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Molecular Structural Flexibility Dependence of Mesomorphism through Ortho-Substituted Nitro Group

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ABSTRACT

A novel homologous series of liquid crystalline materials of schiff's bases viz. $RO-C_6H_4-CH=CH-COO-C_6H_4-N=CH-C_6H_4-NO_2$ is synthesized and studied with a view to understanding and establishing the effect of molecular structure on liquid crystal properties and to provide novel thermotropic LC material to the scientific and technological community of research interest. The novel series consist of 12 homologues. All members of the series are enantiotropically smectogenic character. The transition temperatures were determined by an optical polarizing microscopy equipped with a heating stage. Thermal stability of smectogenic mesophase is 132.2 °C and mesophase length is varies between 4.7 °C to 32.7 °C. The LC behaviour of the novel series compared with a structurally similar known series.

Keywords: Liquid crystal, Smectogenic, Nematogenic, Thermotropic, Mesophase

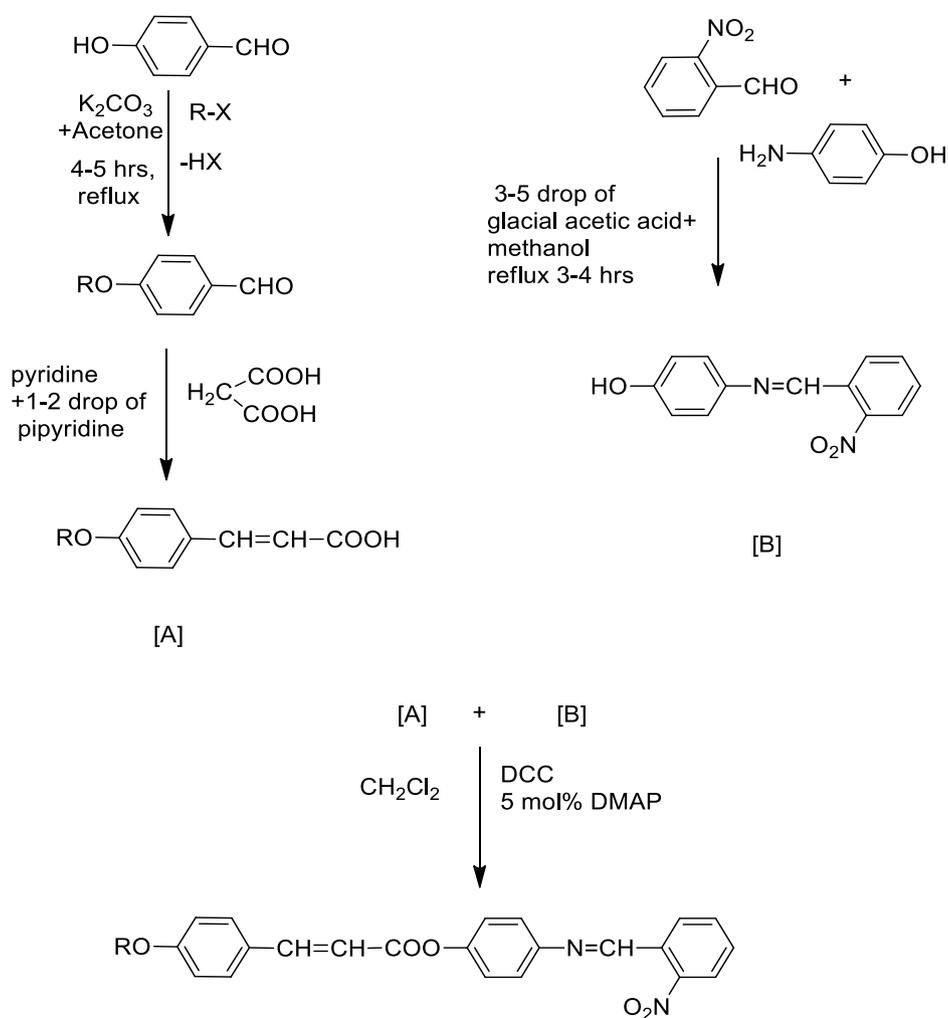
1. INTRODUCTION

The liquid crystalline state of a substance is known since 1888 [1]. The unique properties as to flow as liquid and optically to behave as crystal in display devices [2, 3] and the biologically system including biochemical reactions occurring in the body [4-9] which play an important role in living entities. Thus in order to extend and continue research activities of scientist and

