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Synthesis, Characterization and Phase Transition Studies in Hydrogen Bonded Liquid Crystal *p*- Octyloxy Benzoic Acid: *p*-(*p*'- Ethoxy Benzylidene)- Cyano Aniline (8OBA: EBCA)

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ABSTRACT: Design of novel liquid crystals for suitable applications giving a thrust in recent times. One of the approaches to this new mesogens is through hydrogen bond between compounds whether they are liquid crystals or non-liquid crystals. Hence authors are motivated to synthesize complex liquid crystals via hydrogen bond consisting the type of mesogenic and mesogenic, namely *p*- octyloxy benzoic acid (8OBA) and *p*-(*p*'- ethoxy benzylidene)-cyano aniline (EBCA). Further characterization is also carried out and results are discussed in this communication.

KEYWORDS: 8OBA, EBCA, hydrogen bond, polarizing microscopy, FTIR, Navard and Haudin parameter.

I. INTRODUCTION

The design considerations involve the materials viability in applications. It depends not only on the stability and durability but also on the physical parameters. Systematic studies on different types of liquid crystalline materials reveal their importance in fundamental [1] and applied [2] research. The complex liquid crystals are also important to bring down the high transition temperature, to increase the phase range and to induce new phases. If the complex is formed through inter molecular hydrogen bond, lengthening of rigid rod segments occurs as a property of liquid crystalline behaviour. Much [3-6] attention has been focused on the mesomorphism induced by the non-covalent electrostatic interaction involving intermolecular hydrogen bonding. This work is motivated to synthesize complex liquid crystal via hydrogen bond consisting the type of mesogenic and mesogenic, namely *p*- octyloxy benzoic acid (8OBA) and *p*-(*p*'- ethoxy benzylidene)- cyano aniline (EBCA).

II. RELATED WORK

Kato and Frechet advanced a new concept for the construction of liquid crystalline compounds [7,8] and Srinivaulu [9] which involve the processes of molecular recognition and self – organization. Hydrogen bonding new liquid crystals increased the applications. Hydrogen bonds usually considered as the fifth fundamental type of chemical interactions, have attracted much attention in recent years owing to their importance as powerful tools for self-assembling molecules [10-12]. Despite their low bond energies, these hydrogen bonds have great importance both in biochemical systems and normal chemical reactions. Due to low- bond and activation energies, H- bond complexes

play a significant role in many reactions at normal temperatures. Intermolecular systems have a direct consequence on physical properties like melting and boiling points, enthalpies, sublimation, quenching of phases, inducing of new phases in addition to change in thermal ranges [13,14].

III. EXPERIMENTAL

Synthesis of *p*- octyloxy benzoic acid: *p*- (*p*'- ethoxy benzylidene)- cyano aniline (8OBA: EBCA).

p- Octyloxy Benzoic Acid (8OBA) and *p*-(*p*'- ethoxy benzylidene)- cyano aniline (EBCA) are obtained from M/s Frinton Laboratories Vineland, New Jersey, USA which are mesogens whose chemical structures are given below.

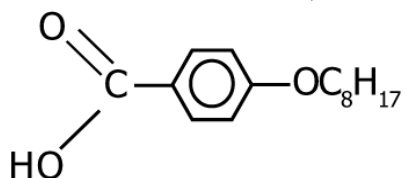


Fig 1 8OBA

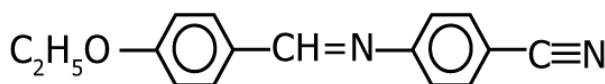


Fig 2 EBCA

Both 8OBA and EBCA are White Crystalline compounds at room temperature. The required sample of each compound is weighed on a single pan Electronic Balance AFCOSET make, ER-180A with an accuracy of 0.01 mg. The complex, *p*- octyloxy benzoic acid and *p*- (*p*'- ethoxy benzylidene)- cyano aniline (8OBA:EBCA) whose structure is given below was synthesized by refluxing together in equimolar ratio(1:1) of *p*-octyloxy benzoic acid (0.4505 gm) and *p*- (*p*'- ethoxy benzylidene)- cyano aniline (0.3914 gm). Further synthesis procedure is reported elsewhere [15] and the structure is given below in fig. 3. The yielding is at about 85 %.

