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## Study of Influence on Mesomorphic Properties by Structural Changes with Additional -CH<sub>2</sub>-CH<sub>2</sub>- Spacer between Two Rigid Cores

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### ABSTRACT

The novel poly mesomorphic homologous series was synthesized to understand the mesomorphic behaviour of compounds with respect to molecular structure. Present series was consisting 11 homologues which were completely smectogenic and along with poly-mesomorphism. In smectogenic texture it shows some broken fane like texture which represent the presence of smectic-A texture along with smectic-C. Mesomorphic behaviour was measured by polarizing optical microscope with heating stage. Average thermal stability of series is 130.8 °C and 140.7 °C for Sm-N/I and N-I respectively along with middle order melting type and the transition curve behaves in normal manner.

**Keywords:** Spacer, Nematic, Smectic, Discotic nematic, Poly mesomorphism, Smectic A

### 1. INTRODUCTION

Since 1888, the world of LC was established, enormous scientist was doing their study on LC compounds with the different aim and objectives or purpose [1-4]. The current work has been designed to study the LC properties in contrast of molecular structure. RO-C<sub>6</sub>H<sub>4</sub>-CH=CH-COO-CH<sub>2</sub>-CH<sub>2</sub>-O-C<sub>6</sub>H<sub>4</sub>-O-CH<sub>2</sub>-C<sub>6</sub>H<sub>5</sub> type of homologues were synthesised and their mesomorphic properties were measured. Generally -CH<sub>2</sub>-CH<sub>2</sub>- linkage provides molecules

good flexibility [5] which provides molecules to arrange in particular angle to form N<sub>tb</sub> mesophase. But in current work we observe that molecule possess poly mesomorphism still absence of N<sub>tb</sub> mesomorphism, all homologues show smectogenic while C<sub>4</sub>, C<sub>5</sub>, C<sub>6</sub> & C<sub>8</sub> homologues show poly mesomorphic in nature. The current work has been compared with the same homologous series but having different linking group and concluded that current linking group increase the mesomorphic behaviour in synthesized series.

## **2. MATERIALS AND METHODS**

4-Hydroxy benzoic acid, n-alkyl bromides (R-Br), K<sub>2</sub>CO<sub>3</sub>, malonic acid, 4-hydroxy benzaldehyde, 4-benzyl phenol, chloro ethanol, IBCF (Isobutyl chloroformate), methanol, acetone, pyridine and other reagents etc required for synthesis were purchased from Spectrochem, Merck and Sigma-Aldrich. All the reagents and solvents were used as received. Purity of all these synthesized compounds was ascertained by TLC. It was accomplished on 0.2 mm pre-coated plates of silica gel G60 F254 (Merck). Visualization was made under UV light (254 and 365 nm). The few homologues of synthesized series were characterized by elemental analysis (Table 1), infrared spectroscopy, <sup>1</sup>H NMR spectra, and mass spectroscopy. Microanalysis was performed on EuroEA Elemental Analyzer. IR spectra were recorded on Shimadzu FTIR Model-IRAffinity-1S (MIRacle 10), <sup>1</sup>H NMR spectra were recorded on Bruker spectrometer using CDCl<sub>3</sub> as a solvent and mass spectra were recorded on Shimadzu GC-MS Model No. QP-2010.

## **3. EXPERIMENTAL**

### **3. 1. Synthesis of 4-n-alkoxy benzaldehyde (Int-1)**

In 250 mL single neck RBF 4-hydroxy benzaldehyde (5.0g, 40.9 mmol, 1.0 eq.) was charged with Acetone and K<sub>2</sub>CO<sub>3</sub> (8.4 g, 61.47 mmol, 1.5 eq.) and n-Alkyl Bromide (61.47 mmol, 1.5 eq.) was added to it and allowed to stir at reflux temperature for 12 hr. After completion of reaction indicated by TLC, reaction mixture was quenched by water and extracted with diethyl ether. The organic layer was washed with 10 % NaOH solution and dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to give yellow oil as SA-A1 (n-Alkoxy benzaldehyde) which was used for the next step without purification [6].

### **3. 2. Synthesis of 3-(4-Alkoxyphenyl) acrylic (Int-A)**

In 250 mL single neck RBF n-Alkoxy benzaldehyde (1.0 eq.) charged with pyridine and malonic acid (1.5 eq.) was added and followed by addition of piperidine catalytic amount allowed to stir at reflux temperature for 12 hr. After completion of reaction indicated by TLC reaction was poured to cold water, white solid was filtered and dried to give 3-(4-methoxyphenyl) acrylic Int-A [7].

