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Dependence of LC state on molecular flexibility

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ABSTRACT

A novel azoester homologous series of liquid crystals (LCs) viz. RO–$C_6H_4COOC_{10}H_6$-N=N(ortho)-C_6H_4–OC_4H_9(para) has been synthesized and studied with a view to understanding and establishing the effect of molecular structure on LC properties. Homologous Series consists of thirteen members (C_1 to C_{18}). C_1 to C_3 members are nonliquid Crystals and the rest of the homologues are LC in an enantiotropic manner. C_7 to C_{18} are smectogenic in addition to nematogenic whereas C_4, C_5, and C_6 are only nematogenic. The Sm-N and N–I transition curves behave in a normal manner with the usual exhibition of an odd–even effect. The Cr-M/I curve also behaves in a normal manner. Analytical and spectral data confirm the molecular structures of homologues. The average smectic and nematic thermal stabilities are 60.31°C and 79.6°C, respectively, with total mesophase length varying minimum to maximum is 21°C to 57°C at the C_6 and C_{14} homologue, respectively. Thus, present novel azoester homologous series is partly smectogenic and predominantly nematogenic with low ordered melting type and useful to construct LC devices workable at low temperatures or room temperature.

KEYWORDS

Azoester; liquid crystal; mesomorphs; smectic; nematic

Introduction

The study of the liquid crystalline (LC) state [1] has attracted scientists and technologists for its unique applications in electronic display devices [1–4] and biological activity [5–9] from thermotropic or lyotropic LC substances. Hence, every scientist and technologist needs novel LC substances to continue and extend their research programs. The present investigation is planned with a view to understanding and establishing the effects of molecular structure [10–13] on LC properties by synthesizing novel LC substances through homologous series of novel unexploited moieties of varying geometrical shape, size, polarities, polarizability, ortho-meta-para substitution, or lateral, and/or terminal substitution etc. of molecule which causes variations in molecular rigidity and/or flexibility as well as thermodynamical properties from one molecular structure to other molecular structure in crystalline or solution state [14–18]. The present proposed homologous series planned to synthesize novel azoester series consisting of naphthyl ring, two phenyl rings bonded to central bridges and two tail groups. Number of ester series have been reported till the date [19–23].Thermometric properties will be determined after characterization of novel homologues and then the data will be recorded and

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interpreted in terms of molecular rigidity and flexibility including derivation of group efficiency order on the basis of thermal stability, Commencement of LC state and the degree of mesomorphism.

Experimental

Synthesis

4-Hydroxy benzoic acid was alkylated using suitable alkylating agent (R-X) to convert it into 4-n-alkoxy benzoic acids (A) by modified method of Dave and Vora [24], Alkylation of Paracetamol using alkylating agent n-C_4H_9Br is carried out to form 4-n-butyloxy acetanilide, which on hydrolysis converted to 4-n-butyloxy aniline is form by usual establish method. Azo dye (B) 2-hydroxy naphthyl azo 4′ butyloxy benzene (m.p. 80°C, yield 72%) was prepared by well-known azotization method, Final azoester products were synthesized by condensation of (A) and (B) [25]. Thus, the azo-ester homologue derivatives were filtered, washed with sodium bicarbonate solution followed by distilled water, dried and purified till constant transition temperatures obtained using an optical polarizing microscope equipped with a heating stage. 4-hydroxy benzoic acid, Alkyl halides, Paracetamol, 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 the series is mentioned in Scheme 1.

Characterization

Representative homologues of a series were characterized by elemental analysis, Infrared spectroscopy, ^{1}H NMR spectra, IR spectra were recorded on 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 analyzer (Table 1). Transition temperature and LC properties (Textures) were determined using an optical polarizing microscopy equipped with heating stage (POM). Texture images of nematic phase were determined by miscibility method (Table 2), thermodynamic quantities enthalpy(ΔH) and entropy($\Delta S = \Delta H/T$) are qualitatively discussed.

