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# Textural, thermal and infrared studies on hydrogen bonded liquid crystal *p*-pentyl benzoic acid:propyl *p*-hydroxy benzoate (5BA:PHB)

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**ABSTRACT:** Mesogen (Liquid crystal) and non-mesogen (non-liquid crystal) combination class of *p*-pentyl benzoic acid:propyl *p*-hydroxy benzoate complex was synthesised. Enantotropic smectic G phase of texture was observed using polarizing microscopic studies. Phase transition temperature investigations were carried out using polarizing microscopy and differential scanning calorimetry (DSC). Transition temperatures of in good agreement with DSC and microscope studies. FTIR studies reveal in solid (KBr) and solution (chloroform) state that there is a hetero dimer formation of acid moieties in the two compounds.

**KEYWORDS:** Texture, transitions temperatures, DSC, FTIR, Intermolecular hydrogen bond,

## I. INTRODUCTION

Aromatic carboxylic acids are the first compounds that exhibit liquid crystalline behaviour due to hydrogen bond formation [1,2]. In recent years, supramolecular liquid crystals have been synthesized through hydrogen bonding of complementary molecules in a large number. The role of H-bonding interactions in the formation and/or stabilization of liquid crystalline phases has also been recognized [3]. If the complex is formed through inter molecular hydrogen bond, lengthening of rigid rod segments occurs as a property of liquid crystalline behaviour. Induced mesomorphism due to non-covalent electrostatic interaction involving intermolecular hydrogen bonding type work is gained momentum [4]. Mesogen and mesogen as well as mesogen and non mesogen systems [5,6,7] were carried out by our group. In continuation of research, this work is taken up to prepare complex liquid crystal via hydrogen bond through the type of mesogen and non-mesogen combination, namely *p* -pentyl benzoic Acid (5BA) and propyl *p*- hydroxy benzoate (PHB)

## II. EXPERIMENTAL

Textural, transition temperatures and infrared studies are carried out using Mopta polarizing optical microscope, M/s Thermal Analysis Instruments Differential Scanning Calorimeter (DSC) and FTIR 5300 JASCO infrared spectrometer respectively. Polarizing optical microscope is equipped with hot stage indigenously designed [8] and Canon colour camera attachment. Temperature is measured with a digital thermometer with an accuracy  $\pm 0.1^\circ\text{C}$ .

### Preparation of *p*-pentyl benzoic acid :-propyl *p*-hydroxy benzoate (5BA :PHB):-

*p* -Pentyl Benzoic Acid (5BA) and propyl *p*- hydroxy benzoate (PHB) are purchased from Frinton Laboratories Vineland, New Jersey, USA. The intermolecular hydrogen (H) bonding complex, *p* – pentyl benzoic acid :propyl *p*-hydroxy benzoate (5BA : PHB) was synthesized by refluxing together equimolar ratio(1:1) of *p*-Pentyl benzoic acid (0.6408 gm) and Propyl *p*- Hydroxy benzoate (0.6007 gm) and 20 ml of pyridine under constant stirring at  $80^\circ\text{C}$  for ~4hrs and the detailed procedure is reported in [6,7]. The yielding is of about 85 %.. The synthesis route is given in Fig. 1.

