycle only, and hence, it is monotropic. Further cooling below 81 °C, a distorted brush texture (Fig. 6b) is observed before it attains crystal phase.

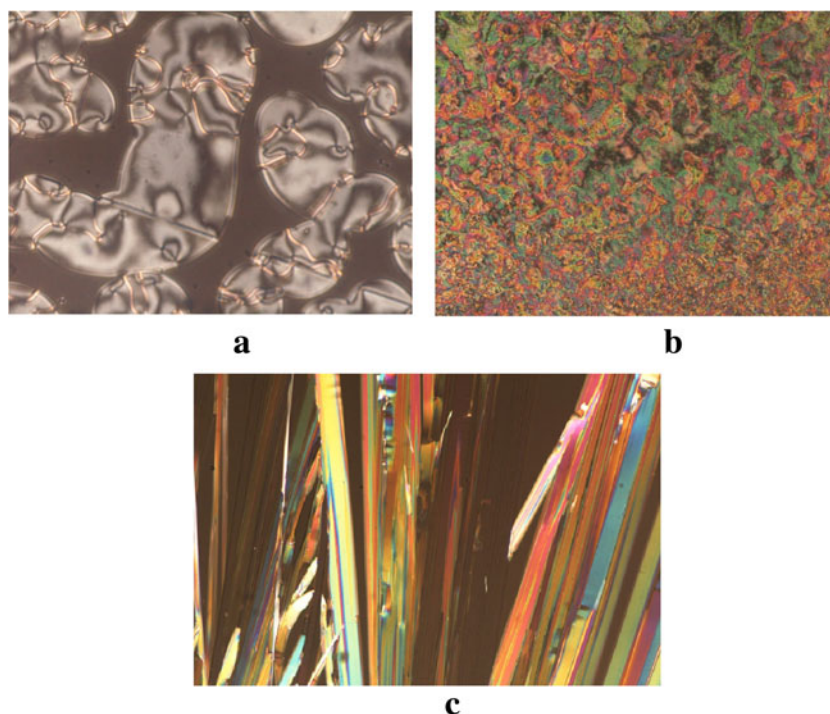
Due to the presence of an aromatic ring in the LTA + 12BAO complex, rich liquid crystallinity has been observed. Due to the presence of aliphatic rings in the LTA + CHA complex, only one mesomorphic phase has been observed as the aliphatic lateral substituent destabilizes the mesophase behavior.

The LTA + 12BAO + CHA ternary complex is found to exhibit characteristic textures (Fig. 7a–c) like droplet nematic texture (N^*), schlieren texture of smectic (C^*), and multicolored marble texture of smectic (G^*). In this ternary complex, N^* is enantiotropic while C^* and G^* are monotropic.

3.3 DSC Studies

The DSC thermograms are obtained with a scan rate of 10 °C min⁻¹ with the heating (Fig. 8) and cooling (Fig. 9)

Fig. 5 Characteristic texture of the LTA + 12BAO binary complex (**a–c**)



cycles of all the binary and ternary mixtures. The respective equilibrium transition temperatures corresponding to the enthalpy values of mesogens are listed in Table 1. POM studies have also confirmed these DSC transition temperatures. An increment in chain length favors the enhancement in the thermal range of mesophases. Hence, an enhanced thermal range around 18 °C of the nematic phase is observed in the ternary mixture when compared to the binary mixture. Because of the attachment of the alkyloxy chain and aliphatic chain on either side of chiral molecules, the ternary mixture shows abundant reduction in phase transition temperature and a lowered melting point when compared to the binary mixtures. At the eutectic point, the melting point reaches its minimum and the clearing point of the liquid crystal complex is a linear average of composition. Thus, the liquid crystal mixture can offer a much larger temperature range that exhibits the nematic phase for display applications.