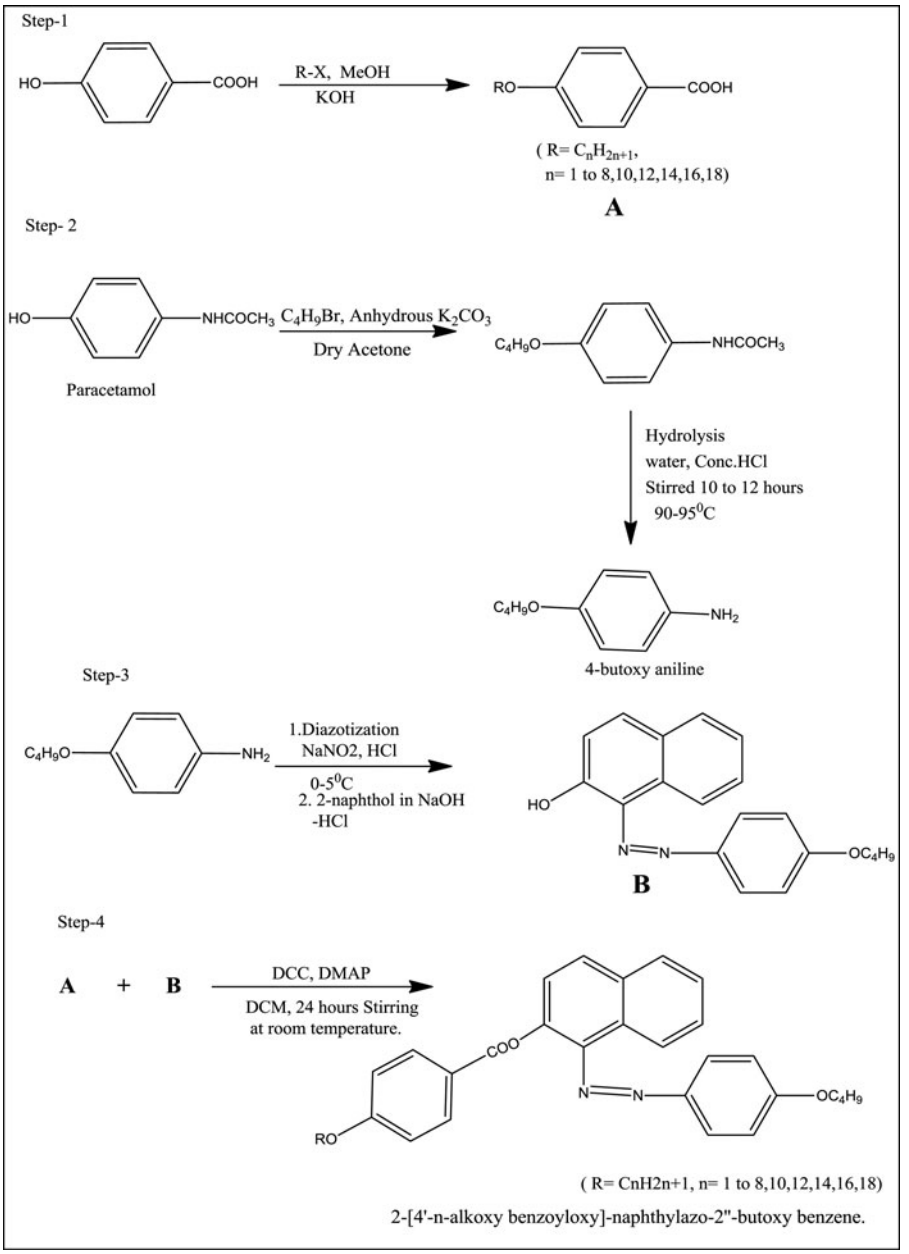
Analytical data

IR Spectra in cm^{-1} for Octyloxy & Dodecyloxy Derivatives:

Octyloxy : 669 Polymethylene (–CH_2–)n of –OC_8H_{17}, 840 (–C–H– def. m di-substituted-Para), 759 Polymethylene (–CH_2–) of –OC_4H_9, 1105 and 1024 (–C–O–) Str 1163,1247(–C–O str in –(CH_2)n chain, 1423 and 1469 (–C–H– def. in CH_2), 1494 (–C=C–)str, 1604(N=N group) and 1735 (–COO– ester group), 2848 and 2918 (–C–H str in CH_3).

