Study of Mesomorphism in Isomeric and Nonisomeric Linear and Nonlinear Chalconyl Series

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ABSTRACT. A novel nonlinear chalconyl homologous series of ester: RO-C₆H₄-COO-C₆H₄ (m)-CO-CH:CH-C₆H₄-OC₁₆H₃₃(n) (para) have been synthesized and studied with a view to understand and establish the relation between thermotropic liquid Crystal (LC) properties and geometrically nonlinear shaped molecular structure. Homologous series consists of thirteen members (C₁ to C₁₈) whose enantiotropic nematogenic mesomorphism commences from C₆ homologue without exhibition of smectic property. C₁ to C₅ homologues are nonmesomorphic. Transition temperatures and textures of homologues were determined by an optical polarising microscopy equipped with a heating stage (POM). Transition curves of a phase diagrams (Cr-N/I and N-I) behaved in normal manner. N-I transition curve exhibited odd-even effect. Textures of a nematic phase are threaded or schlieren. Analytical and spectral data supported molecular structures of homologues. Thermal stability for nematic is 104.5°C whose degree of mesomorphism vary minimum of 13.0°C to maximum of 37.0°C. Group efficiency order for nematic is derived by comparing present novel series with structurally analogous other homologous series. Thus, homologous series of present investigation is nematogenic with absence of smectic property and of middle ordered melting type with considerable degree of mesomorphism.

1. INTRODUCTION

Study of chalconyl ester derivatives as liquid crystals (LC) [1] either of thermotropic or lyotropic type is an important from point of view of their applicability and usefulness in the benefit of mankind [2,3,4,5,6,7,8,9]. Present investigation is planned with a view to understand and establish the effect of molecular structure on LC properties of isomeric and nonisomeric, linear and nonlinear shaped homologous series of thermotropic chalconyl ester derivatives [10,11,12,13,14]. The novel chalconyl esters of nonlinear shaped will be synthesized and characterized by an optical polarising microscopy (POM) equipped with a heating stage, analytical data and spectral data from synthesis of a homologous series, which will consist of three phenyl rings, two central bridges and two terminals -OR and -OC₁₆H₃₃. Number of homologous series with one ester central group and other central bridge -CH=N-, -N=N-, -CO-CH=CH-, -CH=CH-CO-, -COO-CH₂-, -CH=CH-COO-CH₂ etc. are reported till the date [15,16,17,18,19,20,21,22,23,24,25,26]. Some thermometric data of novel homologues will be discussed and interpreted in terms of molecular rigidity and flexibility [27,28,29,30] and then, LC properties and behaviours will be compared with structurally analogous or similar other series. The group efficiency order will be derived from thermal stability, degree of mesomorphism and the commencement of mesophase.

2. EXPERIMENTAL

2.1 Synthesis:

4-n- alkoxy Benzoic acids (A) prepared by modified method of Dave and Vora [31]. α -3-hydroxyl benzoyl β -4'-Hexadecyloxy phenyl ethylene (B) was prepared by usual established method [32]. Esters were synthesized by a literature method [33]. Thus, the Chalconyl - ester

homologue derivatives were filtered, washed with sodium bicarbonate solution, dried and purified till constant transition temperatures, Alkyl halides, 3-hydroxy acetophenone, dicyclohexyl carbodimide, Dimethyl amino pyridine, DCM, MeOH, Acetone required for synthesis were used as received except solvents which were dried and distilled prior to use. The synthetic route to a series is mentioned in Scheme-1.

Reaction Scheme

Scheme 1: Synthetic route to the Series-1.

2.2 Characterization:

Representative homologues of a series were characterised by elemental analysis, Infrared spectroscopy, ^1H NMR spectra. IR spectra were recorded by 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 and LC properties (Textures) were determined using an optical polarising microscopy equipped with heating stage. Texture of some homologues for nematic phase were determined by miscibility method (Figure-1). Thermodynamic quantities enthalpy (ΔH) and entropy ($\Delta S = \Delta H/T$) are qualitatively discussed.

Analytical Data:**Table 1:** Elemental Analysis for (1) Propyloxy (2) Heptoloxo (3) Dodecyloxy and (4) Tetradecyloxy derivatives

Sr. No.	Molecular formula	Elements % found		Elements % Calculated	
		C	H	C	H
1	C ₄₁ H ₅₄ O ₅	78.52	8.60	78.59	8.62
2	C ₄₅ H ₆₂ O ₅	79.13	9.06	79.17	9.09
3	C ₅₀ H ₇₂ O ₅	79.75	9.55	79.78	9.57
4	C ₅₂ H ₇₆ O ₅	79.94	9.72	80.00	9.74

IR Spectra in cm⁻¹ for Hexaloxo & Decyloxy Derivatives:

Hexaloxo : 682 Polymethylene (-CH₂-)_n of -OC₆H₁₃ , 999 (-C-H- def. hydrocarbon in -OC₁₆H₃₃), 1114(-C-O-) Str, 1165, 1249 and 1379 (-C-O str in -(CH₂)_n chain), 1462 (-C-H- def. in CH₂), 1604, 1660 (-C=O group) and (-COO- ester group), 2848 and 2918 (-C-H str in CH₃).