### **3. 3. Synthesis of 2-(4-(benzyloxy) phenoxy) ethan-1-ol (Int-B)**

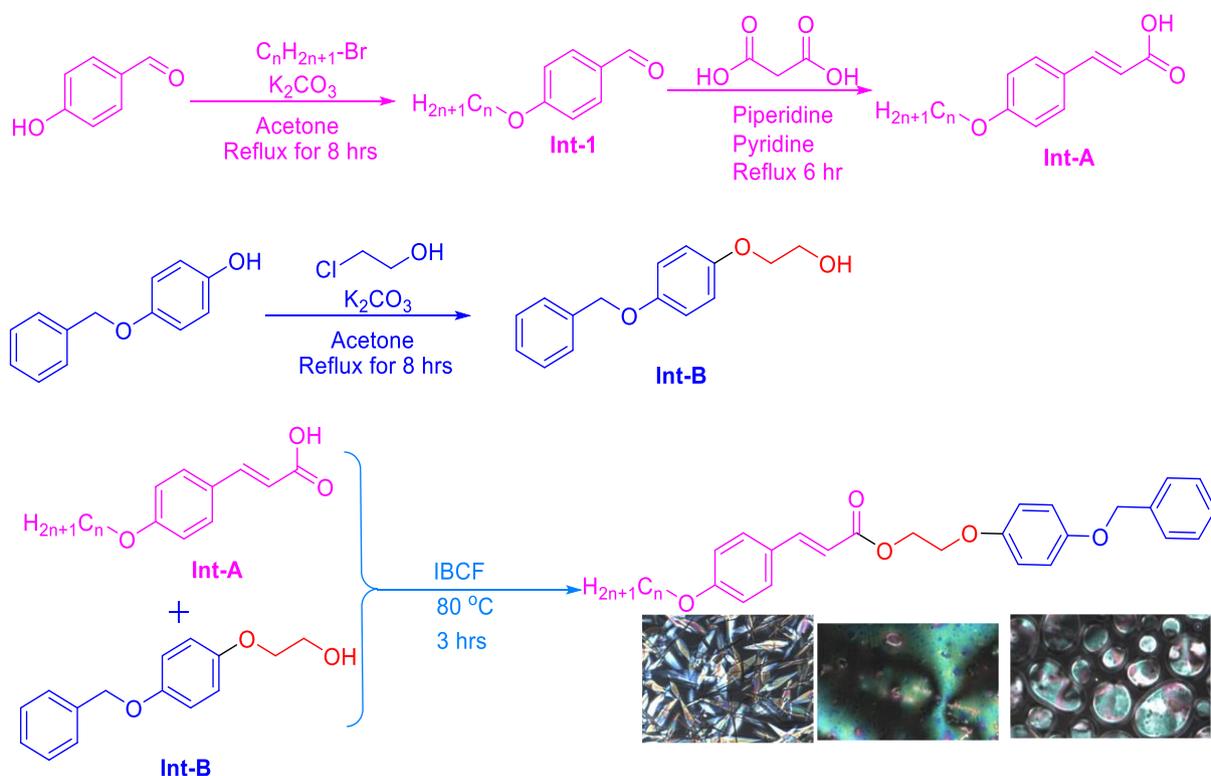
In 250 mL single neck RBF 4-benzyloxy phenol (1.0 eq.) was charged with acetone and K<sub>2</sub>CO<sub>3</sub> (1.5 eq.), 2-chloroethanol (1.2 eq.) was added and reaction was allowed to stir at reflux temperature for 12 hr. After completion of reaction indicated by TLC reaction was quenched

with water and extracted with diethyl ether. Organic layer was washed with 10 % NaOH solution and dried over  $\text{Na}_2\text{SO}_4$  and concentrated to give off white solid which was used for the next step without purification [6].

### 3. 4. Synthesis of 2-(4-(benzyloxy) phenoxy) ethyl -3-(4-Alkoxyphenyl) acrylate

In 25 mL single neck RBF 3-(4-methoxyphenyl) acrylic acid (SA-A) (1.0 eq.) and 2-(4-(benzyloxy) phenoxy) ethan-1-ol (SA-B) (1.0 eq.) was charged with IBCF (Isobutyl chloroformate) and allowed to stir at  $80\text{ }^\circ\text{C}$  for 3 hr. After completion of reaction indicated by TLC reaction was quenched with bicarbonate solution and extracted with diethyl ether. Organic layer was dried over  $\text{Na}_2\text{SO}_4$  and concentrated to give crude product. The crude was purified by crystallisation using acetic acid [8].

Synthesis of ether linkage novel cinnamate is depicted in Scheme 1.



**Scheme 1.** Synthetic route of the series 1

### 3. 5. Analytical data

- $^1\text{H}$  NMR in ppm of ethyloxy derivative

$^1\text{H}$  NMR in  $\delta$ ppm 400 MHz, 1.41-1.45 (t, 3H,  $\text{O}-\text{CH}_2-\text{CH}_3$ ), 4.05-4.11 (q, 2H,  $\text{O}-\text{CH}_2-\text{CH}_3$ ), 4.38-4.69 (t, 4H,  $-\text{O}-\text{CH}_2-\text{CH}_2-\text{O}-$ ), 5.10 (s, 2H,  $-\text{O}-\text{CH}_2-\text{Ar}$ ), 6.89-6.91 (dt, 2H, -Alkyloxy phenyl), 7.02-7.04 (dt, 2H  $-\text{O}-\text{CH}_2-\text{Ar}$ ), 7.09-7.13 (m, 3H,  $-\text{O}-\text{CH}_2-\text{Ar}$ ), 7.45-

7.49 (d, 1H, -CH=CH-), 7.62-7.66 (m, 2H, Alkyloxy phenyl), 7.76-7.79 (d, 1H, -CH=CH-), 7.9-8.05 (m, 4H -O-Ar-O-).