3.4 Optical Tilt Angle Measurements

The angle between the direction of assembly molecules along the optic axis and reference axis is named as the optical tilt angle. The position of molecules in the mesophase with the reference coordinate system in the liquid crystalline complex depends on the tilt angle of the mesophase. To explain this, the direction perpendicular to the layer plane in the smectic phase is taken as the reference axis. The tilt angle of the smectic C* phase in the given LC complex is experimentally determined by optical extinction method [39, 40]. The temperature variation is evaluated by applying the measured data of temperature $\theta(T)$ in the relation given below:

$$\theta(T) \propto \theta_0 (T - T_c)^\beta$$

where, $\theta(T)$ is temperature at which the tilt is measured (°C), T is actual temperature (°C), T_c is transition

Fig. 6 Characteristic texture of the LTA + CHA binary complex (**a, b**)

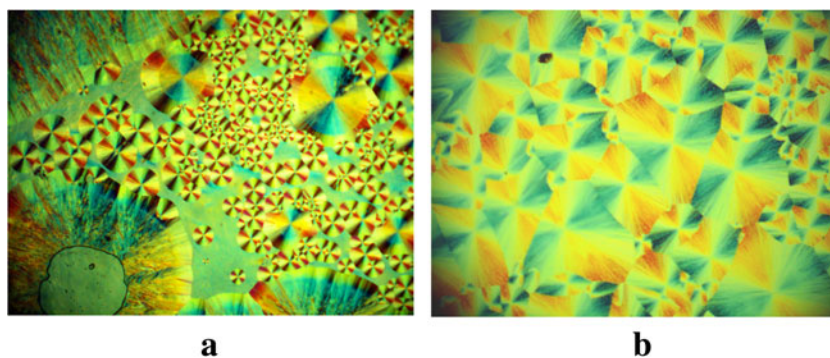
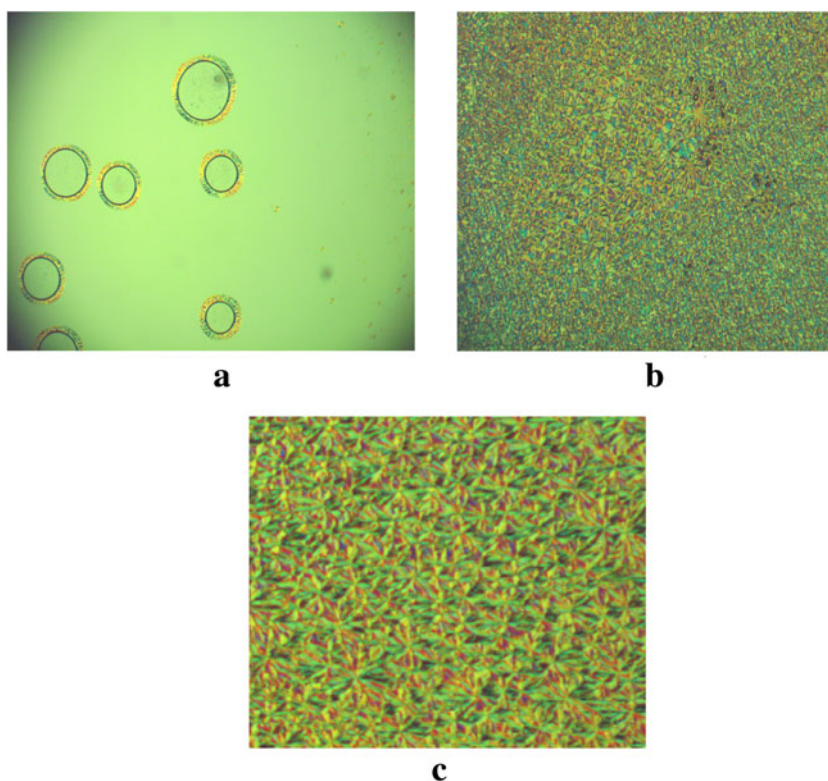


Fig. 7 Characteristic texture of the LTA + 12BAO + CHA ternary complex (**a–c**)



temperature ($^{\circ}\text{C}$), θ_0 is a constant, and β is critical exponent value determined by fitting temperature data to the above expression and it is found to be 0.50 which is in good agreement with the mean field value [41]. Optical tilt angle has been experimentally measured in the smectic C^* phase of the LTA + 12BAO and LTA + 12BAO + CHA mixtures. From the Figs. 10 and 11, it is observed that the tilt angle increases with decreasing temperature and attains a saturation value. The saturated value for the tilt angle in

the smectic C^* of binary and ternary mixtures is found to be 16° and 17° , respectively, as shown in Figs. 10 and 11. This value of the tilt angle is attributed to the direction of soft covalent hydrogen bond interaction which extends along the molecular axis with finite inclination. The dotted line in the graph represents fitted data. The agreement of the β value with the mean field value depicts the long-range interaction of the transverse dipole moment for the stabilization of the tilted smectic C^* phase.

