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## Synthesis, Characterization and Mesomorphic Study of new Homologous Chalcone Series

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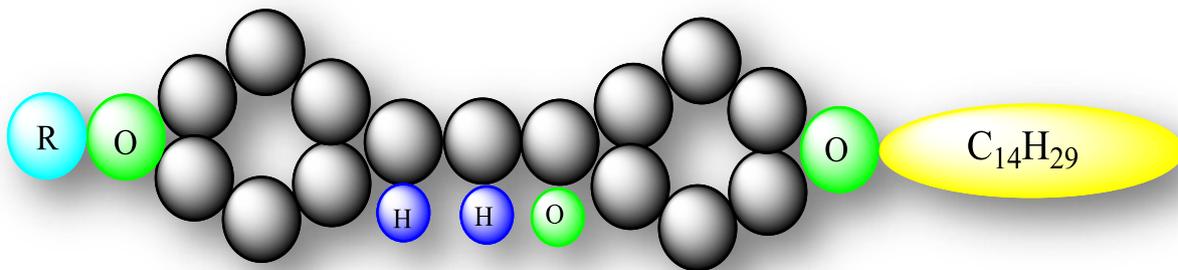
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### ABSTRACT

A new designed mesogenic homologous series of chalconyl derivative with two ring  $\alpha$ -4-Alkoxy benzoyl- $\beta$ -4'-tetradecyloxy phenyl ethylene have been synthesized and characterized by a combination of elemental analysis and standard spectroscopic methods. In series, lower members ( $C_5$  to  $C_8$ ) exhibit only enantiotropic nematic mesophase but higher members ( $C_{10}, C_{12}, C_{14}, C_{16}, C_{18}$ ) exhibit enantiotropic nematic as well as smectic mesophase. The nematic mesophase (Thread type) commence from  $C_5$  homologue and smectic mesophase (smectic-A type texture) commence from  $C_{10}$  homologue as enantiotropic and persists up to the last member synthesized. Textures are conformed by Polarizing Optical Microscope (POM). The mesomorphic properties of present series were compared with other two structurally similar series to study the effects of change of meta and para position on the mesomorphism.

