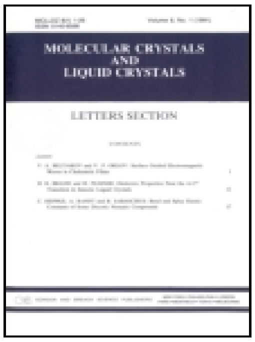

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Molecular structural flexibility dependence of mesomorphism through ortho-substituted bromo group

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ABSTRACT

A novel series of liquid crystal (LC) materials of Schiff's bases viz. RO-C_6H_4–CH=CH–COO–C_6H_4–N=CH–C_6H_4–Br (ortho) is synthesized and studied with a views 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 consists of thirteen members (C_1 to C_8, C_{10}, C_{12}, C_{14}, C_{16} & C_{18}). All the members of a series are enantiotropic liquid crystals. Smectic (C_{10}–C_{18}) and nematic (C_1–C_{18}) property commence from C_{10} and C_1 homologue respectively. Transition temperatures and the textures of homologues were determined using a polarizing optical microscope (POM) equipped with a heating stage. Transition curves Cr-M, Sm-N and N-I of a phase diagram behaved in normal manner. Odd–even effect is absent for Sm-N transition curve, but it is present for N-I curve. Sm-N transition curve is extrapolated to C_8 and C_6 nonsmectogenic homologues to determine and predict their latent ability for exhibition of smectic property. Analytical and spectral data confirms the molecular structures of homologues. Thermal stabilities of smectic and nematic are 85.4°C and 130.1°C °C whose upper and lower total mesophase length vary from 58.0°C to 06.0°C at the C_{10} and $C_1 = C_5 = C_6$ homologue, respectively. Textures of nematic phase are threaded or Schlieren and smectic phase are of type A or C. LC properties of present novel series are compared with structurally series known series. Thus, present novel series is predominantly nematogenic and partly smectogenic with middle ordered melting type and considerable total mesophase length range.

KEYWORDS
Liquid crystal; schiff's base; smectic; nematic; LTT

Introduction

Studies of Liquid Crystal (LC) substances [1] of the type either thermotropic or lyotropic have developed considerable interest due to their applications in the benefit of mankind. The unique properties as to flow as liquid and optically to behave as crystal in display devices [2, 3] and the biological systems including biochemical reactions occurring in the body [4–10] which plays an important role in the living entities. Thus, in order to extend and continue research activities of scientists and technocrats, novel LC substances are required to produce and exploited in the interest of mankind. Present investigation is planned with a view and

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aim to understand and establish the effects of molecular structure [11–14] on LC properties through synthesis of novel homologues series of thermotropic LC, which, provide the source of novel LC materials for researchers working with different aims, objects and views at different angles and ideas as well as skills of developing relations between mesomorphism and the novel molecular structures. Number of homologous series have been reported till the date [15–20]. Among them present novel series will consist of three phenyl rings bonded through –CH=CH–COO– and –N=CH– central bridges and ortho-substituted lateral –Br as tail group with varying –OR group. The effect of flexible lateral group, simultaneously with change of n-alkyl chain will be studied by POM and will be characterized through spectral and analytical data as a consequence of changing molecular flexibility with or without constant molecular rigidity [21–24]. Mesomorphic behaviors of novel series will be evaluated and compared with structurally similar series.

Experimental

Synthesis

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–Bromobenzal-4′-hydroxy aniline (B) was prepared by an established method [27] [M.P.181-183°C, Yield-74.8%]. Coupling of compound A and B is done by Steglich esterification to yield 4-(4′-n-alkoxy cinnamoyloxy) 2‴-bromobenzal aniline [28].

The synthetic route to the novel homologous series of Schiff's base cinnamoyl ester derivatives is under mentioned in Sch. 1.

