

Induced Smectic – G Phase Through Intermolecular Hydrogen Bonding –Study Of Thermal And Phase Behaviour Of (5oba:Ebca) P- Pentyloxy Benzoic Acid:P-(P'-Ethoxy Benzylidene)-Cyano Aniline

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ABSTRACT: Synthesis and characterization by polarizing optical microscope, differential scanning calorimeter and infrared spectroscopy of hydrogen bonded liquid crystalline compound consisting of the type mesogenic + mesogenic, namely, p-pentyloxy benzoic acid: p-(p'-ethoxy benzylidene – cyano aniline (5OBA:EBCA). Induced smectic G phase is observed and discussed the results.

KEYWORDS: polarizing optical microscope, Differential Scanning Calorimetry, hydrogen bond, Infrared Spectroscopy, Smectic –G.

I. INTRODUCTION

The role of hydrogen bonding interactions in the formation and stabilization of mesophases has been recognized and significant work has been turned out [1-6]. The synthesis and characterization of supramolecular liquid crystal obtained through hydrogen bonding interaction of complimentary molecule has been extensively studied [7-11]. The first hydrogen bonded complexes exhibiting liquid crystalline behaviour resulted from the dimerisation of aromatic carboxylic acids [7, 12]. Lengthening of rigid rod segments occurs as a property of liquid crystalline behaviour. The recent attention has been focused on the mesomorphism induced by the noncovalent electrostatic interaction involving intermolecular hydrogen bonding. The significance of H-bonded liquid crystal materials is all together highly realized.

The non-covalent interactions exhibit significant effects on physical and thermal properties i.e., mesomorphic behaviour and transition temperatures [13]. Induced smectic A phase is reported [14] in p-n-alkyl benzoic acids with p-(p'-octyloxy benzylidene)-cyano aniline. A new series of supra molecular liquid crystals (nOBA:TMD n = 4 to 12) were studied by Kalyani et al. [15]. Supramolecule through intermolecular hydrogen bond in 5OBA:TMD liquid crystal was studied [16,17].

It was found that hydrogen bonds are successfully employed for the formation of new mesophase. The hydrogen bond has a striking influence on the phase behaviour as well as transition temperatures. The hydrogen bonded liquid crystal has been synthesized with the combination of mesogenic + mesogenic, mesogenic + nonmesogenic and non- mesogenic + non- mesogenic compounds. In continuation of our work on liquid crystals, synthesis of hydrogen bonded liquid crystalline compound consisting mesogenic + mesogenic, namely, p-pentyloxy benzoic acid: pbenzylidene (p'-ethoxy cyano aniline (50BA:EBCA) is carried out.

II. EXPERIMENTAL Preparation of Hydrogen Bonded Mesogen

p- pentyloxy benzoic acid (50BA) and p-(p'-ethoxy benzylidene) – cyano aniline (EBCA) mesogens were obtained from M/s Frinton Laboratories, Inc., USA

The complex, p-pentyloxy benzoic acid : p-(p'-ethoxy benzylidene) – cyano aniline (5OBA: EBCA) was synthesized by refluxing together in equimolar ratio (1:1) of 5OBA 0.4505gm and EBCA.3914 gm. They were taken individually and mixed in the pyridine solvent (20ml). Now the two solutions were mixed and kept under constant stirring at 80° C for ~4hrs. Then most of the pyridine



is removed by vacuum distillation process. It means the volume of the resultant homogeneous mixture was reduced to almost dryness by removing the excess pyridine under a controlled vacuum filtration. The white crystalline product was dried and recrystallized from hot dichloromethane solution. The yielding is at about 85% with intermolecular hydrogen bonded structure shown in Fig. 1 along with the preparation route.



Pyridine, 4 hrs reflux

And after distillation

Fig 1.Intermolcular Hydrogen Bonded mesogen 50BA: EBCA

Liquid crystal is birefringent and it exhibits different textures for different temperatures under crossed polars. A polarizing optical microscope MEOPTA, MICROSCOPE-DRO-3, (serial number 56512) with a heating stage attached high resolution Canon camera was used to identify the different phases and to determine phase transition temperatures. A -50° C to 300° C range digital thermometer with an accuracy of $\pm 0.1^{\circ}$ C was used to measure the temperature.