- <sup>1</sup>H NMR of in ppm of butyloxy derivative  
0.90-1.00 (t, 3H, -O-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>3</sub>), 1.46-1.52 (m, 2H, -O-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>3</sub>), 1.75-1.82 (m, 2H, -O-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>3</sub>), 3.99-4.02 (t, 2H, -O-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>3</sub>), 4.38-4.69 (t, 4H, -O-CH<sub>2</sub>-CH<sub>2</sub>-O-), 5.04 (s, 2H, -O-CH<sub>2</sub>-Ar), 6.89-6.91 (dt, 2H, -Alkyloxy phenyl) 7.02-7.05 (dt, 2H, -O-CH<sub>2</sub>-Ar), 7.09-7.14 (m, 3H, -O-CH<sub>2</sub>-Ar), 7.46-7.49 (d, 1H, -CH=CH-) 7.62-7.66 (m, 2H, Alkyloxy phenyl), 7.76-7.79 (d, 1H, -CH=CH-) 7.98-8.06 (m, 4H, -O-Ar-O-).
- IR Spectrum in cm<sup>-1</sup> of heptyloxy derivative  
3039.91 (=C-H aromatic Stretching), 2924.18 & 2854.74 (-CH<sub>2</sub> Alkane Starching), 1720.56 (-C=O str. of Ester), 1635.69 (C=C alkenes stretching), 1504.53 (C=C aromatic bending), 1419.66 (bending for -CH<sub>2</sub> of alkyl chine), 1311.64 (bending for -CH<sub>3</sub> of alkyl chain), 1203.62 (C-O-C ether stretching), 833.28 (p-substitution aromatic ring).
- IR Spectrum in cm<sup>-1</sup> of Dodecyloxy derivative  
3032.20 (=C-H aromatic Stretching), 2916.4 (-CH<sub>2</sub> Alkane Stretching), 1720.56 (-C=O str. of Ester), 1635.69 (-C=C- alkene stretching), 1504.53(C=C aromatic bending), 1419.66 (bending for -CH<sub>2</sub> of alkyl chain), 1203.62 (C-O-C ether stretching), 833.28 (p-substitution aromatic ring).
- Mass Spectrum of decyloxy derivative  
m/z (int of %) 530.0 (3.0% M+), 287.0 (83 % ester linkage break), 331.0 (80 % ether linkage break), 91.0 (100% benzyloxy linkage break)
- Mass Spectrum of octyloxy derivative  
m/z (int of %) 457.8, 258.9 (100% ester linkage break), 302.9 (1% ether linkage break), 91.0 (35.0% benzyloxy linkage break).
- Elemental analysis of C1, C3, & C5 homologues (Table 1).

**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 <sub>25</sub> H <sub>24</sub> O <sub>5</sub>	74.19	5.93	19.72	74.24	5.98	19.78
2	C <sub>27</sub> H <sub>28</sub> O <sub>5</sub>	74.99	6.49	18.30	74.98	6.53	18.37
3	C <sub>29</sub> H <sub>32</sub> O <sub>5</sub>	75.59	6.98	17.31	75.63	7.00	17.37

#### 4. RESULT AND DISCUSSIONS

The current series was synthesized to understand LC properties according to molecular structure. The derivative of 2-(4-(benzyloxy) phenoxy) ethyl- 4-n-alkoxycinnamate were synthesised using 4-n alkoxy cinnamic acid and 2-(4-benzyloxy phenoxy) ethanol. New synthesized series was poly mesomorphic in nature. 11 homologues were synthesized, and their phase transition temperature was recorded in polarizing optical microscope Nikon Eclipse 400/ TU Plan ELWD 20 X /0.40 which shown in Table 2. All homologues were smectogenic while C<sub>4</sub>, C<sub>5</sub>, C<sub>6</sub>, & C<sub>8</sub> homologues were enantiotropically smectogenic as well as nematogenic.