Dodecyloxy : 661 Polymethylene (–CH_2–)n of –$OC_{12}H_{25}$, 844 (–C–H– def. m di-substituted-Para), 769 Polymethylene (–CH_2–) of –OC_4H_9, 948 (–C–H– def. hydrocarbon), 1085 and 1031 (–C–O–) Str, 1165,1255 and 1307 (–C–O str in –(CH_2)n chain, 1465 (–C–H– def. in CH_2), 1566 (–C=C–)str, 1604(N=N group) and 1737 (–COO– ester group), 2848 and 2916 (–C–H str in CH_3).

^{1}HNMR spectra in $CDCl_3$ in δ ppm for Hexyloxy & Octadecyloxy Derivative:



Scheme 1. Synthetic route to the series-1.

Hexyloxy: 0.88 (t,–CH_3 of –C_6H_{13}), 1.18-1.37(m, n-poly methylene groups of–OC_6H_{13}), 1.48 (m, n-poly methylene groups of –OC_4H_9), 3.8(s,–OCH_2–CH_2–ofOC_6H_{13}), 3.99 (s,–OCH_2–CH_2–of OC_4H_9), 6.9-7.1(s,naphthalene ring), 7.7-7.99 (s, p-disubstituted phenyl ring).

Octadecyloxy: 0.82 (t,–CH_3 of –$C_{18}H_{37}$), 1.1-1.3(m, n-poly methylene groups of–$OC_{18}H_{37}$), 1.51 (m, n-poly methylene groups of –OC_4H_9), 3.3-3.6(s,–OCH_2–CH_2–of $OC_{18}H_{37}$), 4.0(s,–OCH_2–CH_2–of OC_4H_9), 7.0-7.2(s,naphthalene ring), 7.9-8.1 (s, p-disubstituted phenyl ring).

Table 1. Elemental analysis for pentyloxy, octyloxy, decyloxy, hexadecyloxy, derivatives.

		%Elements found			%Elements calculated		
Sr. No.	Molecular formula	C	H	N	C	H	N
1	$C_{32}H_{34}O_4N_2$	74.00	6.49	6.50	74.70	6.61	6.62
2	$C_{35}H_{40}O_4N_2$	75.45	7.10	5.45	75.53	7.19	5.57
3	$C_{37}H_{44}O_4N_2$	75.92	7.40	5.38	76.02	7.53	5.47
4	$C_{43}H_{56}O_4N_2$	77.01	8.29	4.66	77.24	8.38	4.79

Result and discussion

Smectogenic and nematogenic mesomorphism is induced on condensing dimeric 4-n- alkoxy benzoic acids and an azo dye (m.p. 80°C) derived from 2-napthol and 4- butyloxy aniline. The transition temperatures of novel azoester homologues alternates and are relatively lower than the corresponding dimeric n-alkoxy acids. Nematogenic mesophase formation and Smectogenic mesophase formation commences from C_4 and C_7 homologues, respectively, in an enantiotropic manner upto last C_{18} homologue with absence of LC Phase in C_1,C_2, and C_3. Transition temperatures (Table 3) as determined from an optical polarizing microscopy equipped with a heating stage are plotted versus the number of carbon atoms present in left n-alkoxy (-OR) group. A Phase diagram consisted of Cr-M/I, Sm-N and N–I transition curves is obtained by linking like or related transition points, showing phase behaviors of a novel series as shown in Fig. 1. Cr-M/I transition curve follows a zigzag path of rising and falling with overall descending tendency and behaved in normal manner.

The Sm-N transition curve initially rises and passes through maxima at the C_{14} homologue and then descended with exhibition of odd–even effect from C_7 to C_9 derivative. An N–I transition Curve initially descends and then rises from C_7 homologue to maxima at C_{14}

Table 2. Texture of nematic phase of C_6, C_8, C_{12}, C_{18} by miscibility method.

