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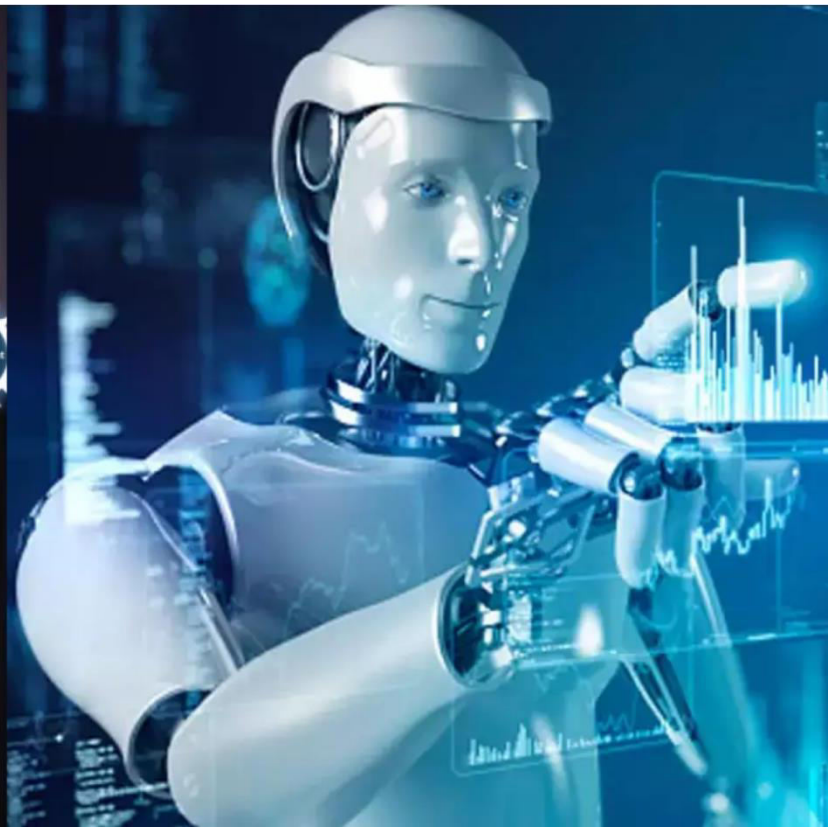

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Liquid Crystalline Phase Behavior in *nOBA:EBCA* through Intermolecular Hydrogen Bond

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ABSTRACT: This study investigates the phase behavior of liquid crystal complexes formed through self-organization of p-n alkoxy benzoic acid mesogens (nOBA where n= 4-10, and 12) with mesogenic material p-(p'-ethoxy benzylidene)-p-cyano aniline (EBCA). nOBA and EBCA exhibit enantiotropic behavior, displaying both a nematic phase and a smectic C phase in their higher homologues. The molecular structure of liquid crystal complexes can also be enhanced through hydrogen bonding, resulting in increased molecular packing density and the formation of the crystal G phase (nOBA:EBCA). The liquid-crystalline complex phase change temperatures are slightly lower than those of the pure systems (nOBA, EBCA). A comparative examination of phase abundance is provided for the pure nOBA and various hydrogen-bonded liquid crystal complexes of nOBA:EBCA. Differential Scanning Calorimetry (DSC) and Polarizing Optical Microscope (POM) are used to determine the complex's thermal and phase behavior. Intermolecular interactions of the molecules lead to the formation of a hydrogen bond between the proton donor (COOH) of nOBA and proton acceptor CN group of EBCA.

KEYWORDS: Liquid crystals, hydrogen bond, phases, transition temperatures, POM, DSC.

I. INTRODUCTION

In recent research, demand for new liquid crystals has increased due to their scientific and technical applications in fields such as displays, electro-optic devices, photonics, health care, photovoltaic cells, fiber communications, and biology. Synthesis of new liquid crystals involving hydrogen-bond formation between molecules has attracted widespread attention. Formation of hydrogen bonds between the components of molecules results the new complex systems with special shapes and structures [1-5]. Moreover, these have a notable impact on the behavior of liquid crystals, including transition temperatures, enthalpies, entropies, phase quenching, and the induction of new phases with a broad temperature range [6–11].