**Keywords:** Liquid Crystal; Smectic; Nematic; Enantiotropic; Mesomorphism

## GRAPHICAL ABSTRACT



where:  $R = C_nH_{2n+1}$      $n = 1,2,3,4,5,6,7,8,10,12,14,16,18$

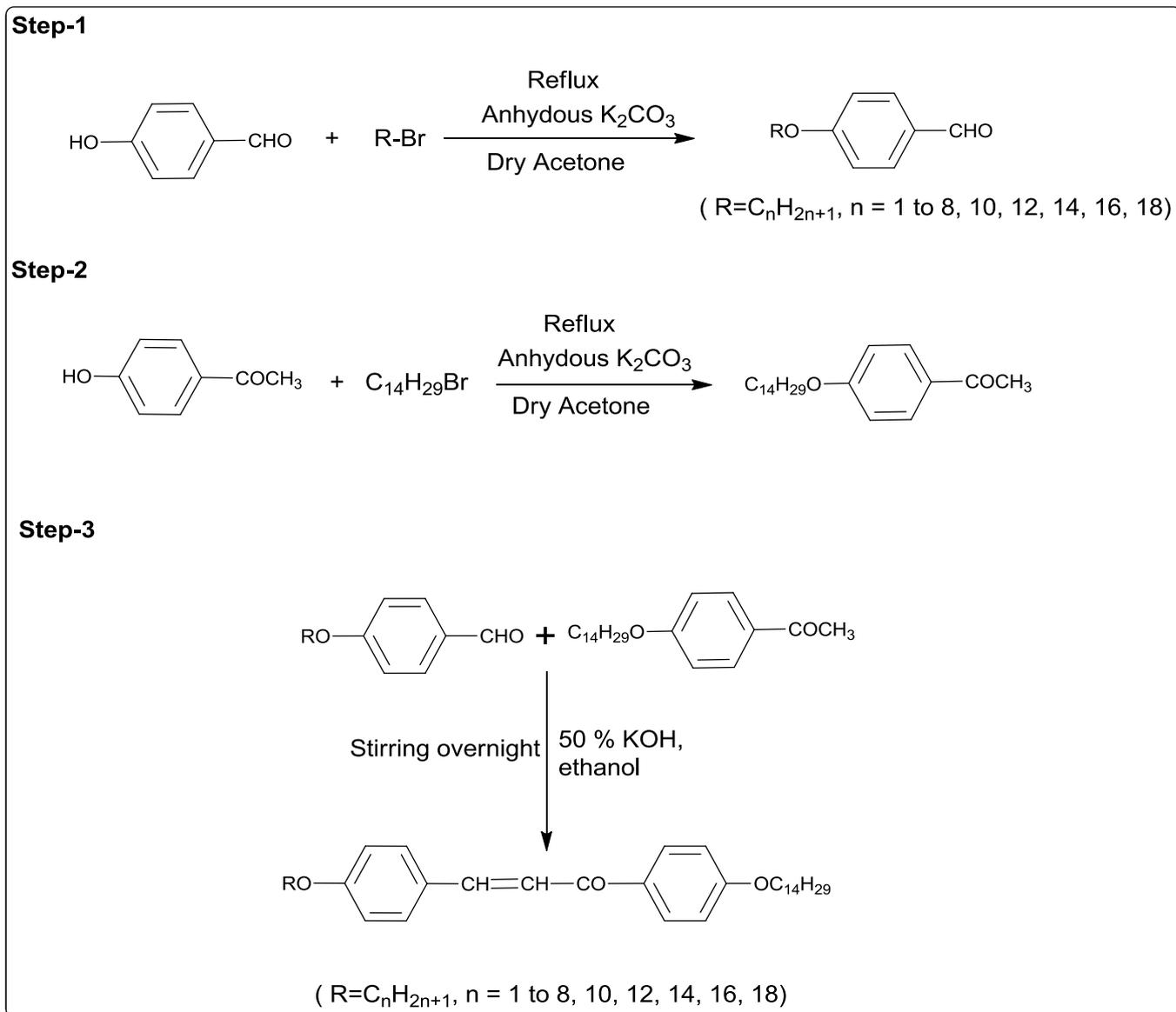
## 1. INTRODUCTION

Liquid crystal (LC) [1] is an intermediate state of a matter in between the solid crystalline and liquid. These kinds of substances are neither fully crystal nor liquid. There are many kinds of LC materials with different molecular structures and shape. The Chalconyl derivatives due to their geometrical shapes may exhibit LC properties with lower thermometric transitions and bioactivity. Chalconyl derivatives with two ring and  $-CH=CH-COO-$  as central linkage exhibit thermotropic liquid crystal property (LC) property with biological activity which is very useful for society in the manufacture of LC devices and the pharmaceutical preparations [2-10]. The present work includes synthesis, characterization by analytical, thermal and spectral data. Result and discussion on the basis of thermometric data and thermal behaviors of novel series derived using Polarizing Optical Microscope (POM). The object of this work to understand the effect of molecular structure on liquid crystal property with reference to changing meta, para position and molecular flexibility through lateral groups of present and analogous series [11-18].

## 2. EXPERIMENTAL

### Synthesis

Alkylation of 4-hydroxy benzaldehyde to give 4-n-alkoxy benzaldehyde is carried out by reported method [19] and 4-n-tetradecyloxy acetophenone is obtained by alkylation of 4-hydroxy acetophenone by reported method [20] Thus, the chalconyl homologue derivatives were prepared by usual establish method [21]. Homologues were filtered, washed with ethanol solution dried and purified till constant transition temperatures obtained using an optical polarising microscope equipped with a heating stage. Alkyl halides, EtOH, KOH, 4-Hydroxy acetophenone, 4-Hydroxy benzaldehyde etc., required for synthesis were used as received except solvents which were dried and distilled prior to use. The synthetic route to the series is mentioned below as Scheme 1.



