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Study of mesomorphism dependence on molecular flexibility of an azoester series containing a napthyl unit

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ABSTRACT

A novel azoester homologous series of liquid crystalline (LC) compounds: RO–C_6H_4-COO–$C_{10}H_6$-N:N-C_6H_4–OC_4H_9(n) without lateral substitution has been synthesized and studied with a view to understanding and establishing the effects of molecular structure on thermotropic LC substances with reference to tailed-end group. The novel homologous series consists of 13 homologs (C_1 to C_{18}) whose nematogenic and smectogenic mesomorphism commences enantiotropically from C_6 and C_{12} members of the series, respectively. The C_{12}–C_{18} homologs are smectogenic and C_6–C_{18} are nematogenic, of which C_{12}–C_{18} homologs are smectogenic plus nematogenic. The C_1–C_5 homologs are nonmesogenic. Transition temperatures and the textures of the homologs were determined and identified by an optical polarizing microscope (POM) equipped with a heating stage. Textures of a nematic phase are threaded or Schlieren and that of the smectic phase are of the type A or C. Transition curves Cr-M/I, Sm-N and N-I of a phase diagram behaved in normal manner except N-I transition temperature of C_{10} homolog which deviated by 9°C–10°C from normal behavior. N-I transition curve exhibited odd-even effect. Analytical, spectral, and thermal data confirms the molecular structures of homologs. Thermal stability for smectic and nematic are 115.5°C and 138.5°C, respectively whose corresponding mesophaselengths are varied from 10.0°C to 16.0°C and 13.0°C to 24.0°C, respectively. Group efficiency order for smectic and nematic are derived from comparative study of structurally similar analogous series; as smectic: $-OC_4H_9$ (n) > $-CH_3$ > −H; Nematic: −H > $-OC_4H_9$ (n) > $-CH_3$

KEYWORDS

Azoester; liquid crystal; LTT; Nematic; Smectic

1. Introduction

Our continuous efforts on the study of thermotropic liquid crystals[1] is aimed at understanding and establishing the effects of molecular structure[2–5] on liquid crystal (LC) properties with reference to terminal and/or lateral group or groups; and on the number of phenyl rings, different functional groups and bridging groups. The LC materials are useful in the benefit of mankind for LC devices workable at low or desired temperature, light emitting diodes and devices of semiconductor materials, optical imaging, etc.[6–10]. Azoester LC dyes find equal importance as useful as other dyeing LC material. Proposed investigation involve synthesis of an azoester homologous series consisting of three phenyl rings bonded through two central

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bridges viz. –COO– and –N=N– and two terminal end groups –OR and $-OC_4H_9$ (n) where *R* is varied from C_1 to C_{18} in P' and P" positions. The thermometric data will be evaluated using an optical polarizing microscopy (POM) equipped with a heating stage and then will be interpreted in terms of molecular rigidity and flexibility[11–14]. The group efficiency order for nematic and smectic will be derived from comparative study of structurally similar analogous series. Number of ester homologous series are reported till the date[15–21].

2. Experimental

2.1. 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[22], 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 formed by usual establish method. Azo dye (B) 4-hydroxy naphthyl azo 4"- butyloxy benzene (m.p. 132°C, yield 71%) was prepared by well-known azotization method[23]. Final azoester products were synthesized by condensation of (A) and (B)[24]. Thus, the azoester homolog 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, 1-Napthol dicyclohexyl carbodimide, Dimethyl amino pyridine, K_2CO_3, NaOH, $NaHCO_3$, $NaNO_2$, KOH, 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 shown inScheme 1.