Hydrogen bonding has recently emerged as an alternative approach in the design of new twist-bend nematogens. Specifically, the twist-bend nematic phase (NTB) exhibited by hydrogen-bonded liquid crystal complexes has been generated from the pairs of acids (like nOBA), yielding bent supramolecular complexes [12-19]. In this study, a unique homologous series of mesogenic materials is generated by forming a hydrogen bond between the COOH of the mesogenic compounds p-n alkoxy benzoic acids, where n = 4 to 10 and 12 (nOBA) and CN of the mesogenic compound p-(p'-ethoxy benzylidene)-p-cyano aniline (EBCA).The resulting homologous series of liquid crystal complexes exhibits a non-covalent intermolecular interaction that induces and stabilizes the liquid crystalline phase [20-28].Here, age-old mesogenic materials, p-n alkoxy benzoic acids and p-(p'-ethoxy benzylidene)-p-cyano aniline (EBCA), are considered for the preparation of liquid crystalline complexes [21]. p-n-alkoxy benzoic acids are good building blocks for self-organized systems via hydrogen-bond formation. They also exhibit unique mesogenic behavior in terms of transitions, phases, and optical anisotropy. Therefore, a systematic investigation of the liquid crystalline behavior, including thermal and phase characteristics, of the complex systems (nOBA:EBCA, where n=4-10 and 12) is conducted using a Polarising Optical Microscope (POM) and Differential Scanning Calorimetry (DSC). Fourier Transform Infra-Red (FTIR) spectroscopy is used to study intermolecular interactions in complexes.

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II. EXPERIMENTAL

Mesogenic materials p-n alkoxy benzoic acids (nOBA, where n=4-10 and 12) and p-(p'-ethoxy benzylidene)-p-cyano aniline (EBCA) purchased with high purity and used. Liquid crystals are optical anisotropic materials and are highly birefringent. The infrared spectra of the compounds were obtained using a Bruker Alpha infrared spectrometer. The thermal and anisotropic nature of the samples is observed and captured using a crossed-polarization condition of a polarizing optical microscope (Olympus). The objective resolution of the polarizing microscope used for the investigation is 40x. A digital thermometer with a range of -50ºC to 300ºC and an accuracy of +/- 0.1ºC is used to observe and record the transitional temperatures. A commercially available ITO-coated liquid crystal cell with homogeneous (planar) alignment, having a thickness of 5 µm, is used as the sample cell for investigation. Using a DSC Q20 V24.2 Build 107 Universal V4.5 TA instrument in a nitrogen atmosphere, transition temperatures and the enthalpies related to liquid crystal phase transitions of pure (nOBA, EBCA) and complex systems (nOBA:EBCA, where n=4 -10 and 12) are measured at a heating/cooling rate of 5ºC/min. During calibration, the transition temperature in DSC is often defined as the point at which peak values intersect the linearly extrapolated baseline.

2.1.Preparative method

Liquid crystalline compounds: p-n-alkoxy benzoic acids and p-(p'-ethoxy benzylidene)-p-cyano aniline(EBCA) (high purity nearly 99.9%), are procured from Frinton Laboratories, Vineland, New Jersey, USA. Double-distilled solvents with AR grade areused while synthesizing liquid crystalline complexes (nOBA:EBCA, where n=4 - 10 and 12).

2.2. p-n-alkoxy benzoic acids: p-(p'-ethoxy benzylidene)-p-cyano aniline (nOBA: EBCA)

The process of synthesizing the intermolecular H-bonding complex nOBA: EBCA involved a reflux method [29-31].The equimolar mixtures of liquid crystal samples nOBA (where n=4 -10 and 12) and EBCA were prepared by co-dissolving pre-weighed amounts of each compound in absolute pyridine. The prepared solution was stirred continuously at 80ºC for approximately 4 hours. The volume of the resultant homogeneous mixture was then reduced to dryness by removing the excess pyridine under a controlled vacuum filtration. The light-yellow crystalline products were dried and recrystallized from hot dichloromethane solution, then dried over calcium chloride in a desiccator at room temperature [32 - 34].

