



Mesogenic Dependence of Chalconyl Schiff's Bases on Molecular Rigidity and Flexibility

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ABSTRACT

A novel homologous series $R_0-C_6H_4.CH:N-C_6H_4.CO.CH:CH-C_6H_4-OC_6H_{13}(n)$ of liquid crystal property is synthesized and studied with a view to understand and establish the effect of molecular structure on liquid crystal properties with respect to Schiff base and chalconyl central bridges as well as changing left n-alkoxy terminal and right n-hexyloxy fixed tail ended group. Homologous series consisted of eleven (C_1 to C_{16}) homologues in which the nematogenic mesomorphism commences from C_3 homologue and continued to last C_{16} homologue without exhibition of smectogenic property. Rest of the C_1 and C_2 homologues are nonmesomorphic, whose Latent transition temperature (LTT) are predicted by extrapolation of N-I transition curves. Odd-even effect is observed for N-I transition curve. N-I transition curve and Cr-N/I transition curve behaved in normal manner. The Nematic thermal stability is $72.2^\circ C$. The minimum and maximum nematogenic mesophase lengths are ranging between $12.0^\circ C$ and $30.0^\circ C$ at C_{16} and C_{14} homologue respectively. Transition temperatures were determined by an optical polarizing microscopy (POM), equipped with a heating stage. Thermal analytical and spectral data confirms the molecular structures of homologues. The novel homologous series of chalconyl Schiff's base is low melting series, whose mesogenic transition temperatures ranges between $43.0^\circ C$ and $79.0^\circ C$.

Keywords: Mesomorphism, Nematic, Liquid Crystals, Enantiotropy, Smetic

1. INTRODUCTION

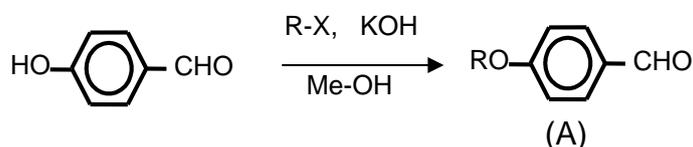
Liquid crystal (LC) property [1] and its degree of LC of a substance is operated by favourable magnitudes of molecular rigidity and/or flexibility [2-5] depending upon molecular structure [6-11] of a substance concerned, which differs with more or less extent from substance to substance in the same or difference series. Therefore present investigation is planned with a view to understand and established the effects of molecular structure on LC property and the degree of liquid crystallinity, by –selecting unexploited and perhaps never exploited moiety of chalconyl schiff's bases homologous series by replacing nonester –COO- or –CH=CH-COO- central group through –CH=N- and chalconyl –CO-CH=CH- group. Number of homologous series with –COO- and –N=N- or –COO- and –CH=N- have –COO- and –CH=CH-CO- or –COO- or –COO-CH₂- or –CH=CH-COO-CH₂- etc have been reported [12-21] till the date.

2. EXPERIMENTAL

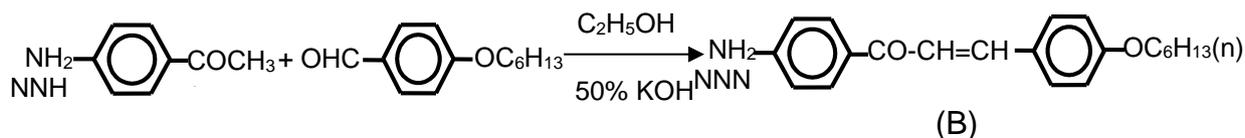
Synthesis

4-n-Alkoxy benzaldehyde (A) were synthesized from 4-hydroxy benzaldehyde using corresponding suitable alkylating agent (RX) by the modified method of Dave and Vora [22]. α -4-Amino benzoyl – β -4'-Hexyloxy phenyl ethylene (B) (Chalcone) was prepared from 4-amino acetophenone and 4-Hexyloxy benzaldehyde by known condensation [23] method. 4-n-Alkoxy benzaldehyde (A) and α -4-Hydroxy benzoyl – β -4'-Hexyloxy phenyl ethylene (B) were condensed in 1,3-dicyclohexylcarbodiimide (DCC), 4-dimethylaminopyridine (DMAP) and CH₂Cl₂ [24]. Synthetic route to series is mentioned below as Scheme 1. Final products were individually filtered, washed, dried and purified till the constant transition temperatures obtained.