Characterization

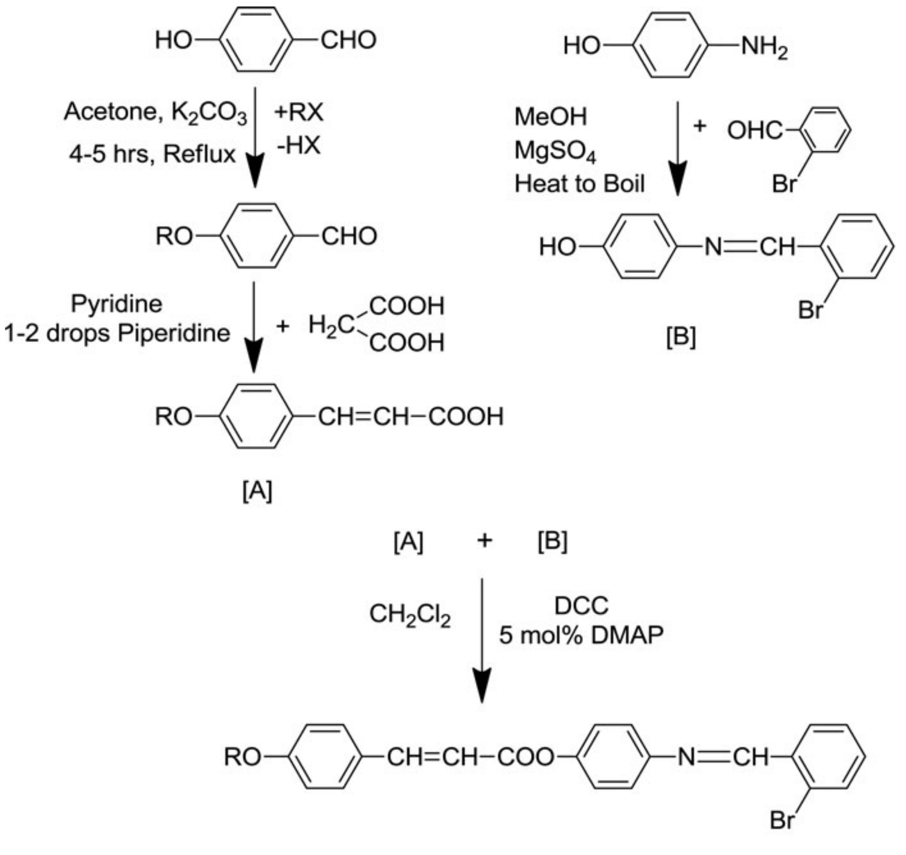
Some of members of a novel series as the representative member of a series were characterized by elemental analysis (Table 1), Infrared spectroscopy, ^{1}HNMR spectra and mass spectroscopy. Microanalysis was performed on EuroEA Elemental Analyzer. IR spectra were recorded on Shimadzu FTIR Model -IRAffinity-1S (MIRacle 10), ^{1}HNMR spectra were recorded on Bruker spectrometer using $CDCl_3$ as a solvent and mass spectra were recorded on Shimadzu GC-MS Model No.QP-2010. The liquid crystal behavior and the type of textures were determined by miscibility method on microscopic observations.

Analytical data

Spectral data

^{1}HNMR in ppm for ethyloxy derivative

1.20–1.35 (t, 3H, $-CH_3$ of $-OC_2H_5$ group), 4.03–4.09 (m,2H,$CH_3\underline{-CH_2}-O$), 7.51–7.53 (d,1H, $\underline{-CH}=CH-COO-$), 6.47–6.51 (d,1H, $-CH=\underline{CH}-COO-$), 8.85 (s,1H, $-N=\underline{CH}-$), 6.87–7.66 (4H, phenyl ring containing ethoxy chain), 7.25–7.33(4H, middle phenyl ring), 7.38–7.85 (4H, phenyl ring containing bromo ring). The NMR data are reliable with the molecular structure.



Where, R=C_nH_{2n+1}, n=1 to 8, 10, 12, 14, 16 and 18

Scheme 1. Synthetic route to the novel series

Table 1. Elemental analysis for (1) Propyloxy (2) Octyloxy (3) Dodecyloxy derivatives.