III. RESULTS AND DISCUSSION

3.1.Thermal and Phase behavior

The first step in studying the liquid crystalline behavior of the synthesized hydrogen-bonded liquid crystals is carried out using thermal analysis tools such as differential scanning calorimetry (DSC) and a polarizing optical microscope (POM). The prepared sample cell of thickness 5µm was placed on the hot stage of POM, and phase transitional behavior of the nOBA, EBCA and *nOBA:EBCA* (where n=4 to 10 and 12) are observed. From optical microscopic studies, it was observed that p-n-alkoxy benzoic acids are enantiotropic liquid crystals, which exhibit the threaded marble nematic mesophases for n=4 to 10 and 12, whereas higher homologs from n=7 to 10, 12 exhibit the schlieren SmC phase [35-37]. p-(p'-ethoxy benzylidene)-p-cyano aniline (EBCA), which is also an enantiotropic liquid crystal, exhibits a unique threaded nematic mesophase [21].

Homologous series of newly synthesized nOBA: EBCA (where n=4 to 10 and 12) complexes exhibit the crystal G phase in addition to the schlieren SmC, threaded marbled nematic phases. The formation of a hydrogen bond between the two liquid-crystalline molecules, nOBA and EBCA, induces the crystal G phase in the series of complexes. In addition to the nematic phase, lower members of series *nOBA:EBCA* also display the crystal G phase. The liquid crystal complexes' induced crystal G phase as a smooth, multicolored mosaic texture. Textures pure and complexes are given in Figs 1 and 3.

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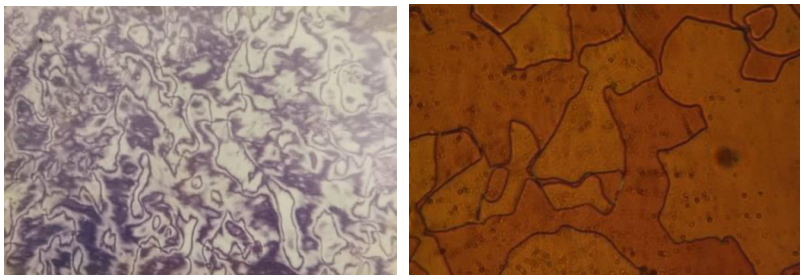


(a) (b)

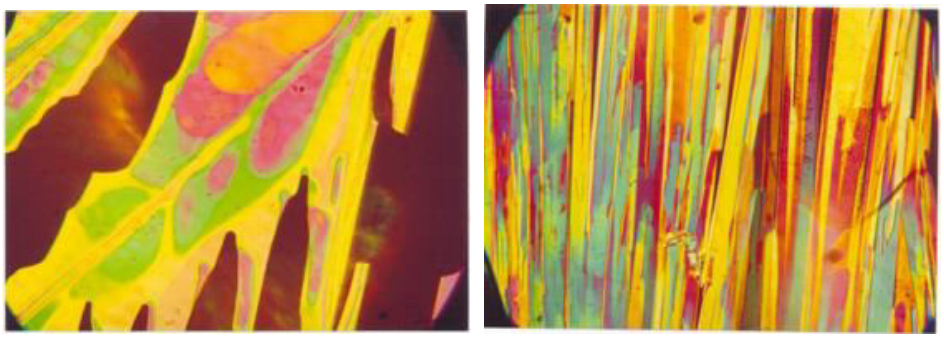
Fig.1. (a) Schlieren S_C Phase in nOBA; (b) Threaded marble nematic in nOBA.



Fig.2. Threaded Nematic texture in EBCA.



(a) (b)



(c) (d)

Fig.3.Crystal G phase of (a) 4OBA:EBCA;(b)7OBA:EBCA;(c)10OBA:EBCA;(d)12OBA:EBCA.

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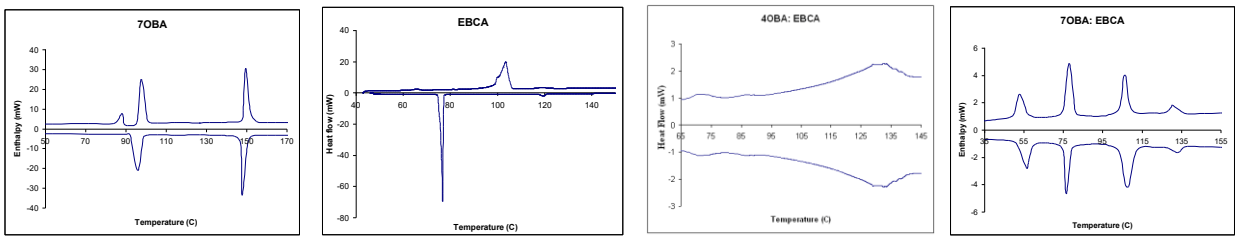
Thermal behavior of liquid crystals nOBA, EBCA and their complexes nOBA:EBCA(where n=4 to 10 and 12) are also evaluated with Differential scanning calorimetry (DSC) measurements. Differential Scanning is used to identify phase transitions and the enthalpies connected to them. The presence of Cr-CrG-SmC -nematic-Isotropic transitions in hydrogen-bonded systems (nOBA:EBCA) is supported by calorimetry studies [21]. DSC thermograms are given in Fig. 4.



