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Dependence of Molecular Flexibility of Lateral Group on Mesomorphism

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ABSTRACT

A Chalconyl homologous series: RO-C_6H_4-CH=CH-CO-C_6H_4-$OC_{12}H_{25}$(n) (meta) of novel liquid crystalline (LC) derivatives of thermotropic variety have been synthesized and studied with a view to understand the effect of molecular structure on LC behaviours with reference to varying flexibility due to tailed end group for the same homologue and from homologue to homologue in the same series. A novel series consisted of thirteen homologues whose first member is nonliquidcrystals and the rest of the homologues are LC in enantiotropic or monotropic condition. Nematogenic mesomorphism commences from C_2 homologue. C_2 to C_5 homologues are enantiotropic nematic and C_6 to C_{18} homologues are monotropic nematic. Smectogenic character commences from C_4 homologue as enantiotropic manner and C_6 to C_{18} derivatives are monotroic smectic. Transition temperatures and textures of mesophases were determined by an optical polarising microscopy (POM) equipped with a heating stage. The transition curves of a phase diagram behaved in normal manner. Spectral, analytical and thermal data supported the molecular structures of homologues. Thermal stability for smectic is low of the order of 56.0 °C and that of the nematic is 70.5 °C. The corresponding mesophaselengths for smectic and nematic are varied minimum to maximum are 1 or 2 °C to 15 °C and 5 to 29 °C respectively. The group efficiency order derived on the basis of thermal stability .

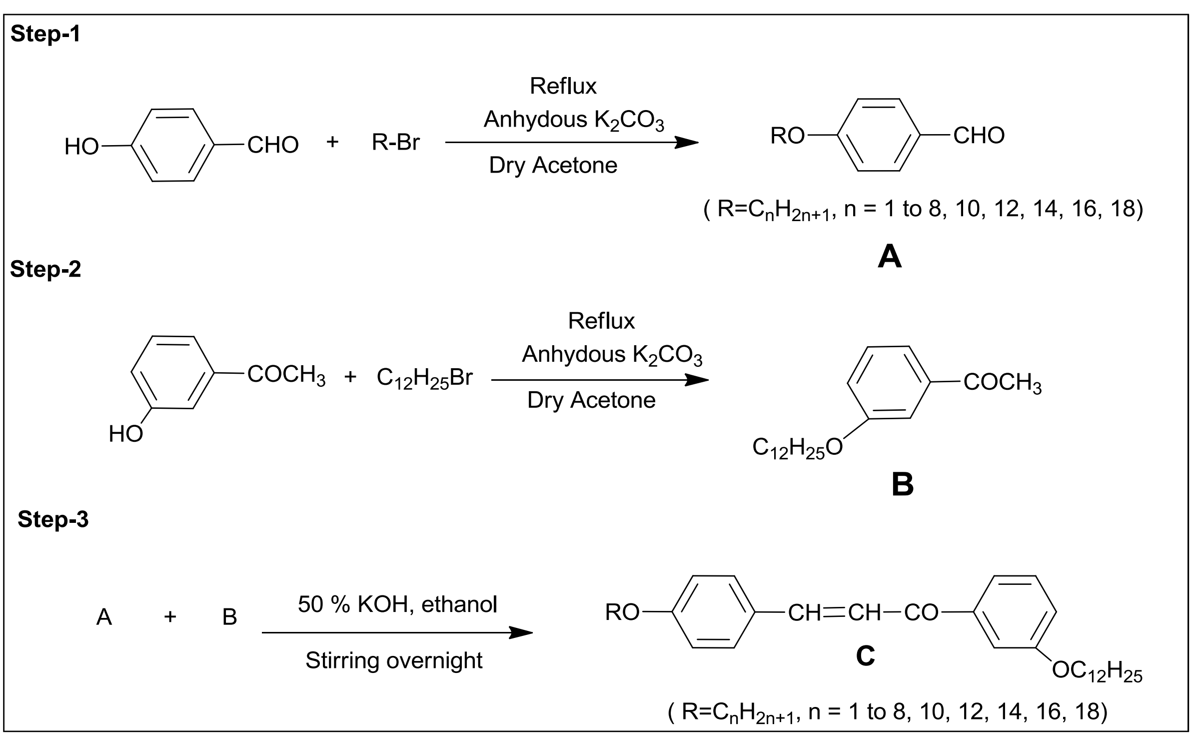
Keywords**:** Liquid Crystals; Smectic; Nematic; monotropy; Enantiotropy

