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RESEARCH ARTICLE

SYNTHESIS AND STUDY OF HOMOLOGOUS SERIES CONTAINING ETHYL SPACER MESOMORPHIC ALONG WITH NTB PHASE COMPOUNDS

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ABSTRACT

According to molecular flexibility, new ethylspacer containing benzoates RO-C₆H₄-COO-CH₂-CH₂-O-C₆H₄-O-CH₂-C₆H₅ were synthesized. As ethylspacer provides good flexibility, this series shows polymorphism. We are introducing an easy one-pot reaction for the esterification of an aliphatic alcohol with the help of isobutyl chloroformate, reaction time and yield as well as liquid crystalline properties of synthesized compounds were studied. As compared to the reported series, the current series is polymesomorphic in nature with a lower transition temperature. Introducing a spacer between two rigid cores enhance the mesomorphic behavior to show N_{tb} mesophase. The setexture and transition temperatures were carried out in temperature controlling polarizing optical microscope. All homologues were confirmed by ¹H NMR, Mass spectrometry and IR spectroscopic technique.

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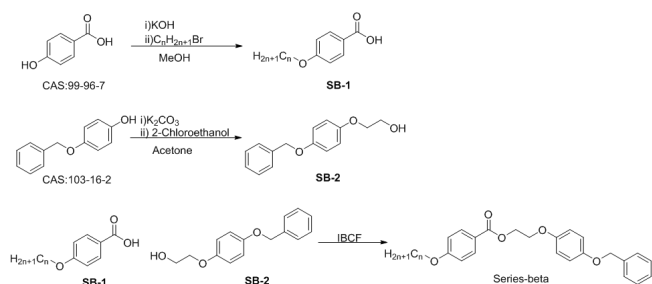
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INTRODUCTION

Since earlier times (1) the study of liquid crystal is very useful for a human being in all ways either in the research field or in daily life in different electronic gadgets (2-6). In current research work flexibility of ethyl-spacer is used to design slight bent shape molecule, albeit flexibility is the most important factor for the mesomorphic property. The majority of the liquid crystalline compound was synthesized by using a terminal alkyl chain, or any other group to utilise the flexibility of the alkyl chain to gain more type of mesomorphic properties or polymesomorphism. In contrast, researchers use a different spacer between two rigid cores to make the molecule flexible albeit this type of spacer provides a quite different type of orientation to the molecule. They show nematic mesophase cause of spacer they align at a particular angle. This type of nematic phase also called the twist-bend nematic phase (N_{TB}) (7-12). This phase was first time described by Dozovin 2001(13) and currently, it is a very striking area in the research of liquid crystals.

Correlation between structure and mesophase plays most important role in the formation of Ntb phase. Many researchers have reported that molecules which have asymmetric structure and polar or nonpolar lateral groups with alkyl terminal chain shows Ntb phase. The parity spacer used in molecule designing provide bent shape which give rise to formation of the twist bent nematic phase. It has been reported that long chain used between to rigid core as spacer it lowers the probability of the formation of twist bent nematic mesophase that can be hidden by smectic blanket (14-18). Here the length of the spacer between two rigid cores is decreased than earlier reported molecules and expected that it will enhance the polymorphism. Different homologues of 2-(4-(benzyloxy)phenoxy)ethyl 4-(alkoxy)benzoates were synthesized and their mesomorphic properties were studied. C₁ to C₅ homologues are non-mesomorphic while the rest of the homologues are polymesomorphic. Polymesomorphic homologues show the smectic mesophase below 100 °C while nematic mesophase is observed above 100 °C except for C₈ and C₁₂. C₈ shows the nematic phase at 97.3 °C while C₁₂ shows at 88.5 °C.