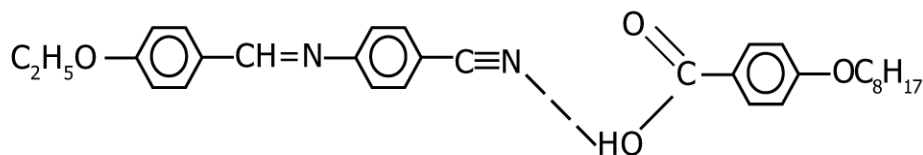


Fig. 3 Structure of 8OBA: EBCA

IV. RESULTS AND DISCUSSION

Thermal polarizing microscopy: -

In heating cycle, the pure 8OBA exhibits smectic-C (plate 1) phase in 102.2°C. On further heating it changes into nematic phase at 102.2°C (plate 2) and this phase remains upto 138.4°C. The nematic phase with droplets is shown in (plate 3). Isotropic phase is observed with the increase of temperature. In cooling cycle, the nematic phase separates

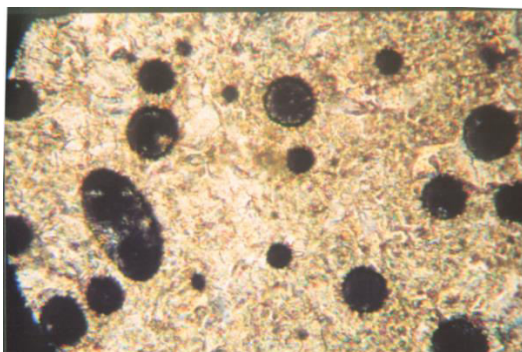


Plate 1: Smectic C phase

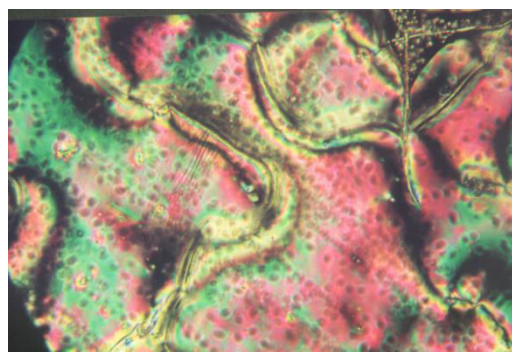


Plate 2 Nematic phase and Schlieren

from the isotropic phase at 136.2°C and this phase remains upto 102.4°C. On further cooling, Schlieren smectic C phase exists in between 102.4°C and 95.1°C. On further cooling, it becomes as solid below 95.1°C.

The transition temperatures corresponding to pure 8OBA reported values according to GRAY *et al* [16] are also given. i.e., At 75°C, pure 8OBA undergoes solid to solid transitions. On further heating, the pure 8OBA exhibits smectic-C phase in between the temperatures 101°C and 108°C. On further heating it changes into nematic phase at 108°C and this phase remains upto 146°C. Afterwards, on further heating, it becomes isotropic

The transition temperatures corresponding to pure 8OBA reported by Swathi *et al* [17] are also given. i.e., In heating cycle, the pure 8OBA exhibits Smectic-C phase in between the temperatures 97.8°C and 101.9°C. On further heating, it changes into nematic phase at 101.9°C and this phase remains upto 147.5°C. Afterwards, on further heating, it becomes isotropic