The transition temperatures of the compounds under investigation were determined using Perkin-Elmer differential scan calorimeter (DSC), modal Diamond in both heating and cooling cycles at a scan rate of 5° C/min. and the sample weights were measured with single pan balance Metler Model AE 240 with an accuracy of 0.01 mg.

The Fourier transform Infrared Spectrometer is useful in obtaining wave numbers of different bonds. In the present study model FTIR 5300 spectrometer of JASCO (Japan) instrument was used

III. RESULTS AND DISCUSSION Polarizing Optical Microscope (POM):

The textural studies were performed in both heating and cooling stages for the individuals as well as the prepared compound.

In heating cycle, pure 5OBA which is enantiotropic exhibits threaded marble nematic (plate 1) phase in between the temperatures 122.8°C and 148°C. On further heating, the nematic phase with droplets is shown in (plate 2). Afterwards, on further heating, it becomes isotropic above 148°C. In cooling cycle, the nematic droplets separated from the isotropic phase at 146.5°C combine to form nematic phase and this phase remains up to 121.6°C. On further cooling, it becomes solid below 121.6°C.

The transition temperatures corresponding to pure 5OBA reported by Rajeswari et al [18] are also given. i.e., in heating cycle, the pure 5OBA exhibits nematic phase in between the temperatures 123.9°C and 151.4°C. On further heating, it becomes isotropic. The first reported values of transition temperatures corresponding to 5OBA reported by Gray et. al., [19] are also given. i.e., the pure 5OBA exhibits nematic phase in between the temperatures 125°C and 153.5°C. On further heating, it becomes isotropic. The transition temperatures corresponding to pure 5OBA reported by Frinton Laboratories [20] are also given. In Table 1

EBCA, which is also an enantiotropic liquid crystal, exhibits nematic phase (plate 3) 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 nematic droplets of EBCA are shown in (plate 4).

The transition temperatures corresponding to pure EBCA reported by Venkata Rao [21] are also given. i.e., the pure EBCA exhibits nematic phase in between the temperatures 106°C and 122°C. On further heating, it becomes isotropic. The transition temperatures corresponding to pure EBCA reported by Frinton Laboratories [20] are also given. i.e., the pure EBCA exhibits nematic phase in between the temperatures 103°C and 125°C. On further heating, it becomes isotropic.

The prepared compound 5OBA: EBCA exhibits two phases i.e., smectic-G, and nematic in between solid phase and isotropic phase. 5OBA: EBCA exhibits an induced smectic-G phase only in cooling cycle, in addition to the nematic phase. In heating, threaded marble nematic phase is observed in between the temperatures 75.5°C and 110.8°C. On further heating, the nematic phase is changed to Isotropic phase at 110.8°C.

On cooling, liquid crystalline nematic droplets separate from Isotropic phase at 111.1° C same as shown in the plate 2. These nematic droplets combine to form threaded marble nematic phase as shown in plate 1. The nematic phase is changed to smectic-G phase at 93.5°C. The smectic-G phase consists of mosaic like structure at temperature of 93.5°C is shown in plate 5. The smectic-G phase at 89°C is also shown in plate 6.



The smectic-G phase is changed to solid phase at $74^{\circ}C$.

Hence the synthesized compound exhibits enantiotropic liquid crystal nature with smectic-G phase only in cooling cycle in addition to the nematic phase in both heating and cooling cycles. The data is depicted in Table 1.



Plate 1Threaded marble nematic texture of 5OBA



Plate 2 Nematic droplets of 50BA



Plate.3 Threaded Nematic texture of EBCA



Plate 4 Nematic droplets of EBCA



Plate 5 Mosaic texture of Smectic G in 50BA:EBCA at 93.5 ^oC



Plate 6 Induced texture of Smectic G in 50BA:EBCA at 89

3.2. Differential Scanning Calorimeter:

The transition temperatures of pure 50BA, EBCA and the compound 50BA: EBCA according to the Differential Scanning Calorimetry (DSC) are given below.

