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Induced Crystal G phase through Intermolecular Hydrogen bonding in *n*OBA: 9HB

S. Sreehari Sastry¹, C. Nageswara Rao¹, T. Vishwam², K. Mallika², B. Gowri Sankara Rao² and Ha Sie Tiong³

¹Department of Physics, Acharya Nagarjuna University, Nagarjunanagar, INDIA ²Gitam University-Hyderabad campus, Rudraram village, Patancheru (Mandal) Medak Dist, AP, INDIA

³Faculty of Science, University Tunku Abdul Rahman, Jalan University, Bandar Barat, Kampar, MALAYASIA

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Abstract

In this present communication, a novel homologous series of supra molecular liquid crystal complexes essentially with long axis is prepared through the formation of hydrogen bonding between mesogenic compounds p,n-alkoxybenzoic acid (nOBA, wheren= 5 to10) and nonmesogenic compound nonylp-hydroxy benzoate (9HB). The formation of hydrogen bonding is confirmed by Fourier Transform Infrared (FTIR) spectroscopy. Thermal and Phase behavior of all complexes are investigated by Polarizing Optical Microscope (POM) and Differential Scanning Calorimetry (DSC). Thermal analysis studies reveal-enantiotropic-crystal G phase with wide thermal span is induced for the entire homologous series of liquid crystal complexes by quenching the nematic phase (n = 5 to 10) and smectic C (n = 7 to 10) phase. Molecular structure of the liquid crystal complexes, in the solid phase was elucidated using powdered x-ray diffraction and proton nuclear magnetic resonance ¹HNMR. Two higher member mesogens of the present homologous series exhibit the monotropic crystal 2 phase. The average thermal stability, mesomorphic characteristics and structural behaviors are compared between present liquid crystal complexes of p,n-alkoxy benzoic acids and similar homologous series p,n-alkyl benzoic acids (nBA, where n = 5 to 10).

Keywords: Supra molecular liquid crystal complexes, Intermolecular hydrogen bonding, *p*, *n*-alkoxy benzoic acids, *p*,*n*-alkyl benzoic acids, mesomorphic characteristics.

Introduction

Non covalent interactions between mesogenic and non mesogenic materials are advantageously used for the design of new supramolecular materials and are a widely discussed topic¹⁻³. Formation of hydrogen bond through the non covalent interactions of molecules is a power tool for self assembling the molecules to form the liquid complexes with different molecular shapes and structures⁴⁻¹⁰. Preparation of these supra molecular compounds leads the generation of new phases and properties which are differ from the origin of the compounds due to its stability and directionality of the molecules¹¹⁻¹⁷. Interest in these hydrogen bonded materials arises from their low bond activation energies, possible commercial applications and also from the organic materials widening in applications. Among such supra molecular hydrogen bonded systems, preparation of liquid crystal complexes based on p,n- alkoxy benzoic acids (nOBA) compounds with other chemical moieties are used for various studies in different fields. Such systems are, *n*OBA with hexadecylaniline used in crystallization studies^{18,19}, *n*OBA with benzoate derivatives used in biological studies²⁰, Estrification of *n*OBA with hydroxyl propyl cellulose results the liquid crystal properties which are useful in electro optic applications²¹, nOBA separately with *p*-*n* alkyl benzoic acid, choloro/ hydroxyl benzaldehyde, succinic acid, levo tartaric acid are used in optical shuttering (optical shutters), light modulation (light modulators)²²⁻²⁵, and nOBA with various nOBAs