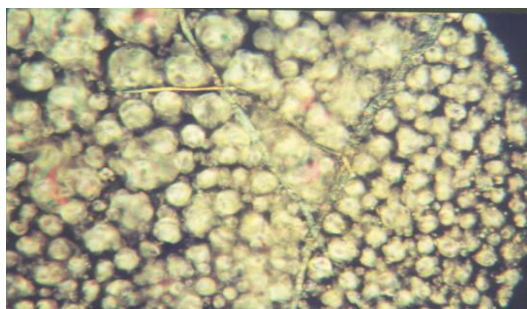


Plate 3 Nematic droplets

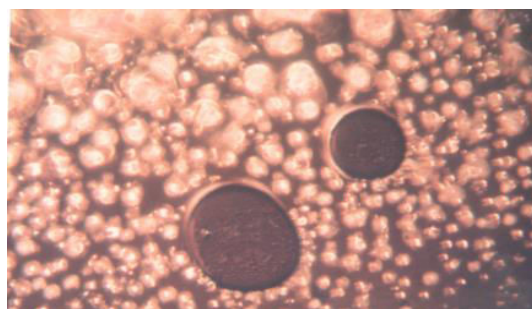


Plate 4 Nematic phase

EBCA, which is also an enantiotropic liquid crystal, exhibits nematic phase (plate4) in between the temperatures 98.5°C and 123.9°C on heating and in between the temperatures 122.5°C and 84.3°C on slow cooling.

The compound 8OBA: EBCA exhibits three phases i.e., Smectic G, Smectic C and Nematic in between solid phase and isotropic phase. The pure compound 8OBA has the Schlieren Smectic C and nematic marble textures. The hydrogen bonded compound 8OBA: EBCA exhibits an induced smectic G phase only in cooling cycle, in addition to the smectic C and nematic phases. In heating, Smectic-C phase is observed in between the temperatures 69.5°C and 93.5°C. On further heating, the smectic C phase is changed to nematic phase in between the temperatures 93.5°C and 132.5°C. On further heating, the nematic phase is changed to Isotropic phase at 132.5°C.

On cooling liquid crystalline nematic droplets separate from Isotropic phase at 133.5°C same as shown in the plate 3. These nematic droplets combine to form marbled nematic phase same as shown in plate 2. The nematic phase is changed to Schlieren smectic C phase at 98.5 °C same as shown in plate 1. The Schlieren smectic C phase is changed to smectic G phase at 90.5°C. The smectic G phase consists of mosaic like structure as shown in plate 5 at temperature of 93.5°C. The smectic G at 81.8°C is shown in plate 6. The smectic G phase is changed to solid phase at 57.1°C.

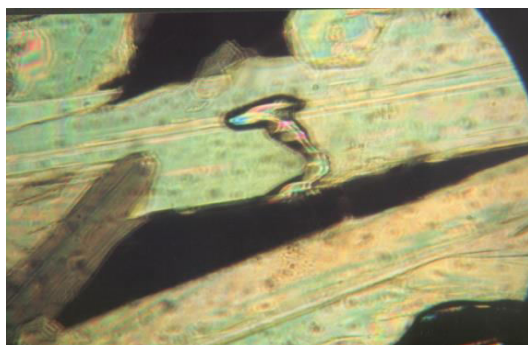


Plate 5 Smectic –G phase at 90.5°C

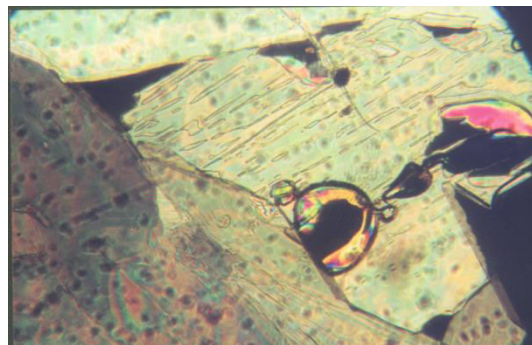


Plate 6 Smectic-G at 81.8°C

Hence the synthesized compound exhibits liquid crystal nature with smectic-G phase in cooling cycle, in addition to Schlieren smectic-C and nematic phases. The data is directed in Table 1.