1. INTRODUCTION

Liquid crystalline [1] property (LC) of a substance is an unique property, which flows on the surface like liquid and possesses optical properties like crystals. Therefore such substances of thermotropic or lyotropic varities are neither fully crystalline nor fully liquidous. The Chalconyl derivatives due to their geometrical shapes may exhibit LC properties with lower thermometric transitions and bioactivity. The aims and object in view to study the effect of molecular structure on LC properties and LC behaviours [2-7] as a result of molecular flexibility keeping molecular rigidity unaltered throughout a series and changing flexibility from series to series for the same homologue at constant rigidity. LC substances have proved their ability for LC devices to be operated at room temperature or desired temperature as well as in the pharmaceutical preparations operating bioactivity as antibacterial, antifungal, antimalarial, anticancer etc. [8-15]. Present investigation will include, synthesis and characterization by analytical, thermal and spectral data. Thermometric data will be derived using an optical polarising microscopy (POM) equipped with a heating stage and will be discussed and interpreted in terms of molecular rigidity and flexibility in relation [16-19] to molecular structure geometry, size, polarity and polarizability etc. LC properties of present novel series will be compared with the structurally similar analogous series. Number of homologous series have been reported till the date [20-26].

2. EXPERIMENTAL

2. 1. Synthesis



Scheme 1. Synthetic route to the series.

Alkylation of 4-hydroxy benzaldehyde to give 4-n-alkoxy benzaldehyde is carried out by reported method [27] and 3-n-alkoxy acetophenone is obtained by alkylation of 3-hydroxy acetophenone by reported method [28] Thus, the chalconyl homologue derivatives (C) were prepared by usual establish method [29] 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, 3-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.

2. 2. Characterization

Selected members of the novel homologous series were characterized by Elemental Analysis, infrared spectroscopy, ^{1}H NMR spectra. IR spectra were recorded by Perkin-Elmer spectrum GX,^{1}H NMR spectra were recorded on Bruker using $CDCl_3$ as solvent. Microanalysis was performed on a Perkin-Elmer PE2400 CHN analyzer. Transition temperature and LC properties(Textures) were determined using an optical polarizing microscopy equipped with a heating stage. Textures of nematic phase determined by miscibility method. Thermodynamic quantity enthalpy (ΔH) and entropy (ΔS) are qualitatively discussed instead of DSC scan.

2. 3. Analytical Data

Table 1. Elmental Analysis for Hexloxy, Octyloxy, Decyloxy and tetradecyloxy derivatives.

Sr. No.	Molecular formula	% Elements found		% Elements Theoretical	
		C	H	C	H
1	$C_{33}H_{48}O_3$	80.48	9.75	80.43	9.72
2	$C_{35}H_{52}O_3$	80.76	10.00	80.71	9.97
3	$C_{37}H_{56}O_3$	81.02	10.21	80.98	10.18
4	$C_{41}H_{64}O_3$	81.45	10.59	81.40	10.54

2. 4. IR Spectra in cm^{-1} for Hexyloxy & Dodecyloxy Derivatives

Hexyloxy: 2953 (C-H str. of alkane), 2847 (C-H str. of –(CH_2-)n group of –OC_6H_{13} group, 1600-1668 (C=O str. of carbonyl group of chalconyl group), 1614 (C=C str. of alkene), 1585 (C=C str. of aromatic ring), 1008, (C-H bending of alkene), 1170 (C-O str. of ether linkage), 1248 (C-O str. of carbonyl group), 778 Polymethylene (-CH_2-) of –$OC_{12}H_{25}$, 822 (–C-H- def. m di-substituted-Para), IR data confirms the molecular structure.

Dodecyloxy: 2956 (C-H str. of alkane), 2848 (C-H str. of –(CH_2-)n group of –$OC_{12}H_{25}$ group, 1600-1685 (C=O str. of carbonyl group of chalconyl group), 1641 (C=C str. of alkene), 1589 (C=C str. of aromatic ring), 1012, (C-H bending of alkene), 1174 (C-O str. of ether linkage), 1251 (C-O str. of carbonyl group), 775 Polymethylene (-CH_2-) of –$OC_{12}H_{25}$,823 (–C-H- def. m di-substituted-Para), IR data confirms the molecular structure.