**Table 2.** Transition temperature in °C.

Sr no.	n-Alkoxy chain	Smectic	Nematic	Isotropic
1	C <sub>1</sub>	142.4	-	150.2
2	C <sub>2</sub>	158.2	-	162.3
3	C <sub>3</sub>	151.5	-	153.9
4	C <sub>4</sub>	138.9	145.0	148.2
5	C <sub>5</sub>	127.6	131.0	141.3
6	C <sub>6</sub>	109.0	124.4	135.5
7	C <sub>7</sub>	104.3	-	118.8
8	C <sub>8</sub>	118.2	123.7	137.9
9	C <sub>10</sub>	81.0	-	104.4
10	C <sub>12</sub>	92.4	-	130.3
11	C <sub>14</sub>	86.6	-	95.3

According to transition temperature graph of LC phase behaviour were plotted transition temperature in °C Vs no. of carbon atom in n-alkyl chain shown in Figure 1. Transition from Cr-Sm was shown by dotted line and it behaves in normal descending manner as series was ascended following partially zig-zag path. Sm-N/I curve flows parallel to Cr-Sm curve up to pentyloxy homologue and from pentyloxy homologue its flows partial cosine pattern. N-I curve show the transition temperature of four homologues which were poly mesomorphic in nature.

##### 4. 1. Effect of structural modification on mesomorphic state

The current synthesized series was compared with the structurally most similar series X [9] and series Y [9] to understand the effect of structural change on LC properties which shown in Table 3.

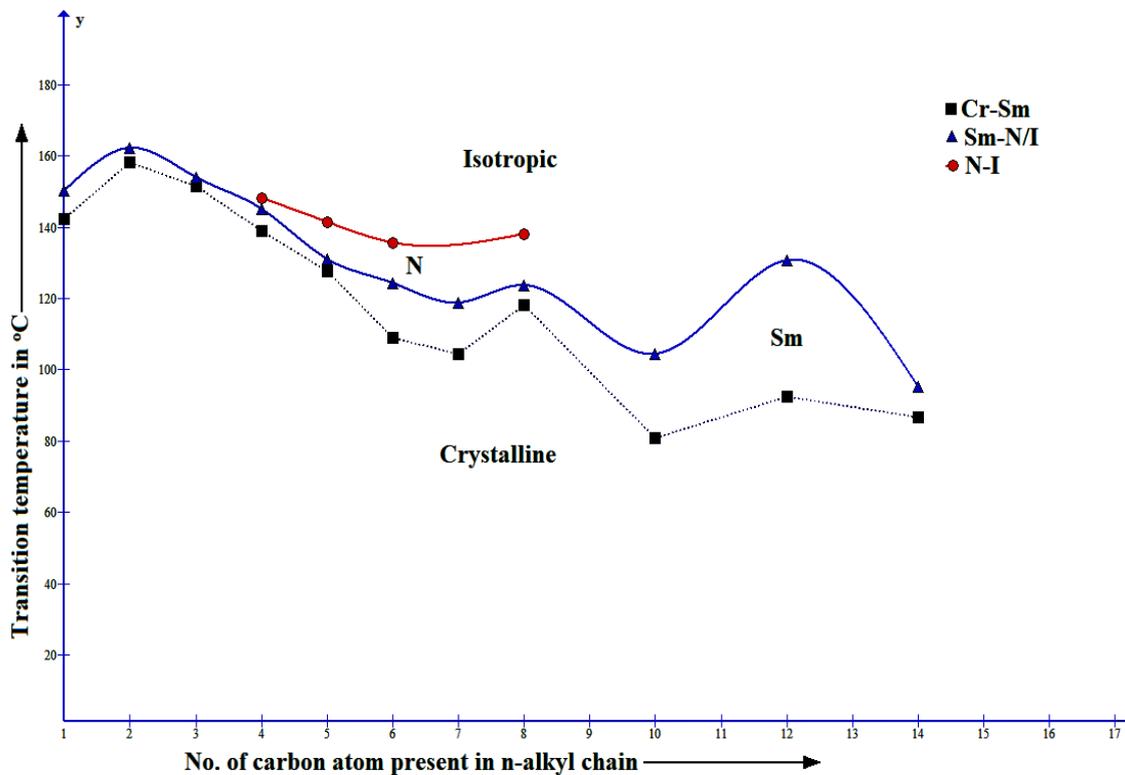
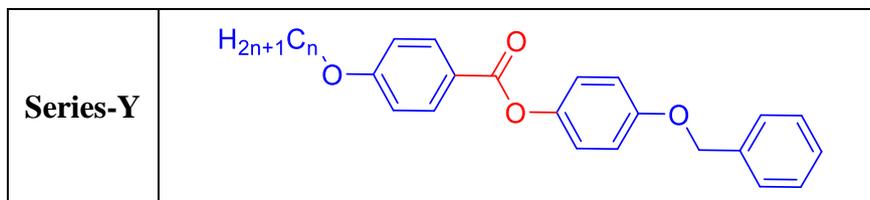


Figure 1. Phase diagram of series 1

Synthesized series was only differing with compared series X in  $-\text{O}-\text{CH}_2-\text{CH}_2-$  linkage and with Y in  $-\text{CH}=\text{CH}-$  &  $-\text{CH}_2-\text{CH}_2-$  linkage but this minor structural change gives tremendous rise to partial poly-mesomorphism in current series. Series-X was partially nematogenic as well as smectogenic in LC manner while in series Y completely opposite observation was observed that it was mesomorphic with the half of homologues having monotropic smectogenic characteristic.