forms the complimentary hydrogen bonded systems are useful in light filtering etc^{23} . Moreover the presences of hydrogen bond in such liquid crystal complexes influence the performance of systems in terms of physical properties which are differ from the original compounds. Such physical properties: rate of crystallization¹⁸, melting and clearing temperatures, enthalpies, entropies, mesophse thermal stabilities, compress the formation of liquid crystal phase, quenching of phases and inducing new phases with wide and narrow thermal spans²⁶⁻³², field induced transitions²⁴, probing different textures, rich polymorphism^{25,33-35}, reentrant liquid crystal phases³⁶, dielectric measurements^{18, 22,37} and material toughness and processability²¹. Keeping view of the above, In this paper a novel homologous series of liquid crystal complexes are prepared through the development of hydrogen bond between COOH of liquid crystal compounds *p*,*n*-alkoxy benzoic acids when n = 5 to 10 and OH of non liquid crystalline compound nonyl-p-hydroxy benzoate (9HB). nOBAs with hydroxy benzoates acts as bioactive molecules and possess the liquid crystal properties are useful in biological, pharmaceutical and analytical field as well as homologous series expected to be of low temperature mesogenic materials which are useful in nonmedical fileds, LCD indicators, electronic screens etc. Therefore, a systematic investigation for the confirmation of hydrogen bonding is done by Fourier Transform Infrared (FTIR) spectroscopy and physical properties of the newly prepared systems is carried out using thermal studies (Polarizing Optical Microscope (POM), Differential Scanning Calorimetry (DSC)) and structural

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studies by powder X- ray diffraction, Proton Nuclear Magnetic Resonance (¹HNMR). A comparative study of thermal and structural behavior between complexes *n*OBA: 9HB (n = 5to 10) and p,n - alkyl benzoic acid: nonyl hydroxy benzoate (*n*BA: 9HB) where n = 5 to 10 reported in by salma Begums etal.³⁸.

Methodology

Experimental: *p,n*- alkoxy benzoic acid (nOBA) with 99% purity and nonyl-*p*-hydroxy benzoate (9HB) were supplied by Firnton laboratory, New Jersey, USA. Infrared spectra were recorded on an Alpha model BRUKER infrared spectrometer. The X-ray diffraction patterns of powder samples were taken from the Philips Diffractometer, type PW710 using CUK α radiation. ¹HNMR spectra of the complexes are also recorded on the BRUKER Avance 400mHZ instrument which was used for the structural studies. Differential scanning calorimetry (DSC) was performed on a DSC Q20 V24.2 Build 107 universal V4.5 TA instruments under nitrogen atmosphere at the heating / cooling rate of 5°c/min. A small quantity of sample is placed on a commercially available glass slide usually which will keep in the hot stage of the polarizing optical microscope (Meopta DRU 3 model) to observe the textures o the samples.

Canon EOS Digital REBEL XS/EOS 1000D is a digital single lens reflex camera with 10.10 mega pixel image sensor is used to record the texture images of the sample through the crossed polarizers of the POM. The image is having 3888x2592 pixel size depicting 24 bit tonal levels in a true color production.

Preparation of p,n - alkoxy benzoic acid (nOBA): nonyl p-hydroxybenzoate (9HB) (where n=5 to 10) complexes: Hydrogen bonded liquid crystalline complexes namely, nOBA: 9HB (where n=5 to 10) are prepared by the following procedure given in scheme 1. Required amount of samples for synthesis are ____ISSN 2320–4796 *Res. J. Physical Sci.*

weighed on a single pan electronic balance Dhona make, ER-180A with an accuracy of 0.01mg.Equimolar (1:1) ratio of *n*OBA and 9HB are used for the preparation of liquid crystal complexes *n*OBA: 9HB, n=5 to 10. Compounds are taken individually and mixed in the pyridine solvent (20ml). Now the two solutions are mixed and kept under constant stirring at 80^oC for 4hrs. Then most of the pyridine is removed by vacuum distillation process. It means 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%.