Scheme 1. Reaction scheme for synthesized series

Table 1. Elemental analysis of C₁, C₃, & C₅ homologues

Sr. No.	Molecular formula	Element % Found			Element % calculated		
		C	H	O	C	H	O
1	C ₂₃ H ₂₂ O ₅	72.89	5.77	21.34	73.00	5.86	21.14
2	C ₂₅ H ₂₆ O ₅	73.71	6.35	19.94	73.87	6.45	19.68
3	C ₂₇ H ₃₀ O ₅	74.62	6.91	18.47	74.63	6.96	18.41

Table 2. Transition temperature in °C

Compound No.	n-Alkyl chain C _n H _{2n+1} (n)	Transition Temperature in °C (Series 1)			
		Sm	Ntb	N	I
1	C ₁	-	-	-	186.5
2	C ₂	-	-	-	201.5
3	C ₃	-	-	-	124.6
4	C ₄	-	-	-	86.4
5	C ₅	-	-	-	76.1
6	C ₆	72.2	102.5	105.4	146.2
7	C ₇	93.2	-	101.2	110.3
8	C ₈	74.1	-	95.0	99.5
9	C ₁₀	88.9	-	111.9	116.6
10	C ₁₂	82.7	-	94.4	106.5
11	C ₁₄	98.2	-	115.5	130.6

Experimental

Synthesis Procedure: General Procedure for Synthesis of 4-Alkoxy benzoic acid (SB-1) As shown in scheme 4-alkoxy benzoic acids (SB-1) were prepared by the well-known method Dave and Vora (19). In 250 mL single neck round bottom flask 4-hydroxy benzoic acid (1.0 eq.) and n-alkyl bromide (1.2 eq.) was charged with MeOH (20 v) and 10% w/v methanolic KOH was added and allowed to stir at reflux for 12 hr. After completion of reaction monitored by TLC.

The reaction was poured into ice-cold water and pH was neutralized with dil. HCl, the white solid was precipitated out, filtered and washed with cold water and dried to give crude product. The crude was purified by crystallization using MeOH to yield 75-80 % of n-alkoxy benzoic acid (SB-1).

Synthesis of 2-(4-(benzyloxy) phenoxy) ethan-1-ol (SB-2): In 250 mL single neck round bottom flask 4-benzyloxy phenol (1.0 eq.) was charged with acetone and K₂CO₃ (1.5 eq.), 2-chloroethanol (1.2 eq.) was added and the reaction was allowed to stir at reflux temperature for 12 hr. After completion of reaction indicated by TLC. The reaction was quenched with water and extracted with diethyl ether. The organic layer was washed with 10 % NaOH solution, dried over Na₂SO₄ and concentrated to the crude product which was purified by crystallization using Ethanol and dried to yield 95 % of SB-2 (20).

General Procedure for the Synthesis of 2-(4-(benzyloxy) phenoxy) ethyl 4-(Alkoxy) benzoates: In 25 mL single neck round bottom flask 4-Alkoxy benzoic acid (SB-1) (1.0 eq.) and 2-(4-(benzyloxy) phenoxy) ethan-1-ol (SB-2) (1.0 eq.) was charged with IBCF (Isobutyl chloroformate) (5 v) and allowed to stir at 80 °C for 3 hr. After completion of reaction indicated by TLC.

The reaction was quenched with a bicarbonate solution and extracted with diethyl ether. The organic layer was dried over Na₂SO₄ and concentrated to give a solid crude product. The crude was purified by crystallization using ethanol and dried to yield 90-95 % of 2-(4-(benzyloxy) phenoxy) ethyl 4-(Alkoxy) benzoates (21)

Characterisation: The few homologues of synthesized series were characterised by elemental analysis (Table 1), Infrared spectroscopy, ¹H NMR spectra, DSC and Mass spectrometry. Micro-analysis was performed on EuroEA Elemental Analyzer. IR spectra were recorded on Shimadzu FTIR Model-IRAffinity-1S (MIRacle 10), ¹H NMR spectra were recorded on Bruker spectrometer using CDCl₃ as a solvent and mass spectra were recorded on Shimadzu GC-MS Model No. QP-2010.