Table 3. Comparison of series 1 with structural similar series X and Y.

Series 1	
Series-X	



In contrast the synthesized series possess the poly-mesomorphic characteristic with C<sub>4</sub>, C<sub>5</sub>, C<sub>6</sub> & C<sub>8</sub> homologues and those homologues which monotropically shows nematogenic characteristic in series X, those homologues show smectogenic nature in series Y & series 1. As comparative to series Y, it concluded that each and every homologue synthesized in current series factually possess the mesomorphic behaviour, so the word monotropy was completely removed after this structural change (Table 3).

The main role in liquid crystal transition was played by the temperature and it was completely depending on the molecular structure and it was observed by giving changes to molecular structure. As changes in molecular structure were given it made to produce the change in molecular polarizability, also in length and its alignment with end to end terminal attraction. These all factors play important role in LC properties. Here compared both the series exhibit LC texture below 100 °C and this was the major change which observed in current comparison after this type of structural change.

Taking close observation to the transition temperature Table 2 it observed that the transition temperature was descended with the ascending of series and it also reveals that the poly-mesomorphism was also increased and it's due to the molecular structural change. Because as series ascended the terminal alkyl chain length was also increase and this give rise to molecular all over length and this supports the overall rising polarizability of the molecule which increase the terminal attraction as the terminal end to end attraction increases molecule align factually preserve layered structure which support the liquid condition and under this condition smectic texture arise and with the increasing temperature give rise to the thermal vibrations which enhance the fluid character of the molecule but still random alignment was prohibited and at actual temperature nematic texture comes in the picture but still end to end parallel attraction remain and this gives rise to the poly-mesomorphic characteristic.

#### 4. 2. DSC Analysis

Synthesized new homologous series possess the smectogenic as well as nematogenic characteristic. This transition changes were supports by the analysis of differential scanning calorimetry. It's used for the detection of the change in compounds physical properties under thermal conditions. In presented work enthalpy of transition of propyloxy and decyloxy homologues were measured by the help of DSC analysis at the rate of 10 °C/min and their phase length were recorded in Table 4. The Figure 3 and 4 shows the graph of DSC analysis for the propyloxy and decyloxy homologue respectively.

From the Figure 2 and 3 we can observe that the very sharp smectic change observed in propyloxy and decyloxy homologues. The DSC spectra of propyloxy homologue shows one sharp peak while two peaks were observed in Figure 4 which represent the sharp smectic change along with isotropic change. The phase length for the propyloxy derivative is very small which could not be observed at the rate of 10 °C/min so that we observed the absence of isotropic peak in propyloxy homologue.