2.2. Characterization

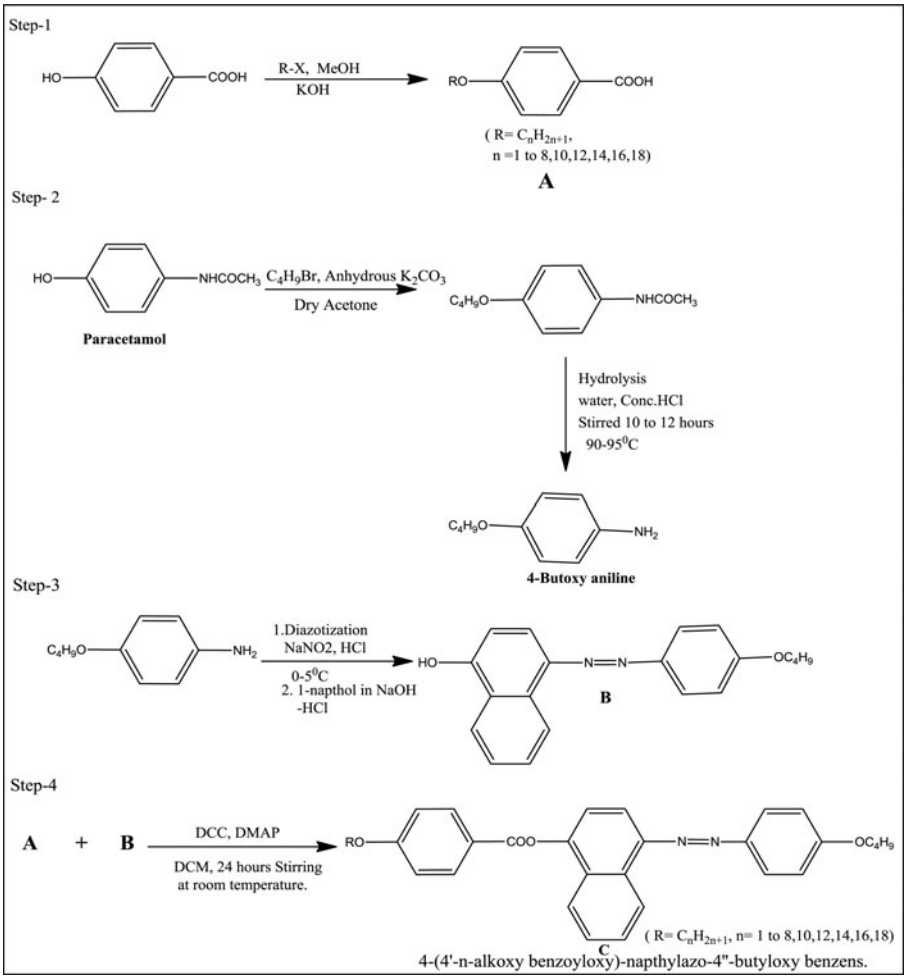
Representative homologs of the series were characterized by elemental analysis, Infrared spectroscopy, ^{1}H NMR spectra. IR spectra were recorded on Perkin-Elmer spectrum GX, ^{1}H NMR spectra were recorded on Bruker using $CDCl_3$ as solvent. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyzer (Table 1). Transition temperatures and LC properties (textures) were determined using an optical polarizing microscopy equipped with heating stage (POM). Texture images of nematic, smectic of selected homologs phase were determined by miscibility method (Table 2).

2.3. Analytical data

2.4. IR Spectra in cm^{-1} for tetradecyloxy & dodecyloxy derivatives

Tetradecyloxy: 653 Polymethylene $(-CH_2-)$n of $-OC_{14}H_{29}$, 813 (–C–H- def. di-substituted-Para), 769 Polymethylene $(-CH_2-)$ of $-OC_{14}H_{29}$, 1139 and 1062 (–C–O–) Str 1163,1247(–C–O str in -(CH_2)n chain, 1377 and 1467 (–C–H- def. in CH_2), 1502 (–C=C–)str, 1598, 1604(N=N group), 1645 (C=O of ester group), and 1735 (–COO– ester group), 2848 and 2918 (–C–H str in CH_3).

Dodecyloxy: 640 Polymethylene $(-CH_2-)$n of $-OC_{12}H_{25}$, 837 (–C–H- def. di-substituted-Para), 761 Polymethylene $(-CH_2-)$ of $-OC_4H_9$, 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, 1602 (N=N group), 1625 (C=O of ester), and 1737 (–COO– ester group), 2848 and 2922 (–C–H str in CH_3).

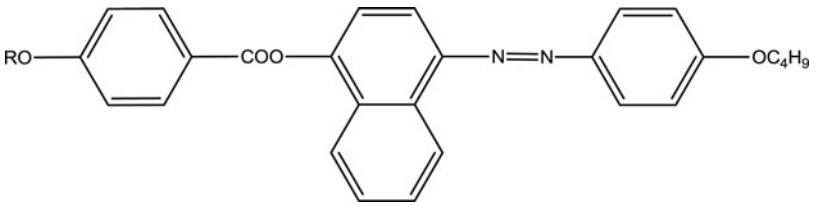


Scheme 1. Synthetic route of series 1.