Analytical data: ¹H NMR (CDCl₃) in ppm of octyloxy derivative 0.87-0.91 (t, 3H, -O-CH₂-(CH₂-CH₂)₃-CH₃, J=8 Hz) 1.25-1.35 (m, 8H, -O-CH₂-(CH₂)₂-(CH₂)₄-CH₃) 1.45-1.48 (m, 2H, -O-CH₂-CH₂-CH₂-(CH₂)₄-CH₃) 1.79-1.83 (m, 2H, -O-CH₂-CH₂-CH₂-(CH₂)₄-CH₃) 4.01-4.04 (t, 2H, -O-CH₂-CH₂-CH₂-(CH₂)₄-CH₃, J=6.8 Hz) 4.34-4.39 (t, 2H, -O-CH₂-CH₂-O-, J=4 Hz) 4.75-4.79 (t, 2H, -O-CH₂-CH₂-O-, J=5.6 Hz) 5.06 (s, 2H, -O-CH₂-Ar) 6.94-7.01 (m, 4H, -CH₂-O-Ar-O-CH₂-) 7.09-7.12 (m, 2H, -O-Ar-COO-CH₂-) 7.33-7.44 (m, 5H, -O-CH₂-Ar) 7.11-7.13 (dd, 2H, -O-Ar-COO-CH₂-, J=8 Hz).

¹H NMR (CDCl₃) in ppm of butyloxy derivative: 0.90-1.05 (t, 3H, -O-CH₂-CH₂-CH₂-CH₃, J=7.2 Hz) 1.39-1.44 (m, 2H, -O-CH₂-CH₂-CH₂-CH₃) 1.68-1.74 (m, 2H, -O-CH₂-CH₂-CH₂-CH₃) 4.00-4.03 (t, 2H, -O-CH₂-CH₂-CH₂-CH₃, J=6.4 Hz) 4.26-5.25 (t, 2H, -O-CH₂-CH₂-O-, J=4.8 Hz) 4.62-4.60 (t, 2H, -O-CH₂-CH₂-O-, J=5.2 Hz) 5.14 (s, 2H, -O-CH₂-Ar) 6.84-6.93 (m, 2H+4H, -O-Ar-COO-O-Ar-O-) 7.99-8.01 (dd, 2H, Ar-COO-, J=7.2 Hz), 7.32-7.44 (m, 5H, -O-CH₂-Ar).

¹³C NMR (CDCl₃) in ppm of butyloxy derivative: 14.2 (O-CH₂-CH₂-CH₂-CH₃) 18.8 (O-CH₂-CH₂-CH₂-CH₃) 31.9 (O-CH₂-CH₂-CH₂-CH₃) 68.1-69.7 (O-CH₂) 115.1-116.1 (O-Ar) 121.8 (CO-Ar) 127.7-130.1 (aromatic C) 139.1 (CH₂-aromatic C) 154.2, 160.6 (O-Aromatic C) 161.6 (C=O).

¹³C NMR (CDCl₃) in ppm of octyloxy derivative: 14.1 (O-CH₂-(CH₂-CH₂)₃-CH₃) 18.8-31.9 (O-CH₂-(CH₂-CH₂)₃-CH₃) 68.1-69.6 (O-CH₂) 115.1-116.1 (O-Ar) 121.9 (CO-aromatic C) 127.7-130.1 (aromatic C) 139.1 (CH₂-aromatic C) 153.4, 160.8 (O-Aromatic C) 161.6 (C=O).

IR Spectrum in cm⁻¹ of octyloxy derivative: 3063.06 (=C-H aromatic Stretching) 2924.18 & 2854.74 (C-H Alkane Stretching) 1728.28 (C=O str. Of Ester) 1512.24 (C=C aromatic bending) 1465.95 (bending for -CH₂ of alkyl chain) 1381.08 (bending for -CH₃ of alkyl chain) 1249.91 (C-O-C ether stretching) 848.71 (p-substitution aromatic ring) 740.69 & 763.84 (aromatic -C-H bending).


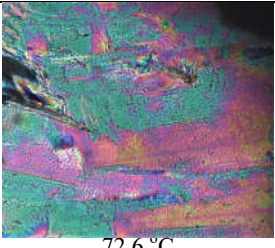
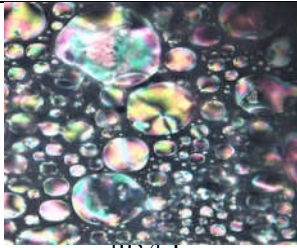
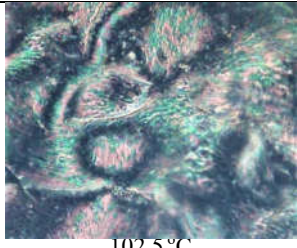
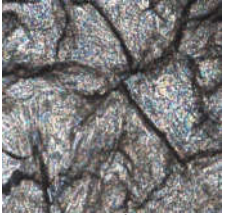
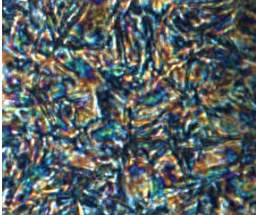
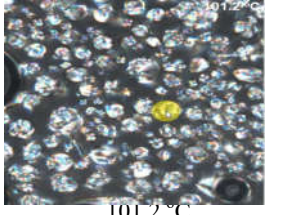