		Elements% Found			Elements% Calculated		
Sr. No.	Molecular formula	C	H	O	C	H	O
1	$C_{25}H_{22}BrNO_3$	64.73	4.75	10.30	64.66	4.78	10.34
2	$C_{30}H_{32}BrNO_3$	67.47	5.99	8.96	67.41	6.03	8.98
3	$C_{34}H_{40}BrNO_3$	69.22	6.79	8.09	69.15	6.83	8.13

^{1}HNMR in ppm for hexyloxy derivative

0.89–0.93 (t, 3H, $-CH_3$ of $-OC_6H_{13}$ group), 1.25–1.29 (m, 2H, $CH_3\text{–}\underline{CH_2}\text{-}(CH_2)_4\text{–}O\text{–}$), 1.32–1.36 (p, 2H, $\underline{\text{–}CH_2}\text{-}(CH_2)_3\text{–}O\text{–}$), 1.43–1.50 (p, 2H, $\underline{\text{–}CH_2}\text{-}(CH_2)_2\text{–}O\text{–}$), 1.66–1.79 (p,2H,$\underline{\text{–}CH_2}\text{–}CH_2\text{–}O\text{–}$), 3.97–4.01 (t,2H,$\text{–}CH_2\text{–}O\text{–}$), 7.52–7.54 (d,1H, $\underline{\text{–}CH{=}}CH\text{–}COO\text{–}$), 6.48–6.52 (d,1H, $\text{–}CH{=}\underline{CH}\text{–}COO\text{–}$), 8.86 (s,1H, $\text{–}N{=}\,CH\text{–}$), 6.91–7.63 (4H, phenyl ring containing hexyloxy chain), 7.28–7.34(4H, middle phenyl ring), 7.39–7.86 (4H, phenyl ring containing bromo ring). The NMR data are reliable with the molecular structure.

IR in cm^{-1} for methyloxy derivative

3064 (C–H str. of alkene disubstituted), 2933 & 2841 (C–H str. of $-OCH_3$), 1734 (C=O str. of carbonyl carbon of ester group), 1639 (C=C str. of alkene), 1597 & 1504 (C=C str. of aromatic ring), 1319 & 1249 (C–H bending of alkene), 1211 (C–O str. of ether linkage), 1141 (C–O str. of ester group), 1029, 985 & 920 (C–H bending of alkene), 671 (C–Br str.). The IR data are consistent with the molecular structure.

IR in cm^{-1} for octyloxy derivative

3059 (C–H str. of alkene disubstituted), 2920 & 2850 (C–H str. of $(-CH_2-)_n$ group of $-OC_8H_{17}$), 1726 (C=O str. of carbonyl carbon of ester group), 1597 (C=C str. of alkene), 1506 (C=C str. of aromatic ring), 1309 & 1267 (C–H bending of alkene), 1190 (C–O str. of ether linkage), 1118 (C–O str. of ester group), 1026, 999 & 970 (C–H bending of alkene), 673 (C–Br str.). The IR data are consistent with the molecular structure.

Mass spectra of propyloxy derivative

m/z (rel.int%): 465 $(M)^+$,276,189,167,147,119,91.

Mass spectra of decyloxy derivative

m/z (rel.int%): 563$(M)^+$, 287,276,196,147,119,91.

Results and discussion

Schiff's base 2-bromobenzal-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 enantiotropic manner. C_{10} to C_{18} homologues are enantiotropically smectogenic in addition to nematogenic whereas, C_1 to C_8 exhibited only nematogenic character with absence of smectogenic property, even in the monotropic condition. Transition temperatures of homologues (Table 2) were plotted against the number of carbon atoms present in n-alkyl chain of the left n-alkoxy group, a phase diagram (Fig. 1) consisting of Cr-M, Sm-N and N-I transition curves are obtained on linking the like or related points. Cr-M transition adopts a zigzag path of rising and falling with overall descending tendency. Sm-N transition curve commences from C_{10} homologue and smoothly rises up to last C_{18} homologue without exhibition of odd–even effect. Sm-N transition curve is extrapolated to [29, 30, 31, 32] C_8 and C_6 homologue to predict their latent transition temperatures (LTT) to judge their latent ability to form smectic mesophase. N-I transition curve continuously descends as series is ascended with exhibition of odd–even effect. Odd–even effect observed up to C_6 homologue and then disappears from and beyond merging of N-I transition curves for longer

Table 2. Transition temperatures in °C.

Compound No.	n-alkyl chain C_nH_{2n+1} (n)	Sm	N	Isotropic
1	1	–	145	151
2	2	–	141	157
3	3	–	109	133
4	4	–	103	143
5	5	–	131	137
6	6	–	130	136
7	7	–	91	133
8	8	–	76	127
9	10	65	81	123
10	12	62	79	118
11	14	61	83	114
12	16	68	91	112
13	18	72	93	108

Sm = Smectic, N = Nematic.