Differential Scanning Calorimeter:

OBA, EBCA and 8OBA: EBCA transitions are carried out with Differential Scanning Calorimetry (DSC). In heating cycle, the pure 8OBA exhibits Smectic-C phase in between the temperatures 75.1°C and 101.1°C. On further heating it changes into nematic phase at 101.1°C and this phase remains upto 143.9°C. Afterwards, on further heating, it becomes isotropic. In cooling cycle, the nematic phase separates from the isotropic phase at 137.1°C and this phase remains upto 90.9°C. On further cooling, Schlieren smectic C phase exists in between 90.9°C and 51.1°C. On further cooling, it becomes as solid below 95.1°C.

Table: 1 Textural changes in heating and cooling through TM for 8OBA, EBCA and 8OBA: EBCA

Compound	Cr to G/C/N		G to C		C to N		N to I	
	Heating	Cooling	Heating	Cooling	Heating	Cooling	Heating	Cooling
8OBA	94.4°C	95.1°C			102.2°C	102.4°C	138.4°C	136.2°C
EBCA	98.5°C	84.3°C					123.9°C	116.6°C
8OBA: EBCA	69.5°C	57.1°C		90.5°C	93.5°C	98.5°C	132.5°C	133.5°C

EBCA exhibits nematic phase in between the temperatures 103.8°C and 126.3°C on heating and 116.3°C and 76.6°C on super-cooling, which is enantiotropic.



In heating cycle, 80BA:EBCA exhibits Schlieren smectic C phase in between the temperatures 73.9°C and 97.8°C. On further heating, it exhibits nematic phase in between the temperatures 97.8°C and 140.9°C. Afterwards, on further heating, it becomes isotropic.

In cooling cycle, the nematic phase separates from the isotropic phase at 139°C. On cooling, nematic phase is changed to Schlieren smectic C phase. But the transition peak corresponding to nematic to smectic C transition is not well resolved in DSC. On further cooling, smectic-G phase separated from Smectic C phase exists in between the temperatures 93.8°C and 58°C. Afterwards, on further cooling, it becomes as solid below 58°C.

Table-2 textural changes in heating and cooling by DSC for 80BA, EBCA and 80BA: EBCA

Compound	Cr to G/C/N		G to C		C to N		N to I	
	Heating	Cooling	Heating	Cooling	Heating	Cooling	Heating	Cooling
80BA	75.1°C	51.5°C			101.1°C	90.9°C	143.9°C	137.1°C
EBCA	103.8°C	76.6°C					126.3°C	116.3°C
80BA:EBCA	73.9°C	58°C		93.8°C	97.8°C		140.9°C	139°C

The DSC is sophisticated technique to detect phase transitions, enthalpy changes at the transitions and useful for qualitative analysis in purity, isothermal crystallization etc., measurements as well as in determining the order of transition whether first order or second order. Navard and Haudin [18] provided a simple method to determine the order of phase transition from the DSC peak heights. It can be measured from the ratio (N) of different enthalpy peak heights h_2/h_1 , performed with two different scan rates (T_1 and T_2), which is a function of the ratio of the scanning rates, ($T_2/T_1 = R$), keeping the weight of the compound constant. In the first order transition, N varies as the square root of R, while for the second order transition, N varies directly proportional to R.

For first order phase transition, let us consider a vapour – liquid mixtures in equilibrium at vapour pressure P and temperature T. Both P and T remain constant throughout. If V and S be the specific volume and entropy, then

$$dG = V dp - S dT$$

Further $G = G(P, T)$

Taking its derivative, we have

$$dG = (\partial G/\partial P)_T dP + (\partial G/\partial T)_P dT$$

Comparing the above two equations we can write,

$$(\partial G/\partial P)_T = V, (\partial G/\partial T)_P = -S.$$

So we observe that the first derivative of G (for liquid) is different from that of G (for vapour) at transition temperature T and pressure P.

In general, if the derivatives $(\partial G/\partial P)_T$, and $(\partial G/\partial T)_P$ are discontinuous at the transition point, i.e., V and S have different values in two phases; the transition is called first order transition.

Therefore, the following statements can characterize first order transition.