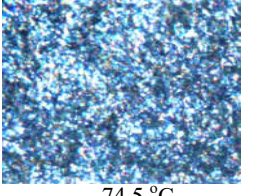
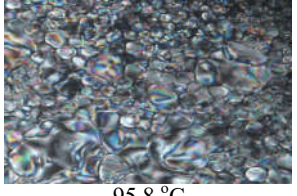
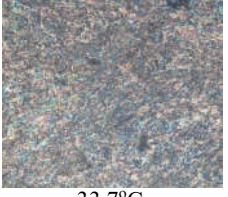
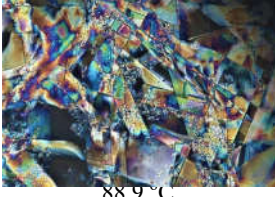
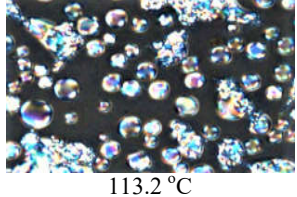

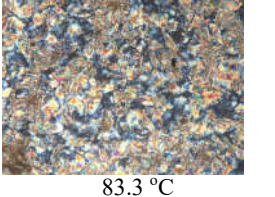
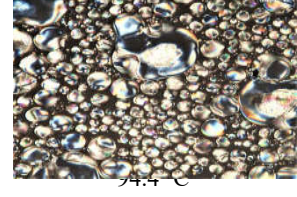
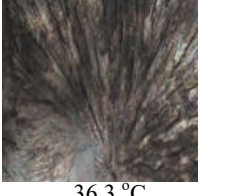
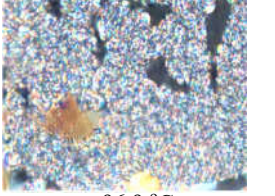
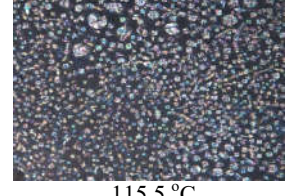
Table 3. Relative thermal stability in °C

Series→	Series 1	Series A
Sm-I or Sm-N	103.3	101.5
Commencement of smectic phase	(C ₆ -C ₁₄) C ₆	(C ₂ -C ₁₈) C ₈
N-I	118.28	-
Commencement of nematic phase	(C ₆ -C ₁₄) C ₆	-
Total mesophase length in °C minimum to maximum	5.0 °C to 74.0 °C	7 to 15 °C

Table 4. Transition enthalpy and entropy change for C₈ and C₁₂ homologues

Compound	Transition	Peak Temp. in °C	$\Delta H/ J g^{-1}$	$\Delta S/ J g^{-1} K^{-1}$	Phase length
C ₈	Cr-SmC	74.19	3.30	0.0095	Sm-20.85
	SmC-N	95.04	33.59	0.0912	N-4.5
C ₁₂	Cr-SmC	82.79	15.61	0.0438	Sm-11.71
	SmC-N	94.41	3.33	0.0090	N-12.1

Table 5. Mesomorphic texture with transition temperature

Homologues	Texture with transition temperature			
	Crystalline	Smectic	Nematic	N _{tb}
C6	 35.0 °C	 72.6 °C	 105.4 °C	 102.5 °C
C7	 33.8 °C	 92.8 °C	 101.2 °C	--
C8	 35.2 °C	 74.5 °C	 95.8 °C	--
C10	 33.7 °C	 88.9 °C	 113.2 °C	--
C12	 31.9 °C	 83.3 °C	 94.4 °C	--
C14	 36.3 °C	 96.9 °C	 115.5 °C	--

IR Spectrum in cm^{-1} of dodecyloxy derivative: 3073.01 (=C-H aromatic Stretching) 2992.18 & 2854.74(-C-H Alkane Stretching) 1720.56 (-C=O str. Of Ester) 1512.24& 1604.21 (C=C aromatic bending) 1465.95& 1427.37 (bending for -CH₂ of alkyl chain) 1378.82(bending for -CH₃ of alkyl chain) 1257.63(C-O-C ether stretching) 848.71(p-substitution aromatic ring).771.55(aromatic -C-H bending).