Fourier Transform Infrared Spectroscopy (FTIR): The hydrogen bonded nOBA: 9HB complexes are white crystalline substances and are stable at room temperature. All the complexes are soluble in chloroform, pyridine, ethyl alcohol and dichloromethane. Structural elucidation of these homologous series complexes which involves the non covalent intermolecular hydrogen bonding interactions are carried out using FTIR spectra. IR spectra of complexes nOBA : 9HB (n= 5-10) and 9HB were recorded at room temperature in solid KBR. Due to the excitation of both molecular vibrations and rotations, absorption of electromagnetic radiation causes the formation of absorption bands in the IR spectra which are useful to explain the bonding interaction of the molecules^{39,40}. The IR spectra of 9HB exhibit the strong characteristic absorptions bands at 1272. 50 cm⁻¹ for C - O stretching along with IPB, OPB modes of (OH)_{phenolic} at 1385.02 cm⁻¹, 618.02 cm⁻¹. nOBA : 9HB complexes show the absorption bands in the range of 3381.81 - 3446.08cm⁻¹ for $(OH)_{phenolic}$ stretching mode, strong intense bands due to C = Omode of benzoic acid moiety in the range of 1679.26-1684.71 cm^{-1} which strongly supports the existence of *n*OBA moieties in monomeric form upon complexation.



p,n - alkoxy benzoic acids (nOBA) + nonyl - p - hydroxy benzoate (9HB)

Pyridine, 4 hrs reflux (at 80° C)

And after distillation



Liquid crystal complex- *n*OBA: 9HB, Where $R = C_n H_{2n+1, and} n = 5$ to 10. Scheme-1 Preparation of hydrogen bonded liquid crystal complexes (*n*OBA: 9HB), where *n*=5 to 10

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The intermolecular hydrogen bonding is interpreted corresponding to functional groups in the nOBA: 9HB complexes where n = 5 to 10 of nOBA: 9HB reveal a bathochromic shift in the (C=O) acid $\simeq 5$ cm⁻¹ and (C-O) acid $\simeq 24$ cm⁻¹ associated with a large shift in the OH of the acid. The reduced nature of C = O stretching of complexes is due to the hydrogen bonding between the C= O of benzoate and the hydroxyl group. Further the hydrogen bonding is supported by the hypso chromic shift in the (C - O) benzoate is $\simeq 25$ cm⁻¹ in IPB and (OH) Benzoate is $\simeq 38$ cm⁻¹ and OPB of (OH) benzoate

is $\simeq 22$ cm⁻¹. The IR spectral analysis signifies that the strong intermolecular hydrogen bond formed between the COOH group of *n*OBA and OH group of 9HB and detailed IR spectral data of 9HB and complexes *n*OBA : 9HB where *n* = 5 to 10 are given in table-1. The vibrations involving the proton donating and accepting groups should show shift in their absorption frequencies towards the lower frequencies confirms the formation of hydrogen bonding in liquid crystal complexes^{41,42}. As s representative case the IR spectra of 9HB and liquid crystal complex 5OBA:9HB is shown in figure-1.



FTIR spectra of compounds (a) 9HB; (b) 5 OBA: 9HB

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As a comparative study between the $nBA:9HB^{38}$ and nOBA:9HB (n=5 to 10), the data given in table -1 reveals that in case of OH (IPB) there is a increased shift of 28 cm^{-1} for nBA:9HB and 38 cm^{-1} for nOBA:9HB. And also there is a increased shift of OH (OPB) 6 cm⁻¹ in nBA: 9HB and 22 cm^{-1} in nOBA:9HB. In case of (C - O) acceptor increased shift is 4 cm⁻¹ in nBA:9HB and 25 cm^{-1} in nOBA:9HB.

Thermal Studies: Phase and thermal studies of homologous series of the liquid crystal complexes nOBA: 9HB are determined from the POM and DSC. Instead of original nematic (n = 5 to 10) and smectic C phases (n = 7 to 10), the novel homologous series exhibits the smectic G phase. The formation of hydrogen bond between COOH of nOBA where n = 5 to10and OH of 9HB improve the molecular arrangement order and induce the occurrence of smectic G phase. Higher members of homologous series n = 8, 9 exhibits the monotropic crystal 2 phase and was confirmed by the DSC. Crystal G phase of the complexes and crystal 2 phase of the 80BA:9HB, 90BA: 9HB are shown in figure-2. Quenching of the original phases (nematic, smectic C) and induction of new phases (smectic G) with wide thermal spans for the series of nOBA: 9HB complexes are attributed to the formation of hydrogen bond through non covalent interaction of the molecules. All these

mesogens are melting at temperature around 110° C. Thermal stability of liquid crystallinity exists for the homologous series: *n*OBA: 9HB compounds are in the range of about (28–61°C) which is relatively high when compared to the individual compounds (25–58°C). The benzoate family at the terminal of the alkoxy series attributed to the formation of crystal G phase and adversely affects the mesophase thermal stability by decreasing the transition temperatures low enough to maintain layered arrangement.