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

### Characterization

Representative homologues of a series were characterised by elemental analysis, Infrared spectroscopy,  $^1H$  NMR spectra, IR spectra were recorded on Perkin-Elmer spectrum GX,  $^1H$  NMR spectra were recorded on Bruker using  $CDCl_3$  as solvent. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyser (Table 1). Transition temperature (Table 3) and LC properties (Textures) were determined using an optical polarising microscopy equipped with heating stage and digital camera (POM). Texture images of nematic phase were determined by miscibility method (Table 2), thermodynamic quantities enthalpy ( $\Delta H$ ) and entropy ( $\Delta S = \Delta H/T$ ) are qualitatively discussed.

**Analytical Data**

**Table 1.** Elemental Analysis for (1) Hexyloxy (2) Octyloxy (3) Dodecyloxy and (4) Tetradecyloxy derivatives.

Sr. No.	Molecular formula	% Elements found		% Elements Theoretical	
		C	H	C	H
1	C <sub>35</sub> H <sub>52</sub> O <sub>3</sub>	80.76	10.00	80.74	10.03
2	C <sub>37</sub> H <sub>56</sub> O <sub>3</sub>	81.02	10.21	81.01	10.19
3	C <sub>41</sub> H <sub>64</sub> O <sub>3</sub>	81.46	10.60	81.43	10.59
4	C <sub>43</sub> H <sub>68</sub> O <sub>3</sub>	81.64	10.76	81.60	10.75

**Table 2.** Texture of Nematic Phase of C<sub>6</sub>, C<sub>10</sub>, C<sub>14</sub>, C<sub>18</sub> by miscibility method.

Sr. No.	Homologue	Texture
1	C <sub>6</sub>	Threaded
2	C <sub>10</sub>	Threaded
3	C <sub>14</sub>	Schlieren
4	C <sub>18</sub>	Schlieren

**IR Spectra in cm<sup>-1</sup> for Heptyloxy&Dodecyloxy Derivatives**

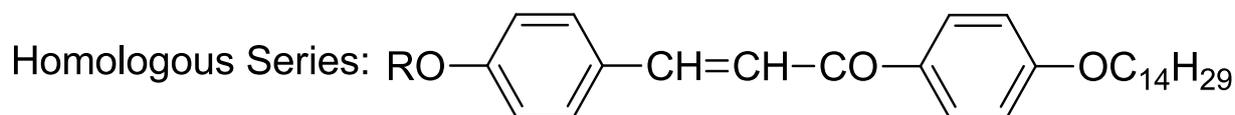
**Heptyloxy:** 2912 (C-H str. of alkane), 2848 (C-H str. of -(CH<sub>2</sub>)<sub>n</sub> group of -OC<sub>7</sub>H<sub>15</sub> alkyl chain, 1604-1654 (C=O str. of carbonyl carbon of chalconyl group), 1612 (C=C str. of alkene), 1512, 1564 (C=C str. of aromatic ring), 995 (C-H bending of alkene), 1182 (C-O str. of ether linkage), 1285, 1244 (C-O str. of carbonyl group), 776 Polymethylene (-CH<sub>2</sub>-) of -OC<sub>14</sub>H<sub>29</sub>, 682 Polymethylene (-CH<sub>2</sub>)<sub>n</sub> of -OC<sub>7</sub>H<sub>15</sub>, 830 (-C-H- def. m di-substituted-Para), IR data confirms the molecular structure.

**Dodecyloxy:** 2914 (C-H str. of alkane), 2850 (C-H str. of -(CH<sub>2</sub>)<sub>n</sub> group of -OC<sub>12</sub>H<sub>25</sub> group, 1602-1656 (C=O str. of carbonyl group of chalconyl group), 1610 (C=C str. of alkene), 1514, 1568 (C=C str. of aromatic ring), 1002 (C-H bending of alkene), 1184 (C-O str. of ether linkage), 1280, 1252 (C-O str. of carbonyl group), 774 Polymethylene (-CH<sub>2</sub>-) of -OC<sub>14</sub>H<sub>29</sub>, 828 (-C-H- def. m di-substituted-Para), IR data confirms the molecular structure.