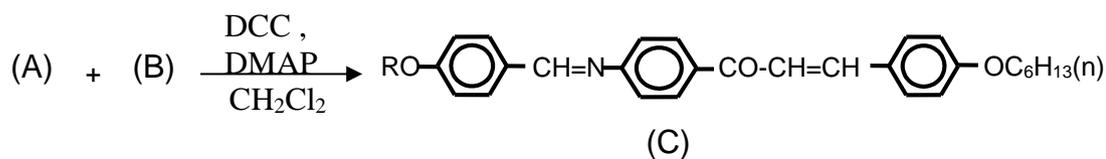
(1) Synthesis of n-Alkoxy Benzaldehyde [22]



(2) Synthesis of α -4-Amino benzoyl – β -4'-Alkoxy phenyl aldehyde [23]



(3) Synthesis of α -4-[4'-n-Alkoxy Benzoyloxy] β - benzoyl - 4''- Animo phenyl Ethylenes [24]



where: R = C₁, C₂, C₃, C₄, C₅, C₆, C₇, C₈, C₁₀, C₁₂, C₁₄, C₁₆.

Scheme 1. Scheme of synthesis (Figure 1)

Characterisation

Some selected members of the titled series were characterized by elemental analysis, ¹H NMR spectra and IR spectra, Textures of mesophases were characterized by miscibility method. Microanalysis for C, H, N, elements were performed on Perkin Elmer PE 2400 analyzer (Table 1). ¹H NMR spectra were obtained on Bruker spectrometer using CDCl₃ as solvent. IR spectra were recorded on a Perkin- Elmer spectrum GX. Transition temperatures and liquid crystal properties were investigated Using optical polarizing microscope with heating stage.

Table 1. Elemental analysis for the butyloxy, pentyloxy and hexyloxy derivatives

Molecular formula	Elemental % found (Compared with % calculated)	
	C	H
C ₃₂ H ₃₇ O ₄ N	79.35 (79.50)	7.28 (7.66)
C ₃₃ H ₃₉ O ₄ N	79.39(79.67)	7.30 (8.84)
C ₃₄ H ₄₁ O ₄ N	79.62 (79.84)	7.88 (8.02)

Analytical Data

¹H NMR: in ppm. For Octyloxy homologue.

Ethylenes: (200 MHz) δ (CDCl₃) (ppm) 1.10 (-CH₃-CH₂ of -C₈H₁₇), 1.40 (long -CH₂- chain), 3.0 -OCH₂ of (-OCH₂ of C₈H₁₇), 6.88 & 6.50, 8.60 & 8.18 (p-sub. benzene rings)

¹H NMR: in ppm. For Decyloxy homologue.

Ethylenes: (200 MHz) δ (CDCl₃) (ppm) 1.05 (-CH₃-CH₂ of -C₁₀H₂₁), 1.59 (long -CH₂- chain), 3.53 -OCH₂ of (-OCH₂ of C₈H₁₇), 6.29 & 6.45, 8.45 & 8.20 (p-sub. benzene rings)

IR in cm⁻¹,

IR Spectrum For Octyloxy homologue

Ethylenes: ($\nu_{\max}/\text{cm}^{-1}$): 2989, 2879, 1475, 1340 (-C-H, aliphatic), 1737, 1266 (ester group), 1744 (>C=O group), 1764 (-C=N-), 1030,1248 (ether group), 857.2 (p- sub. benzene ring), 1622,1483, 1455 (Aromatic ring).