- (i) There exist changes in entropy, volume and latent heat.
- (ii) The density changes are discontinuous at transition temperature and pressure.

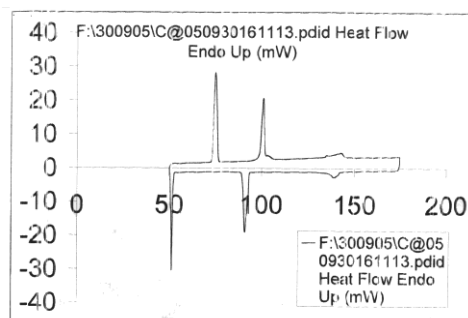


Fig 4 DSC thermogram of 8OBA

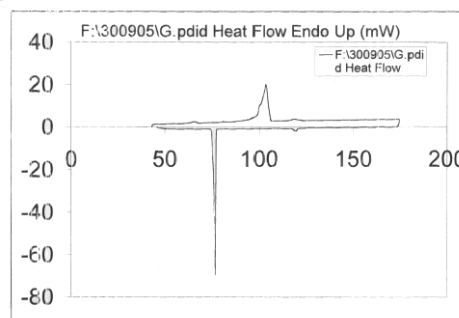


Fig 5 DSC thermogram of EBCA

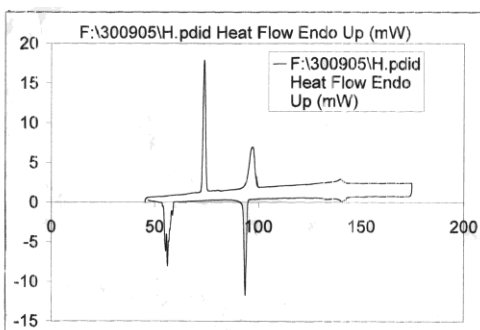


Fig 6 DSC thermogram of 8OBA:EBCA

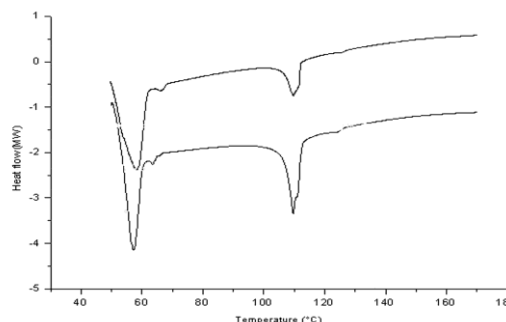


Fig 7 DSC thermogram of 8OBA:EBCA for scan rates of 5 °C/min and 10 °C/min in cooling.

For second order phase transition the phase transition is continuous in the sense that the state of the body changes continuously. It means latent heat is absorbed while heating or evolved while cooling when the system under goes first order transition. Here from the DSC curve we observe continuous absorption of heat energy at transition. This indicates the second order transition. The DSC thermogram of 8OBA is shown in figure 4 and of EBCA fig 5 respectively in both heating and cooling cycles. The DSC thermogram of 8OBA: EBCA is shown in figure 6 in both heating and cooling cycles at a scan rate of 5°C/min, the DSC thermogram of 8OBA: EBCA is shown in figure 7 in both heating and cooling cycles at a scan rate of 10°C/min and the transition temperatures are shown in Table 2. From the thermal microscope observations of phase transition temperatures and also from DSC studies reveals the phase transition temperatures are coinciding. For the synthesized *p*-propoxy benzoic acid: *p*- Chloro benzoic acid compound, we have used $R = 2$ (i.e., keeping the mass of the sample constant) the DSC thermograms are obtained for scan rates 10 °C and 5 °C. DSC thermogram scanned at 5 °C /min (h_1) and 10 °C / min (h_2) are shown in figure 7. The height calculated for 5 °C /min (h_1) and 10 °C /min (h_2) are 1.70 and 1.949 respectively. The Nevard and Haudin parameter is $h_2/h_1 = 1.146$. This value suggests that the order of isotropic - nematic transition in this compound is the first order. The thermogram of 8OBA;EBCA for a scan rate of 5° C/minute and 10°C/minute while cooling is given in fig 7.