Phase transition temperatures and enthalpy values of the liquid crystal complexes *n*OBA: 9HB are evaluated with DSC measurements. Temperatures corresponds to the heat flow peaks in the DSC thermogram consistent with the occurrence of Cr – smectic G – Isotropic transitions and vice versa obtained from the POM studies with $\leq 5^{0}$ C variation. Transition temperatures and corresponding enthalpy values of liquid crystal complexes are shown in table-2. From table-2, it is clear that enthalpy values of the complexes are increased with increasing their chain length. As a representative case DSC thermogram of the compound 80BA: 9HB and 90BA: 9HB are shown in figure - 3. In some systems the peaks corresponds to the liquid crystal – crystal phase are not well resolved.

Table–1		
Solid State IR spectra data of NHB, <i>n</i> BA: 9HB and <i>n</i> OBA:9HB (<i>n</i> =5 to10)		

Comment			OII domon	OH acceptor		(\mathbf{C},\mathbf{O}) = = = = \mathbf{O}
Compound	C=O donar	C.O donor	OH donor	IPB	OPB	(C.O) acceptor
9HB				1385.02	618.02	1272.50
5BA 9HB	1680	1278	3380	1396	638	1278
50BA 9HB	1679.26	1261.47	3383.79	1429.8	644.8	1261.47
6BA 9HB	1679	1277	3379	1397	638	1277
60BA 9HB	1682.79	1280.80	3382.87	1465.26	638.8	1280.50
7BA 9HB	1681	1278	3380	1424	637	1278
70BA 9HB	1681.32	1258.39	3381.81	1429.38	643.23	1258.39
8BA 9HB	1680	1279	3380	1423	639	1279
80BA 9HB	1684.33	1255.26	3381.98	1427.90	624.93	1255.76
9BA 9HB	1681	1279	3380	1424	633	1279
90BA 9HB	1681.78	1257.71	3392.71	1431.18	645.83	1257.71
10BA 9HB	1680	1281	3381	1423	639	1281
100BA 9HB	1684.71	1256.07	3446.08	1429.64	646.42	1256.09



Figure-2 (a) Crystal G phase of *n*OBA: 9HB; (b) Crystal 2 phase of 8OBA:9HB at temperature 43^oC on heating ; (c) Crystal 2 phase of 9OBA:9HB at temperature 60^oC on cooling

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A phase diagram is drawn based on the data obtained from the POM and DSC which is shown in table - 2. The homologous series of liquid crystal complexes *n*OBA:9 HB exhibits the liquid crystal stability $\leq 110^{\circ}$ C. The observed slope changes in the phase diagram curves corresponds to the transitions are found that, the temperatures obtained from the DSC are in good agreement with the temperatures obtained from POM with

variation of $\leq 5^{0}$ C. From figure-4, it is clear that liquid crystal complexes *n*OBA: 9HB and individual compounds (*n*OBA) exhibits the weak even – odd effect at the crystal G – isotropic phase transition. The compounds with even number of carbon atoms exhibit the high transition temperatures (Crystal G–I) corresponding odd number in the series.

