Effect of Equipolar Endgroup of a Molecule on Mesomorphic-Isotropic Transition Curve

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ABSTRACT: A Homologous series:RO-C₅H₄-CH=CH-COOC₆H₄-CO-CH=CH-C₆H₄-OC₁₈H₃₇(n) (para) of chalconyl novel ester of dual character as thermotropic and lyotropically bioactive is synthesized and studied with a view to understand and establish the relation between mesomorphism and molecular structure on the basis of molecular rigidity and flexibility through thermotropic liquid crystalline(LC) state. Novel series consists of thirteen homologues of which four homologues (C₁ to C₄) are non liquid crystal (NLC) and the rest of the homologues (C₅ to C₁₈) are thermotropically enantiotropic nematic without exhibition of smectic property. Thermometric transition temperatures and textures of nematogenic phase are determined by an optical polarizing microscopy equipped with a heating stage (POM).Textures of nematic phase are threaded or schlieren. Transition temperatures of novel homologues are relatively lower than the transition temperature of corresponding dimeric trans n-alkoxy cinnamic acid. Cr-N/I and N-I transition curves behaves in normal manner with negligible abnormality at C₁₈ homologue in phase diagram of a series.Analytical and spectral data conforms the molecular structure of homologues. Thermal stability for nematic is 121.88°C whose degree of mesomorphism vary minimum from 4.0°C to a maximum of 34.0 °C, and hence novel series is of middle ordered melting type. Group efficiency order for -OC₁₈H₃₇ tail groups (series-1&Y) is highest irrespective of type of ester group –COO- and CH=CH-CO derived for nematic.

1. INTRODUCTION

The study of LC [1] property of a substance have played an important role from the point of view of utility in LC devices and pharmaceutical preparations. Thus, chalconyl derivatives of thermotropic LC may be used for the manufacture of LC devices which are to be operated at room temperature or at desired temperature [2,3,4] and their bioactive nature can be exploited against pharmaceutical preparation as antibacterial ,antimalarial, anticancer etc [5,6,7].

Hence, present investigation is planned to synthesize novel chalconyl derivatives through novel homologous series and to characterize homologues by thermometric, elemental analysis, IR and H¹NMR spectra, LC properties like thermal stability, degree of mesomorphism etc. with a view to understand the effect of molecular structure on LC properties [8,9,10,11].The novel chalconyl derivatives may be further studied by the groups of researchers working with bioactivity of chalconyl ester derivatives, because present planned work will include only the study of thermotropic LC derivatives of chalcone as novel series. Number of homologous series of ester including azoester, chalconyl ester, carboxy and vinyl carboxy ester have been reported till the date [12,13,14,15,16,17,18,19,20] .The results of novel thermotropic LC derivatives will be discussed and interpreted in terms of molecular rigidity and flexibility [21,22,23,24,25] and the LC behaviours of present study will be compared with other analogous or structurally similar series for deriving group efficiency order for nematic.
2. EXPERIMENTAL

Synthesis:

4-Hydroxy cinnamic acid was alkylated using suitable alkylating agent (R-X) to convert it into 4-n-alkoxy cinnamic acids (A) by modified method of Dave and Vora[26], α-4-Hydroxy benzoyl β-4-octadecyloxy phenyl ethylene (B) was prepared by usual established method [27]. Acid chloride component of each 4-n-alkoxy cinnamic acids (A) were condensed with 4-hydroxy derivative of chalcone (B) in dry cold pyridine by usual established method [28] carefully. Thus, chalconyl ester homologue derivatives were finally decomposed, filtered, washed, dried and purified till constant transition temperatures obtained using an optical polarising microscope equipped with a heating stage. 4-Hydroxy cinnamic acid, Alkyl halides, Thionyl chloride, MeOH, EtOH, KOH, H$_2$SO$_4$, Acetone, Pyridine, 4-Hydroxy acetophenone, 4-Hydroxy benzaldehyde, Octadecyl halide 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](image-url)
3. CHARACTERIZATION:

Representative homologues of a series were characterised by elemental analysis, Infra red 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 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. Textures of nematic phase determined by miscibility method (Table-2) thermodynamic quantities enthalpy ($\Delta H$) and entropy ($\Delta S=\Delta H/T$) are qualitatively discussed.