3.3. FTIR SPECTRA:

Due to the excitation of both molecular vibrations and rotations, the absorption bands in the infrared region of the electro- magnetic radiation are obtained. The IR spectrum of a particular free organic compound is the characteristic of the entire molecule and it is true that certain functional groups of the molecule give rise to the bands at or near the same frequency regardless of structure of the rest of the molecule. The IR spectra are very useful in structural

information. When an organic compound binds to another molecule, at least one additional atom is introduced into the vibrational system of the other compound. As a consequence the bond angles, bond lengths and inter atomic force constants associated with the individual compounds would be expected to alter slightly in their homologue complexes. The IR spectrum of a coordinated compound differs from that of a free molecule and thus, it should be possible to correlate the changes in the form of band shifts observed in the spectra with the changes in bonding. Thus the information regarding the bonding nature of the constituent molecule in a particular complex may be obtained. Our aim is to establish the existence of inter molecular hydrogen bonding using this IR spectra.

p- Octyloxy benzoic acid (8OBA):-

The IR spectrum of *p*-octyloxy benzoic acid recorded at room temperature in solid (KBr) state is shown in figure 8 3.f. The IR data of 8OBA in solution (CHCl₃) state is taken from the literature [19]. The spectra of *p*-Octyloxy benzoic acid show a sharp peak at 1691.72cm⁻¹ due to KBr and 1712 cm⁻¹ due to chloroform for ν (C = O) mode. Another strong intense band assigned to ν (OH) mode of carboxylic acid group is obtained at 2926.28cm⁻¹ due to KBr and 3032cm⁻¹ due to chloroform. To avoid further complications due to such intermolecular interactions, comparison of spectra of complexes is made with the solution state spectra of free 8OBA.

p- (*p*'- ethoxy benzylidene)- cyano aniline (EBCA) :-

The IR Spectrum of *p*- (*p*'- ethoxy benzylidene)- Cyano aniline is recorded at room temperature in solid (KBr) (shown in figure 9 3.g). The spectrum of ethoxy benzylidene *p*- cyano aniline shows a sharp peak at 2223.52cm⁻¹ for ν (CN) mode in KBr.

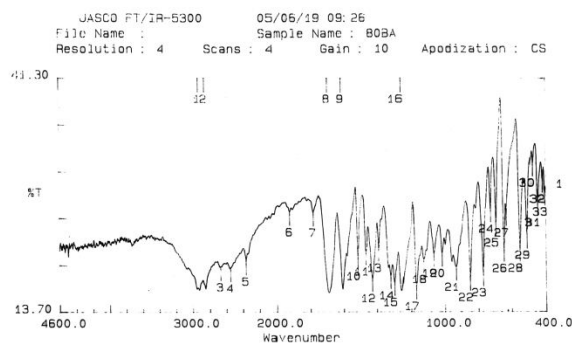


Fig 8 FTIR Spectrum of 8OBA

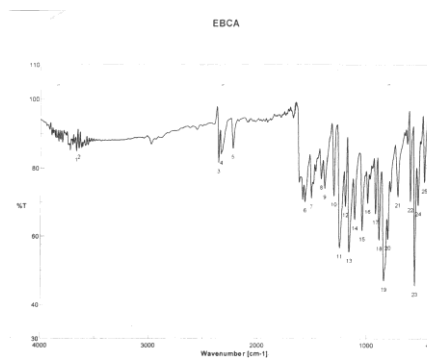


Fig. 9 FTIR Spectrum of EBCA

p- Octyloxy benzoic acid: *p*- (*p*'- ethoxy benzylidene)- cyano aniline (8OBA : EBCA):-