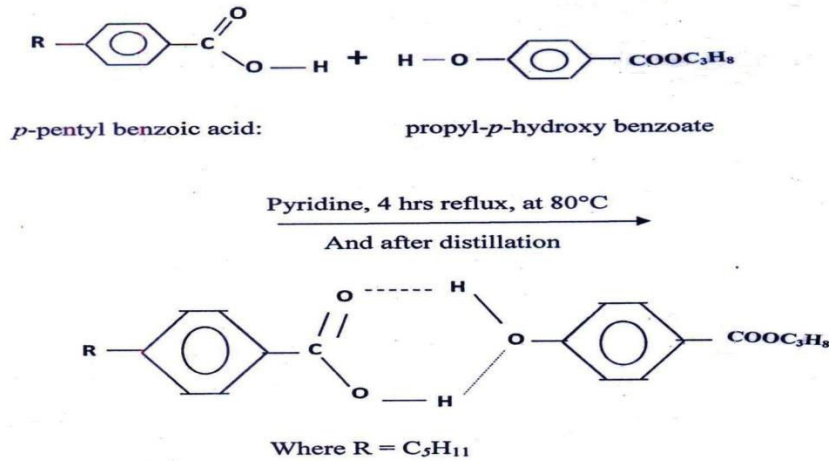


Fig. 1: Synthesis of 5BA:PHB

### III. RESULTS AND DISCUSSION

#### Thermal polarizing microscopy: -

In heating cycle, the pure 5BA exhibits nematic (Fig. 2) phase in between the temperatures 82.8°C and 118.8°C. The nematic droplets formed just before the isotropic phase is shown in Fig. 2. On further heating, it becomes isotropic above 118.8°C. In cooling cycle, the nematic droplets, separated from the isotropic phase at 116.2°C, combine to form nematic phase and this phase remains up to 80.6°C. On further cooling, it becomes solid below 80.6°C.

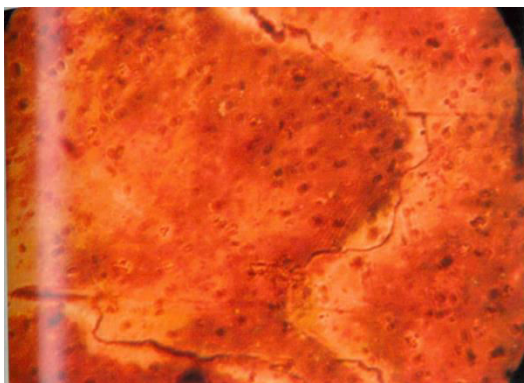


Fig. 2: Nematic texture of 5BA: PHB

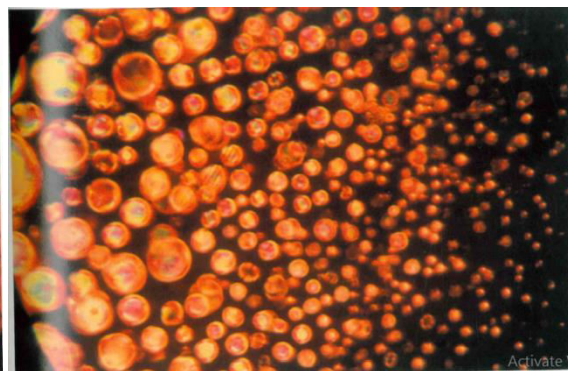


Fig. 3: Nematic droplets of 5BA

The transition temperatures corresponding to pure 5BA reported by Venkata Rao [9] are given i.e., in heating cycle, the pure 5BA exhibits nematic phase in between the temperatures 82°C and 115°C. On further heating, it becomes isotropic.

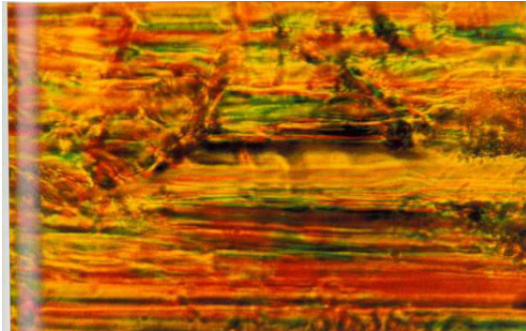
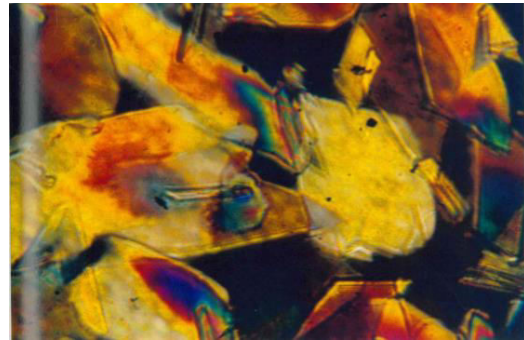
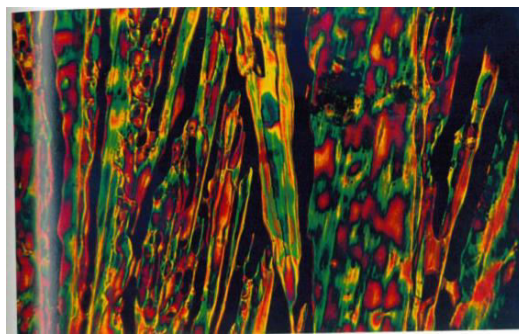
The transition temperatures corresponding to pure 5BA given by Frinton Laboratories is also given. i.e., the pure 5BA exhibits nematic phase in between the temperatures 86°C and 122°C and becomes isotropic on further heating.