Spectrum For Decyloxy homologue

Ethylenes: ($\nu_{\max}/\text{cm}^{-1}$): 2935, 2832, 1437, 1355 (-C-H, aliphatic), 1708, 1238 (ester group), 1745 (>C=O group), 1725 (-C=N-), 1023,1242 (ether group), 844.6 (p- sub. benzene ring), 1632,1462, 1480 (Aromatic ring)

Table 2. Transition Temperatures in °C

Compound No	n-alkyl ($\text{C}_n\text{H}_{2n+1}$) group	Smectic	Nematic	Isotropic
1	C_1	-	-	89.0
2	C_2	-	-	72.0
3	C_3	-	48.0	63.0
4	C_4	-	43.0	69.0
5	C_5	-	50.0	68.0
6	C_6	-	59.0	75.0
7	C_8	-	52.0	76.0
8	C_{10}	-	61.0	77.0
9	C_{12}	-	60.0	79.0
10	C_{14}	-	43.0	73.0
11	C_{16}	-	58.0	10.0

3. RESULT AND DISCUSSION

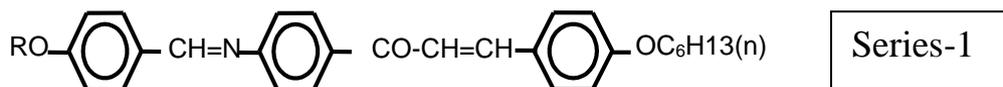
A novel chalconyl Schiff's base homologous series consisting of eleven homologues was synthesized using 4-n-alkoxy benzaldehydes, 4-amino acetophenon and 4-n- hexyloxy benzaldehyde. C_3 to C_{16} homologues are enantiotropically nematogenic with absence of smectogenic character. Transition temperatures of homologues as determined by POM were plotted versus the number of carbon atoms present in n-alkyl chain[-R] of left n-alkoxy[-OR] group. Cr-N/I transition curve followed a zigzag path of rising and falling manner with overall falling tendency and behaves in normal manner. N-I transition curve initially ascended and then, descended from and beyond C_{12} homologue. Thus, N-I transition curve also behaved in normal expected manner. N-I transition curve was extrapolated [25-30] to nonmesomorphic homologues C_1 and C_2 to predict their probable LTT transition temperatures to exhibit mesomorphism below respective isotropic temperatures. Thus, probable LTTs predicted for C_1 and C_2 homologues may be 59.0 °C and 63.0 °C respectively in monotropic manner. Odd-even effect is observed for a novel series upto C_7 member of a series at which N-I transition curve for odd and even members of a series matches into each other and then, a single N-I transition curve propagated from and beyond C_7 homologue. Thermal, analytical and spectral data supported molecular structures of the homologues. Thermal stability in average for nematic is 72.2 °C and the total mesophase length ranges between 12.0 °C to 30.0

°C at the C₁₆ and C₁₄ homologues of a series. Thus, present novel series is a low melting series and the shorter mesophaselengths.

The commencement and the exhibition of mesomorphism depend upon the suitable magnitudes of anisotropic forces of intermolecular attractions as a result of favourable magnitudes of molecular rigidity and the flexibility, which disaligns the molecules at an angle ninety degree or less than ninety degree with the plane of their floating surface under the influence of exposed thermal vibrations. In present novel homologous series, the molecular are disaligned at an angle less than ninety degree resisting exposed thermal vibrations reversibly on heating and cooling process throughout the series except C₁ and C₂ homologues. Therefore, the only statistically parallel orientational order of molecules by end to end attractions is facilitated in reversible manner. Therefore, only enantiotropically nematogenic mesophase formation facilitated from C₃ to C₁₆ homologues.

The absence of smectogenic property throughout the series under present investigation is attributed to the absence of lamellar packing of molecules and hence, the of appearing sliding layered molecular arrangement either in enantiotropically or the monotropically due to inadequate value of dipole-dipole interactions and low magnitude of dispersion forces failst. Thus, in their preoccupied crystal lattices appearance of smectogenic mesophase formation is to be eliminated or not facilitated. The absence of smectic and nematic mesophase or nonmesomorphic property in case of C₁ and C₂ is attributed to the low magnitudes of dipole-dipole interactions and the low magnitudes of dispersion forces, which causes unsuitable magnitudes of anisotropic forces of intermolecular end to end attractions even in monotropic condition to exhibit mesophase formation. The appearance of odd-even effect and alternation of transition temperature is attributed to the presence of even and odd numbered -CH₂- unit or number of carbon atoms in n-alkyl (-R) chain bonded to phenyl ring through oxygen atom. Such effectiveness appears upto C₇ homologue.