Phase	I ransition temperatures	(C) of $nOBA$; $nOBA$: 91	1B (<i>n</i> =5 to 10) ; P	OM (DSC)
Compound	Phase	Phase transition temperature POM (DSC)		
		Cr-N	Cr-N	N to I
50BA	Ν	121.6(113.6)	-	146.5(150.4)
60BA	Ν	101.9 (106.8)	-	150.5(148.8)
70BA	NC	89.5(0*)	97.5(95.9)	144(147.8)
80BA	NC	95.1(91.1)	102.4(100.9)	136.2(137.1)
90BA	NC	86.3(88.9)	116.5(0*)	135.2(141.3)
100BA	NC	94(91.9)	114(116.6)	136.5(141.8)
Compound	Phase	Phase Transition	on Temperature P	OM (DSC (?HJ/g))
		Cr. to Cryst	al G	Cr. to Crystal G
50BA: 9HB	Crystal G	44.3(41.95(7.8	893))	105.8(110.05(2.629))
60BA: 9HB	Crystal G	72.3(72.12(12	07))	101 (103.83(6.583))
70BA: 9HB	Crystal G	42.1(43.03(21	.46))	88.2(91.29(8.052))
80BA: 9HB	Crystal G	38.3(41.61(27	7.04)	96.2(99.03(6.310))
90BA: 9HB	Crystal G	44.2(44.46(29	9.54)	90.2(93.28(26.94))
100BA: 9HB	Crystal G	87.8(86.05(70.51))		93.5(97.05(38.47))

Table-2	
Phase Transition temperatures (⁰ C) of <i>n</i> OBA; <i>n</i> OBA : 9HB (<i>n</i> =5 to 10) ; POM (D	SC

 $Cr - Crystal, C - Smectic C, N - Nematic, I - Isotropic, (0*) - peaks are not well resolved? Indicates \Delta$



Figure-4 Phase diagrams for (a) *n*OBA; (b) *n*OBA: 9HB on heating process

Phase diagrams shown in figure-4 (b), 5 reflects upon the features of the odd even effect in transition temperatures which were used for the comparative thermal study of the *n*OBA: 9HB and *n*BA : 9HB³⁸. Transition temperatures of the complexes are given in table - 3. Homologous series of both complexes quenches the original phase of individual compounds (*n*BA, *n*OBA) and induces the crystal G phase. *n*BA: 9HB mesogens are melted at temperature around $\leq 90^{\circ}$ C which is less than *n*BA:9HB ($\leq 110^{\circ}$ C). Liquid crystal phase existing temperature range for *n*BA: 9HB where *n* = 5 to10 complexes is 44 – 90°C which is less than the *n*OBA: 9HB in the range from 38–106°C and hence *n*OBA:9HB complexes are more stable than *n*BA:9HB complexes. The relative mesophase length of *n*BA:

9HB varies in the range of about $(23-40^{\circ}\text{C})$ which was small compared to the *n*OBA: 9HB is in the range of about $(28-61^{\circ}\text{C})$ except for 10OBA: 9HB. This can be attributed to the polarity and polarizability of the molecules in the complexes⁴³.

*n*BA: 9HB complexes exhibit the strong even – odd effect at the crystal – crystal G transition. Complexes with even no.of carbon atoms exhibit the high transition temperatures for Cr – Crystal G tansition. But in *n*OBA: 9HB complexes, weak even – odd effect is observed for crystal G – isotropic phase transition. The compounds with even number of carbon atoms exhibit the high transition temperatures (Crystal G–I) corresponding odd number in the series.

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Phase Transition temperatures of nBA: 9HB [38] (n=5 to 10); POM (DSC)				
Compound	Phase	Cr-Crystal G	Crystal G to I	
5BA: 9HB	Crystal G	44.3 (37.98)	68.2 (66.4)	
6BA: 9HB	Crystal G	53.6 (46.20)	79.3 (77.9)	
7BA: 9HB	Crystal G	50.8 (55.79)	83.1(80.9)	
8BA: 9HB	Crystal G	52.6 (47.66)	89.7 (87.5)	
9BA: 9HB	Crystal G	49.2 (46.60)	88.9 (85.5)	
10BA: 9HB	Crystal G	54.2 (50.68)	85.2 (83.4)	

Table-3
hase Transition temperatures of nBA: 9HB [38] (n=5 to 10); POM (DSC)