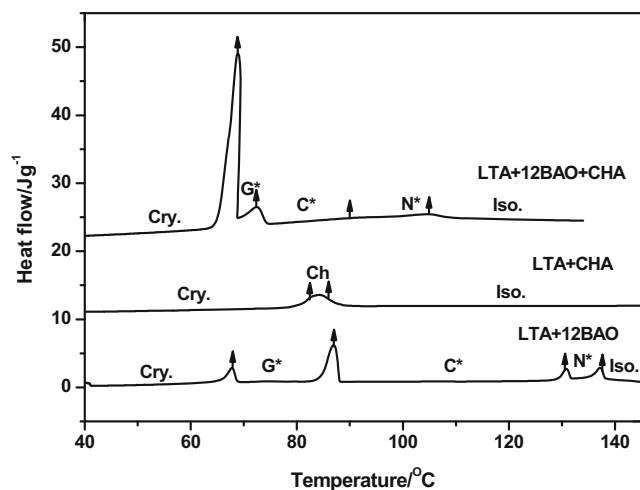


Fig. 8 DSC thermograms of binary and ternary complexes during heating run ($10^{\circ}\text{C min}^{-1}$)

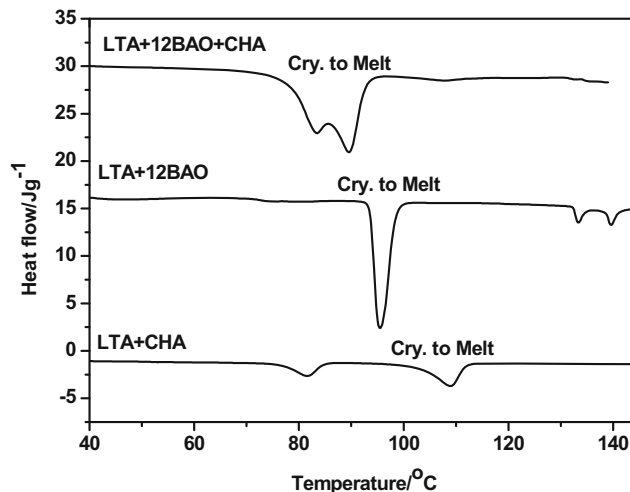


Fig. 9 DSC thermograms of binary and ternary complexes during cooling run ($10^{\circ}\text{C min}^{-1}$)

Table 1 Transition temperatures in centigrade obtained by DSC techniques along with enthalpy values given in J g⁻¹

LC mixture	Phase variance	Technique	Cycle	Crystal-melt	N*/Ch	Sm C*	Sm G*	Crystal
(LTA + 12BAO)	N*C*G*	POM	Heating	98.6	140.3	132.7	^b	
			Cooling		142.1	131.2	85.8	64.1
		DSC	Heating	97.4 (101.12)	141.1 (1.60)	131.5 (2.12)	^b	
			Cooling		139.1 (1.64)	130.7 (0.99)	84.1 (5.20)	63.51 (38.06)
(LTA + CHA)	Ch	POM	Heating	105.8	^b	^a	^a	
			Cooling		81.3	^a	^a	80.6
		DSC	Heating	106.1 (158.3)	^b	^a	^a	
			Cooling		81.0 (merged with crystal)	^a	^a	80.3 (73.9)
(LTA + 12BAO + CHA)	N*C*G*	POM	Heating	83.7	108.0	^b	^b	
			Cooling		107.8	89.7	68.9	63.4
		DSC	Heating	83.5 (27.58)	107.9 (2.44)	^b	^b	
			Cooling		107.8 (0.42)	89.5 (26.80)	68.9 (0.39)	63.2 (33.24)