(a) **(b)** **(c)** **(d)**

Fig. 4. DSC thermograms of (a) 7OBA; (b) EBCA;(c) 4OBA:EBCA;(d)7OBA:EBCA.

Transition temperatures of nOBA, EBCA samples obtained from POM and DSC values are given in Table 1.

Table 1. Transition temperatures of nOBA: EBCA observed through POM & DSC

Compound	Phase	Solid –G/N	G-C/N	G/C-N	N-I
n=3 (TM) (DSC)	G	118 120.9			142 145.7
n=4 (TM) (DSC)	NG	68.5 71.2		133 133.6	140 137.1
n=5 (TM) (DSC)	NG	74 [a]		93.5 101	111.1 106.1
n=6 (TM) (DSC)	NG	68 65.9		87 90	121.8 125.4
n= 7 (TM) (DSC)	NCG	55 56.1	75 76.4	105.5 108.2	125.6 132.8
n=8 (TM) (DSC)	NCG	57.1 58	90.5 93.8	98.5 [a]	133.5 139
n=9 (TM) (DSC)	NCG	61.1 58.6	83.6 [a]	113.5 [a]	120.2 [a]
n=10 (TM) (DSC)	NCG	74 75.8	91 84.2	106 92.3	122 [a]
n=12 (TM) (DSC)	NCG	71 69.5	85 94	116 123.6	130 137.1

[a] –DSC peak is not well resolved [103]

There are some differences between the transition temperatures obtained from the POM, DSC techniques. The differences are in the range of approximately 8ºC (less than 10ºC) for p-n alkoxy benzoic acid mesogens (nOBA, where n=4-10 and 12) and hydrogen bonded liquid crystals (nOBA:EBCA, where n=4-10 and 12). In case of p-(p'-ethoxy benzylidene)-p-cyano aniline (EBCA) crystal to liquid crystalline phase transition temperature difference between the POM, DSC is very high. In present work, transition temperatures of EBCA determined using POM is 104.5ºC – 122.5ºC and from DSC it is 76.6ºC – 116.3ºCon heating. Transition temperatures for liquid crystal to isotropic phase transition determined from DSC is differing by 13ºC. According to literature found in [40] transition temperature of EBCA is 105ºC - 129ºC for crystalline phase to isotropic transition via liquid crystalline phase (on heating). This is due to the super cooling effect of sample. And also, transition temperatures of liquid crystal samples may depend on the purity, heating element of the instrument, experimental and instrumental conditions (like model)

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while finding the transition temperatures, thickness of the samples and rate of cooling and heating effect. All these conditions, may lead to the differences in transition temperatures obtained from DSC and POM. Transition temperatures of pure nOBA, EBCA and *nOBA:EBCA* are given in Table 2.

Table 2. **Transition temperatures of *nOBA:EBCA* observed through POM and DSC (enthalpy j/g).**

Compound	Phase sequence	Solid –CrG/N	CrG/SmC/N	SmC-N	N-I
n=4 (TM) (DSC)	CrG N	68.5 71.2 (…)		133 133.6(…)	140 137.1(…)
n=5 (TM) (DSC)	CrG N	74 (a)		93.5 101(…)	111.1 106.1(…)
n=6 (TM) (DSC)	CrG N	68 65.9 (8.4)		87 90 (51.4)	121.8 125.4 (36.6)
n= 7 (TM) (DSC)	CrGSmC N	55 56.1(1.29)	75 76.4(22.8)	105.5 108.2(23.9)	125.6 132.8(1.29)
n=8 (TM) (DSC)	CrGSmC N	57.1 58(…)	90.5 93.8(...)	98.5 (a)	133.5 139(…)
n=9 (TM) (DSC)	CrGSmC N	61.1 [a]	83.6 [a]	113.5 [a]	120.2 [a]
n=10 (TM) (DSC)	CrGSmC N	74 75.8 (0.7)	91 84.2(34.2)	106 92.3(16.9)	122 [a]
n=12 (TM) **(DSC)**	**CrG**SmC N	71 **69.5 (1.6)**	85 **94 (61.7)**	116 **123.6(13.3)**	130 **137.1(4.6)**

[a],(…) peaks are not resolved; CrG –Crystal G; SmC – Smectic C; N- Nematic ; I-isotropic.