technocrats, novel LC substances are required to produce and exploited in the interest of mankind. The present investigation is planned with a view to understanding and establishing the effects of molecular structure on mesomorphism of a substance through the synthesis of novel homologous series. Several homologous series have been reported [10-17, 31] of varying moieties by changing the molecular rigidity and flexibility [18-21] through variation of the number of phenyl, heterocyclic or hydrocarbon rings, the central groups the terminal or lateral groups, which change the shape, size, length, polarity and polarizability [22-24, 32-38]. Evaluated results can be useful to the other groups of research's who are working with LC substances because every researcher in LC's required novel substances.

2. EXPERIMENTAL

2. 1. Synthesis



Where $R=C_nH_{2n+1}$, $n= 1$ to 8,10,12,14&16

Scheme 1. Synthetic route to the series

4-*n*-Alkoxybenzaldehydes were synthesized by refluxing 4-hydroxybenzaldehyde (1 equiv.) with corresponding *n*-alkyl bromides (1 equiv.) in the presence of potassium carbonate (1 equiv.) and acetone as a solvent [25]. The resulting 4-*n*-alkoxybenzaldehydes were reacted with malonic acid (1.2 equiv.) in the presence of 1–2 drops piperidine as catalyst and pyridine as solvent to yield corresponding *trans*-4-*n*-alkoxy cinnamic acids (A) [26]. 2-Nitrobenzyl-4'-hydroxy aniline (B) was prepared by an established method [27] M.P. 140-145 °C, Yield 76.5%. Coupling of compound A and B is done by Steglich esterification to yield 4-(4'-*n*-alkoxy cinnamyloxy)-2''-nitro benzal aniline [28, 29]. The synthetic route to the novel homologous series of Schiff's base cinnamoyl ester derivatives is under mentioned in Scheme 1.

2. 2. Characterization

Some of the members of novel series were characterized by elemental analysis (Table 1), Infrared spectroscopy, ¹H NMR spectra, and mass spectroscopy. Microanalysis was performed using a EuroEA Elemental Analyzer. IR spectra were recorded on Shimadzu FTIR Model-IRAffinity-1S (MIRacle 10). ¹H NMR spectra were recorded on a Bruker spectrometer using CDCl₃ as a solvent and mass spectra were recorded on Shimadzu GC-MS Model No.QP-2010. The liquid crystal behaviour and the type of textures were determined by a miscibility method on microscopic observations by using modern optical polarizing microscope Rankem Nikon model NO. LV100NPOL.

Analytical data

Table 1. Elemental analysis for (1) Butyloxy (2) Heptyloxy (3) Octyloxy derivatives.

Sr. No.	Molecular formula	Element % found				Element % calculated			
		C	H	N	O	C	H	N	O
1	C ₂₆ H ₂₄ N ₂ O ₅	70.23	5.42	6.38	18.02	70.26	5.44	6.30	18.0
2	C ₂₉ H ₃₀ N ₂ O ₅	71.76	6.56	5.56	15.98	71.84	6.63	5.58	15.95
3	C ₃₀ H ₃₂ N ₂ O ₅	72.18	6.82	5.46	15.48	72.21	6.84	5.43	15.51

Spectral Data

¹H NMR in ppm for Pentyloxy Derivative

0.91-0.96 (t, 3H, -OC₅H₁₁ group), 1.36-1.47(m, 4H, CH₃-(CH₂)₂-CH₂-CH₂-O-), 1.74-1.80 (m, 2H, CH₃-(CH₂)₂-CH₂-CH₂-O-), 3.96-4.02 (t, 2H, CH₂-O-), 6.48-6.52 (d, 1H, -CH=CH-COO-), 7.82-7.86(d, 1H, -CH=CH-COO-), 8.96 (s, 1H, -CH=N-), 6.88-6.92 & 7.52&7.55 (d, 4H, phenyl ring containing pentyl chain), 7.22-7.26 & 7.32-7.36 (d, 4H, middle phenyl ring), 8.08-8.10 (d, 1H, O-NO₂ containing phenyl), 7.61-7.66 (dd, 1H, m-NO₂ containing phenyl), 7.73-7.77 (d, 1H, p-NO₂ containing phenyl), 8.30-8.32 (d, m-NO₂ containing phenyl).