Decyloxy : 721 Polymethylene (-CH₂-)_n of -OC₁₀H₂₁ ,829(-C-H- def. m di-substituted-meta), 999 (-C-H- def. hydrocarbon of -OC₁₆H₃₃), 1168 and 1257 (-C-O str in -(CH₂)_n chain, 1467(-C-H- def. in CH₂), 1510 (-C=C-)str, 1607 and 1722 (-C=O group) and (-COO- ester group), 2848 and 2972 (-C-H str in CH₃).

¹HNMR spectra in CDCl₃ in δ ppm for Octyloxy & Decyloxy Derivative:

Octyloxy : 0.87 (t, -CH₃ of -C₈H₁₇), 1.2-1.6(m, n-poly methylene groups of-OC₈H₁₇),1.8(m, n-poly methylene groups of -OC₁₆H₃₃), 3.5-3.8 (s,-OCH₂-CH₂- of -OC₁₆H₂₉), 4.01 (s,-OCH₂-CH₂-of -OC₈H₁₇), 6.51(d,-CH=CH-), 6.9-7.4 (s,-CO-CH=CH), 8.09 (s,-disubstituted phenyl ring).

Decyloxy : 0.87 (t,-CH₃ of -C₁₀H₂₁), 1.5- 1.6 (m, n-poly methylene groups of-OC₁₀H₂₁),1.81 (m, n-poly methylene groups of -OC₁₆H₃₃), 3.3-3.8(s,-OCH₂-CH₂-of -OC₁₆H₃₃),4.0 (s,-OCH₂-CH₂-of OC₁₀H₂₁), 6.8 (d,-CH=CH-) 6.9-7.4(s,-CO-CH=CH), 8.1(s,-disubstituted phenyl ring).

Table 2: Textures of Nematic phase by miscibility method for C₅, C₈, C₁₄, C₁₆.

Sr. No.	Homologue	Texture
1	C ₅	Schlieren
2	C ₈	Threaded
3	C ₁₄	Droplets of nematic
4	C ₁₆	Schlieren

Texture by POM image

Figure 1: (A) Nematic phase of C_6 at 81°C . (B) Nematic phase of C_7 at 75°C . (C) Nematic phase of C_{14} at 83°C . (D) Nematic phase of C_{12} at 80°C .

Table 3: Transition Temperatures in $^\circ\text{C}$ for Series-1.

Homologous Series: α -3-[4'-n-alkoxy benzoyloxy] phenyl- β -4''-hexadecyloxy phenyl ethylenes

Sr.no	R= n-alkyl group	Transition temperatures in $^\circ\text{C}$		
		Smectic	Nematic	Isotropic
1	C_1	-	-	134.0
2	C_2	-	-	139.0
3	C_3	-	-	142.0
4	C_4	-	-	129.0
5	C_5	-	-	132.0
6	C_6	-	81.0	118.0
7	C_7	-	75.0	107.0
8	C_8	-	82.0	110.0
9	C_{10}	-	88.0	101.0
10	C_{12}	-	80.0	106.0
11	C_{14}	-	83.0	107.0
12	C_{16}	-	72.0	96.0
13	C_{18}	-	67.0	91.0

3. RESULT AND DISCUSSION

α -3-Hydroxy benzoyl- β -4'-hexadecyloxy phenyl ethylene (m.p. 78°C , yield, 69.81 %) on condensation with dimeric 4-n-alkoxy benzoic acid yielded chalconyl esters (C_1 to C_{18}), of which C_6 to C_{18} homologues are enantiotropically nematogenic with absence of smectogenic character. Rest of the homologues (C_1 to C_5) are nonliquid crystals. The transition temperatures of novel homologues (table-2) as determined by an optical polarizing microscopy (POM) equipped with a heating stage are lower than the corresponding dimeric aromatic n-alkoxy acids. A phase diagram (Figure-2) of a novel series consisted of Cr-N/I and N-I transition curves showing phase behaviours of series are obtained by linking like or related points of the graph plotted for numbers of carbon atoms present in n-alkyl chain R of left n-alkoxy group -OR versus transition temperatures of homologues. The Cr-N/I transition curve adopts a zigzag path of rising and falling with overall descending tendency and behaved in normal manner. The N-I transition curve initially rises and falls for odd and even members till C_{10} homologue, where curves for odd and even members are