It is also noted that, thermal span of nematic phase is high for lower alkoxy chain length compounds. It decreases with increasing chain length from n=7 to higher members of the series since they exhibit the SmC mesophase. It was observed that, thermal range of liquid crystal phase is high for the homologous series of nOBA: EBCA when compared to the pure nOBA, EBCA compounds. Thermal span of the liquid crystal phases in complexes is in the range of 37°C–76°C. Liquid crystal complex 8OBA:EBCA exhibit the high thermal range for liquid crystallite phase of 76^{0}C.When compared to pure nOBA series, in the synthesized compounds, the clearing temperatures are lowered and the stability of liquid crystalline phases has also been increased. The flexibility of the molecules can be changed from either side of the liquid crystal molecule by varying the n value in nOBAs. Pure nOBAs have two flexible end chains at both ends of the molecule. However, a variable flexible chain is coupled to one end of hydrogen bound liquid crystal complexes, and the other end is attached to the EBCA, where its length is fixed. Rigid rods' longer chains are reported to produce the crystal G phase in complexes. In the current series, the induction of the crystal G phase and the occurrence of new phase sequences, crystal –CrG, CrG–SmC, and SmC -nematic, may be attributed to a variety of potential molecular contributions arising from non-covalent interactions associated with terminal functional residues, such as the –OH group of the nOBA series and the CN of the EBCA. The imino group and terminal bulky phenyl ring causing the delocalized electron cloud may also play an effective role on the origin of inducement of new phases in addition to these non-covalent interactions. Furthermore, the variation in structural phases between short and long-range homologous series is driven by the competing influences of intercalation, encouraged by the strong interactions among mesogenic cores, and self-organization facilitated by the cyano and alkoxy terminal groups. Based on data on phase transition temperatures and phases of alkoxy benzoic acids and hydrogen-bonded systems, a phase diagram is drawn as a function of different alkoxy chain lengths and shown in Fig 5. This phase diagram helps understand the phase behavior in liquid crystal systems with varying alkoxy chain lengths.

The slope of the interfaces and thermal stabilities corresponding to the crystal - SmC/nematic, SmC - nematic, and nematic - isotropic transitions of alkoxy benzoic acids and crystal - CrG, CrG–SmC, SmC - nematic, and nematic – isotropic transitions were found to vary with increasing alkoxy chain length (n) value. Figure 5(a) shows that, Liquid crystal nOBA exhibits the odd-even effect for Cr- SmC phase transition from n=7 to 10. But, the smectic phase stability of even (8OBA, 10OBA and 12OBA) and odd compounds (7OBA,9OBA) increases with increasing carbon chain

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length. Phase stability of compounds with odd carbon atoms (n=7,9) is in the range of (8ºC-31ºC) for even numbers it is in the range of (7ºC to 35ºC). As the alkoxy chain length increases from n=7 to 12, the nematic phase decreases and is in the range from (47ºC – 20ºC).

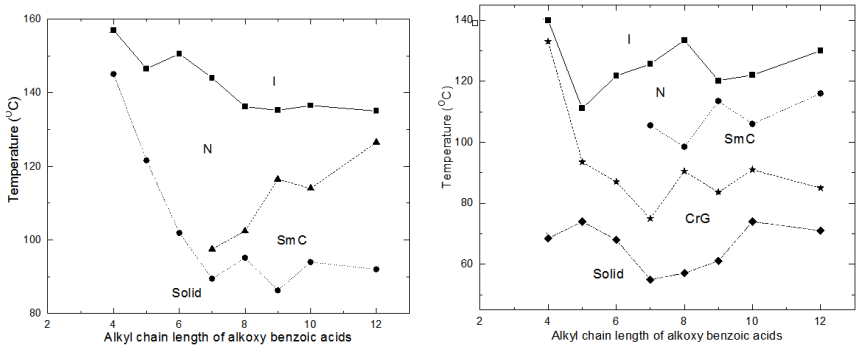

Fig. 5. Phase diagram of (a) nOBA; (b) equimolar mixtures of nOBA:EBCA, where (n= 4 to 10 and 12)