Mass Spectrum of hexyloxy derivative

m/z (int of %) 448.1(0.05% M⁺) 205.0 (1.0% ester linkage break) 249.1(7.0%) 138.0(100%)

Mass Spectrum of Heptyloxy derivative

m/z (int of %) 461.8(0.04% M⁺) 219.0 (100% ester linkage break) 121.1 (78%) 91.0 (82.0%)

RESULTS AND DISCUSSION

4-Alkoxy benzoic acid and 4-benzyloxy phenol are non-mesomorphic, but in current work both are linked with ester linkage with -O-CH₂-CH₂-O- spacer which provides good flexibility to the molecule and due to this linkage among all homologues C₆ to C₈, C₁₀, C₁₂ and C₁₄ homologues are poly mesomorphic. All transition temperature (Table 2) were carried out in a polarizing optical microscope with a hot plate. The phase diagram was plotted by the transition temperature in °C against the number of carbon atom present in the alkyl chain, denoted by 'n' in C_nH_{2n+1}. The transition curve Cr-Sm/I, Sm-N, and N-I were obtained by linking with related transition dots as shown in the phase diagram (Fig 1) which shows the phase behaviour of synthesized novel series. Cr-Sm/I transition curve of novel homologue series RO-C₆H₄-COO-CH₂-CH₂-O-C₆H₄-O-CH₂-C₆H₅ adopts the partially zigzag path of rising and falling as in descending manner as series is ascended.

Sm-N curve initially move downwards and then rise slowly and passes through maxima at C₁₀ homologue and then follows thenormal manner as expected. rising at C₁₄ homologue make it wave-like shape. Odd even effect was not observed and the thermal stability for the smectogenic behaviour is 103.3 °C following by 33.2 °C and 2.8 °C of maximum and minimum phase length respectively. While for the nematogenic behaviour thermal stability is 118.2 °C following by 40.8°C and 2.2°C is the maximum and minimum mesophase length respectively (Table-3). Total mesophase length (Sm+N) is 74.0 °C and 5.0 °C of maximum (C₆) and minimum (C₈) respectively. According to Dozov(13), all uniform bend shaped molecules must be accompanied by other deformations of local director and give rise to twist bend nematic phase. As we design a slight bent-shaped molecule by adding a spacer of two carbons linking with benzoate. This linkage favours the molecule alignment in a bent shape. So here in the current work, we show texture of the Ntb phase by the comparison (Fig-2) of previously published(22) texture of Ntb and other references which suggest that it shows rope-like texture.

C₆ homologue shows smectic phase at 72.2 °C and nematic phase at 105.4 °C while Ntb was observed at 102.5°C. In series-1, 11 homologues are synthesized, C₆ possess the Ntb phase (Table-6) while C₇, C₈, C₁₀, C₁₂, and C₁₄, are enantiotropically smectogenic as well as nematogenic (Table-5). As the length

of alkyl chain increases terminal attraction also increase and molecular alignment switch towards pure nematic texture, so N_{tb} texture disappears with the ascending of series. All six homologue shows smectic texture below 100 °C while nematic observed above 100 °C. mesophase lengths for the smectic texture are in the range of 2.8 – 33.2°C. A series is ascended phase length decreases for smectogenic behaviour. The same pattern also follows for nematogenic behaviour.