merging into each other and then N-I transition curve propagated as a single transition curve from and beyond C₁₀ homologue of longer n-alkyl chain R of left –OR flexible group and behaved in normal manner. The N-I curves for odd and even member are extrapolated to C₅ and C₄ homologues to highlight odd-even effect and to predict their probable latent transition temperatures (L.T.T.) 107.0°C and 123.0°C respectively; for nematic phase formation. The textures of nematic phase are threaded or schlieren. Analytical and spectral data confirms the structures of homologues. Thermal stability for nematic is 104.5 in average, whose, degree of mesomorphism is 13.0°C to 37°C, Thus, novel series under discussion is nematogenic and of middle ordered melting type.

Homologues series

Figure 2: Phase behaviours of Series

The lowering of transition temperatures and the disappearance of dimerization of corresponding n-alkoxy aromatic acids are attributed to the process of esterification by breaking of hydrogen bonding between two molecules of n-alkoxy benzoic acids. The nonmesomorphicity of C₁ to C₅ homologues is attributed to their high crystallising tendency which arises due to low magnitudes of dispersion forces and low dipole-dipole interaction which results into unsuitable magnitudes of anisotropic forces of intermolecular end to end or lateral attractions as a consequence of unfavourable combined effects of molecular rigidity and /or flexibility. Therefore, the molecules

of nonmesomorphs sharply transform into isotropic liquid from crystalline solid to isotropic liquid with bypassing of liquid crystalline state formation. However, the disaligned molecules of C_6 to C_{18} homologues at an angle less than ninety degree with surface of floating; resists the exposed thermal vibrations to arranges themselves in stastically parallel orientational order for definite temperature range under floating condition. Thus, nematic mesophase formation persisted for C_6 to C_{18} homologue under microscopic examination of a sample. The absence of lamellar packing of molecules in their crystal lattices causes absence of smectogenic property throughout the entire series. The molecules are randomly oriented in all possible directions from and beyond isotropic temperature, with high order of disorder or high entropy ($\Delta S = \Delta H/T$); but, on cooling the same from and below isotropic temperature in reversible manner, the nematogenic mesophase reappears at the same temperature at which mesophase was persisted on heating. Thus, a present series is enantiotropically nematogenic.

The odd-even effect observed for N-I transition curve for lower homologues is attributed to shorter n-alkyl chain 'R' by sequentially added methylene unit at the left -OR group. However, the disappearance of odd-even effect from and beyond C_{10} homologue is attributed to the unusual status of longer n-alkyl chain R of -OR due to its coiling or bending or flexing or coupling with the major axis of the core structure which affects to the molecular rigidity and/ or flexibility and, the end to end attractions. Extrapolation of N-I transition curve to the left in a phase diagram predicts LTT for C_4 and C_5 nonmesomorphs as monotropic temperatures 123°C and 107°C respectively [34,35,36,37], but, they are not realizable in actual practice because of their high crystallizing tendency. The molecular rigidity due to three phenyl rings and two central bridges are constant throughout the novel series for homologues, but the changing molecular length from homologue to homologue in the same series due to changing number of methylene units in n-alkyl 'R' chain of -OR group induces different magnitudes of flexibility contribution to total flexibility including flexibility of common $-C_{16}H_{33}$ n-alkyl chain 'R' present at the tail end group $-OC_{16}H_{33}$ at the other (right) end. Thus, effective molecular flexibility occurred due to difference of polarities between two end groups plays the role for nematic mesophase formation or in general, mesophase formation. Thus changing trends in mesomorphic behaviours of a present series from homologue to homologue is attributed to the effective flexibility of each chalconyl ester nonlinear homologue molecule. The mesomorphic behaviours of present novel series are compared with the structurally analogous series as shown below in figure-3.

Figure 3: Structurally Similar Series

Homologous series-1 and X are identical with respect to three phenyl rings, two central bridges, joining phenyl rings and two flexible terminal end groups (-OR and $-OC_{16}H_{33}$). But, they differ with respect to their geometrical shapes linear and nonlinear respectively for the same homologue from series to series. Therefore they differ with respect to combined effects of molecular rigidity and flexibility, including intermolecular distance and molecular polarizability

which operates LC behaviours of series. Homologous series-1 and Y are identical with respect to geometrical shapes, three phenyl rings, two central bridges and the left –OR group for the same homologue, but they differ with tail ended –NO₂ and –OC₁₆H₃₃ groups. Therefore, the combined effects of molecular rigidity and flexibility for the same homologue from series to series and from homologue to homologue in the same series causes difference. Thus, variations in mesomorphic (LC) properties are attributed to the magnitudes of differing features of (shapes and terminal end groups) each series for the same homologue and from homologue to homologue in the same series. Following table-4, represents some thermometric mesogenic properties of each series in comparative manner.