2. 5. ^{1}HNMR spectra in $CDCl_3$ in δ ppm for Decyloxy & Tetradecyloxy Derivative

Decyloxy: 0.88 (t, -CH_3 of polymethylene –$C_{10}H_{21}$ and –$C_{12}H_{25}$), 1.80 (p, CH_3-CH_2-CH_2-CH_2-CH_2- of –$OC_{10}H_{21}$ and –$OC_{12}H_{25}$), 1.28 (m, –CH_2-CH_2-CH_2- of –$OC_{10}H_{21}$ and –$OC_{12}H_{25}$), 1.48 (q, -CH_2-CH_3), 4.06 (t,-OCH_2-CH_2-), 7.59 (d,-CH=CH-), 7.43, 7.28 & 7.83 (meta substituted phenyl ring), 7.55 & 7.60 (phenyl ring with alkoxy chain). NMR data confirms the molecular structure.

Tetradecyloxy: 0.86 (t, -CH_3 of –$C_{14}H_{29}$and –$C_{12}H_{25}$), 1.78 (CH_3-CH_2-CH_2-CH_2-CH_2-CH_2 of –$OC_{14}H_{29}$ and –$OC_{12}H_{25}$), 1.31 (polymethylene –CH_2-CH_2-CH_2- of –$OC_{14}H_{25}$ and –$OC_{12}H_{25}$), 1.43 (q,-CH_2-CH_3), 4.04 (t, -OCH_2-CH_2-), 7.54 (d, -CH=CH-), 7.40, 7.28 & 7.80 (meta substituted phenyl ring), 7.52 & 7.62 (phenyl ring with alkoxy chain). NMR data confirms the molecular structure.

Table 2. Texture of Nematic Phase of C, C_{10}, C_{14}, C_{16} by miscibility method.

Sr. No.	Homologue	Texture
1	C_6	Threaded
2	C_{10}	Threaded
3	C_{14}	Schlieren
4	C_{16}	Droplets type

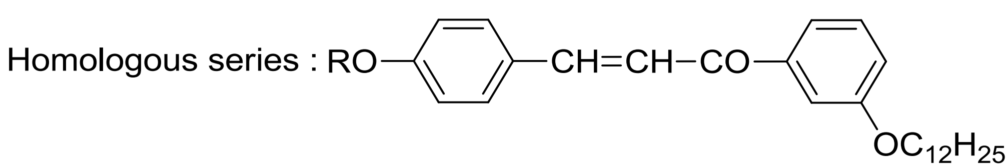


Table 3. Transition Temperature of Homologous series.

Sr.no	R= n-alkyl group	Transition temperatures in °C		
		Smectic	Nematic	Isotropic
1	C1	-	-	85.0
2	C2	-	50.0	74.0
3	C3	-	44.0	68.0
4	C4	43.0	58.0	72.0

5	C5	40.0	54.0	68.0
6	C6	(39.0)	(47.0)	62.0
7	C7	(42.0)	(48.0)	61.0
8	C8	(40.0)	(46.0)	59.0
9	C10	(44.0)	(49.0)	58.0
10	C12	(43.0)	(52.0)	63.0
11	C14	(46.0)	(51.0)	61.0
12	C16	(45.0)	(54.0)	60.0
13	C18	(42.0)	(48.0)	63.0