The propyl-p-hydroxy benzoate is a non-mesogen and its melting point is 98°C

The synthesized compound 5BA: PHB exhibits an enantiotropic smectic-G phase with smooth multi-coloured mosaic texture. In heating cycle, the 5BA: PHB compound exhibits mosaic smectic G phase (Fig. 4) in between the



temperatures 73°C and 83°C. On cooling the isotropic melt, an induced smectic G phase (Figs. 5 and 6) is observed in between the temperatures 68.3°C and 63°C. Below 63°C, it becomes solid. The thermal spans of smectic G phase in 5BA: PHB are 10°C and 5.3°C in both heating and cooling respectively.

Fig. 4L  $S_G$  phase at 63.8 °C in 5BA: PHBFig. 5.:  $S_G$  phase at 67.5 °C in 5BA: PHBFig. 6:  $S_G$  phase at 80.2 °C in 5BA: PHB

#### Differential Scanning Calorimeter:

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

In heating cycle, the pure 5BA exhibits nematic phase at 89°C and this phase remains up to 126°C. Afterwards, on further heating, it becomes isotropic. In cooling cycle, the nematic phase separates from the isotropic phase at 124°C and this phase remains up to 84°C. On further cooling, it becomes as solid below 84°C. The DSC thermogram of 5BA is shown in Fig. 7. in both heating and cooling cycles.

For 5BA: PHB, in heating cycle, the peak corresponding to solid to smectic G phase is not well resolved in DSC. It becomes isotropic at 80.6°C. In cooling cycle, the smectic-G phase is separated at 71.13°C from the isotropic melt and retains up to 64.9°C. Afterwards, on further cooling, it becomes as solid below 64.9°C. The phase transition temperatures observed through thermal polarizing microscope are almost coinciding with DSC studies. The thermal span of smectic-G phase in 5BA: PHB is 6.2°C in cooling cycle.

The transition temperatures obtained from the DSC studies are shown in Table 1. The DSC thermogram of 5BA: PHB is shown in Fig 8 in both heating and cooling cycles at a scan rate of 5°C/min. Temperature hysteresis is observed as a predominant characteristic in this investigation. DSC study also confirms the feature which is observed through polarizing microscopy.