**<sup>1</sup>HNMR spectra in CDCl<sub>3</sub> in δ ppm for Hexyloxy & Hexadecyloxy Derivative:**

**Hexyloxy:** 0.88 (t, -CH<sub>3</sub> of polymethylene -C<sub>6</sub>H<sub>13</sub> and -C<sub>14</sub>H<sub>29</sub>), 1.78 p, CH<sub>3</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>- of -OC<sub>6</sub>H<sub>13</sub> and -OC<sub>14</sub>H<sub>29</sub>), 1.25 (m, -CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>- of -OC<sub>6</sub>H<sub>13</sub> and -OC<sub>14</sub>H<sub>29</sub>), 1.34 (q, -CH<sub>2</sub>-CH<sub>3</sub>), 4.02 (t, -OCH<sub>2</sub>-CH<sub>2</sub>-), 7.64 (d, -CH=CH-), 7.42, 7.28 & 7.80 (para substituted phenyl ring), 7.50 & 7.98 (phenyl ring with alkoxy chain). NMR data confirms the molecular structure.

**Hexadecyloxy:** 0.87 (t, -CH<sub>3</sub> of -C<sub>16</sub>H<sub>33</sub> and -C<sub>14</sub>H<sub>29</sub>), 1.80 (CH<sub>3</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub> of -OC<sub>16</sub>H<sub>33</sub> and -OC<sub>14</sub>H<sub>29</sub>), 1.28 (polymethylene -CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>- of -OC<sub>16</sub>H<sub>33</sub> and -OC<sub>14</sub>H<sub>29</sub>), 1.32 (q, -CH<sub>2</sub>-CH<sub>3</sub>), 4.06 (t, -OCH<sub>2</sub>-CH<sub>2</sub>-), 7.58 (d, -CH=CH-), 7.40, 7.26 & 7.83 (para substituted phenyl ring), 7.54 & 7.96 (phenyl ring with alloy chain), NMR data confirms the molecular structure.

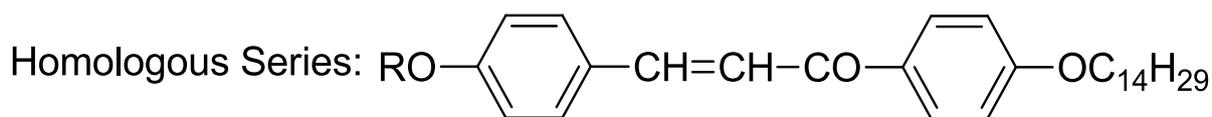


**Table 3.** Transition temperature in °C

Sr. No	R = n-alkyl group	Transition temperatures in °C		
		Smectic	Nematic	Isotropic
1	C <sub>1</sub>	-	-	92.0
2	C <sub>2</sub>	-	-	96.0
3	C <sub>3</sub>	-	-	88.0
4	C <sub>4</sub>	-	-	84.0
5	C <sub>5</sub>	-	67.0	80.0
6	C <sub>6</sub>	-	59.0	74.0
7	C <sub>7</sub>	-	60.0	73.0
8	C <sub>8</sub>	-	63.0	76.0
9	C <sub>10</sub>	44.0	57.0	71.0
10	C <sub>12</sub>	48.0	62.0	75.0
11	C <sub>14</sub>	50.0	59.0	69.0
12	C <sub>16</sub>	45.0	60.0	72.0
13	C <sub>18</sub>	42.0	57.0	70.0