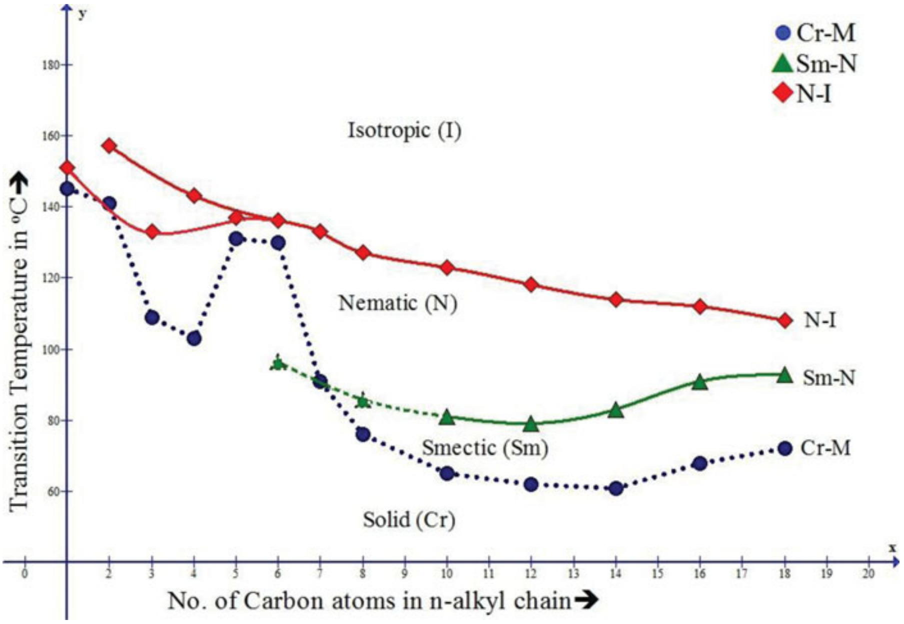


Figure 1. Phase behavior of series

n-alkyl chain of higher homologues. The textures of nematic phase are threaded or Schlieren and that of smectic phase are of the type A or C. LC properties of novel series varies from homologue to homologue in the same series due to changing molecular structure from homologue to homologue keeping right sided laterally substituted –Br group unchanged. The smectogenic mesophase length ranges from 16.0°C to 23.0°C and nematogenic mesophase length range is 06.0°C to 58.0°C. Thus, series is predominantly nematogenic and partly smectogenic of middle ordered melting type and considerable degree of mesomorphism.

Nonmesomorphic component Schiff's base on linking with n-alkoxy aromatic acid increases molecular length, permanent dipole moment across the long molecular axis, dispersion forces, dipole-dipole and electron-electron interactions, molecular polarizability, aromaticity, lateral attractions due to ortho substituted –Br group etc. which disalign the molecules of all the homologues of a series at an angle ninety degree or less than ninety degree under the influence of exposed thermal vibration as a consequence of molecular rigidity and flexibility. The suitable magnitudes of anisotropic forces of intermolecular end to end attractions for relatively lower (C_1 to C_8) n-alkyl chain of varying flexibility maintain only statistically parallel orientational order of molecules in floating condition but, molecular polarizability factor gradually strengthen the intermolecular cohesive forces by simultaneously increasing n-alkyl chain length from and beyond C_{10} homologue, whose magnitudes are capable of building lamellar packing of molecules in rigid crystal lattices and three dimensional networking, which adopts sliding layered molecular arrangement under exposed thermal vibrations to exhibit smectogenic mesophase formation and then, above the smectogenic mesophase appearance, the same molecules floated and adopts statistically parallel orientational order of molecules by residual inter molecular cohesion for another range of temperature. Thus, smectogenic property is induced for a range of temperature (t_1 to t_2) prior to nematogenic mesophase formation of another range (t_2 to t_3) of temperature. None of the homologues have exhibited monotropic mesophase formation. The alternation of transition temperatures of relatively shorter n-alkyl chain length resulted into exhibition of odd–even