¹H NMR in ppm for Dodecyloxy Derivative

0.86-0.90 (t, 3H, -OC₁₂H₂₅ group), 1.25-1.34 (m, 18H, CH₃-(CH₂)₉-(CH₂)-CH₂-O-), 1.78-1.80 (m, 2H, CH₃-(CH₂)₉-(CH₂)-CH₂-O-), 3.98-4.02 (t, 2H, CH₂-O-), 6.48-6.52 (d, 1H, -CH=CH-COO-), 7.82-7.86 (d, 1H, -CH=CH-COO-), 8.96 (s, 1H, -CH=N-), 6.92-6.94 & 7.53-7.55 (d, 4H, phenyl ring containing dodecyl chain), 7.22-7.26 & 7.33-7.35 (d, 4H, middle phenyl ring), 8.08-8.10 (d, 1H, O-NO₂ containing phenyl), 7.63-7.65 (dd, 1H, m-NO₂ containing phenyl), 7.73-7.75 (d, p-NO₂ containing phenyl), 8.30-8.32 (d, 1H, m-NO₂ containing phenyl).

IR in cm⁻¹ for Hexyloxy Derivative

3109-3039 (C-H str. of alkene disubstituted), 2862-2931 (C-H str. of (-CH₂)_n group of -OC₆H₁₃), 1728 (C=O str. of carbonyl carbon of ester group), 1627-1604 (C=C str. of alkene), 1592 & 1574 (C=C str. of aromatic ring), 1309 & 1265 (C-H bending of alkene), 1226 (C-O str. of ether linkage), 1134 (C-O str. of ester group), 979 (C-H bending of trans alkene), 1342 & 1512 (N-O stretching), 1288-1257 (C-H bending), 833 (para disubstituted Ar ring). The IR data are consistent with the molecular structure.

IR in cm⁻¹ for Decyloxy Derivative

3039 (C-H str. of alkene disubstituted), 2924 & 2854 (C-H str. of (-CH₂)_n group of -OC₁₀H₂₁), 1735 (C=O str. of carbonyl carbon of ester group), 1629 (C=C str. of alkene), 1597 & 1512 (C=C str. of aromatic ring), 1342 & 1311 (C-H bending of alkene), 1203 (C-O str. of ether linkage), 1134 (C-O str. of ester group), 979 (C-H bending of trans alkene), 856 (para disubstituted Ar ring). The IR data are consistent with the molecular structure.

Mass spectra of Ethyloxy Derivative

m/z (rel.int%): (415.9M⁺), 175 (base peak), 240.9, 147.

Mass spectra of Tetradecyloxy Derivative

m/z (rel.int%): (584M⁺), 343 (base peak), 240.9, 147.

3. RESULTS AND DISCUSSION

Schiff's base 2-nitrobenzal-4'-hydroxy aniline is a nonmesomorphic, which on condensation with trans 4-n-alkoxy cinnamic acids yielded mesomorphic novel homologues throughout the entire series in enantiotropically smectogenic with absence of nematogenic property. Transition temperature of homologues (Table 2) were plotted against the number of carbon atom present in n-alkyl chain of the left n-alkoxy group, a phase diagram (Fig. 1) consisting of Cr-Sm and Sm-I transition curve are obtained on linking the like or related points. Cr-Sm transition adopts a zigzag path of rising and falling with overall descending tendency. Sm-I transition behaved normal manner with exhibition of odd-even effect. Sm-I transition curve for odd and even numbers merges into each other at the octyloxy (C₈) homologue of series and then it prolong as a single curve till C₁₆ homologue. Sm-I transition curve for odd member's occupied lower position as compared to a curve for even number. Thus, Cr-Sm and Sm-I transition curved behaved in normal manner.