### 3. RESULT AND DISCUSSION

The novel homologous series have been synthesized by condensation reaction between 4-n-alkoxy benzaldehyde and 4-decyloxy acetophenone which include thirteen homologues. In present series C<sub>1</sub> to C<sub>4</sub> homologues are nonmesogenic, C<sub>5</sub> to C<sub>8</sub> homologues are enantiotropicallynematic and C<sub>10</sub>, C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub> C<sub>18</sub> homologues are enantiotropicallynematic plus smectic. The transition temperature table-3 determined with the help of polarizing optical microscope (POM) equipped with the hot stage and plotted against the number of carbon atoms present in n-alkyl chain 'R' of -OR group and then transition curves Cr-M/I, N-I and Sm-N are obtain on linking like or related transition points as shown in a phase diagram (Figure 1). Transition Curve for Cr-M/I adopted zigzag path of rising and falling manner with overall descending manner and behaved in normal manner. Sm-N transition curve slightly rises from C<sub>10</sub> to C<sub>12</sub> and descended till the last homologue C<sub>18</sub> with absence of odd-even effect. N-I transition curve descended from C<sub>5</sub> to C<sub>10</sub> and ascended at C<sub>12</sub>, and descended till the last homologue C<sub>18</sub> with exhibition of very narrow and short odd-even effect upto C<sub>9</sub> homologue. Thermal stabilities are 59.0 °C and 73.0 °C for smectic and nematic respectively whose maxima to minimum phase lengths are 09 °C to 15.0 °C and 12 °C to 15.0 °C respectively. Thus, novel homologous series is low melting type series. Analytical, spectral and thermal data supported molecular structures of homologues. The variations in mesogenic properties and behaviours from homologue to homologue in the same series is observed with changing number of carbon atoms in n- alkyl chain 'R' of -OR group, keeping rest of the molecular part unaltered throughout a series.



*α*-4-Alkoxybenzoyl*β*-4'-tetradecyloxy phenyl ethylene

The condensation of nonmesomorphs 4-n-alkoxy benzaldehydes with 4-n-tetradecyloxy acetophenone extends molecular lengths, polarity and polarizability, rigidity and flexibility, permanent dipole moment across the long molecular axis, intermolecular end to end and lateral attractions, suitable magnitude of dispersion forces and dipole-dipole interaction etc. Which induces resistivity and disalignment of molecules at an angle ninety degree or/and less than ninety degree with the plane of floating surface under the influence of exposed thermal vibrations to maintain the floating with statistically parallel orientational order and/or with sliding layered molecular arrangement for different range or ranges of temperatures to facilitate only nematic or smectic plus nematic mesophase formation for C<sub>5</sub> to C<sub>8</sub>, C<sub>10</sub>, C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, C<sub>18</sub> homologues. The variations in liquid crystal (LC) properties and the degree of mesomorphism from homologue to homologue in the same series or from series to series for the same homologue is attributed to the changing magnitudes of molecular rigidity and flexibility due to varied molecular polarity and polarizability and other related parameters concerning suitable magnitudes of intermolecular cohesions and closeness.