In heating cycle, the pure 5OBA exhibits transitions at 121.2° C and 151.5° C. Afterwards, on further heating, it becomes isotropic. In cooling cycle, phase transition temperatures at 150.4° C and 113.6° C. On further cooling, it becomes solid below 113.6° C. The DSC thermogram of 5OBA is shown in figure 2. in both heating and cooling cycles.

EBCA, which is enantiotropic liquid crystal, 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. The DSC thermogram of EBCA is shown in figure 3. in both heating and cooling cycles.

In heating cycle, 5OBA: EBCA exhibits nematic phase in between the temperatures 77.9° C and 112.6° C. Afterwards, on further heating, it becomes isotropic. In cooling cycle, the nematic phase separates from the isotropic phase at 106.1°C. Further, on cooling, nematic phase is changed to smectic-G phase at 101.1°C. The transition peak corresponding to smectic-G to solid transition is not well resolved in DSC. The DSC thermogram of 50BA: EBCA is shown in figure 4.



The transition temperatures obtained from the DSC studies are shown in Table 1. The phase transition temperatures observed through thermal polarizing microscope are almost coinciding with DSC studies.

The results of 5OBA: EBCA are compared with that of 8OBA: EBCA, 5OBA: MBCA and 5BA: EBCA which are tabulated in table 1.

The thermal span of the nematic phase in 50BA: EBCA is decreased when compared with that of 50BA: MBCA, 80BA: EBCA and 5BA: EBCA. The thermal span in 50BA: EBCA is about 18°C whereas the thermal spans in 50BA: MBCA, 80BA: EBCA and 5BA: EBCA are 37°C, 35°C and 40°C respectively.

The thermal span of the smectic-G phase in 5OBA: EBCA is increased when compared with that of 5OBA: MBCA and decreased when compared with that of 8OBA: EBCA and 5BA: EBCA. The thermal span in 5OBA: EBCA is about 20°C whereas the thermal spans in 5OBA: MBCA and 8OBA: EBCA are 17°C, 34°C and 14°C respectively.

Hence the synthesized complex exhibits enantiotropic liquid crystal nature with inducement of smectic-G phase in addition to the nematic phase. The data is depicted in **Table 1**.

Differential Scanning Calorimeter:

The transition temperatures of pure 5OBA, EBCA and the compound 5OBA: EBCA according to the Differential Scanning Calorimetry (DSC) are given below.

In heating cycle, the pure 5OBA exhibits nematic phase at 121.2°C and this phase remains up to 151.5°C. Afterwards, on further heating, it becomes isotropic. In cooling cycle, the nematic phase separates from the isotropic phase at 150.4°C and this phase remains up to 113.6°C. On further cooling, it becomes as solid below 113.6°C. The DSC thermogram of 50BA is shown in figure 2. in both heating and cooling cycles.



Fig.2. Thermogram of 5OBA

EBCA, which is enantiotropic liquid crystal, 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. The DSC thermogram of EBCA is shown in figure 3. in both heating and cooling cycles.

Table 1: POM in Heating and Cooling Cycles for50BA and 50BA:EBCA

JODA allu JODA.EDCA							
Name of	Expt.	Solid	N to				
The	-	to N	Iso				
Publisher/			ropic				
Present			1				
Study							
A V Raies	ТМ	123.9°	$151 4^{\circ}$				
wari	1.111.	C	151. ч С				
[108]		C	C				
C W Grou	тм	125°C	152 50				
0.w. 01ay	1.111.	125 C	155.5 C				
[190]		102.00	L 140.0C				
Frinton		123°C	149°C				
Laboratori							
es							
[194]							
Present	T.M	122.8°	148°C				
Study	(H)	С					
	T.M(C)	121.6°	146.5°				
		С	С				
	DSC(H	121.2°	151.5°				
)	С	С				
	DSC(C	113.6°	150.4°				
)	С	С				
Frinton	,	103°C	125°C				
Laboratori							
es							
[194]							
V Venkata	ТМ	106°C	122°C				
Rao	1.1111.	100 0	122 0				
[100]							
Drosont	тм	08 5°C	123.00				
Study		90.3 C	123.9 C				
Suuy	(Π)	94 2 ⁰ C	1166^{0}				
	1.M(C)	84.5 C	110.0				
			C				



	DSC(H	103.8°	126.3°
)	С	С
	DSC(C	76.6°C	116.3°
)		С
		Solid	S _G to
		to	Ν
		S _G /N	
Present Study	T.M	75.5°C	
	(H)		
	T.M(C)	74°C	93.5°C
	DSC(H	77.9°C	
)		
	DSC(C		101.1°
)		С



Fig. 3. Thermogram of EBCA

In heating cycle, 5OBA: EBCA exhibits nematic phase in between the temperatures 77.9° C and 112.6° C. Afterwards, on further heating, it becomes isotropic.