The N-I transition curves for add and even members of a series matches at the C₇ member of a series and a single N-I transition curve follows from and beyond C₇ member of a series. Thus, the disappearance odd-even effect of higher homologues of longer n-alkyl(R) chain is attributed to the unusual status of n-alkyl chain(-R) by coiling or bend or couple to lye with core structure of a molecule, Which, causes uncertainty in the status of molecular length and the relative intermolecular cohesive forces responsible to facilitate mesophase formation. The variations of mesomorphic properties or mesophaselength from homologue to homologue in the same series is attributed to the changing number of carbon atoms in n-alkyl(-R) chain keeping the rest of the molecular part unaltered which, affects the length to breadth ratio, molecular polarity and polarizability, favourable magnitudes of molecular rigidity and flexibility, permanent dipolemoment across the long molecular axis, dipole-dipole interactions etc. Such effects respond variations in the mesomorphic behaviours, properties and the degree of mesomorphism, from one homologue to another in the same series. The mesomorphic properties like thermal stability, commencement of mesophase, total mesophaselength range or the degree of mesomorphism etc. of present novel series (1) are compared with structurally similar analogous series-X[31] and Y[32] mentioned below in Figure 2.



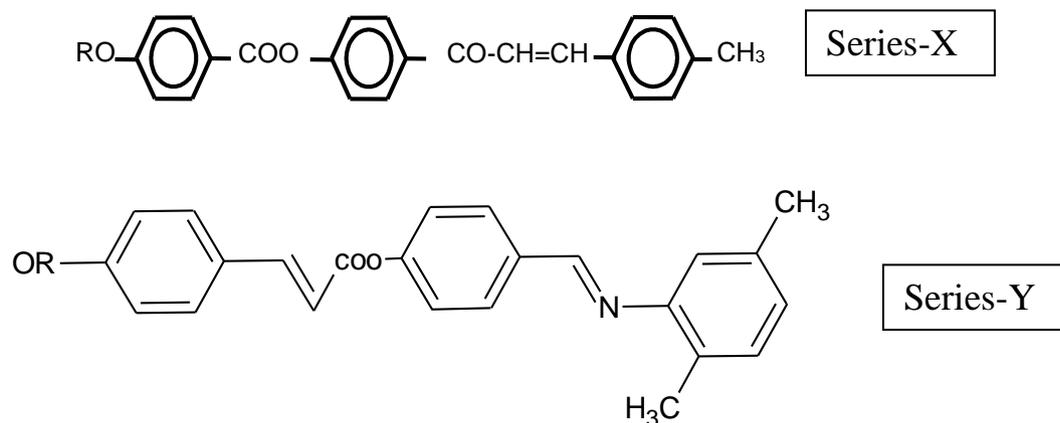


Figure 2. Structurally similar homologous series

Homologous series-1 of present investigation and the homologous Series-X and Y chosen for comparison are identical with respect to three phenyl rings, left n-alkoxy terminal end group for the same homologue and one of the central group either $-\text{CH}=\text{N}-$ or $-\text{CO}-\text{CH}=\text{CH}-$ which are identically similar partly in their molecular rigidity. However they differ with respect to one of the central groups to which $-\text{CH}=\text{N}-$ is replaced by $-\text{COO}-$ or $-\text{CO}-\text{CH}=\text{CH}-$ is replaced by $-\text{CH}=\text{CH}-\text{COO}-$ which affects total molecular rigidity. Moreover, the molecular flexibility from homologue to homologue in the same series and the changing tail ended terminal or lateral group from series to series are varied drastically. Therefore, the variations in mesomorphic properties and the degree of mesomorphism can be correlated with the variations caused by varying magnitudes of molecular flexibility and rigidity as a result of more or less alternation corresponding to respective molecular structures. Following table-3 represents comparatively some liquid crystal properties for Series-1, X and Y as mentioned above.