Thermal stability for smectic is 132.2 °C and the smectogenic mesophase length ranges minimum of 4.7 °C at the hexadecyloxy (C₁₆) homologue to a maximum of 32.7 °C at Hexyloxy (C₆) homologue. Thus, present novel series is smectogenic and middle order melting type. The mesogenic property from homologue to homologue varies in present series with changing molecular flexibility by –OR end group.

Table 2. Transition temperatures in °C

Compound No.	R=n-alkyl chain C _n H _{2n+1}	Transition temperatures in °C		
		Sm	N	Isotropic
1	1	158.6	-	168.0
2	2	148.5	-	173.5
3	3	136.3	-	150.1
4	4	133.2	-	150.4
5	5	118.1	-	123.0
6	6	109.8	-	142.5
7	7	100.5	-	112.8
8	8	107.5	-	118.0
9	10	102.7	-	109.8
10	12	98.8	-	128.4
11	14	96.7	-	105.3
12	16	103.8	-	108.5

Sm - Smectic; N - Nematic;

The mesomorphic properties commence from the very first member of the novel series and vary from homologue to homologue due to the changing the length of left n-alkoxy terminal end group. None of the homologues show monotropic mesomorphism and nonmesomorphic behaviors. The absence of nematogenic mesophase formation and the exhibition of only smectic mesophase formation very first member of a present series is attributed to the unsuitable magnitudes of anisotropic force of intermolecular end-to-end attractions as a consequence of combined effect of unfavourable molecular rigidity and flexibility which are not sufficiently

strong enough to maintain statically parallel orientational order of molecules in floating condition to induce nematic property. However, the favourable molecular rigidity displayed by the central bridges which link the phenyl ring through at least one conjugated double or multiple bond as well as overall sufficient molecular polarizability which favour the formation of lamellar packing of molecules in their crystal lattices from very first member (C_1) of the series to its last member (C_{16}).

Thus, the molecule of C_1 - C_{16} homologues with suitable magnitude of intermolecular anisotropic forces are sufficient to facilitate originally layered order in their crystal lattice which acquire sliding layered molecular arrangement under exposed thermal vibration for certain definite range of temperatures; depending upon molecular resistivity toward heat or exposed thermal vibrations or the force holding the plane of the layers together in the rigid crystal which are weakened relative to the attraction among the molecule within a given layer at soled mesomorphic transition temperature and the layers may become free to slide and rotate over one another giving rise to smectic mesophase formation and then on heating the smectic phase to a higher temperature the intermolecular force between the slides of the molecule weakened to such an extent that resultant intermolecular force do not permit molecules to slide out of the layer and statically parallel orientational order is not maintained at all; but entire sample of a substance transform into isotropic liquid.

Conversely on cooling the isotropic melt, the smectic phase reappears exactly at a temperature at which mesophase was disappeared in reversible manner in case of all the homologues (C_1 - C_{16}) of the present novel series.