Analytical data:

| Sr. No. | Molecular formula | Elements found % | Elements calculated % |
|---------|-------------------|------------------|-----------------------|
|         |                   | C    | H    | C    | H    |
| 1       | C$_2$             | 76.34| 8.85 | 76.27| 8.70 |
| 2       | C$_5$             | 79.01| 9.05 | 79.66| 9.036|
| 3       | C$_{12}$          | 80.01| 9.25 | 80.39| 9.67 |
| 4       | C$_{16}$          | 81.80| 10.8 | 81.49| 10.07|

IR Spectra (KBr) in cm$^{-1}$ for Heptoxy, Octadecyloxy Derivatives:

**Heptoxy:** 710 Polymethylene (-CH$_2$)$_n$ of –OC$_3$H$_{15}$, 845( –C-H- def. m di-substituted-Para), 778 Polymethylene (-CH$_2$-) of –OC$_{18}$H$_{37}$, 920 &910(-CH=CH-), 941 (–C-H- def. hydrocarbon), 1046 and 1010(-C-O-) Str, 1365 and 1303 and 1255, 1180 (-C-O str in –(CH$_2$)$_n$ chain), 1427 and 1465 (-C-H- def. in CH$_3$)1530 (-C=C-)str, 1610 and 1670 (-C=O group), 1730 (-COO- ester group), 2865 and 2935 (-C-H str in CH$_3$).

**Octadecyloxy:** 765 Polymethylene (-CH$_2$)$_n$ of –OC$_{18}$H$_{37}$, 850( –C-H- def. m di-substituted-Para),915,925(CH=CH-Group), 948 (–C-H- def. hydrocarbon), 1050 (-C-O-) Str, 1370 and 1310 and 1249, 1165 (-C-O str in –(CH$_2$)$_n$ chain, 1388 and 1460 (-C-H- def. in CH$_2$),1530 ( -C=C-)str, 1635 and 1681 (-C=O group), 1735 (-COO- ester group), 2854 and 2916 (-C-H str in CH$_3$).

$^1$H NMR spectra in CDCl$_3$ in $\delta$ ppm for Decyloxy & Dodecyloxy Derivative:

**Decyloxy:** 0.88(t, -CH$_3$ of –C$_{16}$H$_{21}$), 1.26-1.20(m, n-poly methylene groups of-OC$_{10}$H$_{21}$),1.55-1.76 (m, n-poly methylene groups of-OC$_{18}$H$_{37}$),3.8-4.0(s,-OCH$_2$-CH$_2$-ofOC$_{18}$H$_{37}$),4.0-4.2(s,-OCH$_2$-CH$_2$-of OC$_{16}$H$_{21}$),6.31-6.50(s,-CH=CH-group),7.1-7.4(s,-CO-CH=CH),8.8-8.9 (s, p-di substituted phenyl ring).

**Dodecyloxy:** 0.83(t, -CH$_3$ of –C$_{18}$H$_{37}$),0.96(t,-CH$_3$ of –OC$_{12}$H$_{25}$),1.3-1.4(m, n-poly methylene groups of -OC$_{18}$H$_{37}$),1.5 -1.7(m, n-poly methylene groups of- OC$_{12}$H$_{25}$),3.6(s,OCH$_2$ ),3.9(s,-OCH$_2$-CH$_2$-of OC$_{12}$H$_{25}$),4.1-4.4(s,-OCH$_2$-CH$_2$-of OC$_{18}$H$_{37}$),6.21-6.30(s CH=CH-),6.9-7.2(s, -CO-CH=CH),7.5-7.7(s, p-substituted phenyl ring).