^a Not resolved^b Monotropic transition

3.5 Thermal Stability Factor

If the liquid crystalline molecules have two symmetric end chains, then the phase transition temperatures and the thermal span ranges are affected [42, 43]. The molecular weights of the 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 essential parameters that govern the utility of the mesogen. As a representative case, the phase stability of smectic C* for ternary mixture has been discussed. The term phase stability can be attributed to the nematic N* to smectic C* transition temperature as well as to the temperature range of the smectic C* phase. It is

reasonable to define a parameter called stability factor (S) after considering both the above factors. The stability factor for smectic C* phase is

$$S_{C^*} = T_{\text{mid}} \times \Delta T_{C^*}$$

where T_{mid} is the mid-smectic C* temperature and ΔT_{C^*} is the smectic C* thermal range. The thermal stability of smectic C* exhibited by ternary mixture complex is calculated as 1631. By following the same procedure, the thermal stability of other phases for binary and ternary mixtures is calculated and the same is appended in Table 2. It can be observed that the combination in the LC mixture alters the thermal stability of the phases with respect to their thermal span.

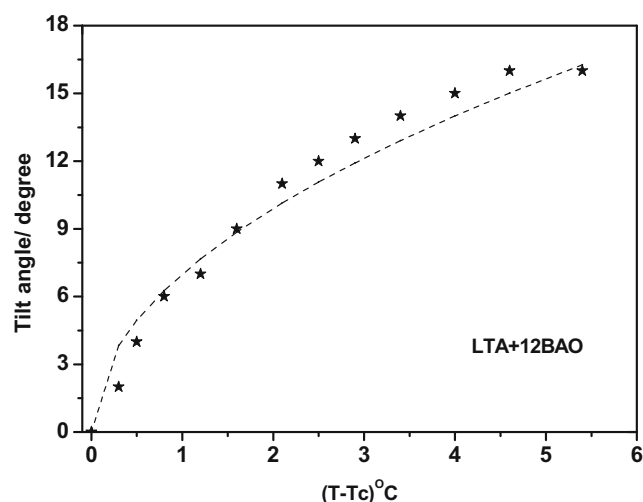
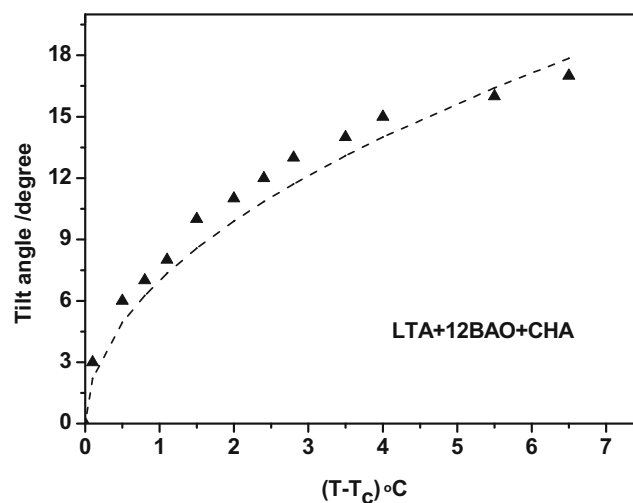
**Fig. 10** Tilt angle in the LTA + 12BAO binary complex**Fig. 11** Tilt angle in the LTA + 12BAO + CHA ternary complex

Table 2 Thermal stability factor for binary and ternary LC mixtures for different phases

LC mixture	N*/Ch	Sm C*	Sm G*
LTA + 12BAO	1512	5004	1520
LTA + CHA	32	–	–
LTA + 12BAO + CHA	1825	1631	376

4 Conclusions

Thermal and optical properties of binary and ternary mixtures of HBLCs have been analyzed. This molecular design strategy gave a fabulous method to modulate the phase behavior and transition temperatures of HBLC mesogenic complexes. The textures (POM) and transition temperature along with enthalpy values are observed by DSC studies. Abundant reduction in phase transition temperature and lowered melting point in the ternary mixture is noticed. The HBLC complex (LTA + 12BAO + CHA) possesses a nematic phase with a wider thermal span when compared to the binary mixture. It is a more suitable material for optical devices such as optical (light) modulator, memory, and shuttering devices.

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