		Texture	
Sr. No.	Homologue	Smectic	Nematic
1	C_6	Sm-A	Threaded
2	C_8	Sm-A	Threaded
3	C_{12}	Sm-C	Schlieren
4	C_{18}	Sm-C	Schlieren

Table 3. Transition temperatures in °C for series-1.

		Transition Temperatures in(°C)		
Compound No	n-alkyl chain C_nH_{2n+1}	Smectic	Nematic	Isotropic
1	C_1	—	—	111.0
2	C_2	—	—	101.0
3	C_3	—	—	98.0
4	C_4	—	52.0	78.0
5	C_5	—	49.0	75.0
6	C_6	—	44.0	65.0
7	C_7	40.0	48.0	69.0
8	C_8	44.0	56.0	78.0
9	C_{10}	45.0	58.0	75.0
10	C_{12}	56.0	67.0	89.0
11	C_{14}	41.0	68.0	98.0
12	C_{16}	49.0	59.0	88.0
13	C_{18}	52.0	66.0	81.0

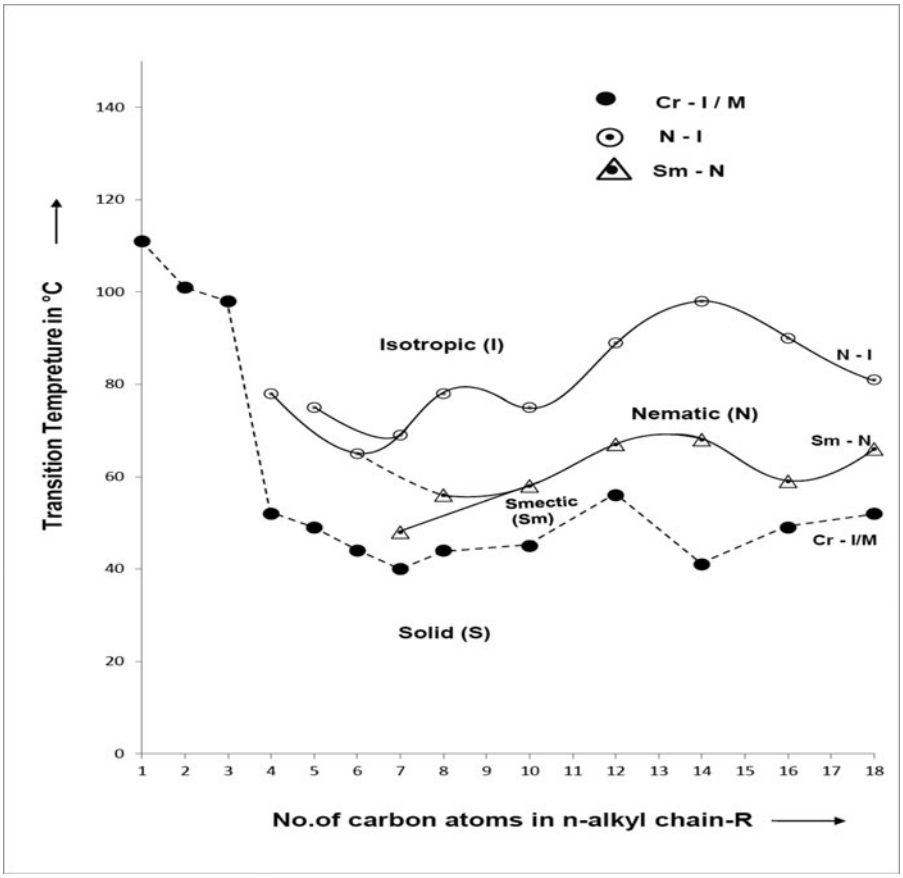


Figure 1. Phase diagram of series 1.

homologue and then descended as series is ascended upto C_{18} homologue with exhibition of odd –even effect from C_4 to C_7 derivative. Odd-even effect for Smectic and Nematic appears upto C_9 and C_7 homologue, respectively, and then disappears for higher homologues of longer n-alkyl chain of left n-alkoxy group. Thus, Cr-M/I, Sm-N and N–I transition curve behaved in normal manner with negligible abnormality. Sm-N transition curve is extrapolated to C_6 homologue [27–29] to determine the absence of smectogenic character in C_6 homologue. Textures of a Nematic phase are threaded or schlieren and that of smectogenic homologues are

Table 4. Relative thermal stability in °C.