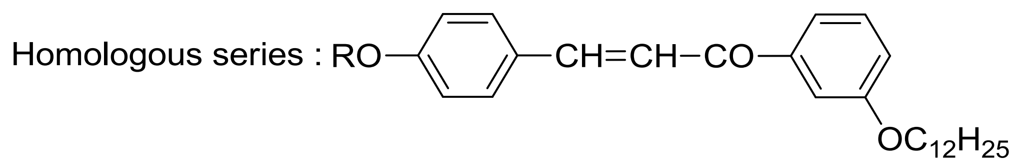
( ) indicates monotropy

3. RESULT AND DISCUSSION

A novel chalconyl series consisted of two phenyl rings, one terminal and one lateral group bonded through one central bridge is liquid crystalline in nature (C_2 to C_{18}) except first member C_1 of a series. Condensation of 4-n-alkoxy benzaldehyde with 3-n-Dodecyloxy acetophenone yielded thirteen (C_1 to C_{18}) homologues. Mesomorphism commenced from C_2 homologue and continued upto C_{18} homologue. C_4 and C_5 homologues are enantiotropic smectic and C_6 to C_{18} members of a series are monotropically smectogenic. Moreover C_2 to C_5 homologues are enantiotropic nematic and C_6 to C_{18} homologues are monotropic nematic. Thus all the members of a series except C_1. The C_2 and C_3 members of a series are only nematogenic, and C_4 to C_{18} homologues are nematogenic plus smectogenic in either enantiotropic (C_4 and C_5) or monotropic (C_6 to C_{18}) condition.

Transition temperatures of homologues (Table 2) were plotted against the number of carbon atoms present in n-alkyl chain 'R' of –OR group and then on linking like or related transition points, transition curves Cr-N/I, N-I or I-N and N-Sm or Sm-N are obtained, which shows the phase behaviours of series in figure-1. The Cr-N/I transition curve adopted zigzag path of rising and falling with overall descending manner. Sm-N or N-Sm transition curve initially descended and then ascended and finally descended to C_{18} homologue with exhibition of odd-even effect from C_4 to C_9 derivatives of a series. N-I or I-N transition curve descended from C_2 to C_6 and then ascended by negligible magnitudes and finally descended by about 4 to 5 degree at C_{18} homologu with exhibition of odd-even effect from C_2 to C_5 homologue.

Thus, transition curves behaved in normal manner. Odd member's transition curve for nematic occupied lower position than even members and reversal of occupation of transition curves for smectic i.e. odd member's transition curve for smectic occupies higher position than even member's curve. The sequential order of mesophase appearance of monotropy and enantiotropy is reversed or deviated from normal order as observed for other homologous series of esters or azoesters or chalconyl esters. Odd-even effect diminishes as series is ascended for higher homologues of longer n-alkyl chain 'R' of –OR group.