Texture determination by miscibility method:
C$_{10}$,C$_5$ and C$_7$,...thread nematic
C16 and C18 schlieren nematic.
4. RESULTS AND DISCUSSION

Homologous series of present investigation is formed by the condensation of n-alkoxy cinnamic acids and the α-4-Hydroxy benzoyl β-4''-octadecyloxy phenyl ethylene (M.P 74°C, Yield 72%). The transition temperatures of novel chalcony ester derivatives of a series are nematogenic LC from and beyond Pentyloxy homologue (C_5 to C_18) with absence of smectic property. C_1 to C_4 homologue are nonliquidcrystal. Transition temperatures (table-2) as determined from an optical polarizing microscopy were plotted versus the number of carbon atoms present in n-alkyl chain bonded to first phenyl ring through oxygen atom (-OR). A phase diagram is obtained from Cr-N/I and N-I transition curves by linking like or related transition points. Cr-N/I transition curve adopted zigzag path of rising and falling with descending tendency and behave in normal manner.

Table 2: Transition Temperatures in °C for Homologous series:

| Compound No. | n-Alkyl chain C_{n}H_{2n+1} | Transition Temperatures in(°C) |
|--------------|-----------------------------|--------------------------------|
|              |                             | Smectic | Nematic | Isotropic |
| 1            | C_1                         | -       | -       | 132.0     |
| 2            | C_2                         | -       | -       | 138.0     |
| 3            | C_3                         | -       | -       | 134.0     |
| 4            | C_4                         | -       | -       | 130.0     |
| 5            | C_5                         | -       | 120.0   | 128.0     |
| 6            | C_6                         | -       | 114.0   | 123.0     |
| 7            | C_7                         | -       | 112.0   | 124.0     |
| 8            | C_8                         | -       | 107.0   | 118.0     |
| 9            | C_10                        | -       | 116.0   | 120.0     |
| 10           | C_12                        | -       | 108.0   | 118.0     |
| 11           | C_14                        | -       | 96.0    | 120.0     |
| 12           | C_16                        | -       | 88.0    | 122.0     |
| 13           | C_18                        | -       | 100.0   | 124.0     |

Homologous Series: RO—CH=CH—COO—CO—CH=CH—OC_{18}H_{37} (n)

α-4-(4’-n-Alkoxy cinnamoyloxy) benzoyl-β-4’’-octadecyloxyphenyl ethylenes
N-I transition curves for odd (C₅, C₇) and even (C₆…C₈) members are matching into each other at the C₁₀ homologue and than gradually ascended up to C₁₈ homologue as a single transition curve from and beyond C₁₀ homologue. Thus, it exhibits odd-even effect and deviated negligibly from and beyond C₁₂ homologue from normal descending tendency. Textures of nematogenic homologues are threaded or schlieren as determined by miscibility method. Analytical and spectral data confirms the molecular structures of homologues. Odd member’s N-I transition curve occupied higher position as compared to even member’s transition curve. The mesogenic (LC) properties from homologue to homologue cause variations keeping –OC₁₈H₃₇ tail end constant and changing –OR tail group at the left. Thermal stability for nematic is 121.88 °C, with low degree of mesomorphism, which varied from 4.0°C to 34.0°C. Disappearance of dimerization of aromatic acids and the lowering of transition temperatures of resultant chalconly esters derivatives are attributed to the breaking of hydrogen bonding between acid molecules by way of esterification process. The inexhibition of any sort of mesophase formation by C₁ to C₄ homologues is attributed to their inability to resist exposed thermal vibration due to unsuitable magnitudes of intermolecular anisotropic end to end or/and lateral forces of cohesion and closeness as emerging from low dispersion forces and low dipole-dipole interactions, which induces high crystallizing tendency in a substance to transform into isotropic liquid sharply from crystalline state on heating and on cooling the same from isotropic liquid to directly solid crystalline state without passing through an intermediate state of existence called as LC state. However, the molecules of C₅ to C₁₈ homologues
disalign at an angle less than ninety degree as a consequence of favorable molecular flexibility, which resisted exposed thermal vibrations due to suitable magnitudes of anisotropic end to end forces of intermolecular attractions, and causes to arrange the molecules in statically parallel orientational order under floated condition on surface, depending upon enthalpy ($\Delta H$) value of LC state within definite range of temperature, called as mesophase length of a homologue. Thus, nematogenic mesophase formation occurs excluding the possibility of smectic mesophase formation which is supposed to form prior to nematic mesophase occurrence. However due to insufficient magnitude of intermolecular lateral forces of attractions as required for lamellar packing of molecules in their crystal lattices of rigid crystalline state is deficient and hence, smectogenic mesophase formation is missing through out to series under dictions Odd-even effect observed for N-I transition curve of phase diagram is attributed to the odd and even number of carbon atoms present in n-alkyl (R) chain of the left n-alkoxy group. The disappearance of odd-even effect from and beyond C$_{10}$ homologue of the longer n-alkyl chain is attributed to the unusual status of the alkyl chain, which may coil or bend or flex or couple to lie with the major axes of the core structure of molecules, resulting into end to end attraction and intermolecular distance almost equivalent. Hence a single transition curve appears with diminishing tendency of odd-even effect. The changing trend in mesomorphic properties from homologue to homologue in same series is attributed to the sequential addition of methylene units at the left n-alkoxy terminal (-OR) end groups which alters molecular length, molecular polarity and polarizability, molecular flexibility, permanent dipole moment across the long molecular axis, dipole dipole interaction, magnitude of intermolecular dispersion forces of cohesion and closeness etc. including uncertainty in the status of an alkyl chain(R) of –OR group. The LC properties of present novel series are compared with structurally analogous series as mentioned below in figure-2.