In cooling cycle, the nematic phase separates from the isotropic phase at 106.1°C. On cooling, nematic phase is changed to smectic-G phase at a temperature of 101.1°C. The transition peak corresponding to smectic-G to solid transition is not well resolved in DSC. The DSC thermogram of 50BA: EBCA is shown in figure 4. in both heating and cooling cycles at a scan rate of 5°C/min.



Fig. 4. Thermogram of 5OBA:EBCA

The transition temperatures obtained from the DSC studies are shown in Table 1. The phase transition temperatures observed through thermal polarizing microscope are almost coinciding with DSC studies.

The POM results (cooling) of 50BA: EBCA are compared with that of 80BA: EBCA, 50BA: MBCA and 5BA: EBCA which are tabulated in table 2 .in $^{\circ}C$

Table 2 Comparison of transition temperatures of 50BA: EBCA with 50BA: MBCA, 80BA: EBCA

and SDA: EDCA III C						
Compo	Iso-N	N-	N/ S _{C/}	S _G -		
und		S _C	SG	Solid		
50BA:	111.1		93.5	74		
EBCA						
50BA:	99.5		62.6	45.4		
MBCA						
80BA:	133.5	98.5	90.5	57.1		
EBCA						
5BA:	110.8		69.8	55.6		
EBCA						

The thermal span of the nematic phase in 50BA: EBCA is decreased when compared with that of 50BA: MBCA, 80BA: EBCA and 5BA: EBCA. The thermal span in 50BA: EBCA is about 18°C whereas the thermal spans in 50BA: MBCA, 80BA: EBCA and 5BA: EBCA are 37°C, 35°C and 40°C respectively.

The thermal span of the smectic-G phase in 5OBA: EBCA is increased when compared with that of 5OBA: MBCA and decreased when compared with that of 8OBA: EBCA and 5BA: EBCA. The thermal span in 5OBA: EBCA is about 20°C whereas the thermal spans in 5OBA: MBCA and 8OBA: EBCA are 17°C, 34°C and 14°C respectively.

FTIR Spectra:

Infrared spectroscopy is an important and powerful technique for structural elucidation of organic molecules, which involve both intra and inter molecular hydrogen interactions. 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- pentyloxy benzoic acid (5OBA) :

The IR spectrum of p-pentyl benzoic acid recorded at room temperature in solid (KBr) state is shown in figure 5. The spectrum of p-pentyl benzoic acid shows a sharp peak at 1678.22cm⁻¹ for v (C = O) mode due to KBr. Another strong intense bond assigned to v (OH) mode of carboxylic acid group is obtained at 2955.21cm⁻¹due to KBr.

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 6). The spectrum of ethoxy benzylidene p- cyano aniline shows a sharp peak at 2223.52 cm^{-1} for v (CN) mode in KBr.

p -pentyloxy benzoic acid:p- (p'- ethoxy benzylidene) - cyano aniline (5OBA: EBCA) :

The infrared spectrum of 5OBA: EBCA is recorded in KBr (solid) state and the spectrum is shown in figure 7. The infrared frequencies of 5OBA: EBCA show the presence of a strong intense band due to v (C = O) mode of benzoic acid moiety at ~1681.62cm⁻¹. This strongly supports the existence of 5OBA moiety in a monomeric form upon complexation. The formation of inter molecular hydrogen bonding between COOH group of 5OBA and CN group of EBCA can be invoked on the basis of hypsochromic shift in v(CN) mode (~5-6cm⁻¹) of EBCA and v (OH) mode (~20 cm⁻¹) of 5OBA in KBr upon complexation which strongly supports the existence of H– bonding. The existence of H-bonding is strongly confirmed due to the induced frequency of ~3420cm⁻¹, lying in the N----H stretching frequency range due to H- bonding.