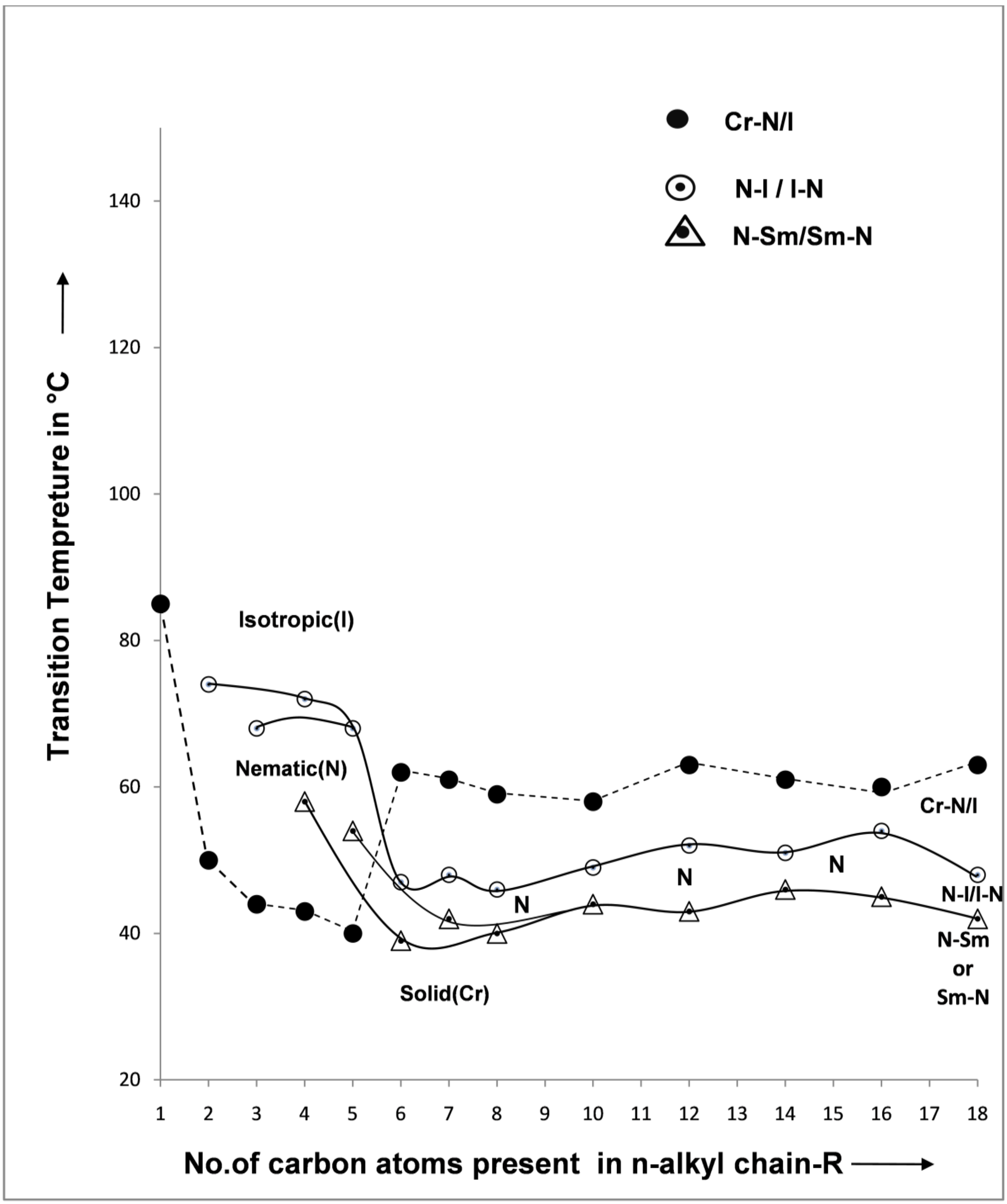


Figure 1. Phase behaviours of Series.

The changing trend in mesogenic properties from homologue to homologue in the same present novel series is observed. The exhibition of mesomorphism by C_2 to C_{18} 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 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 laterally meta substituted $–OC_{12}H_{25}$(n) tailed end unaltered.

Thus, variations in mesomorphic properties from homologue to homologue in a present novel series can be linked to the varied flexibility due to sequentially added methylene unit at the n-alkyl chain 'R' of –OR left terminal end group. The suitable magnitudes of anisotropic forces of intermolecular end to end attractions operated by dispersion forces and dipole-dipole as well as electron-electron interactions are favorably suitable to arrange the molecules of C_2 to C_5 homologues in statistically parallel orientational order above isotropic temperature and then the molecules of C_6 to C_{18} homologues arranges themselves in identically manner below isotropic temperature under floating condition to induce nematic phase.

Similarly C_4 to C_5 homologues and C_6 to C_{18} homologues arrange themselves to float with sliding layered molecular arrangement above and below isotropic temperature under floating condition respectively to induce smectogenic character. Appearance of odd-even effect upto definite homologue is due to the presence of odd and even number of carbon atoms present in n-alkyl chain 'R' of –OR group which normally remain in normal manner.