2.5. ^{1}HNMR spectra in $CDCl_3$ in δ ppm for hexyloxy & hexadecyloxy derivative

Heptyloxy: 0.88 (*t*,–CH_3 of –C_7H_{15}), 1.18–1.37(m, n-poly methylene groups of–OC_7H_{15}), 1.48 (, n-poly methylene groups of –OC_4H_9),3.8(s,–OCH_2–CH_2-of –OC_7H_{15}), 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).

Hexadecyloxy: 0.82 (*t*,–CH_3 of –$C_{16}H_{33}$), 1.1–1.3(m, n-poly methylene groups of–$OC_{16}H_{33}$), 1.51 (m, n-poly methylene groups of–OC_4H_9), 3.3–3.6(s,–OCH_2–CH_2-of $OC_{16}H_{33}$), 4.0(s,–OCH_2–CH_2-of –OC_4H_9), 7.0–7.2(naphthalene ring), 7.9–8.1 (s, p-disubstituted phenyl ring).



4-[4'-n-Alkoxy benzoyloxy]-naphthylazo-4"-butyloxy benzenes

Table 1. Elemental analysis for hexyloxy, octyloxy, dodecyloxy, hexadecyloxy derivatives.

		%Elements found			%Elements calculated		
Sr. No.	Molecular formula	C	H	N	C	H	N
1	$C_{33}H_{36}O_4N_2$	75.50	6.82	5.30	75.57	6.87	5.34
2	$C_{35}H_{40}O_4N_2$	75.42	7.12	5.50	75.53	7.19	5.57
3	$C_{39}H_{48}O_4N_2$	76.94	7.83	4.54	76.97	7.89	4.60
4	$C_{43}H_{56}O_4N_2$	77.15	8.20	4.69	77.24	8.38	4.79

Table 2. Texture of nematic phase of C_5, C_6, C_{14}, C_{16} by miscibility method.

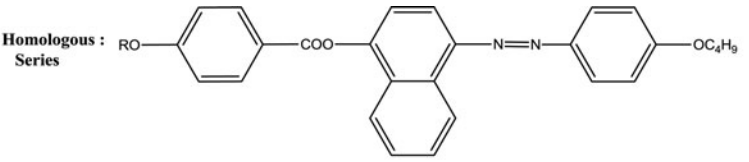
		Texture	
Sr. No.	Homolog	Smectic	Nematic
1	C_{12}	Sm-C	Threaded
2	C_{14}	Sm-A	Threaded
3	C_{16}	Sm-A	Threaded
4	C_{18}	Sm-C	Schlieren

3. Results and discussion

The novel homologous series is derived from condensation of dimeric 4-*n*-alkoxy benzoic acids and 4-hydroxy napthyl azo 4'-*n*-butyloxy benzene (m.p. 132°C, yield 71%) azo dye. The transition temperatures of novel mesomorphic azoester homologs are relatively lower than corresponding *n*-alkoxy acids. C_1–C_5 homologs are nonmesomorphic. C_6–C_{18} homologs are enantiotropically mesomorphic. C_6–C_{10} homologs are only nematogenic, whereas C_{12}–C_{18} homologs are smectogenic in addition to nematogenic. Transition temperatures of homologs (Table 2) were determined by POM. Transition temperatures of homologs were plotted versus the number of carbon atoms present in *n*-alkyl chain 'R' of –OR terminal group. Transition curves, Cr-M/I, Sm-N, N-I were obtained by linking like or related points as depicted in a phase diagram (Fig. 1) showing their phase behaviors for present series. Cr-M/I transition curve adopts a zigzag path of rising and falling tendency with overall descending tendency and behaves in normal expected manner. The Sm-N transition curve continuously descends as the series is ascended without exhibition of odd-even effect, and behaves in normal manner. The N-I transition curve initially rises and descends abnormally at C_{10} homolog and then rises

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	–	–	179.0
2	C_2	–	–	158.0
3	C_3	–	–	150.0
4	C_4	–	–	148.0
5	C_5	–	–	151.0
6	C_6	–	132.0	146.0
7	C_7	–	127.0	140.0
8	C_8	–	130.0	150.0
9	C_{10}	–	120.0	138.0
10	C_{12}	116.0	132.0	146.0
11	C_{14}	108.0	124.0	144.0
12	C_{16}	104.0	112.0	126.0
13	C_{18}	84.0	94.0	118.0



4(4'-n-Alkoxy benzoyloxy)napthyl azo-4"-Butoxy benzens.