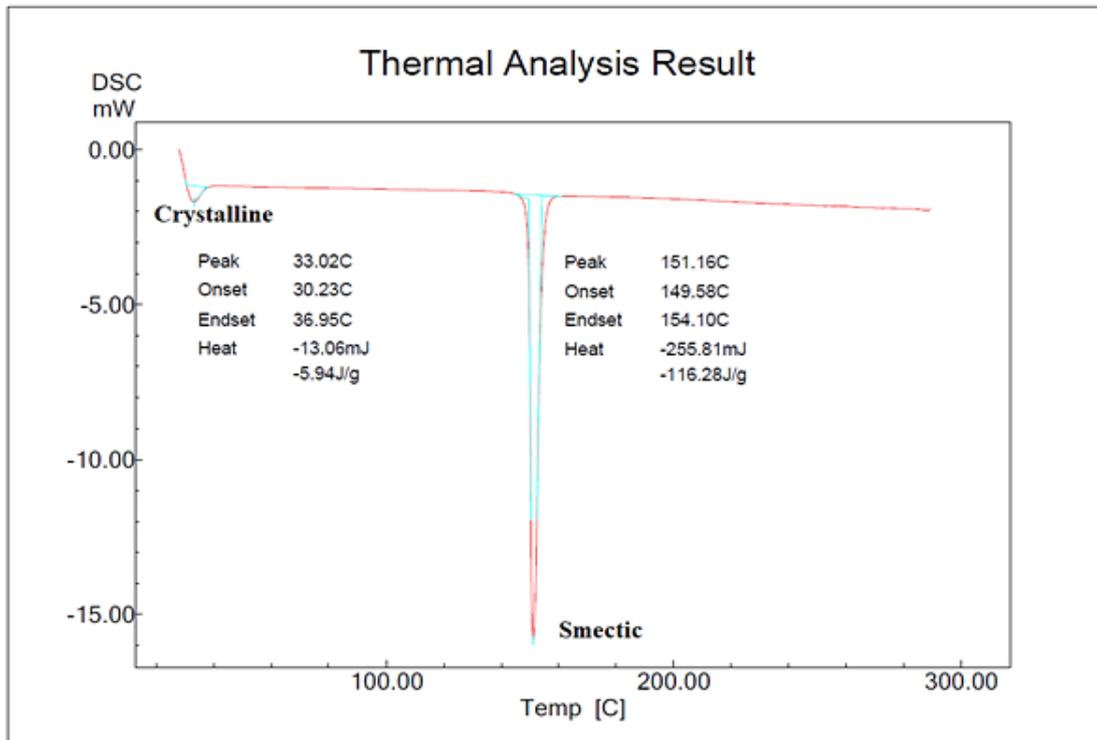


Figure 2. DSC analysis of propyloxy homolog

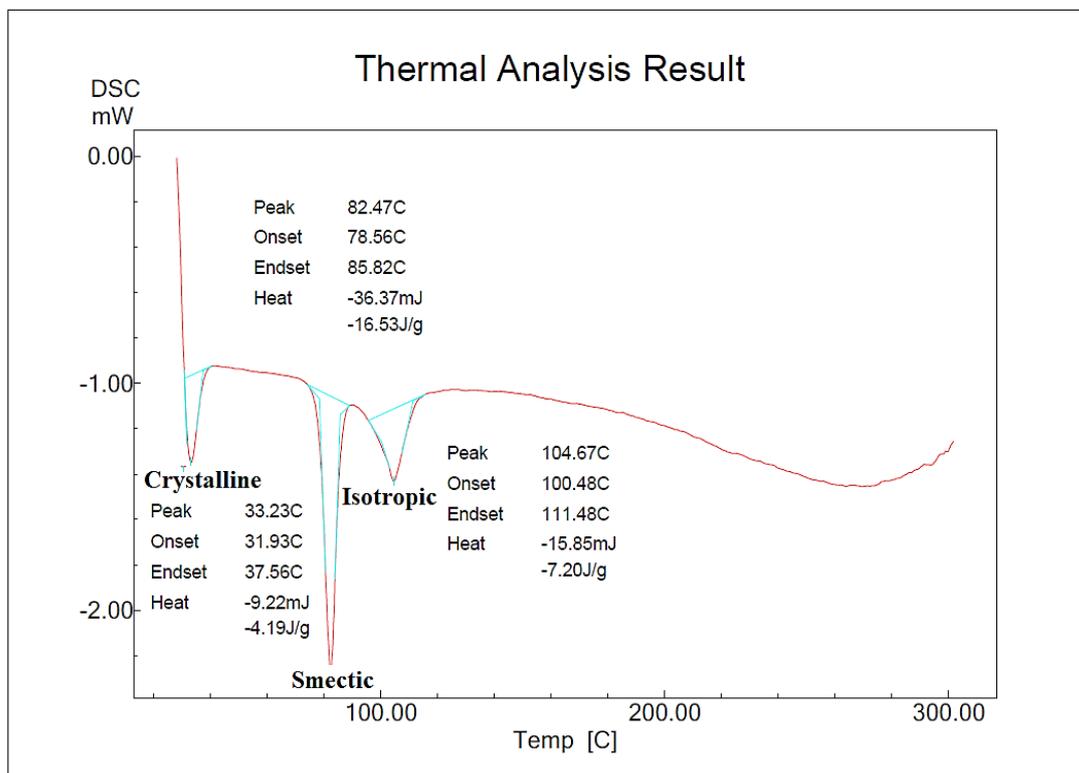


Figure 3. DSC analysis of decyloxy homologue

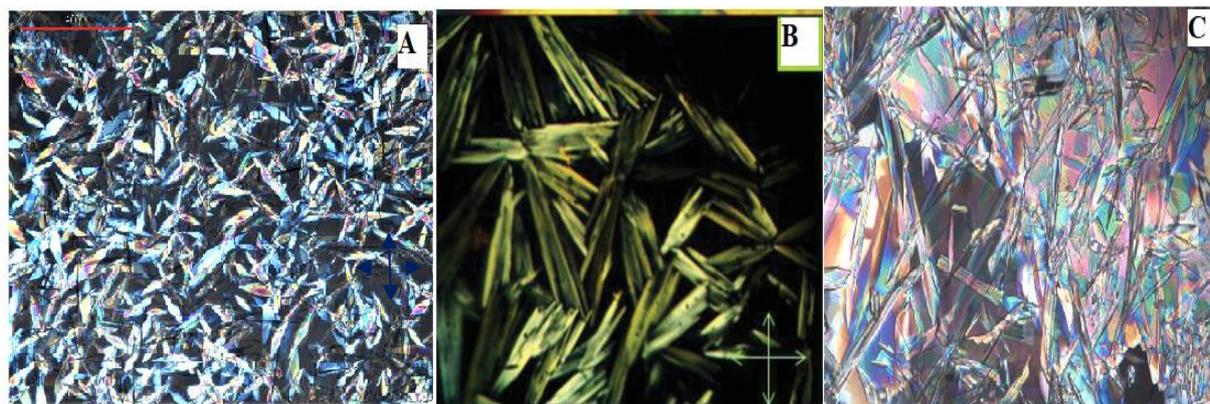
From the Table 4 we can conclude that the mesophase length for the propyloxy homologue is very less as compared to decyloxy homologue. Here the higher enthalpy changes for the propyloxy homologue give rise to the entropy change which lead to unstable smectic calamitic consequently low mesophase length. While decyloxy homologue shows completely opposite observation with low enthalpy change along low entropy which enhance the stability of smectic phase as so that it possesses the long mesophase length.