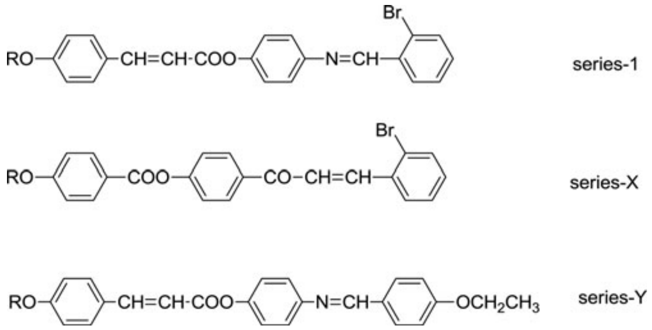


Figure 2. Structurally similar series.

effect in N-I transition curve. The disappearance of odd–even effect from and beyond hexyloxy (C_6) homologue is attributed to the unusual status of longer n-alkyl chain beyond C_6 homologue which may coil or bend or flex or couple to lye with major axis of the core structure. The extrapolated Sm-N transition curve from C_{10} to C_6 through C_8 do not facilitate smectic mesophase formation because of the absence of the focal conic networking layered molecular arrangement, because of the inadequate and unsuitable magnitudes of intermolecular anisotropic forces of cohesion and closeness, which fails to build up sliding layered molecular arrangement in floating condition. The variations in mesomorphic properties like thermal stability, commencement of smectic and/or nematic phase, degree of mesomorphism, etc form homologue to homologue in the same series are attributed to the sequentially added methylene unit or units causes variations in molecular rigidity and flexibility, keeping, rest of the molecular part unchanged throughout the series. The changing behaviors in thermotropic mesogenic properties of present Schiff's base series are compared with structurally similar other known homologous series as shown in Fig. 2.

Homologous series-1 of present investigation and series-X [33] selected for comparison are identical with respect to three phenyl rings, ortho bromo lateral group at the third phenyl ring and the left n-alkoxy (–OR) terminal end group for the same homologue from series to series. But they differ with central bridges linking three phenyl rings. Therefore, molecular flexibility for the same homologue remains unaltered, but molecular rigidity due to differing central bridges alters for the same homologue. Homologous series-1 and series-Y [34] chosen for comparative study are identical with respect to three phenyl rings and two central bridges –CH=CH–COO– and –N=CH– as well left n-alkoxy (–OR) terminal end group for the same homologue, but differed with respect to polar groups –Br and $-O-CH_2-CH_3$ substituted at an ortho or para position respectively of series-1 and series-Y. Thus, molecular rigidity of series-1 and Y 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 magnitudes 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 represents some thermometric property of presently investigated novel series-1 and the series-X and Y selected for comparative study.

Table 3 indicates that,

- Series-1 and Y are smectogenic plus nematogenic while series-X is only smectogenic.
- Smectic property commences form C_{10}, C_8 and C_{12} homologues respectively for series-1, X and Y.
- Nematic property for series-1 and Y commences from C_1 homologue, while, it does not commence till the last homologue of series-X.

Table 3. Relative thermal stabilities in °C.

Series →	1	X	Y
Smectic-Isotropic or Smectic-Nematic	85.4	100.6	122.5
Commencement of smectic mesophase	(C_{10}–C_{18}) C_{10}	(C_{14}–C_{16}) C_8	(C_{12}–C_{18}) C_{12}
Nematic-Isotropic	130.1	—	270.7
Commencement of nematic phase	(C_1–C_{18}) C_1	—	(C_1–C_{18}) C_1
Total Mesophase length range (Sm+N)	06.0 (C_1,C_5,C_6) to 58.0(C_{10})	2.9(C_{14}) to 15.3 (C_{16})	111.0 (C_{18}) to 194.0 (C_7)

- Smectic thermal stabilities are in decreasing order from series-Y to series-X to series-1.
- Nematic thermal stability of series-Y is more than a series-1.
- Total mesophase length is the highest for series-Y and the lowest for series-X whereas it is more than a series-X and less than series-Y for present series-1.