There exists inter-molecular hydrogen bonding between partially positively charged hydrogen atom of -COOH group present in 50BA and partially negatively charged highly electro negative nitrogen atom of -CN group present in EBCA. Due to this inter-molecular hydrogen bonding, it exist an associated liquid crystal. 50BA has acidic nature because it readily looses proton to form pentyloxy benzoate ion. It has several resonating structures and it gets more stability. Hence 50BA acts as Louis acid. Nitrogen atom of the -CN group of EBCA donates a lone pair of electrons to the hydrogen ion of the carboxylic acid and a co-ordinate covalent bond is formed between these two compounds. Here EBCA acts as Louis base. So, the above reaction is considered as neutralization [22-25].

These variations in chemical shifts indicate the involvement of the OH group of acid and the NH group at the tail end. The hydrogen bonding expected between the involved groups by self assembling process leading the small molecular aggregates to form lamellar texture. The larger variations emphasis the local field variations within the molecule to change the order of transition and the large tilt angle in inducement of smectic G phase.



Fig.6: FTIR spectrum of EBCA





Fig 7: FTIR Specturm of 50BA:EBCA

From observations of stretching frequencies of $(C=O)_{amide}$, $(OH)_{acid}$ and NH, the hydrogen bonding may be expected between $(C=O)_{amide}$ and $(OH)_{acid}$ that lead to structure as illustrated in **Figure 7** The IR spectra of the complexes were recorded in chloroform which reveal the increased stretching frequencies of (OH) resulting in the destruction of hydrogen bonding in solution.

The importance of hydrogen bonds greatly influenced in great many physical, chemical, and biological processes is broadly accepted. Typical hydrogen bonds are those where the hydrogen atom lies between two electronegative atoms and further hydrogen bonds that involve donors and/or acceptors which are unconventional involving non covalent interactions gained importance.

Hydrogen-bonding liquid crystals [26] and their complexes that induced crystal G phase in mixtures have interesting properties due to the intermolecular hydrogen bonds. Liquid crystal substances constituted by hydrogen bond in dimer molecules are p-n-alkoxy benzoic acids with n=10 that diplay both nematic and smectic C phase. The occurrence of phase is due to presence of a sufficiently high concentration of dimmers with open dimer corresponds to nematics and closed dimer corresponding to smectics[18]. These sufficiently influence on the microscopic scale in properties of phase, linked with inter-molecular hydrogen bonding.

The energy of closed dimmers is destroyed by temperature variation that resulted in variation in shifts participating in hydrogen bonding and further with variation in intensity in hydrogen bonded complexes. It is due to self assembling process and concentration of hydrogen bonded complex resulted in evolution of new growth of liquid crystalline phase. It is substituent is present which may be supporting the formation of smectic phase by reducing the transition temperature low enough to maintain the layered arrangement by intermolecular hydrogen bonding. These intermolecular hydrogen bonded carboxylic acid dimers aid the formation of networks which exhibit smectic G phase due to the dynamic nature of the non-covalent interaction.

IV. CONCLUSIONS

The thermal polarizing microscope observations show that the three compounds namely, i) p-pentyloxy benzoic acid (50BA), ii) p-(p'- ethoxy benzylidene)-cyano aniline (EBCA) and iii) p-pentyloxy benzoic acid: p- (p'-ethoxy benzylidene)-cyano aniline (50BA: EBCA), are enantiotropic liquid crystals. The phase transition temperatures observed through polarizing microscopic studies are in reasonable agreement with the transition temperatures obtained from Differential Scanning Calorimetry and the transition peak corresponding to smectic-G to solid transition is not well resolved in DSC. An induced Smectic-G phase is observed only in cooling cycle. The induced Smectic-G phase may be due to the intermolecular hydrogen bond. The intermolecular hydrogen bonding between the OH group of ppentyloxy benzoic acid and N=C of p- (p'- ethoxy benzylidene) - cyano aniline (EBCA) is confirmed through IR studies.

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