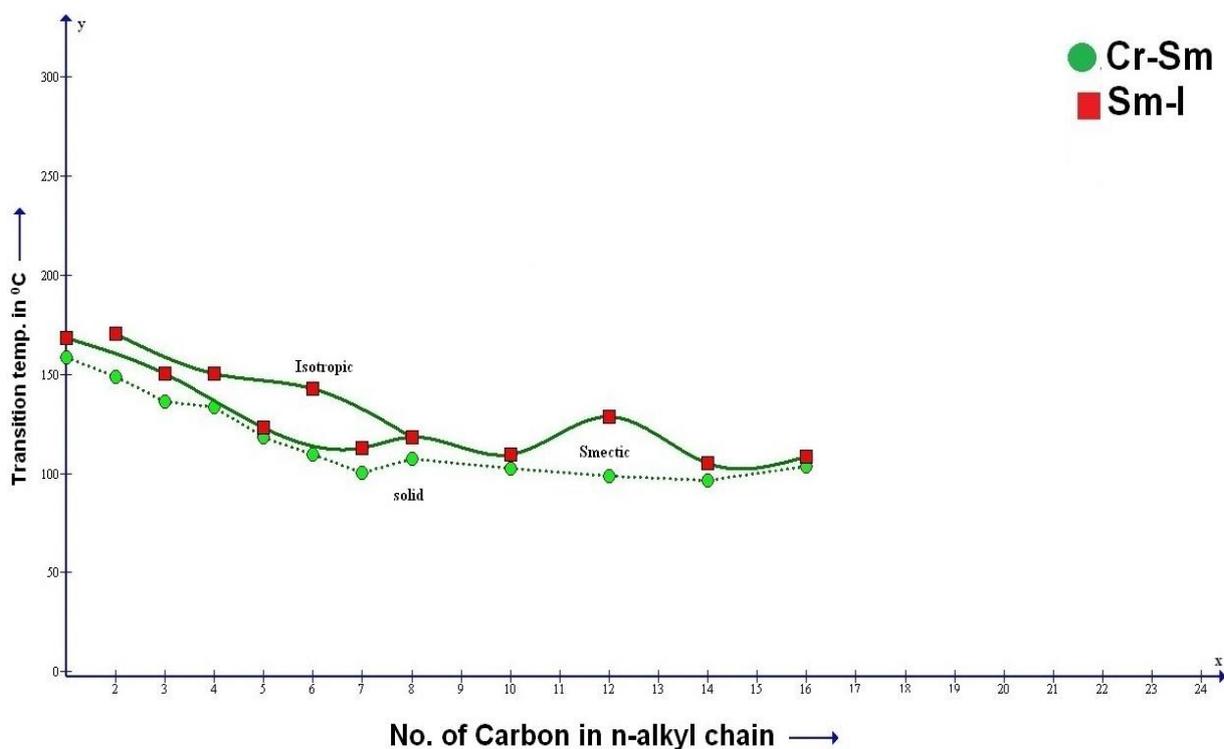


Figure 1. Phase behavior of series

The shorter n-alkoxy terminal end group lies with the major axis of the core, and the end-to-end contact ultimately differs for odd and even homologues through C₁-C₇. Therefore, an odd-even effect is observed. However, the odd-even effect disappears from C₈ because the longer alkyl chain may coil, flex or bend to lie with the major axis of the core and the end-to-end contact would then be the same for odd and even homologues. The sequential addition of a methylene unit contributes toward molecular flexibility and the resultant combined effect of molecular rigidity and flexibility, which vary from homologue to homologue causing changing trend in mesomorphic behaviors.

Some mesomorphic properties of presently investigated novel mesogenic series 1, are compared with the structurally similar other known series X [30] as mentioned in Figure 2.

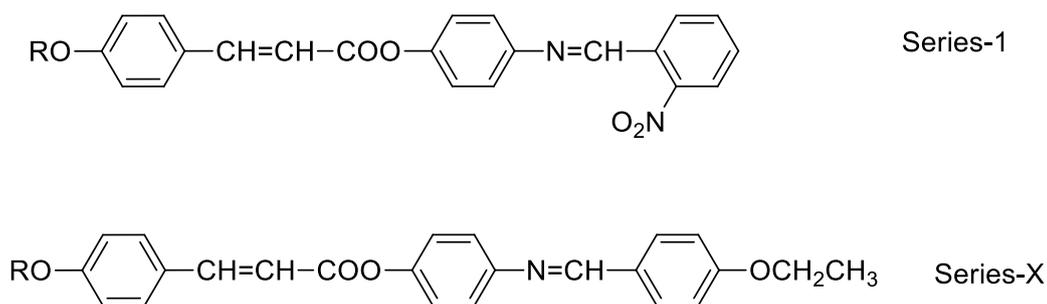


Figure 2. Structurally similar series.

Homologous series 1 and X chosen for comparative study are identical with respect to three phenyl rings and two central bridges $-\text{CH}=\text{CH}-\text{COO}-$ and $-\text{N}=\text{CH}-$ as well as the left n-alkoxy (-OR) terminal end group for the same homologues, but differ with respect to polar groups $-\text{NO}_2$ and $-\text{OCH}_2\text{CH}_3$ substituted at an ortho or para position respectively of series 1 and X. Thus molecular rigidity of series 1 and X remains unaltered, but molecular flexibility alters for the same homologue from series to series. Thus, the mesogenic properties and the degree of mesomorphism as depending on the suitable magnitude of combine effect of molecular rigidity and flexibility, which varies with molecular structure for the same homologue from series to series and from homologue to homologue in the same series. Table 3 represent some thermotropic property of presently investigated series 1 and the series X selected for comparative study.