Series	1(–OC_4H_9)	X(–$COCH_3$)	Y(–CH3)
Smectic-Isotropic Or Smectic-Nematic Commencement of Smectic phase	60.31 (C_7–C_{18}) C_7	—	—
Nematic–Isotropic Commencement of Nematic phase	79.6 (C_4–C_{18}) C_4	144.0 (C_6–C_{14}) C_6	127.27 (C_5–C_{14}) C_1
Total mesophase length range (Sm+N)	21.0 to 57°C	47.0 to 60.0°C	38.0 to 41.0°C
Ci to Cp	C_6 C_{14}	C_8 C_{12}	C_{14} C_2

focal conic of the type Smectic A or C. Analytical and Spectral data confirms the molecular structures of homologues.

Disappearance of dimerization and the lowering of transition temperatures of novel homologues are attributed to the breaking of hydrogen bonding between two molecules of 4-n-alkoxy benzoic acids through esterification process. The absence of LC property in C_1, C_2, and C_3 homologues is attributed to the unsuitable magnitudes of anisotropic forces of intermolecular attractions and closeness occurred as a consequence of unfavorable molecular rigidity and flexibility, which induced high crystallizing tendency due to their low dipole–dipole interactions and dispersion forces by interactions between instantaneous dipoles produced by spontaneous oscillations of electron clouds of the molecules. Thus, crystal lattices of molecules of C_1 to C_3 derivatives breaks abruptly and directly transform into isotropic liquid from solid crystalline state under the influence of exposed thermal vibrations, without exhibition of LC state. The exhibition of LC state by C_4 to C_{18} is attributed to disalignment of molecules at an angle ninety or $< 90°$ by suitable magnitudes of anisotropic forces of intermolecular attractions and closeness as a consequence of a favorable molecular rigidity and flexibility caused by resultant permanent dipole moment across the long molecular axis and dispersion forces. The molecules of C_3 to C_{18} homologues adopted either only statistically parallel orientational order of molecules (C_3 to C_6) or after exhibition of Smectic phase (C_7 to C_{18}) under floating condition due to favorable end to end intermolecular cohesion, within definite range of temperature, which, shows nematic type of textures under microscopic (POM) examinations. However, the molecules of C_7 to C_{18} homologues exhibited smectogenic character prior to nematic mesophase formation for lower mesogenic temperature range by maintaining sliding layered molecular arrangement under floating conditions with showing texture, either smectic-A or smectic–C.

All the mesogenic transitions are enantiotropic and none of them is monotropic. The odd member's transition temperatures being higher than even members up to the end of odd-even effect at (C_7) homologue. Thus, odd–even effect disappeared from and beyond C_7 homologue for longer n-alkyl chain from C_8 to C_{18} homologues, because, longer n-alkyl chain may coil or bend, or flex or couple to lye with the principal axis of a molecular core structure. Hence, addition of $-CH_2-$ units becomes an important. Odd-even effect observed due to odd and even number of methylene units present in n-alkyl chain. Sometimes uncertainly in the status of longer n-alkyl chain causes deviations in the normal behavior of a transition curve or curves of a phase diagram. The variations in the mesomorphic behaviors or LC properties from homologue to homologue in the same series are attributed to the increasing molecular length due to progressive and sequential addition of $-CH_2-$ unit or units at the left n-alkoxy terminal end group, contributing to suitable magnitudes of intermolecular anisotropic forces of adhesion and closeness. The changing trend in left n-alkoxy terminal, keeping rest of the molecular part intact in the same series causes variation in molecular characteristics. The properties of present thermotropic LC series are compared with structurally similar known series X [30] and series Y [31] as shown below in Fig. 2.