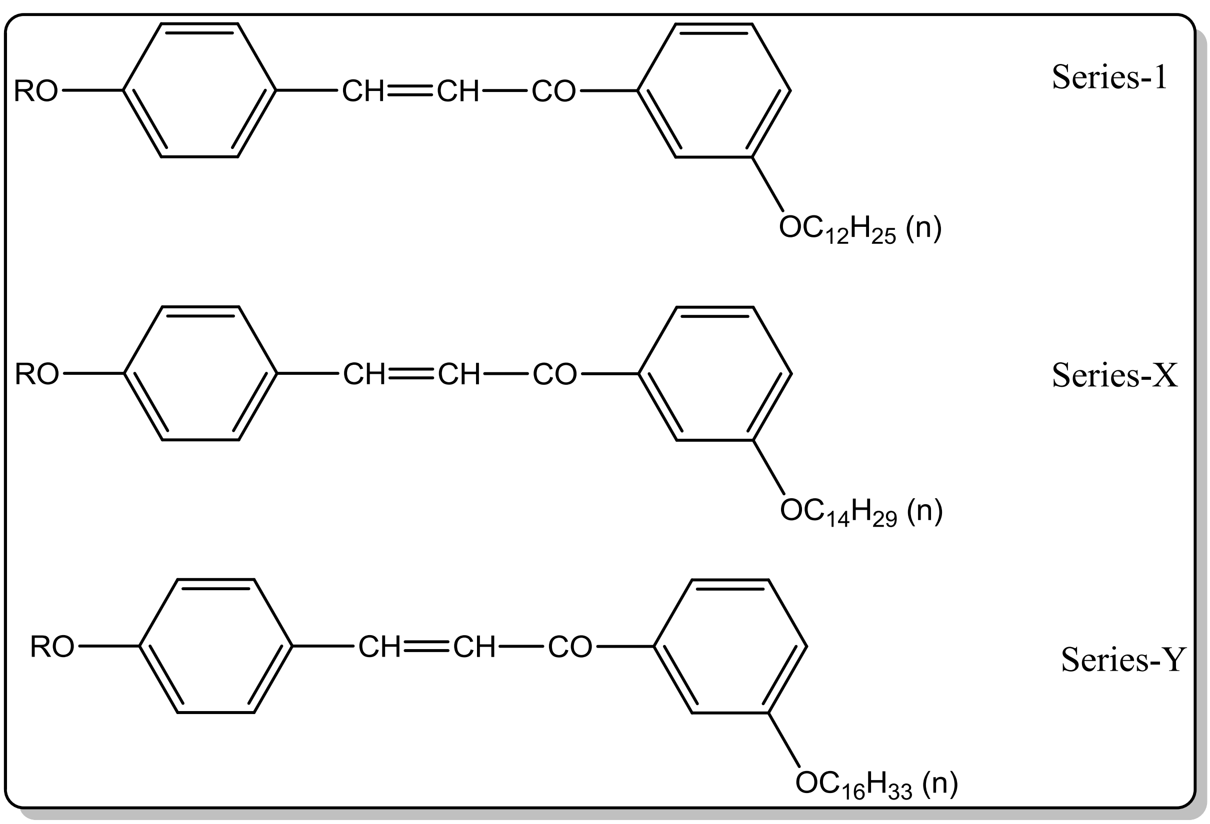


Figure 2. Structurally analogous series.

However, disappearance of odd-even effect for higher homologues of longer n-alkyl chain 'R' of –OR group and $–OC_{12}H_{25}$(n) tailed lateral group is attributed to the coiling or bending or flexing or coupling of n-alkyl chain with the major axis of core structure of a molecule. Thus, unusual status of n-alkyl chain may deviate the magnitudes of anisotropic forces of intermolecular attractions and may favour or disfavour the exhibition of mesomorphism and may alter the normal sequential order of enantiotropy and monotropy in a homologous series. The variations in thermometric mesogenic properties of presently investigated novel series -1 are compared with other structurally similar analogous series -X [30] and Y [31] as mentioned below in Figure 2.

Homologous series (1), X and Y are identical with respect to total molecular rigidity played by two phenyl rings and one central bridge –CH=CH-CO-. The identical left alkoxy terminal end group –OR for the same homologue from series to series which partly contributes to total molecular flexibility. Therefore variations in molecular flexibility arises for the same homologue from series to series is due to the changing meta substituted $–OC_{12}H_{25}$, $-OC_{14}H_{29}$ and $-OC_{16}H_{33}$ tailed ends of series 1, X and Y respectively. Therefore variations in mesogenic properties and behaviours and the degree of mesomorphism can be linked with the changing magnitudes of molecular flexibility due to changing polarity, polarizability, dipolemoments across the long molecular axis, vanderval forces etc. of n-alkoxy meta substituted tail ends from series to series for the same homologue. Some thermometric properties of series 1, X and Y are mentioned below in Table 4 as under.

Table 4. Thermal Stability in °C.

Series ⟶	Series-1 ($-OC_{12}H_{25}$)	Series-X ($-OC_{14}H_{29}$)	Series-Y ($-OC_{16}H_{33}$)
Sm-N or N-Sm Commencement of Smectic phase	56.0 ($C_4 – C_5$) C_4	Few degree 1.o to 2.0 ($C_6 – C_{18}$) C_6	1.0 or 2.0 (few degrees) ($C_7 – C_{18}$) C_7
N-I or I-N Commencement of Nematic phase	70.5 ($C_2 – C_5$) C_2	67.5 ($C_2 – C_5$) C_2	72.6 ($C_2 – C_6$) C_2
Total mesophase lengths in °C	05.0 to 29.0 $C_{10}/C_{14}C_4$	5.0 to 12.0 $C_8C_3/C_5/C_{18}$	05.0 to 21.0 $C_{18}C_4$