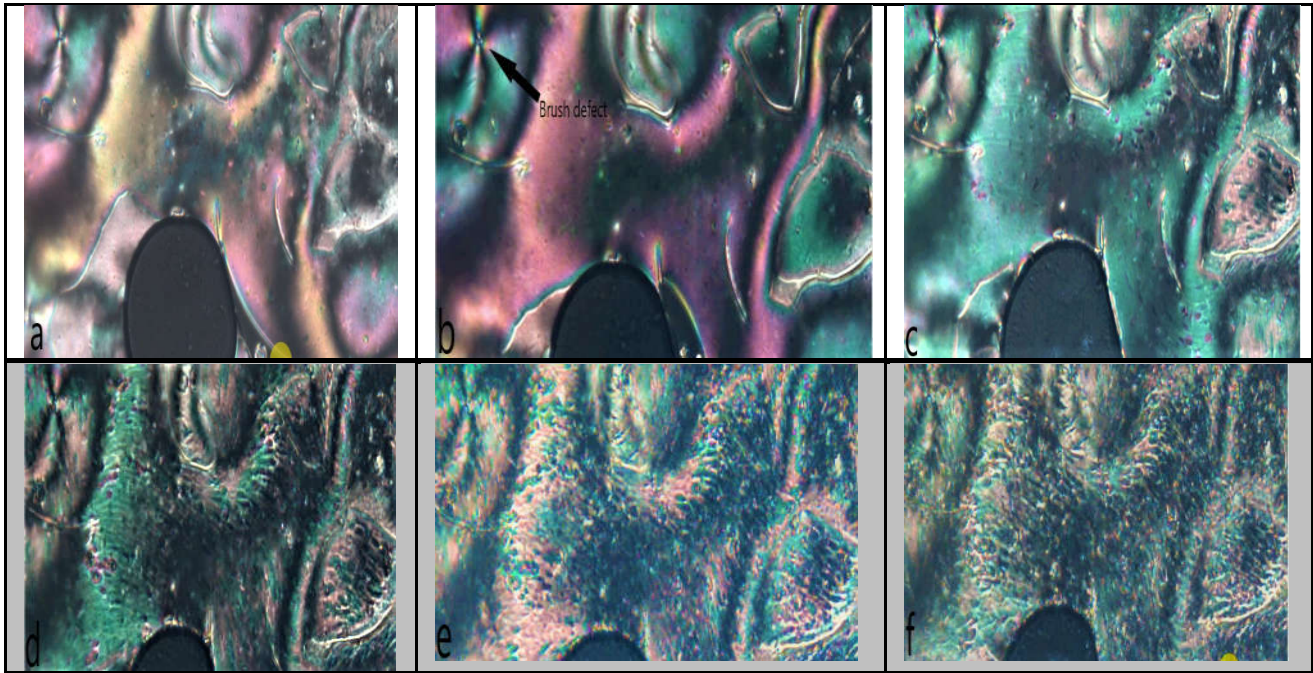
The difference in mesogenic properties is due to the factor of molecular polarizability, as the length of the alkyl chain or terminal chain increases overall polarizability also enhanced with ascending of series. The terminal attraction is increased as series is ascended and molecule under this condition will comply factually preserve layered structure with the partially fluid condition. This condition gives rise to the smectic texture but as the temperature increases smectic texture or preserved layered texture's strength decreases due to thermal vibration increases and molecules alignments move towards more fluidity, albeit they do not gain random arrangement at accurate temperature, smectic texture convert into nematic orientation. Albeit end to end parallel, orientation is preserved by this way all homologues reveal polymorphism at a definite temperature. The bubble type nematic and smectic C texture were judged by the help of the POM heating top by observing the same specimen. C₁-C₅ was non-mesomorphic due to the low diploidal interaction as well as lower magnitudes of dispersion forces also.

The present novel series is compared with almost structurally similar non-isomeric analogues series to evaluate the difference of liquid crystal properties by changing molecular structure concerning group efficiency order and polarizability for the same homologous series. Figure 3 shows the comparison between synthesized homologous series 1 and structurally most similar series X (23). Synthesised series have three phenyl rings same as reported series 1 but it possesses extra ether linkage. this link provides good flexibility to the molecule and helps to align at a particular angle to show poly mesomorphism. By comparing both the series following things are clear.