**Figure 2 Structurally Similar Series**

Homologous series 1, X and Y are identical with respect to three phenyl rings, central bridge –CO–CH=CH and left n-alkoxy terminal end group for the same homologue from series to series. Homologous series 1 and X are identical with respect to first central bridge –CH=CH-COO- including all above other, respect except right side flexible tail end group –OC$_{18}$H$_{37}$ and -OC$_{14}$H$_{29}$ respectively, which differs in molecular flexibility for the same homologue from series to series. Homologous series 1 and Y are identical in all respect for the same homologue except a central bridge linking first and middle phenyl rings viz. –CH=CH-COO- and –COO- respectively contributing to the total molecular rigidity. Thus, the difference of LC properties and the degree of mesomorphism can be correlated with differing features and differing magnitudes of molecular rigidity and/or flexible of molecular structure among the series 1, X and Y respectively for the same homologue from series to series and homologue to homologue in the same series. Some thermotropic LC properties are mentioned below for the comparative study in table-3.
Table 3:-Relative thermal stability in °C

| Series                          | I       | X       | Y       |
|--------------------------------|---------|---------|---------|
| Sm-I or Sm--N                  |         |         |         |
| Commencement of Smectic phase   |         |         |         |
| Nematic-isotropic (N-I)        | 121.88  | 99.75   | 122.0   |
| Commencement of nematic phase  | (C₅-C₁₈)| (C₆-C₁₈)| (C₅-C₁₈)|
|                                 | C₅      | C₆      | C₅      |
| Total degree of mesomorphism   | 04.0 to 34.0 | 19.0 to 28.0 | 7.0 to 38.0 |
| From Ci to Cp                  | C₁₀     | C₆      | C₇      |
|                                | C₁₆     | C₈      | C₁₂     |

Table-3 indicates that,

- Homologous series 1, X and Y under comparison are nematogenic only with absence of smectogenic properties from series to series.
- Mesomorphic properties (Nematic) commences from C₅ homologue for series1 and Y where as, it commence from C₆ homologue of a series-X.
- Thermal stability of series-1 and Y are almost nearer (21.88≈22.0) to each other ,but it is lower for series-X than the a series-1 and Y.
- Degree of mesomorphism of series 1 and Y are almost equivalent but it is lower for a series-X than series 1 and Y.
- N-I transition curves deviates from normal descending behavior in a negligible manner at the homologue C₁₈, C₁₄ and C₁₉ in which both terminal end groups are equipolar and polarizable for series 1, X and Y respectively.