for odd number compounds (7OBA, 9OBA) and for even number compounds(8OBA, 10OBA, and 12OBA), it is in the range from (34ºC – 8ºC). More pronounced reduction is observed for compounds with the longer even-numbered chains. And also, compounds with odd number of carbon atoms exhibit the lower transition temperatures compared to even numbers. Similar to nOBA compounds, from Figure 1 (b) it was observed that transition to CrG – SmC phase exhibits odd-even effect, except n=12. The phase stability compounds with odd carbon atoms (n=7,9) is high (30ºC) compared to the even number complexes (8OBA:EBCA, 10OBA:EBCA) is in the range of (8ºC -15ºC). And also, the complexes n= 7 to 10 and 12 exhibits odd-even effect for the transition nematic – isotropic. Nematic phase stability for both even and odd complexes decreases with increasing alkoxy chain length and more pronounced decrement is observed for the even number complexes with increasing carbon chain length (34ºC to 14ºC) compared odd complexes (20ºC to 16ºC). The odd-even effect indicates the intra chain constraints on the tail-chain flexibility. Liquid crystal complexes *nOBA:EBCA* where (n=4 - 9) exhibits the weak odd even effect for Cr- CrG phase transition. The higher transition temperatures are seen in complexes with even numbers of carbon atoms. These results from the primary influence of the extended terminal chain length, which dilutes interactions among the larger mesogenic units as the volume fraction of the alkyl chains increases. Compared with the pure nOBA series, the synthesized compounds show lower clearing temperatures and an increased liquid-crystalline range. The increase in the induction of CrG phases with alkoxy chain length demonstrates the influence of non-polar tails on the stiffness of the cores. Specifically, the semi-flexible tail chains provide sufficient disorder to maintain the cores' ability to derive partial positional order from the isotropic phase, thereby leading to the CrG phase. Moreover, extending the terminal chain length significantly enhances microphase segregation, driven by enthalpic and entropic factors that favor the formation of smectic and soft-crystal phases. As a result of thermal motion, the terminal groups of nearby dimers converge, and H-bonds are rearranged into open structures, providing substantial stabilization in the solid state as well.

3.2.Fourier Transform Infrared spectroscopy (FTIR)

For the purpose of clarifying the structure of organic compounds involving intra- and intermolecular hydrogen interactions, infrared spectroscopy is a significant and potent approach. Since electromagnetic radiation excites both molecular rotations and vibrations, it can be used to describe the bonding relationship between molecules by producing absorption bands in the infrared spectra. The spectra show differences in the form of band shifts (positions) and band shapes that provide information about the bonding in liquid crystal complexes. Table 3 displays the underlying functional groups that the homologous series of pure (nOBA) and liquid crystal complexes (nOBA:EBCA, where n=4–10 and 12).

All compounds (except compound 6) containing pure p-n-alkoxy benzoic acids have a multiplet structure in the C=O vibrational band. This stake-shaped band is in the middle of the spectrum with frequencies around1670 cm^{-1} - 1694 cm^{-1} for the nOBA compounds.A small shift in the C=O stretching band from ≈1680 cm^{-1} (cyclic dimer) to ≈1672–