**Table 4.** Transition enthalpy and entropy change for C<sub>3</sub> and C<sub>10</sub> homologues

Homologue	Transition	Peak Temp. in °C	$\Delta H/ J g^{-1}$	$\Delta S/ Jg^{-1} K^{-1}$	Phase length
C <sub>3</sub>	Cr-SmC	151.16	116.28	0.2741	Sm-2.4
C <sub>10</sub>	Cr-SmC	82.47	16.53	0.04650	Sm-23.3

### 4. 3. POM texture Study

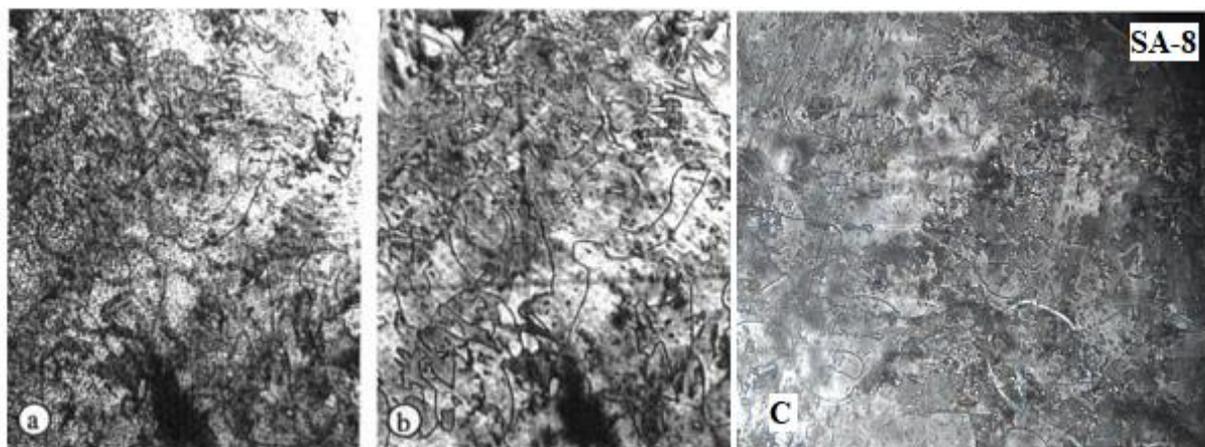
The current synthesized series possess poly mesomorphic characteristic and it confirmed by polarising optical microscopic texture. Present series comparison was carried out with all well published mesogenic textures which were proved by different analytical supports. This comparison makes very precise conclusion about each and every homologues present in series. The series was exclusively possessing nematic characteristic but in nematic mesophase it shows some different textures at certain temperature and those texture were compared with reported LC textures and identification of the probable alignment for particular texture was as followed.



**Figure 4.** Comparison of Sm-A texture with reported texture B

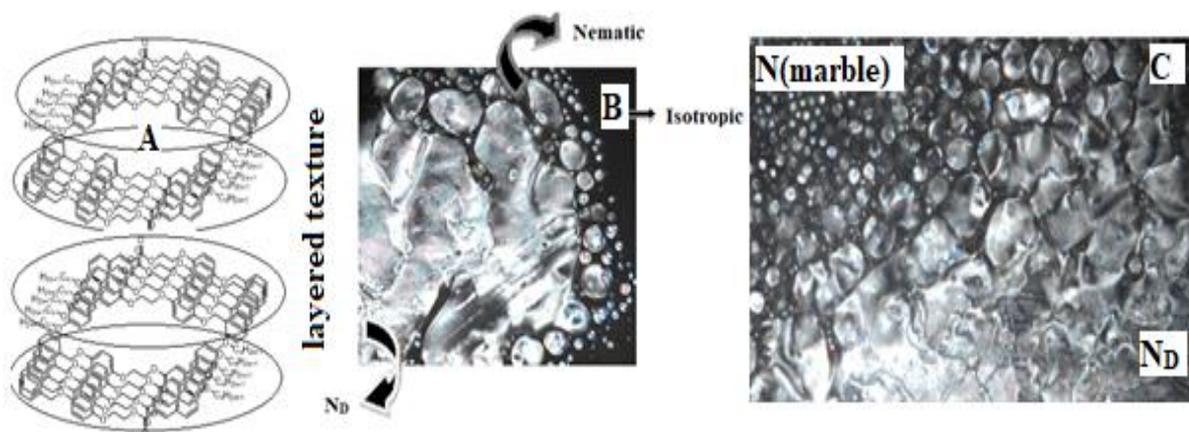
C<sub>7</sub> and C<sub>10</sub> show smectic A which was shown in Figure 4 and those texture were compared with reported smectic A texture (B) [10-13] here C<sub>7</sub> (C) and C<sub>10</sub> (A) reveals the smectic A texture at 104.3 °C and 81.0 °C respectively. The alignment of molecules itself arranged with parallel to each other to form layered or like calamitic preserved long range as well as short range orientational ordering results this type of smectic A texture. In contrast with smectic

texture except with C<sub>4</sub>, C<sub>5</sub>, C<sub>6</sub> and C<sub>8</sub>. These homologues were poly-mesomorphic in nature and it shows the discotic type nematic texture which was shown below (Figure 5).