Cr – Crystal, C – Smectic C, N – Nematic, I - Isotropic



Figure-5 Phase diagram for *n*BA: 9HB on heating process

Table-4 Xrd data for 9HB, nBA: 9HB and nOBA: 9HB				
Compound	Intensity Counts	Bragg Angle (20)	FWHM In radians	Crystallite size (t) in A.U.
5BA 9HB	99	8.96	0.03142	46.23
50BA 9HB	587.7	5.691	0.01744	86.81
6BA 9HB	860	20.60	0.07298	20.16
60BA 9HB	793.3	5.737	0.0137	110.72
7BA 9HB	891	9.24	0.06477	22.43
70BA 9HB	874.5	5.441	0.01509	105.08
8BA 9HB	1796	9.22	0.02619	55.47
80BA 9HB	575.5	5.636	0.01470	103.86
9BA 9HB	2866	48.138	0.01833	86.53
90BA 9HB	934	5.676	0.00935	162.17
10BA 9HB	36.49	10.033	0.02304	63.09
100BA 9HB	1133.5	8.340	0.00399	702.4

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X-ray Diffraction Studies: Powder X-ray diffraction patterns of homologous series of liquid crystal complexes *n*OBA: 9HB (n = 5to10) were recorded at room temperature and as a representative case 5OBA:9HB is shown in figure-6. X-ray diffraction analysis shows that the molecules in the liquid crystal are arranged triclinically. The parameter values obtained from the analysis are used to calculate the crystallite size of liquid crystal complexes from the Scherrer equation⁴⁴. Results obtained from the X- ray diffraction analysis are listed in table - 4.

The data given in table-4 reveals that the crystallite size is more for nOBA: 9HB than nBA:9HB. Interplanar spacing of nOBA: 9HB complexes is more compared to the nBA: 9HB complexes as the bragg angle is increased.

¹**HNMR studies for structural confirmation:** The proton Nuclear Magnetic resonance ¹HNMR confirms the structural studies of liquid crystal complexes, *n*OBA: 9HB where *n*= 5 to 10. The peaks at 1.2 to 1.8 multiplets were attributed to $(CH_2)_n$ of both groups. The chemical shift δ ppm at 0.9 triplets is attributed to CH₃. The peaks at 6.8-6.9 ppm quadralet contribute to aromatic proton (OH) of *n*OBA and the peak at 8 ppm for aromatic proton (OH) of 9HB. The results obtained from ¹HNMR spectra of complexes *n*OBA: 9HB confirms the formation of Hydrogen bond. The ¹HNMR spectrum ^{39,40, 45} of the compound 5OBA:9HB is shown in figure-6.



¹HNMR spectra of 5OBA:9HB

When the results of *n*OBA:9HB (n=5 to 10) are compared with *n*BA:9HB (n=5 to 10)³⁸ it is observed that in both the cases the Chemical shift at 0.9 triplet is attributed to CH₃ and peaks at 4.3 ppm indicate the OH of bonded complexes.

From the above studies, the formation of hydrogen bond through intermolecular liquid crystal complexes has profound impact on thermal properties such as lowering transition temperatures, wide thermal span of liquid crystal range, and mesomorphic behaviour like quenching of nematic phase and inducement of Crystal G phase. Spectral studies FTIR, ¹HNMR and powder X-ray diffraction studies confirm the formation of hydrogen bond between the compounds (*n*OBA, 9HB) and molecular structure in the solid phase.

Conclusion

The attempt of designing the novel homologous series of thermotropic liquid crystal complexes nOBA: 9HB through the formation of hydrogen bond between nOBA and 9HB is successful. Clearing point of entire series is reduced compared to the pure compounds (*n*OBA) with quenching the original nematic and smectic C phase. Crystal G phase is observed for the series of complexes while monotropic crystal 2 phase is observed for the higher members of the series (n = 8,9) and was confirmed by the DSC studies. Thermal stability of the series is less compared to the pure compounds and is high for the similar complexes (*n*BA: 9HB) which can be attributed to the high polarity and polarizability of the molecules.

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