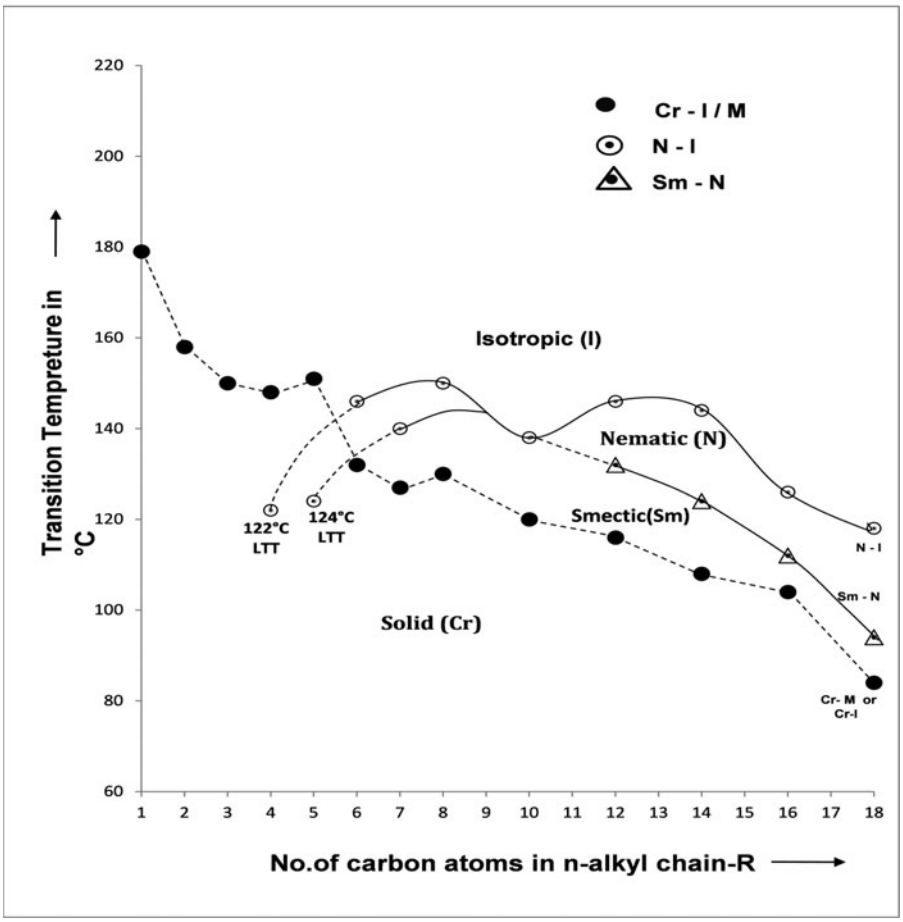


Figure 1. Phase behaviors of series.

through C_{12} and C_{14} and descends in normal manner with exhibition of odd-even effect from C_6 to C_9 homolog. N-I transition curves for odd and even members from C_6 to C_9 homologs merged into each other at C_9 homolog and then it disappears from and beyond C_9 for higher homologs of longer *n*-alkyl chain 'R' of –OR group. N-I transition curve is extrapolated to the left of the curves for[25–28] odd and even members to predict and determine the latent transition temperature (LTT) for nematic for C_4 and C_5 nonmesomorphic homologs; as 122°C and 124°C, respectively. Thermal stabilities for smectic and nematic are 115.5°C and 138.5°C, respectively whose total mesophase length are ranging from 13.0°C to 36.0°C. Textures of a nematic phase are threaded or Schlieren and that of the smectic are A or C. Analytical and spectral data supported molecular structures of homologs. The mesomorphic properties of homologs varied from homolog to homolog in present novel series.