Table 3. Average Thermal Stability in $^{\circ}\text{C}$.

Series	Series-1 ($-\text{OC}_6\text{H}_{13}$)	Series-X ($-\text{CH}_3$)	Series-Y (2,5-Dimethyl)
Sm-I or Sm-N Commencement of smectic phase	--	139.42 ($\text{C}_1 - \text{C}_{16}$) C_1	100.16 ($\text{C}_8 - \text{C}_{16}$) C_8
N-I Commencement of nematic phase	72.2 ($\text{C}_3 - \text{C}_{16}$) C_3	--	110.83 ($\text{C}_5 - \text{C}_{10}$) C_2

Total mesophaselengths in °C	12.0 to 30.0 C ₁₆ C ₁₄	3.5 to 79.6 C ₁ C ₄	4.0 to 41.0 C ₁₈ C ₁₂
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Above table indicates that,

- Homologous Series-1 and X are only nematogenic or Smectogenic respectively without exhibition of smectic or nematic property respectively; whereas a homologous series Y is smectogenic as well as nematogenic in character.
- Thermal stability for smectic decrease from Series-X to Y to 1 and for nematic increases from Series-X to 1 to Y.
- Smectogenic mesophase commences from C₁ and C₈ homologues in Series-X and Y respectively but it does not commence till the last C₁₆ homologue of present Series-1.
- Nematogenic mesophase commences from C₃ and C₂ homologues for present Series-1 and a Series-Y respectively, but, it does not commence till the last member of a series-X.
- The total lower mesophaselengths decreases from Series-1 to Series-Y to Series-X whereas the upper mesophaselength increases from Series-1 to series-Y to Series-X.

The exhibitin of only nematogenic mesophase by a homologous Series-1 of present investigation and the only smectogenic mesophase by the homologues Series-X chosen for comparison is attributed to the disalignment of molecules at an angle less than ninety degree or ninety degree under exposed thermal vibrations while floating on the surface, depending upon resistivity offered towards exposed heat, due to combined effect of individual molecular rigidity and flexibility for the same homologue from series to series; as a result of induced molecular polarity and polarizability. Weaker intermolecular attractions of Series-1 arranges its molecules only in statistically parallel orientational its order, whereas the intermolecular forces resulted from molecular polarity and polarizability provides lamellar packing of molecules under identical condition for the same homologue of Series-X and favours layered molecular net working arrangement which occupy sliding layered molecular arrangement on heating for definite range of temperatures, giving rise to formation of smectic mesophase only. However the magnitudes of residual intermolecular attractions are insufficient for the including effect due to the presence of -COO- central bridge molecules of Series-X; which do not allow to facilitate the formation of nematic mesophase or ester homologous series are (carboxy ester -COO-) generally smectogenic. However, the molecular polarity and polarizability offered by 2,5 dimethyl tail ended group in which both laterally substituted methyl groups of Series-Y are more polarizable as compared to the polarizability offered by para substituted only one methyl group of Series-X and -C₆H₁₃ (n) group of Series-1 under comparison. Therefore, residual force of intermolecular attractions offered by the same homologue of Series-Y which is optimum and more suitable to induce nematic mesophase and smectic mesophase respectively as compared to same homologue of Series-1 and X which are nonsmectogenic and nonnematogenic respectively. Thus, a homologous Series-Y facilitated the formation of enantiotropic nematic mesophase at higher temperature in addition to smectic mesophase as compared to the enantiotropically facilitation and formation of only

nematic mesophase as compared to the enantiotropically facilitation and formation of only nematic or smectic mesophase. The para substituted tail ended methyl group of Series-X adopts geometrical linear lath like shape and relatively less polarizable as compared to the a Series-Y; bearing 2,5 dimethyl tail ended laterally substituted methyl groups; which increases the molecular width and decreases intermolecular attractions as compared to the intermolecular attractions exerted by the molecules of linear lath like molecules of Series-X for the same homologue. Thus, Sm-N transition temperatures of Series-X being higher than a Series-Y. therefore, the Sm-N average thermal stability of Series-X is higher than a Series-Y selected under comparative study. The lowest Sm-N thermal stability or absence of lamellar packing and smectic mesophase formation by series-1 fails to acquire any Sm-N or Sm-I values of thermal stability. The N-I transition temperature of Series-Y are higher than a series-1, because shorter $-\text{CH}=\text{N}-$ central bridge of Series-1 of present investigation is replaced by longer and more polar and polarizable $-\text{CH}=\text{CH}-\text{COO}-$ central bridge of Series-Y, which comparatively raises N-I transition temperatures for the same corresponding homologues.