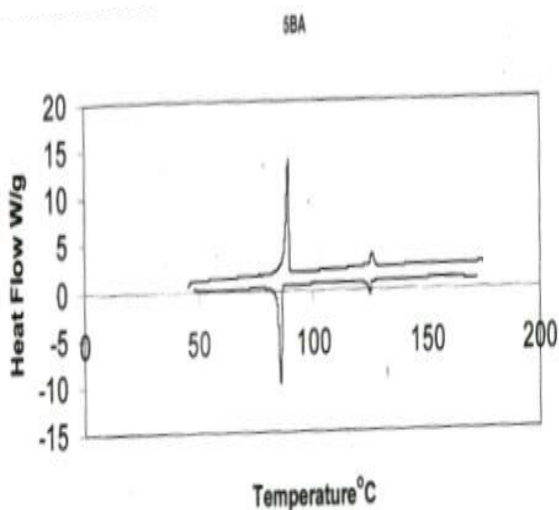


Fig. 7: DSC thermogram of 5BA

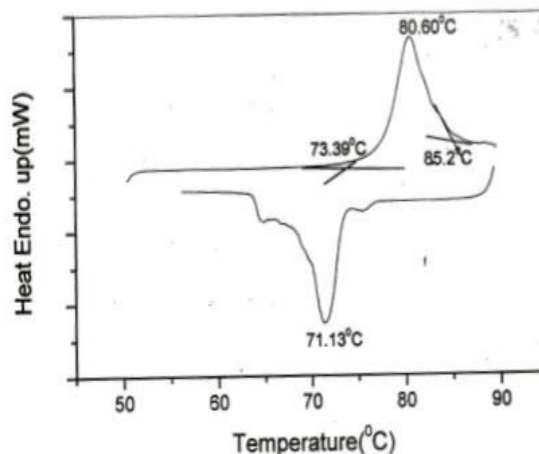


Fig. 8: DSC thermogram of 5BA:PHB

\\ Table 1: T.M. and DSC data on 5BA:PHB in °C

p- 5BA (p-Pentyl Benzoic Acid)			
Name of the reporter// Present study	Experiment	Solid to Nematic	Nematic to Isotropic
Friton Laboratories [10]		86	124
V, Venkata Rao [ 9]	T.M.	82	115
Present study	T.M (H)	81.6	118.8
	T.M. (C)	80.6	116.2
	DSC (H)	89	126
	DSC (C)	84	124
Synthesized compound p-5BA:p-PHB (p-pentyl benzoic acid and propyl p-hydroxy benzoate)			
		Solid to Smectic G	Smectic G to Isotropic
	T.M (H)	73	83
	T.M. (C)	63	683
	DSC (H)	* peak not well resolved.	80.6
	DSC (C)	64.9	71.1

**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. So, to probe the existence of inter molecular hydrogen bonding in the synthesized compound 5BA:PHB using IR spectra.

**p-pentyl benzoic acid (5BA):-**

The IR spectrum of p-pentyl benzoic acid recorded at room temperature in solid (KBr) state is shown in Fig 9. The spectrum of p-pentyl benzoic acid shows a sharp peak at 1680.66cm for  $\nu(C = O)$  mode and 1290.14 cm for  $\nu(CO)$  mode due to KBr. Another strong intense bond assigned to  $\nu(OH)$  mode of carboxylic acid group is obtained at 2927.41cm due to KBr.

**Propyl-p-hydroxy benzoate (PHB) :-**

The IR Spectrum of propyl-p- hydroxy benzoate is recorded at room temperature in solid (KBr) (shown in Fig . 10) The spectrum of propyl-p- hydroxy benzoate shows sharp peaks at 3270.68cm for  $\nu(OH)$  mode and 1281.47cm for  $\nu(C-O)$  in KBr.

**p-Pentyl benzoic acid: Propyl-p-hydroxy benzoate (5BA: PHB):-**

The infrared spectrum of 5BA: PHB is recorded in KBr (solid) state and the spectrum is shown in Fig 11. The infrared frequencies of 5BA: PHB show the presence of a strong intense band due to (C = O) mode of benzoic acid moiety at ~1679.69cm<sup>-1</sup>. This strongly supports the existence of 5BA moiety in a monomeric form upon complexation. The formation of inter molecular hydrogen bonding between COOH group of 5BA and OH group of PHB can be invoked on the basis of bathochromic shift in  $\nu(\text{OH})_{\text{phenolic}}$  mode (~2cm<sup>-1</sup>) and of PHB in KBr and  $\nu(\text{OH})_{\text{acid}}$  mode of carboxylic acid group also suffered a hypsochromic shift of (~26cm<sup>-1</sup>) and  $\nu(\text{C-O})_{\text{acid}}$  mode (9cm<sup>-1</sup>) suffered a bathochromic shift upon complexation which strongly supports the existence of H-bonding. The IR spectral data corresponding to 5BA, PHB and 5BA: PHB are given in Table 4.