- Comparative series X is only smectogenic while series 1 shows poly mesomorphism.
- Commencement of smectogenic mesophase is at C₈ homologue (series X) in contrast series 1 commence smectogenic mesophase earlier at C₆ homologue.
- The nematic phase commences at C₆ homologues in series 1 while series X is completely non-nematogenic.
- The thermal stability of series 1 for the smectogenic texture is also higher. i.e., Series 1 > Series X
- Mesophase length for maximum and minimum for smectic texture is For maximum Series 1 > Series X and minimum Series X > Series 1

The difference in molecular rigidity and molecular flexibility causes a difference in the suitable magnitudes of anisotropic forces of intermolecular attraction, which are liable in mesogenic behaviour from series to series, here we give little change in structure and observe the major difference in mesomorphic behaviour of series. Generally, the physical properties of a molecule are depending on the intermolecular or intramolecular attractions, molecular flexibility, rigidity and polarizability of the molecule.

Table-6 POM texture of Ntb and Nematic mesophase.



a) The nematic texture at 110 °C b) The nematic texture at 108.5 °C c) The initiation of Ntb and nematic phase at 107.4 °C d) The Ntb phase at 104.9 °C e) The Ntb phase at 102.7 °C f) The Ntb phase at 101.1 °C

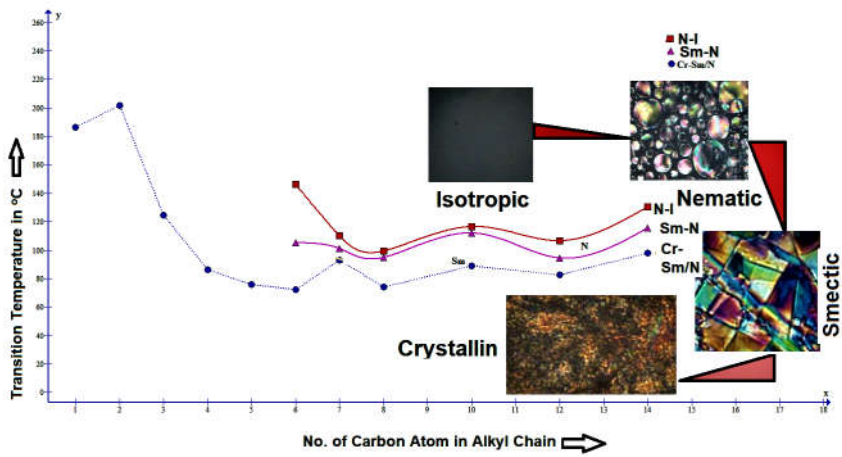


Fig 1. Phase diagram of series

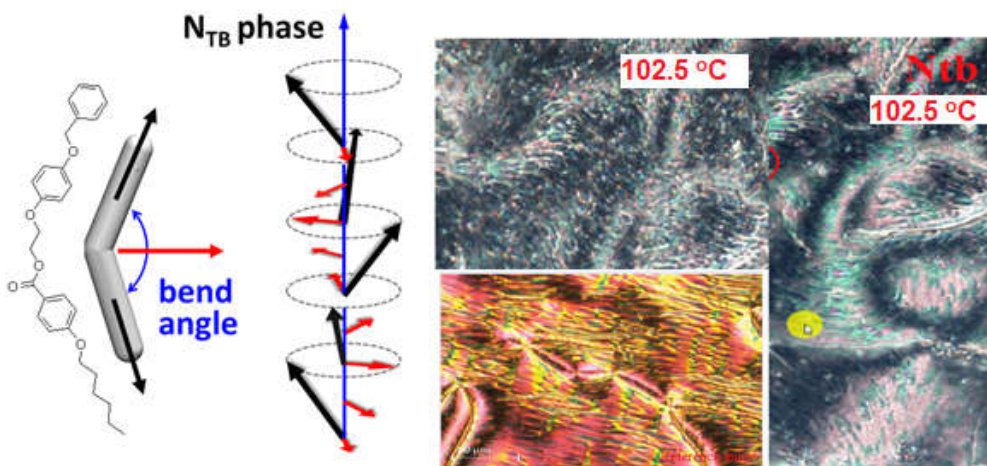


Fig 3. Structurally similar homologous series

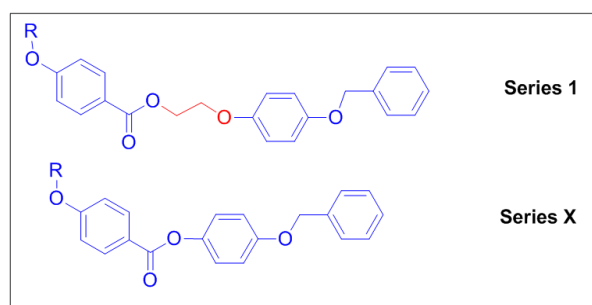
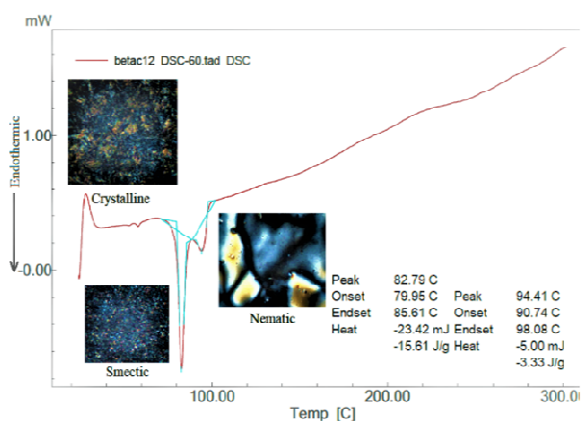
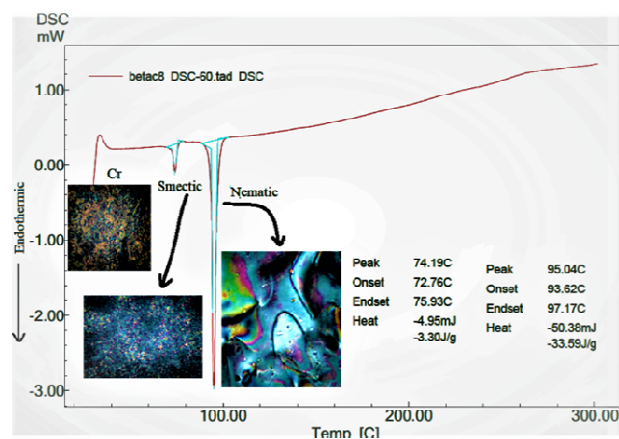


Fig 4. DSC analysis of C8 homologue

Fig 5. DSC analysis of C₁₂ homologue

Thus, two contraries influence of intramolecular attraction due to substituted long n-alkyl tail plays role in facilitating nematogenic or smectogenic phase behaviour through stoutness of either polarizability or molecular polarity. Hence, the molecule aligns at different angle either perpendicular or less than 90° on the fluid surface, which shows smectic or nematic or poly mesomorphism in normal or irreversible