Table 3. Relative thermal stability in °C.

Series →	1	X
Smectic-Isotropic Or Smectic-Nematic	132.2 (C ₁ -C ₁₆)	122.5 (C ₁₂ -C ₁₈)
Commencement of smectic mesophase	C ₁	C ₁₂

Nematic-Isotropic	-	270.7 (C ₁ -C ₁₈)
Commencement of nematic phase	-	C ₁
Total Mesophase length range (Sm+N)	4.7 (C ₁₆) to 32.7 (C ₆)	111.0 (C ₁₈) to 194.0 (C ₇)

Table 3 indicate that,

- Series X is smectogenic plus nematogenic phase while series 1 is only smectogenic.
- Smectic property commences from C₁ and C₁₂ homologues respectively for series 1 and X.
- Nematic property for series X commence from C₁ homologue while it does not commences till the last homologue of series 1.
- Smectic thermal stability are in decreasing from series 1 to series X.
- Nematic thermal stability of series X is 270.7 °C, while nematic mesophase formation did not stabilise and facilitate for series 1.
- Total mesophase length of series X is higher than series 1.

On comparing thermal stability and other thermotropic properties of series 1 than series X, which are relatively higher for series X than series 1 as dependent on molecular flexibility only, keeping molecular rigidity unchanged or constant for the same homologue from series to series. The molecular structure including central bridge and number of phenyl rings for the same homologue, contributing to molecular rigidity are same but, molecular flexibility induced by ortho substituted –NO₂ of series 1 and para substituted –OCH₂CH₃ group of series X. whose polarities and polarizability differed to induced liquid crystalline state in differing suitable magnitudes of anisotropic forces of intermolecular attractions.

The ortho substituted –NO₂ group increases intermolecular distances which lower transition temperatures, resulting into reduction of thermal stability of present series 1, where as para substituted –OCH₂CH₃ tail group maintain linearity of a molecule, which strengthens intermolecular cohesion and closeness. Therefore transition temperature and restitivity toward exposed thermal vibrations are rised more for the same homologue from series 1 to X. Therefore thermal stability and mesophase length ranges for the series X are relatively higher than the present series 1. As regard to the commencement of smectic phase this depends upon the extent of molecular noncoplanarity which varied with positional difference of functional group of different polarities and polarizability for the same homologue from series to series.

The intermolecular dispersion and dipole-dipole interaction of residual forces beyond upper transition of smectic phase induces nematic mesophase formation of different magnitudes of suitable magnitudes, which causes nematic mesophase formation from very first member C₁ to last member C₁₈ of series X, But it fails to induce nematic phase in case of series 1 depending upon thermodynamical quantity (ΔH) associated with molecular structure. Thus, thermotropic properties of mesomorphic substances purely depend upon magnitudes of favourable molecular

rigidity and flexibility as a result of even minor difference of molecular structure, either for same homologue for series to series or from homologue to homologue in the same series.

4. CONCLUSIONS

- Novel homologues series of Schiff's base is entirely smectogenic whose smectic thermal stability are relatively high and of middle order melting type.
- The group efficiency order derived on the basis of (i) thermal stabilities (ii) commencement of mesophase and (iii) Total upper and lower degree of mesophase length range for smectic and nematic are as under.
 - i) Smectic: series 1 > series X
Nematic: series X > series 1
 - ii) Smectic: series 1 > series X
Nematic: series X > series 1
 - iii) (Smectic+Nematic): series X > series 1
- Suitable magnitudes of combined effect of molecular rigidity and flexibility can induce mesomorphism.
- A phenomenon of mesomorphism is very sensitive and susceptible to molecular structure as a consequence of resultant molecular rigidity and flexibility.
- Study is useful to researchers working a liquid crystalline state as a source of LC material.

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