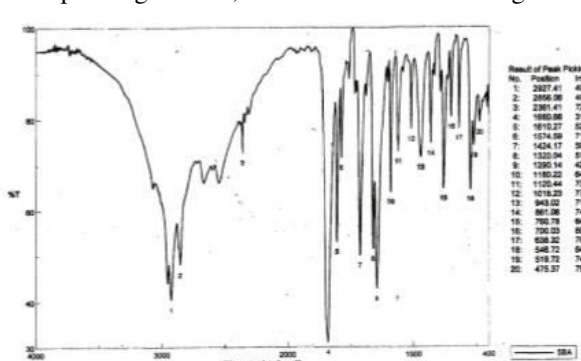


Fig.9: FTIR Spectrum of 5BA in KBr

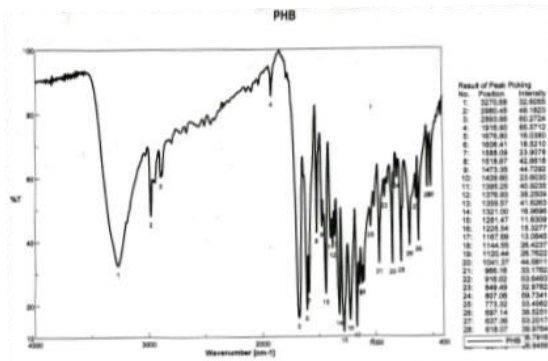


Fig 10: FTIR Spectrum of PHB in KBr

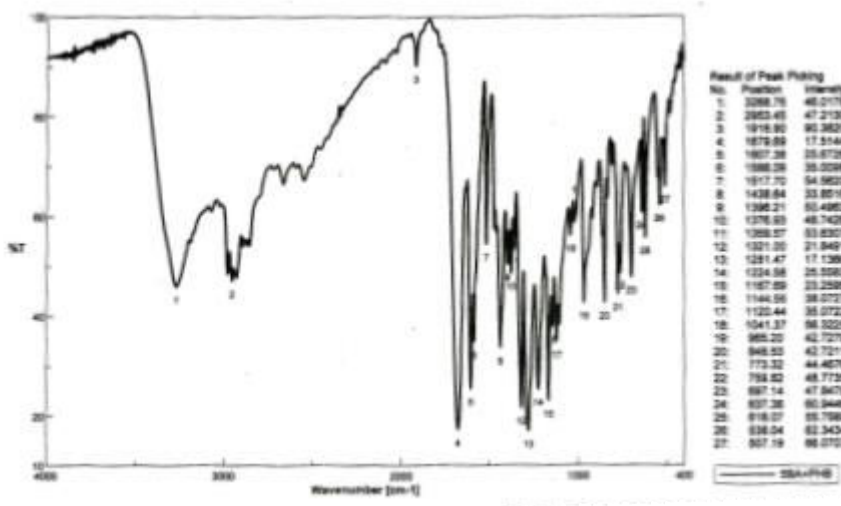


Fig 11: FTIR Spectrum of 5BA:PHB in KBr

There exists intermolecular hydrogen bonding between partially positively charged hydrogen atom of -COOH group present in 5BA and partially negatively charged highly electro negative oxygen atom of -OH group present in PHB. Due to this intermolecular hydrogen bonding, it exists as an associated liquid crystal. 5BA has acidic nature because it readily loses proton to form pentyl benzoate ion. It has several resonating structures and it gets more stability. Hence 5BA acts as Lewis acid. Oxygen atom of the -OH group of PHB donates a lone pair of electrons to the hydrogen ion of the carboxylic acid and a hydrogen bond is formed between these two compounds. Here PHB acts as Lewis base. So, the above reaction is considered as neutralization [11-14]

A detailed IR spectral investigation of 5BA:PHB in solid state (KBr) suggests that the acid and phenol groups act as both proton donor and proton acceptor respectively as reported by Koga et al [15,16] and Rajeswari [17]. Similar result

of the inducing of smectic G phase and quenching of the nematic phase in the synthesized complex which is present in the pure 5BA compound has also been reported [17] in 5OBA: PHB.

Comparing the thermal span of smectic-G phase in 5BA: PHB is 5.3°C whereas in 5OBA: PHB, it is 44°C. The thermal span of smectic G phase in 5BA: EBCA is 14.2°C whereas in 5OBA: EBCA, it is 19.5°C. The thermal span of smectic G phase decreased [17,18] in hydrogen bonded liquid crystals which were synthesized with alkyl benzoic acids when compared with that of alkoxy benzoic acids. Hence, the synthesized complex 5BA: PHB is an enantiotropic liquid crystal with the induced smectic G phase and the quenched nematic phase.

#### IV. CONCLUSIONS

The thermal polarizing microscope observations show enantiotropic 5BA exhibit nematic but enantiotropic 5BA:PHB smectic G phase due to intermolecular hydrogen bond. The phase transition temperatures observed through microscopic studies are in reasonable agreement with the transition temperatures obtained from DSC. Intermolecular hydrogen bond is also confirmed through FTIR study formed between COOH of *p*-pentyl benzoic acid and COOH of propyl *p*-hydroxy benzoate due to hetero dimer formation Comparing the thermal span has 5BA:PHB compound 5.3 °C and 5OBA:PHB hydrogen bond compound 44 °C.

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