**Figure 5.** Comparison of Discotic mesophase with C<sub>8</sub> homologue

As per Figure 6 both texture a and b were well reported as columnar and discotic mesophase texture [14-17] and current synthesized series possess this type of texture, but as reported work was on polymers, but this type of bent core molecules how possess N<sub>D</sub> phase!!!



**Figure 6.** Close transition of marble nematic texture to N<sub>D</sub> texture

Commonly mesophase of columnar or discotic texture was formed by molecular alignment more or less orientational order positionally parallel but long-range positional order was not maintained to form layered texture and this possibility was shown in Figure 6 for the synthesized homologues. Among all the 11 homologues 4 homologues show the discotic nematic texture, initially shows marble type nematic texture and by gradually decreasing temperature made to form more thick fluid texture and reveals the discotic texture which can be easily identified by its disclination lines shown in Figure 6(C) which was the characteristic of the discotic nematic texture.

**Table 5.** Thermal stability of series and mesophase length.

Series→	Series 1	Series X	Series Y
Sm-N/I	130.8 (C <sub>1</sub> -C <sub>14</sub> )	96.0 (C <sub>8</sub> -C <sub>12</sub> )	101.5 (C <sub>8</sub> -C <sub>18</sub> )
Commencement of smectic phase	C <sub>1</sub>	C <sub>7</sub>	C <sub>2</sub>
N-I	140.7 (C <sub>4</sub> -C <sub>6</sub> , C <sub>8</sub> )	153.5 (C <sub>1</sub> -C <sub>2</sub> )	-
Commencement of nematic phase	C <sub>4</sub>	C <sub>1</sub>	-
Total mesophase length in °C minimum to maximum	2.4 to 37.9 °C (C <sub>3</sub> ) (C <sub>12</sub> )	4 to 9 °C (C <sub>2</sub> , C <sub>12</sub> ) (C <sub>1</sub> )	7.0 to 15.0 °C (C <sub>16</sub> , C <sub>18</sub> ) (C <sub>12</sub> )

Table 5 was showing the thermal stability and total mesophase length of series 1 with compared to series X & Y. From Table 5 following conclusions was drawn.

- All over thermal stability of series 1 was comparatively high for the smectogenic phase behaviour.
- Commencement of smectogenic mesophase was earlier at methoxy homologue while it observed at C<sub>7</sub> and C<sub>2</sub> in Series X and Series Y respectively.
- Thermal stability of the nematogenic transition was comparatively middle order at 140.7 °C for series 1 & 153.5 °C for series X while series Y is non-nematogenic in nature.
- Commencement of nematic texture observed at C<sub>4</sub> homologue for series 1 while in series X it observed at C<sub>1</sub> homologue.
- Total mesophase length of synthesised series 1 was higher than series X & series Y.
- The lowest mesophase length observed in-middle of both series.
- Series 1 have the propyloxy homolog with lowest mesophase length while series x and series y have the ethyloxy and hexadecyloxy homologs respectively.
- The higher phase length of series 1 and series y observed at dodecyloxy homolog while it observed at methyloxy homolog in series x.

Thus, the series 1 was smectogenic as well as nematogenic with poly mesomorphic homologues except few homologues with middle order transition temperature, while series X partially smectogenic as well as nematogenic and series Y was the smectogenic with lower transition temperature.

## 5. CONCLUSIONS

- The aim of this study was to elucidate effectiveness of structural modification on the mesomorphism.

- The current series was modified by little ethyl linkage between two rigid cores and it made series to show poly mesomorphism.
- This series shows discotic nematic texture and having middle order transition.
- By the study of comparison of POM texture, it illustrates that heptyloxy and decyloxy homologues were smectogenic with smectic A calamitic and rest of (C<sub>4</sub> to C<sub>6</sub> and C<sub>8</sub>) possess the marble texture of nematic and as well as discotic nematic mesophase.
- The mesomorphic properties were completely influenced by the structural change.
- Central group of molecules affect to the flexibility of the molecule and it also increase the end to end orientational order to form preserved layered texture.
- 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 ethyl spacer provides high thermal stability along with earlier commencement of poly mesomorphism.
- Introducing ethyl spacer reduce the mesophase initially and as no. of carbon increase in n-alkyl chain the mesophase length of molecules also increase respectively.

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