It is clear from Table 4 that,

- A homologous novel series - 1 is enantiotropically and monotropically smectogenic plus nematogenic whereas homologous series-X and Y are enantiotropically and monotropically only nematogenic plus only monotropically smectogenic.
- Nematogenic mesomorphism uniformly commences from C_2 member of series 1 , X and Y whereas smectogenic mesomorphism commences from C_6 and C_7 members of

the series X and Y respectively in monotropic manner and for series-1 it commences from earliest C_4 homologue in enantiotropic (C_4 and C_5) manner and then continue in monotropic (C_6 to C_{18}) manner. i.e. smectogenic mesomorphism commences later in increasing order of homologue i.e. earliest homologue in series 1 and then series X and Y.

- Thermal stability for series-1 is more than series X and Y which is very very short.
- Thermal stability and the mesophaselengths alternates from series-1 to X to Y or series Y to X to 1.

The suitable magnitudes of anisotropic forces of intermolecular attractions caused by suitable magnitudes of dispersion forces, dipole-dipole interactions, molecular polarity and differing polarizability offered by differing meta substituted $–OC_{12}H_{25}$(n), $-OC_{14}H_{29}$(n) and $–OC_{16}H_{33}$(n) groups which induces magnitudes of molecular flexibility to facilitates and stabilise the molecular arrangement required to exhibit nematic and smectic mesophase formation either in enantiotropic or/and monotropic manner under floating condition.

The early or late mesophase or mesophases appearances are attributed to the extent of molecular noncoplanarity exerted from respective molecular structures and the status of n-alkyl chains of the $–OC_{12}H_{25}$(n), $-OC_{14}H_{29}$(n) and $–OC_{16}H_{33}$(n) lateral groups of series 1, X and Y respectively, though their linearity due to the rest of the molecular part except meta substituted groups for the same homologue are identically same.

Thus, differing flexibility offered by varying lateral groups, alters the extent of molecular noncoplanarity for smectic. However, the extent of molecular noncoplanarity is ineffective for commencement of nematic phase from C_2 homologue of all the series 1, X and Y, because of its optimum magnitudes of dispersive forces and dipole-dipole interactions required to cause commencement of nematic phase. The low magnitudes of dispersion forces and same homologue C_1 of the series 1, X and Y under comparative study.

The alternation of nematic thermal stability or mesophaselengths are related with the combined effects of molecular rigidity in combition 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 or monotropic manner. Thus, present chalconyl homologous series is predominantly nematogenic and partly smectogenic of low melting type and shorter mesophaselengths.

4. CONCLUSIONS

- Novel homologous series of single central bridge linking two phenyl rings and n-alkoxy terminal/lateral groups is enantiotropically and monotropically nematogenic and smectogenic in which the sequential order of monotropy and enantiotropy is reversed than expected normal order.
- 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.

(a) Smectic

$-OC_{12}H_{25}(n) > -OC_{14}H_{29}(n) = -OC_{16}H_{33}(n)$

Nematic

$-OC_{16}H_{33}(n) > -OC_{12}H_{25}(n) > -OC_{14}H_{29}(n)$

(b) Smectic

$-OC_{12}H_{25}(n) > -OC_{14}H_{29}(n) > -OC_{16}H_{33}(n)$

Nematic

$-OC_{12}H_{25}(n) = -OC_{14}H_{29}(n) = -OC_{16}H_{33}(n)$

(c) Smectic + Nematic

Upper: $-OC_{12}H_{25}(n) > -OC_{16}H_{33}(n) > -OC_{14}H_{29}(n)$

Lower: $-OC_{12}H_{25}(n) = -OC_{14}H_{29}(n) = -OC_{16}H_{33}(n)$

- Mesomorphism is very sensitive and susceptible to the molecular structure as a consequence of molecular rigidity or/and molecular flexibility.
- Present novel compounds may be useful for the devices to be operated at room temperature or low temperature and their biological activity as antifungal or antibacterial etc. may be exploited in agricultural production to control and reduce the consumption of insecticides and pesticides.
- Present investigation supported and raised creadibility to the conclusions drawn earlier, as well as suggested the possibility of sequential order appearing mesophase as monotropy and enantiotropy in a homologous series.

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