Thus, suitable magnitudes of anisotropic forces of intermolecular attractions of the molecules of Series-Y are higher than the corresponding molecules of Series-1. Consequently average thermal stability for nematic(N-I) of Series-Y is more than a Series-1 or the N-I thermal stability of series-1 is lower than the N-I thermal stability of series-Y. Hence, thermal stability of mesophase represents thermal resistivity of a substance, towards exposed thermal vibrations or the magnitudes of (thermodynamically) internal energy of a system stored or possessed by a molecule which is used or silent against externally exposed thermal energy depending upon its characteristics and mass involved. Early or late commencement of smectic mesophase depends upon the extent of molecular noncoplanarity.

The molecules of para methyl tail ended Series-X are more coplanar or less noncoplanar than corresponding smectogenic homologues of Series-Y with reference to tail ended 2,5-dimethyl group, which reduces the possibility of commencing smectic mesophase late from C_8 homologue as compared to commencement of smectic mesophase from C_1 homologue of Series-X. The geometrical shape of molecular structure of Series-1 bearing longer n-alkyl i.e.- $\text{C}_6\text{H}_{13}(\text{n})$ bonded through oxygen, eliminates the possibility of occurring smectogenic mesophase formation.

Therefore, smectic mesophase does not commence till the last C_{16} homologue of presently investigated Series-1. Decrease in the lower mesophase length and increase in the upper mesophase length for the same homologue from series to series are in the order of Series-1 to Series-Y to Series-X which are depended upon the mesophase stabilization or destabilization as a result of individual molecular polarity and polarizability induced as a result of individual molecular polarity and polarizability induced a consequence of their individual molecular rigidity and flexibility, as occurred from dipole-dipole interactions, permanent dipole moment across long molecular axes, molecular length and breadth, ratio of the polarity to polarizability shape of molecule etc. which operates or control the suitable(not more or less than optimum value) magnitudes of anisotropic forces of intermolecular end to end or lateral attractions for the stabilization or destabilization and the degree of mesomorphism.

Thus, above parameters collectively causes variation in mesomorphic property and the degree of mesomorphism for the same homologue from series to series as well as from homologue to homologue in the same series.

4. CONCLUSIONS

- Presently investigation novel chalconyl schiff's base homologous series is thermally mesomorphic as nematic only with low thermal stability and low melting series whose mesophase length vary between 12.0⁰C to 30.0⁰C and transition temperatures of mesomorphism vary minimum of 43.0⁰C to the maximum of 79⁰C at the C₄ and C₁₂ homologues respectively.
- The group efficiency order derived on the basis of (i) thermal stability(ii) early commencement of smectic and/or nematic phase and the (iii) total mesophase length are as under.

(i) Smectic

Series-X > Series-Y > Series-1

Nematic

Series-Y > Series-1 > Series-X

(ii) Smectic

Series-X > Series-Y > Series-1

Nematic

Series-Y > Series-1 > Series-X

(iii) Total mesophase length-

Lower: Series-1 > Series-Y > Series-X

Upper: Series-X > Series-Y > Series-1

- Mesomorphism is very sensitive and susceptible to molecular structure including isomeric molecular structures.
- LC compounds of present study are applicable for the devices to be operated at room temperature or at desired temperatures using study of binary systems.
- Molecular rigidity and flexibility controls the suitable magnitudes of anisotropic forces of intermolecular attractions.
- Present study may contribute as novel study in the field of biological activity against bodily disordered health of mankind.
- Thus, present investigation raises credibility to the conclusions drawn earlier and adds some contribution to the existing literature of LC and biological activity.

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