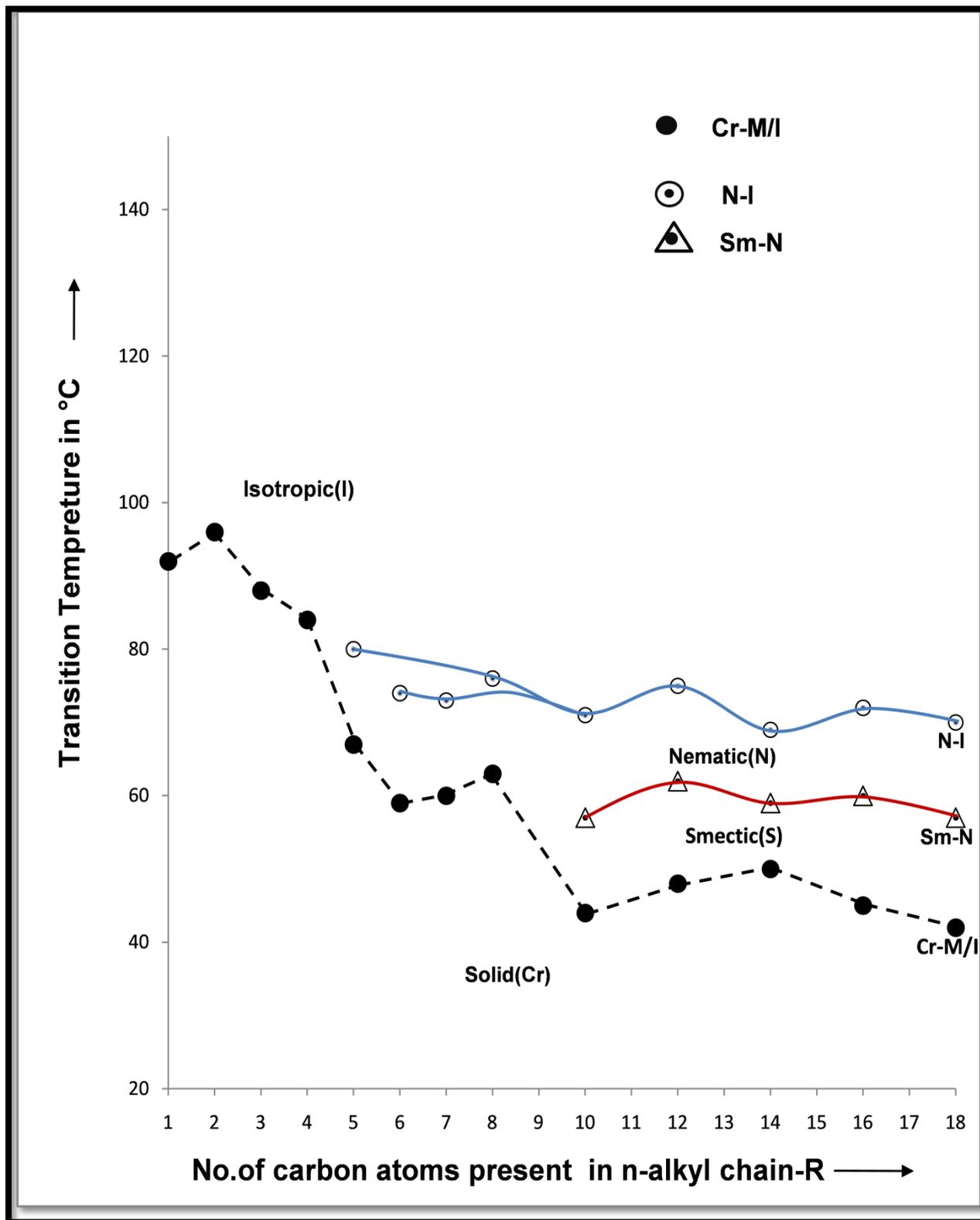
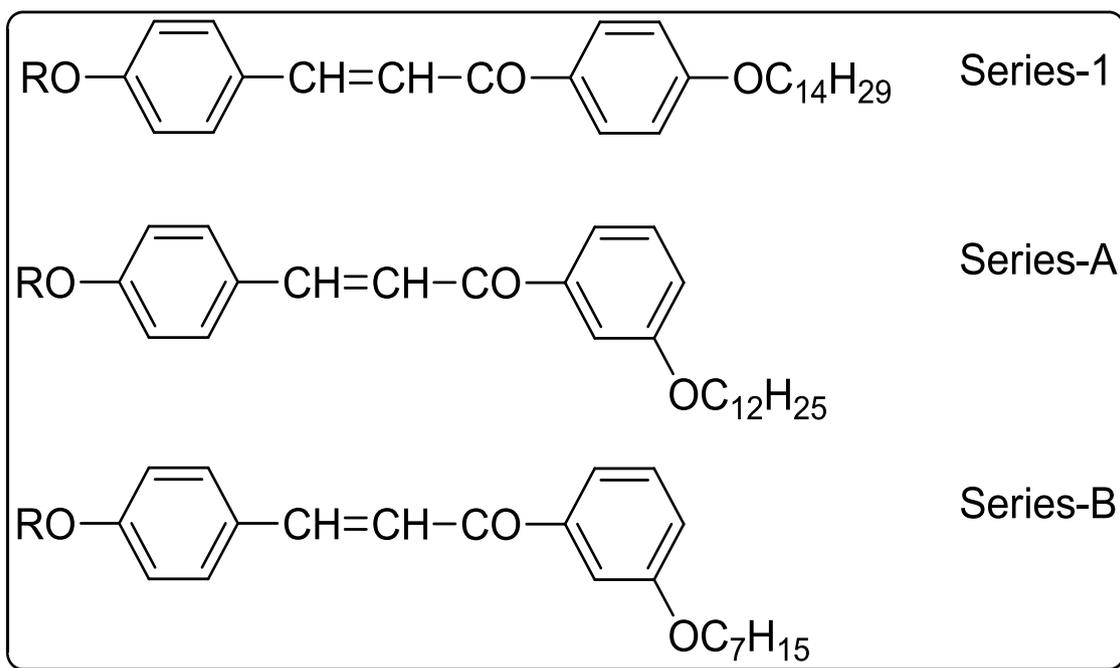