manners to facilitate favourable in enantiotropic mode. The present series only differ by ethyl spacer, which decreases the rigidity of the molecule and increase the flexibility, hence the transition temperature of the synthesized series is low comparative to series X because the transition temperature depends on the central group attached with the molecule which affects the polarity, polarizability, molecular rigidity and flexibility types of physical properties. The length of n-alkyl chain -R in '-OR' group increase as series ascended which also promote the flexibility as well as lowering the transition temperatures of the molecule.\

DSC Study: Synthesised new homologues series possess mesomorphic properties is supported by the DSC analysis.

DSC is a useful tool that complements optical methods in the study of liquid crystal phase transition. In current research work, the enthalpy of transition of octyloxy and dodecyloxy homologues was measured by differential scanning calorimetry (DSC) at the rate of temperature 10°C/min and their phase length recorded in Table-4. The following graph (Fig4&5) shows that dodecyloxy shows the smectogenic behaviour sharp at 82.7 °C while the nematic phase observed at 94.41 °C. According to DSC phase length for smectic and nematic phase behaviour 11.71 °C and 12.1 °C respectively. In octyloxyhomolog smectic and nematic texture is observed at 74.19 °C and 95.04 °C respectively (Fig-4). This homolog possesses phase length 20.85 °C for smectic and 4.5 °C for nematic.

CONCLUSION

Synthesise series is poly mesomorphic and have a low transition temperature. Among all 11 homologues, C₆ shows a twist bend nematic phase which opens the new area in the field of liquid crystals. Introducing ethyl spacer in reported molecules convert it into poly mesomorphic which reveal that with the help of different spacer, a different alignment of the molecules as well as lowering of transition temperature can be obtained. The use of spacer provides high thermal stability as well as earlier commencement of poly mesomorphism.

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