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1674 cm^{-1} and the appearance of a weak band near 1600–1620 cm^{-1} can be attributed to the perturbation of the carbonyl group due to interaction with the C≡N group of EBCA.In the freestate, the O-H vibrational band typically occurs at 3500 cm^{-1}. In H-bonding complexes, it is very broad band around 2500 cm^{-1} to 3100 $cm^{-1}$and broadening in the nOBA complex, indicating the presence of an additional N···H–O hydrogen bond alongside the existing cyclic dimers.The observations on pure compounds clearly indicate that they exist as dimers. In every compound under investigation, the C=O and (O-H) IPB vibrations are nearly constant, demonstrating the stability of the dimer structure. The vibrational band associated with CN stretching show a prominent band around 2223 cm^{-1}in pure EBCA. This band has a sharp, pointed shape. High polar nature of this CN triple bond exhibits the stronger bands.The C≡N stretching band of EBCA shows a noticeable shift to lower wavenumber (≈2210 cm^{-1}) in the complex spectrum, confirming interaction between proton acceptor groups (NC) and proton donor groups (O-H). H-bonding within the complexes may have contributed to the dimers of alkoxy compounds separating out as monomers in the complexes produced with EBCA. This demonstrates that during complexation, the p-n-alkoxy benzoic acid molecule exists in a monomeric form. The following altered frequencies (Table 1) shows that, vibrations involving the proton acceptor groups (CN) and donor groups (O-H) that shift their absorption frequencies towards lower frequencies are indicative of the development of hydrogen bonding in liquid crystal complexes.The stake shaped band corresponding to the C=O stretch mode of the benzoic acid moiety in complex systems shows hypsochromic shifts in their vibrational frequencies with high intensities. These strong and intense bands are responsible for the formation of hydrogen bond in nOBA: EBCA. Shifts in the (C=O) mode of benzoic acid moiety is in the range of 1680 cm^{-1} – 1692 cm^{-1}. The CN mode of EBCA in complex systems show the bathochromic shift in the vibrational frequencies (around 9cm^{-1}) with strong absorption intensities supports the formation of hydrogen bond in complex systems. Comparatively moderate bathochromic shift of 6 cm^{-1} is observed in the complex systems with shorter chain lengths (n=4). The bathochromic shift is in the range of 7 -9 cm^{-1} for complexes with medium chain lengths (n = 5 to 10 and 12), indicating higher hydrogen bonding in these complex systems.

From FTIR studies on nOBA:EBCA, (where n=4 - 10 and 12) complexes the bathochromic shift trend is 7-9 cm^{-1} and change with n is minimal. With the chain length n, even odd effect in complexes is also observed. and as observed just like in pure nOBA. And also, hypsochromic shift of 9cm^{-1} in the (C–O) mode of acid in KBr during complexation be used to infer the creation of intermolecular H-bonding between the COOH group of benzoic acid and the CN group of EBCA. In this instance, the stretching of the O-H group as a result of intermolecular H-bonding was established by the rather sharp vibrations that all of the complexes told around 3370 cm^{-1}. The formation of inter molecular hydrogen bonding between COOH group of nOBA and CN group of EBCA can be invoked not only on the basis of stretching vibrations but also different bending vibrations in the finger print regions that strongly supports the existence of H-bonding [20-25].

Table 3: **IR spectral observations of liquid crystal complexes nOBA, EBCA and nOBA: EBCA, where n=4 – 10 & 12.**

Compounds	$\bar{\nu}(C=O)_{acid}$ cm^{-1}	$\bar{\nu}(C-O)_{acid}$ cm^{-1}	$\bar{\nu}(C \equiv N)$ cm^{-1}	$\bar{\nu}(OH)_{acid}$ cm^{-1}
EBCA	--------		2223.52	--------
4OBA	1680	1290		
4OBA: EBCA	1685.66	1294.15	2217.83	3369.47
5OBA	1678.22	1290.14		
5OBA: EBCA	1680.15	1293.04	2215.81	3370
6OBA	1693.19	1294		
6OBA: EBCA	1692.0	1297.87	2214.23	3370
7OBA	1681.44	1306.54		
7OBA: EBCA	1686.92	1303.38	2214.10	3369.70
8OBA)	1691.72	1294.35	--------	
8OBA: EBCA	1691.72	1296.28	2216.41	3358.37
9OBA	1681.62	1278.57		
9OBA: EBCA	1686.44		2216.41	3370.96
10OBA	1680.08	1281.28		
10OBA: EBCA	1685.00	1300	2216	3370
12OBA	1670	1310		
12OBA: EBCA	1685.33	1306.6	2215.79	3367.32

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IV.CONCLUSION

Liquid crystalline behavior of the homologous series of *nOBA:EBCA* (where n=4 to 10 and 12) are studied successfully using thermal analysis and FTIR spectroscopic techniques and computational analysis. The intermolecular interactions yield the hydrogen bond formation between the terminal functional groups (O-H) of nOBA molecule and CN group of EBCA. Their molecular packing, or the regular arrangement of each molecule head-to-tail with neighboring EBCA molecules in the same layers and inducing crystal G phase for the entire homologous sequence nOBA:EBCA, is influenced by the resulting liquid crystalline complex system. The decrease trend of clearing temperatures is observed for the complexes compared to the pure systems. Polarizing optical microscopy observations of the phase transition temperatures of the nOBA: EBCA series of compounds and EBCA show a reasonable agreement with the DSC results.

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