Figure 1. Phase behavior of series

The changing style of liquid crystal (LC) properties from homologue to homologue in the present novel series is observed. The exhibition of mesomorphism by C<sub>5</sub> to C<sub>8</sub>, C<sub>10</sub>, C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, C<sub>18</sub> homologues of a present novel series is attributed to the suitable magnitudes of anisotropic forces of intermolecular cohesions and closeness as a consequence of fittest molecular rigidity, polarizability and favourable flexibility. The flexibility from homologue to homologue in the same series undergo variations keeping molecular rigidity and the part of flexibility due to para substituted –OC<sub>14</sub>H<sub>29</sub> right end tailed unaltered. The nonmesomorphic behaviours of C<sub>1</sub> to C<sub>4</sub> homologues is due to the low magnitudes of dispersion forces and low magnitudes of dipole-dipole interactions leading to high crystallising tendency and inducing unsuitable magnitudes of anisotropic forces of end to end attractions. Diminishing of odd-even effect for higher homologues beyond C<sub>9</sub> homologue of longer n-alkyl chain ‘R’ of –OR and –OC<sub>14</sub>H<sub>29</sub> right end flexible groups is attributed to coiling, bending, flexing and coupling with the major axes of core structure of a molecule. The liquid crystal (LC) properties of presently investigated homologous Series-1 are compared with structurally similar series A [22] and B [23] as under in Figure 2.



**Figure 2.** Structurally analogous series.

Homologous Series-1, A and B are identical with respect to two phenyl rings and one central bridge –CH=CH-CO- contributing to total molecular rigidity and the left n-alkoxy terminals –OR for the same homologue from series to series which partly contributes to the total molecular flexibility for same homologue and homologue to homologue in the same series. However, they differ with tailed end group viz. para –OC<sub>14</sub>H<sub>29</sub>, meta –OC<sub>12</sub>H<sub>25</sub>, meta –OC<sub>7</sub>H<sub>15</sub> for the same homologue from series to series and homologue to homologue in the same series which partly contributes to the total molecular flexibility. Therefore, variations in

liquid crystal (LC) properties are due to differing features of their molecular structures which differs with respect to flexibility of tailed end groups differing by  $-\text{CH}_2-\text{CH}_2-$  and their position of substitutions para or meta as shown in Figure 2. Following table-4 represents some evaluated thermometric data indicating mesogenic behaviours and the degree of mesomorphism for the series-1, A and B under comparative study.

**Table 4.** Thermal Stability in °C.

Series →	Series-1 Para $-\text{OC}_{14}\text{H}_{21}$	Series-A Meta $-\text{OC}_{12}\text{H}_{25}$	Series-B Meta $-\text{OC}_7\text{H}_{15}$
Sm-I or Sm-N Commencement of smectic phase	59.0 ( $\text{C}_{10}-\text{C}_{18}$ ) $\text{C}_{10}$	56.0 ( $\text{C}_4 - \text{C}_5$ ) $\text{C}_4$	Fraction of 1 °C or 1 °C ( $\text{C}_{10} - \text{C}_{18}$ ) $\text{C}_{10}$
N-I Commencement of nematic phase	73.0 ( $\text{C}_5-\text{C}_{18}$ ) $\text{C}_5$	70.5 ( $\text{C}_2 - \text{C}_5$ ) $\text{C}_2$	66.0 ( $\text{C}_7 - \text{C}_8$ ) $\text{C}_7$
Total mesophase lengths in °C minimum to maximum	13.0 to 27.0 $\text{C}_{5/7}$ $\text{C}_{10/16}$	05.0 to 29.0 $\text{C}_{10/14}$ $\text{C}_4$	07.0 to 15.0 $\text{C}_{10/14/16}$ $\text{C}_{12}$