Homologous series1, X and Y under comparison contain identically two phenyl rings and one naphthyl ring bonded through –COO– and –N=N– central bridges on identical position which contribute to almost identical molecular rigidity and common (-OR) left n-alkoxy terminal end group for the same homologue from series to series. However, they differ with respect to right ended uncommon terminal $-OC_4H_9(n)$/ $-O-CH_2-CH_2-CH_2-CH_3$, $-COCH_3$ or $-CH_3$ polar groups; from series to series. Thus, left terminal –OR vary from homologue to homologue in the same series, keeping right handed terminal unchanged, as well as right handed terminal end group changes for the same homologue from series to series

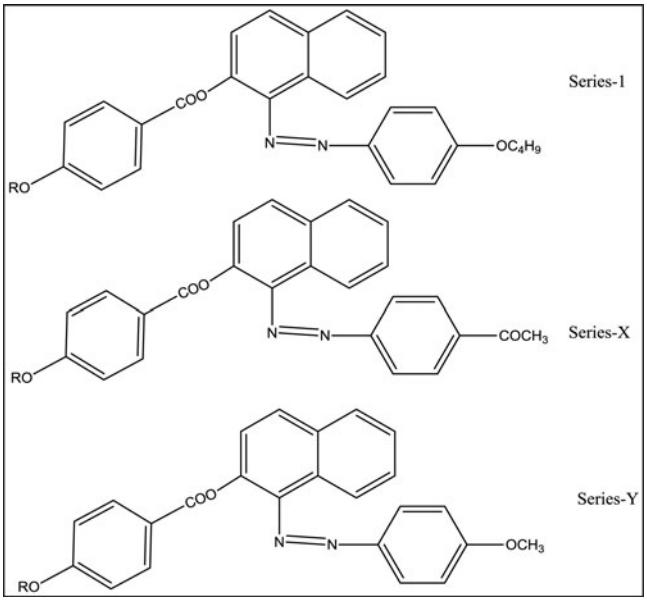


Figure 2. Structurally similar series.

keeping rest of the molecular part unchanged. Therefore, changing trend in LC properties and the degree of mesomorphism will depend upon the differing magnitudes of intermolecular anisotropic forces occurring as a consequence of changing molecular rigidity and molecular flexibility due to respective molecular structure of individual homologue of same series or for the same homologue from series to series. The extraploted Sm-N transition curve matches with N–I transition point of C_6 homologue, which rejects the possibility of smectogenic character in C_6 homologue. Table 3 represents some LC properties of thermotropic present novel series-1 and structurally similar known series X and Y chosen for comparison.

From Table 4, it is clear that,

- Homologous novel series-1 of present investigation is smectogenic plus nematogenic, but homologous series X and series Y selected for comparative study are only nematogenic.
- Smectogenic mesophase commences from C_7 homologue of a series-1, whereas, it does not commence till the last homologue of series X and series Y.
- Nematogenic mesophase commences from C_4, C_6, and C_1 for series-1, X and Y respectively.
- Smectic thermal stability is 60.31 for series-1 whereas, it does not stabilize for series X and Y.
- Thermal stability for nematic are 79.6, 144.0, and 127.27 for series-1, X and Y, respectively.
- The upper and lower mesophase lengths are decreasing from series-X to series-1 to series-Y.