Table 4: Average thermal stability in °C

Series →	1	X	Y
Sm-N or Sm-I Commencement of Smectic phase	—	—	—
N-I Commencement of Nematic phase	104.5 (C ₆ - C ₁₈) C ₆	113.14 (C ₆ - C ₁₈) C ₆	158.25 (C ₃ - C ₁₆) C ₃
Degree of mesomorphism in °C from minimum to maximum	13.0 to 37.0 C ₁₀ C ₆	10.0 to 26.0 C ₈ C ₁₀	16.0 to 60.0 C ₃ C ₆

Table-4 indicates that,

- The homologous series 1, X and Y under comparison are nematogenic with absence of smectogenic property
- Mesomorphism commences earliest from C₃ homologue of a series-Y, but it commences identically from C₆ homologue for linear and nonlinear series-1 and X.
- Thermal stability for nematic are in increasing order from series-1 to series-X to series-Y.
- The degree of mesomorphism of series-1 is lower than a nonlinear series-Y and it is higher than a linear series-X.
- N-I transition curves exhibited odd-even effect for series-1 and Y except series-X.

The exhibition of nematic property and missing of smectic property by the homologous series 1, X and Y are commonly understandable by the suitable magnitudes of anisotropic forces of intermolecular end to end attractions and favourable molecular polarities and polarizability induced by terminal groups which are sufficient to arrange molecules to exhibit nematic mesophase formation under floated condition but, the same intermolecular dispersion forces and dipole-dipole interactions are insufficient to maintain sliding layered molecular arrangement in floating condition, which fails the exhibition of smectogenic character in all the series under comparison. The thermal stability of series-X is higher than a present novel series because, the intermolecular distance between any two molecules for series-X of linear shape are closer than a nonlinear series-1 i.e. end to end attractions are stronger enough to facilitate higher transition temperatures and higher mathematical value of thermal stability. A homologous series being geometrically nonlinear, the intermolecular distance factors predominates against induced polarizability factor acting against each other for the same reason of intermolecular closeness. However, the degree of mesomorphism of series-1 of present investigation is a little bit more than a series-X. This difference of

nematogenic degree of mesomorphism is attributed to the magnitudes of resistivity offered by particular homologue of favourable or unfavourable status of longer or shorter n-alkyl chain under exposed thermal vibrations. The commencement of mesomorphic phase formation takes place from C₆ homologue which suggest that, the extent of molecular noncoplanarity of series-1 and X are identically equivalent i.e. the extent of molecular noncoplanarity are ineffective by linearity of molecules for the same homologue of same terminal groups or for the isomeric series-1 and X. Now on comparing nonlinear analogous series-1 and Y, the polarity difference of terminal groups for the same homologue between –OR and –NO₂ raises or vary thermal stabilities, the degree of mesomorphism and the extent of molecular noncoplanarity which commences the mesophase formation worth earlier from C₃ homologue (series-Y) instead of C₆ homologue, which occurs in case of series-1 or series-X.

4. CONCLUSIONS

- Homologous series of present investigation of nonlinear shape, whose transition temperatures are lower than an isomeric series of linear shape. Consequently the average value of N-I thermal stability is reduced for nonlinear series than an isomeric linear series.
- Degree of mesomorphism may fluctuate depending upon the status of n-alkyl chain 'R' of –OR terminal or the group polarity situated at other end of a molecule.
- The group efficiency order derived on the basis of (a) thermal stability (b) early commencement of mesophase (c) Degree of mesomorphism for nematic are as under for the same homologue.

(a) **Nematic**

–NO₂ > linear series (–OC₁₆H₃₃) > nonlinear series (–OC₁₆H₃₃)

(b) **Nematic**

–NO₂ > linear series (–OC₁₆H₃₃) = nonlinear series (–OC₁₆H₃₃)

(c) **Nematic**

–NO₂ > nonlinear series (–OC₁₆H₃₃) > linear series (–OC₁₆H₃₃)

- Combined effect of molecular rigidity and flexibility of suitable magnitudes induces mesomorphism; respect of shape or size.
- A phenomena of mesomorphism is very sensitive and susceptible to molecular structure.
- LC property of thermotropic chalconyl derivatives can be brought down by twenty degree celsius by study of binary systems and their biological activity can be useful as antibacterial, antimalarial, anticancer etc in the pharmaceutical preparation.
- Present investigation supports and raises the credibility to the conclusions drawn earlier.

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