From Table 4, it is clear that,

- Smectic mesomorphism commences from  $\text{C}_{10}$  homologue in series-1 whereas it commences  $\text{C}_4$  and  $\text{C}_{10}$  homologue in series-A and B respectively
- Nematic mesomorphism commences from  $\text{C}_5$  homologue in series-1 whereas it commences from  $\text{C}_2$  and  $\text{C}_7$  homologue in series –A and B respectively.
- Homologous series under comparison are predominantly nematic and partly smectic in enantiotropic and/or monotropic condition.
- Lower mesophaselength of Series-1 is longest and of equal magnitudes for Series-A and B as well as upper mesophaselength increases from Series-B to 1 to A.
- Thermal stability for smectic is less for the series -1 as compared to series A and B.
- Thermal stability for nematic is more for series-1 as compared to series A and series-B.

In present novel homologous series-1 the alternation of nematic thermal stability or mesophaselengths are related with the combined effects of molecular rigidity in combination with flexibility and the unexpected status of both ended n-alkoxy end groups, which may fluctuate with the status of flexible groups and their polarity or polarizability. Therefore, it may acquire increasing or decreasing or alternating order of facilitating or stabilizing mesophase or mesophases in enantiotropic manner. Series-1 has para substituted  $-\text{OC}_{14}\text{H}_{29}$

group whereas Series -A and B have meta substituted lateral  $-\text{OC}_{12}\text{H}_{25}$  and  $-\text{OC}_7\text{H}_{15}$  groups so there are difference in the geometry of series-1 as compared to series-A and B. Thus, the two opposing effects of intermolecular attractions due to para and meta substituted tail ended n- alkoxy groups play role in facilitating smectic and/or nematic mesophase formation through predominancy of either molecular polarity or polarizability factor. Therefore length to breath ratio, intermolecular closeness, molecular and functional groups polarities and polarizabilities, permanent dipole moment across the long molecular axis, magnitudes of dipole-dipole interactions, magnitudes of dispersion forces, molecular rigidity and flexibility, end to end and lateral attractions vary in more or less in magnitudes for same homologue from series to series , operating a phenomina of mesomorphism and the degree of mesomorphism. The increasing order of nematic thermal stability is attributed to the increasing order of intermolecular cohesions corresponding to increasing number of carbon atoms present from Series-1 ( $-\text{OC}_{14}\text{H}_{29}$  (p)) to series-A ( $-\text{OC}_{12}\text{H}_{25}$ (m)) to Series-B ( $-\text{OC}_7\text{H}_{15}$ (m)) due to increasing order of polarities and polarizability caused by their para substituents or lateral groups for the same homologue in series 1, A and B respectively.

#### 4. CONCLUSIONS

- Novel homologous series of single central bridge linking two phenyl rings and n- alkoxy terminal groups is enantiotropically nematic and smectic.
- The group efficiency order derived on the basis of (a) thermal stability (b) early commencement of mesophase (c) Mesophaselengths for smectic and nematic are as under.

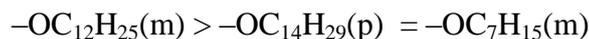
##### (1) Smectic



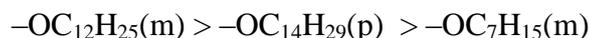
**Nematic**



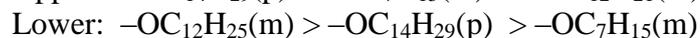
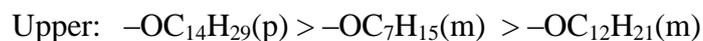
##### (2) Smectic



**Nematic**



##### (3) Sm + N



- Mesomorphism is very sensitive and susceptible to a molecular structure.

- Mesomorphism is depended on suitable magnitudes of molecular rigidity and flexibility.
- Reversal of sequential order of monotropy and enantiotropy in a chalconyl group containing series of two phenyl rings is observed as compared to other homologous series.
- Present investigation may be useful for LC devices, Their pharmaceutical or biological activity may be exploited for pharmaceutical preparation and agricultural production to reduce the consumption of insecticides and pesticide as they are antibacterial and antifungal.
- Present study supports and raises creadibility to the conclusions drawn earlier.

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