The molecular rigidity due to phenyl rings and some central bridges with same geometrical molecular shape being equivalent from homologue to homologue in the same series and for the same homologue from series to series in case of present comparative study (Series 1, X,Y). However, the molecular flexibility is varied from homologue to homologue in the same series and for the same homologue from series to series depending upon the difference of group polarities of left and right sided end groups as well as molecular polarizability difference due to geometrical shapes of molecules. Thus, magnitudes of dispersion forces related

to permanent dipole moment across the long molecular axis, dipole–dipole and/ or electronic interactions and hence the molecular flexibility vary from homologue to homologue in the same series or from series to series; because, the vector sum of the bond polarities of $-OC_4H_9(n)$,

$-COCH_3$ and $-CH_3$ are different from each other for the same homologue from series to series and from homologue to homologue in the same series. The function group

$-CH_3$ of series Y is less polar than $-COCH_3$. Therefore molecular polarity and polarizability of series-X will be higher than a series-Y for corresponding homologue of each other. But the terminal end group $-OC_4H_9$ or $-O-CH_2-CH_2-CH_2-CH_3$ is longer than $-COCH_3$ and $-CH_3$ which induces lamellar packing of molecules in rigid crystal lattices which causes sliding layered molecular arrangement in floating condition under the influence of exposed thermal vibrations prior to commencement of nematic phase relatively at lower temperature up to below 45°C to 40°C and stabilizes smectogenic character in addition to nematogenic character. The commencement of mesophase either smectic or nematic depends upon the extent of molecular noncoplanarity which differs from series to series due to magnitudes of group polarity and polarizability difference and reflects to early or late commencement of mesophase formation. Lower or higher transition temperatures resulting low or high average thermal stability depend upon the resistivity offered by homologue or homologues of an individual series constituting enthalpy change (ΔH) value which is related with magnitudes of resistivity towards the exposed thermal vibrations to facilitate molecular arrangement in floating condition maintaining either of only nematic phase or smectic phase or both, one after another. Thus, $-COCH_3$ terminal being relatively more polar and polarizable causes highest nematic thermal stability, but fails to facilitate lamellar molecular packing in their crystal lattices due to relatively shorter –C–C–linkage with third phenyl ring and does not exhibit smectogenic character throughout a series. Same in the case with $-CH_3$ terminal end group which bears shortest –C–C– linkage with third phenyl ring of series-Y. However, a terminal end group which is relatively longer and bonded through oxygen atom by phenyl ring $-O-CH_2-CH_2-CH_2-CH_3$ bears etherial linkage with the last phenyl ring, facilitated smectic mesophase formation. Hence, type of linkage between tail group and last phenyl ring can also affect (ΔH) value of a molecular structure and hence the molecular flexibility or thermal stability through molecular polarity and polarizability as well as the degree of mesomorphism.

Conclusions

- Two phenyl rings and Naphthyl ring containing substances are not necessarily be always nematogenic, but may be smectogenic in addition to nematogenic, if proper tail groups at proper position are substituted.
- Nonlinear azoester homologous series of present investigation is partly smectogenic and predominantly nematogenic with low ordered melting type and of sufficient range of liquid crystallity.
- The group efficiency order derived for smectic and nematic on the basis of (1) thermal stability (ll) early commencement of mesophase (lll) total upper mesophase length in case of azoester series are as under.

(i)Smectic

$-OC_4H_9(n) > -COCH_3 = -CH_3$

Nematic

$-COCH_3 > -CH_3 > -OC_4H_9$ (n)

(ii) Smectic

$-OC_4H_9$ (n) > $-COCH_3 = -CH_3$

Nematic

$-CH_3 > -OC_4H_9$ (n) > $-COCH_3$

(iii) Total Mesophase length

$-COCH_3 > -OC_4H_9$ (n) > $-CH_3$

- The magnitudes of molecular flexibility of a molecule depends upon the difference of vector sum of bond polarities of two end groups or tail group polarities, contributing to total molecular polarities and polarizability.
- Suitable magnitudes of molecular rigidity and flexibility can induce LC property.
- Molecular rigidity and flexibility are very sensitive and susceptible to molecular structure.
- The LC state of homologue of present series stabilizes at low as 40 to 45°C which can be brought to low as 20 to 25°C or at room temperature by the study of binary systems.
- Room temperature LCs are useful for the manufacture of LC devices to be operated between 20°C to 50°C.
- Present investigation very well support and raises the credibility to the conclusions drawn earlier.

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