The infrared spectrum of 8OBA: EBCA is recorded in KBr (solid) state and the spectrum is shown in figure 10. The infrared frequencies of 8OBA: EBCA show the presence of a strong intense band due to (C = O) mode of benzoic acid moiety at ~1691.72cm⁻¹. This strongly supports the existence of 8OBA moiety in a monomeric form upon complexation. The formation of inter molecular hydrogen bonding between COOH group of 8OBA and CN group of EBCA can be invoked on the basis of hypsochromic shift in ν (CN) mode (~7-8 cm⁻¹) of EBCA in KBr and ν (C=O) mode of carboxylic acid group also suffered a hypsochromic shift of (~20cm⁻¹) upon complexation which strongly supports the existence of H – bonding. The existence of H-bonding is strongly confirmed due to the induced frequency of 3358.37 cm⁻¹, lying in the frequency range of stretching OH- group due to H- bonding, which is not present in the original *p*- octyloxy benzoic acid. presence of a strong intense band due to (C = O) mode of benzoic acid moiety at ~1691.72cm⁻¹. This strongly supports the existence of 8OBA moiety in a monomeric form upon

complexation. The formation of inter molecular hydrogen bonding between COOH group of 8OBA and CN group of EBCA can be invoked on the basis of hypsochromic shift in $\nu(\text{CN})$ mode ($\sim 7\text{-}8\text{ cm}^{-1}$) of EBCA in KBr and $\nu(\text{C}=\text{O})$ mode of carboxylic acid group also suffered a hypsochromic shift of ($\sim 20\text{cm}^{-1}$) upon complexation which strongly supports the existence of H – bonding. The existence of H-bonding is strongly confirmed due to the induced frequency of 3358.37 cm^{-1} , lying in the frequency range of stretching OH- group due to H- bonding, which is not present in the original p- octyloxy benzoic acid.

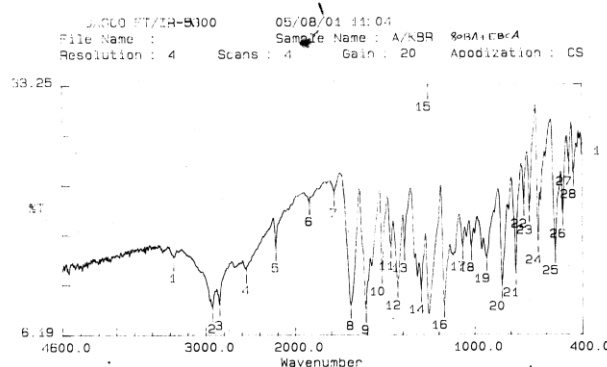


Fig. 10 FTIR Spectrum of 8OBA: EBCA

Table – 3: IR Spectra data cm^{-1} of 8OBA, EBCA and 8OBA: EBCA

S.No.	Compound	$\nu(\text{C} = \text{O})_{\text{acid}}\text{ cm}^{-1}$	$\nu(\text{OH})_{\text{acid}}\text{ cm}^{-1}$	$\nu(\text{C} - \text{N})\text{ cm}^{-1}$
1.	8OBA (CHCl_3)	1712	3032	-----
2.	8OBA (KBr)	1691.72	2926.28	-----
4.	EBCA (KBr)	-----	-----	2223.52
6.	8OBA: EBCA (KBr)	1691.72	3358.37	2216.41

V. CONCLUSIONS

The three compounds 8OBA, EBCA and 8OBA: EBCA, are enantiotropic liquid crystals. The phase transition temperatures observed through polarizing optical microscopy and DSC studies are in reasonable agreement. The transition peak corresponding to nematic to Smectic C phase transition is not well resolved. An induced Smectic G phase is observed in cooling cycle only. The induced Smectic G phase may be due to the intermolecular hydrogen bond. The intermolecular hydrogen bonding between the OH group of p-octyloxy benzoic acid and $\text{N}\equiv\text{C}$ of p- (p'-ethoxy benzylidene)- Cyano aniline (EBCA) is confirmed through IR studies. The Nevard and Haudin parameter is 1.146. This value suggests that the order of isotropic - nematic transition in this compound is of the first order

ACKNOWLEDGEMENTS

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