On comparing homologous series-1 and X, they differ with molecular rigidity contributed by central bridges, keeping molecular flexibility identical or equal by $-C_6H_4Br$ (ortho) unit at the tail end for the same homologue from series-1 to series-X. The vinyl carboxy central bridge –CH=CH–COO– is longer and stronger than carboxy –COO– central bridge by –CH=CH– unit bearing conjugated double bond linking first and middle phenyl rings. The second central bridge linking middle and tail end phenyl rings through –N= CH– and –CO–CH=CH– in series-1 and X respectively. The carbon atom of a middle phenyl ring bonded to nitrogen i.e. –C–N= is polar bond in series-1, whereas, carbon atom of phenyl ring bonded to carbon of –CO–CH= i.e. –C–C– bond is a nonpolar in case of series-X. Therefore the variation in molecular rigidity can be attributed to –CH=CH– unit of a first central bridge and bond polarity difference between –C–N= and –C–C– bond causes the difference in molecular rigidity for the same homologue from series-1 to series-X. More or less magnitudes of anisotropic forces of intermolecular attractions as compared to suitable magnitudes of anisotropic forces of intermolecular attractions fail to induce mesomorphism in a substance. Therefore, suitable magnitudes of intermolecular end to end cohesion commences from C_1 and then end to end as well as lateral cohesion together of suitable magnitude of favorable molecular rigidity and constant flexibility induces smectic and/or nematic mesophase formation for the same homologue in case of series-1 of present investigation. However, because of the low magnitudes of dispersion forces and the dipole-dipole interactions the mesomorphic tendency is absent till C_7 homologue and suitable magnitudes of anisotropic forces of intermolecular attractions commences from C_8 homologue as monotropic smectic in case of series-X. The variations in the bond polarities of –C–N= and –C–C–affects the extent of noncoplanarity, which results in the commencement of smectic phase from C_{10} and C_8 homologues for series-1 and X, respectively. Thermal stability of series-1 is lower than a series-X but its total upper and low mesophase length range is higher than a series-X because, combine effect of suitable magnitudes of molecular rigidity plus flexibility as emerged from respective molecular structures for the same homologue is effectively induces smectic and nematic phase in case of series-1. But, it fails to induce nematic phase in case of series-X depending upon thermodynamical quantity (ΔH) associated with molecular structure. Thus, thermal resistivities towards exposed thermal vibrations are relatively more of present novel series-1 than series-X.

Now, on comparing thermal stability and other thermometric properties of series-1 than a series-Y, which are relatively higher for series Y than series-1 as dependent on molecular flexibility only, keeping molecular rigidity unchanged or constant for the same homologue from series-1 to series-Y. The molecular structures including central bridges and number of phenyl rings for the same homologue, contributing to molecular rigidity are same but, molecular flexibility induced by ortho-substituted −Br of series-1 and para substituted $-OCH_2CH_3$ group of series-Y whose polarities and polarizability differed to induce liquid crystalline state in differing suitable magnitudes of anisotropic forces of intermolecular attractions. The ortho substituted −Br group increases intermolecular distances which lower transition temperatures, resulting into reduction of thermal stability of present series-1, whereas, para substituted $-OCH_2CH_3$ tail group maintains linearity of a molecule, which strengthens intermolecular cohesion and closeness. Therefore transition temperatures and resistivity towards exposed thermal vibrations are raised more for the same homologue from series-1 to Y. Therefore thermal stability and mesophase length ranges for the series-Y are relatively higher than the present novel series-1. As regards 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 C_1 to last member C_{18} of series-1 and Y of different degree of mesomorphism. Thus, thermometric properties of mesomorphic (LC) substances purely depend upon magnitudes of favorable molecular rigidity and flexibility as a result of even minor difference of the molecular structure, either for same homologue for series to series or from homologue to homologue in the same series.

Conclusions

- Novel homologous series of Schiff's base is entirely nematogenic and partly smectogenic whose smectic and nematic thermal stabilities are relatively low and of middle ordered 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-Y > Series-X > Series-1
  Nematic: Series-Y > Series-1 > Series-X
  (ii) Smectic : Series-X > Series-1 > Series-Y
  Nematic: Series-1 = Series-Y > Series-X
  (iii) (Smectic + Nematic): Series-Y > Series-1 > Series-X
- 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 on liquid crystalline state as a source of LC material.
- Study has supported and raised credibility to the conclusions drawn earlier.

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