Exhibition of only nematic property in case of series.1 and X is depended upon the difference of polarities of flexible tail end group –OR, -OC₁₄H₂₉. Hence magnitude of flexibility difference occurred due to n-alkoxy (-OR where R’=C₁₄H₃₇ and C₁₄H₂₉) tail end group for the same homologue from series-1 to series-X which reduces nematic thermal stability and the degree of mesomorphism. The extent of molecular noncoplanarity difference as occurred due to unusual status of n-alkyl chain of tail end groups –OC₁₈H₃₇ and –OC₁₄H₂₉ have operated early (C₅) and late (C₆) commencement of series-1 and X respectively. Now on comparing the molecular structure of series-1 and Y in which the central bridge linking first and middle phenyl ring –CH=CH-COO- and –COO- differs keeping rest of the molecular parts including both terminal tail end groups(-OR and –OC₁₈H₃₇) unaltered. Therefore, central bridge –CH=CH-COO- of series-1 is replaced by –COO- of series-Y which contributes partly to the difference of molecular rigidity. The vinyl carboxylate central group of present novel series-1 is relatively longer than carboxylate central.bridge of series-Y, which causes more noncoplanarity but the presence of another common central bridge –CO-CH=CH- which bears conjugated double bond and maintains almost equal noncoplanarity of molecules of both series-1 and Y under comparison. Thus, the effect of vinyl carboxylate group against carboxylate group toward the extent of noncoplanary of the molecular operate equally for commencement of LC phase from C₅ homologue of series 1 and Y.The negligible difference of thermal stability (121.88 and 122.0°C) is also attributed to the negligible difference of molecular rigidity due to presence of common central group-CO.CH=CH- which minimize the difference of effectivity of lengths between –CH=CH-COO- and –COO- group as a consequence of minor difference molecular rigidity, due to replacement of central ester group. In all these homologous series under comparative study, the molecules of mesogenic homologues exhibited nematogenic mesophase formation is attributed to the disalignment of molecules at an angle less than ninety degree which resists exposed thermal vibrations and arrange molecules to float on the surface with statistically parallel orientational order .But the angle of molecular disalignment with floating surface which vary from series to series and homologue to homologue as a consequence of the magnitudes of changing molecular rigidity and /or flexibility of different magnitudes which affects...
to the molecular resistivity toward exposed thermal vibrations. Thus, nematogenic property vary for
the same homologue from series to series and from homologue to homologue in the same series. The
negligible deviation from normal descending behaviours of N-I transition curve for a homologue,
bearing same polar group present at the both terminal end groups is attributed to the vector sum of
polarities of all bonds in equipolar unique group which is equal in magnitudes but opposite in
directions which nullifies the effect of each other. This factors affects flexibility of a homologue and
induces more or less or negligible deviation in N-I or M-I transition curve.

Conclusions:

- Homologous series of present investigation is nematogenic only with normal degree of
  mesomorphism and of middle order melting type.
- An unique homologue of a chalconyl ester series with equipolar both terminal end groups show
  more or less or negligible deviation from normal descending tendency.
- The group efficiency order derived on the basis of (a) thermal stability (b) early commencement
  LC phase and (c) Degree of mesomorphism for nematic are as under.

Nematic
(a) Series1 ≈ series-Y > series-X
(b) series-1 = series-Y > series-X
(c) series-1≈ series-Y > series-X

- A phenomena of mesomorphism is very sensitive and susceptible to molecular structure
  depending upon the suitable magnitude of molecular rigidity and/or flexibility.
- Study of binary systems of present investigation may prove its utility in LC devices and
  exploitation of biological activity of present compounds may prove their importance in
  pharmaceutical preparation.
- Present investigation supports and raises the credibility to the conclusions drawn earlier.

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