The lowering of transition temperatures of novel homologs as compared to the corresponding dimeric *n*-alkoxy benzoic acids is attributed to the breaking of hydrogen bonding by esterification process. Alternations of transition temperature and exhibition of odd-even effect by N-I transition curve observed in a phase diagram is due to the presence of odd and even numbered carbon atom in *n*-alkyl chain 'R' of left –OR group. The exhibition of mesogenic characteristic commencing from C_6 homolog is attributed to the disalignment of molecules at an angle less than ninety degree and/or perpendicular to the plane of floating surface depending upon thermal resistivity against exposed thermal vibrations as a consequence of favorable magnitudes of molecular rigidity and flexibility, induced by suitable magnitudes of dispersion forces and dipole-dipole interactions. The disalignment of molecules perpendicular to the plane of floating surface under the influence of exposed thermal vibrations organized molecules in lamellar fashion in their crystal lattices and occupy layered molecular arrangement which under the influence of exposed heat to float with two dimensional ordered sliding layered molecular arrangement within definite temperature range to show exhibition of smectogenic character, from C_{12} to C_{18} homolog. However the group of homologs which exhibited smectogenic character have residual end to end intermolecular attractions of suitable magnitudes with their disalignment less than ninety degree which facilitate the floating of the molecules of same C_{12}–C_{18} homologs with statistically parallel orientational order for another higher range of temperature to show nematogenic mesophase formation of higher entropy (ΔS). Then the same molecules on continued heating from and beyond isotropic temperature randomly orient in all possible directions with high order of disorder or high entropy in reversible manner. The molecules of C_6–C_{10} homologs having their only disalignment less than ninety degree with floating surface, float With only statically parallel orientational order of suitable magnitudes of end-to-end attractions facilitating formation of nematic phase only for definite range of temperature excluding formation of smectic phase in reversible manner. The nonmesomorphicity of C_1–C_5 homolog is due to the low magnitudes of dispersion forces and low magnitudes of dipole-dipole interactions leading to high crystallizing tendency. The diminishing tendency of odd-even effect from and beyond C_9 homologs of longer in alkyl chain 'R' of higher homologues may coil or bend or flex or couple to lye with major axes of core structures of a molecule. Therefore, the uncertainty in the status of *n*-alkyl chain may modify permanent dipole moment across the long molecular axes, end-to-end dispersion forces or molecular rigidity and/or flexibility and can induce abnormality in the behaviors of transition curve to more or less extent depending upon the magnitudes and type of modifications in the status of longer *n*-alkyl chain R beyond merging of curves for odd-even members of mesogenic homologs, as observed for C_{10} homolog of N-I transition curve of present investigation. The magnitudes of intermolecular cohesion and closeness vary in present series which depends upon difference of molecular polarity and polarizability for –OR and $-OC_4H_9$(n) end groups keeping rest of the molecular part other than *R* remain unaltered from homolog to homolog in the same series. Thus, changing molecular length, permanent dipole moment across long molecular axis, polarity and polarizability of terminal or lateral groups, molecular rigidity and/or flexibility, suitable magnitudes of end-to-end or/and or lateral cohesion etc., which vary from homolog to homolog in the same series altering magnitudes of mesomorphism or the degree of mesomorphism. The extrapolated N-I transition curve for C_4 and C_5 members are not realizable due to their high crystallizing tendency. The changing trend in mesomorphic behaviors of present novel series 1 are compared with structurally similar analogous series X[29] and Y[30] selected for the study; as shown below in the Fig. 2.

Above homologous series 1, X, and Y under comparative study are identical with respect to two phenyl rings and one (middle) naphthyl ring as well as two central bridges –COO– and

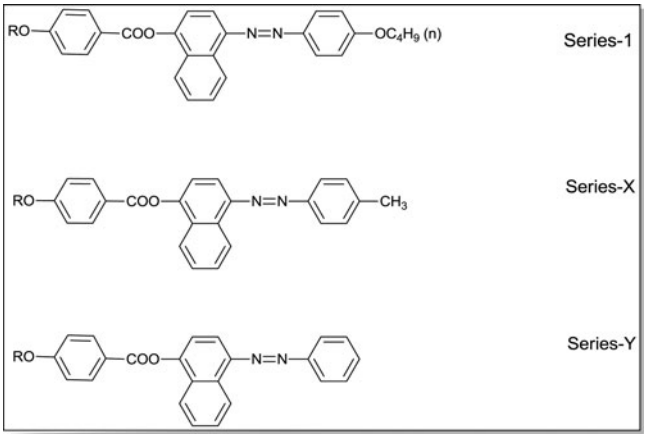


Figure 2. Structurally similar analogous series.

–N=N– which contributes to the total molecular rigidity. Left *n*-alkoxy groups are identically same for all the three series for the same homolog from series to series but differ from homolog to homolog in the same series which contributes partly to the total molecular flexibility. The tailed groups $-O-CH_2-CH_2-CH_2-CH_3$, $-CH_3$ and –H differs from series to series for the same homolog but remain unaltered from homolog to homolog in the same respective series, which contributes partly to the total molecular flexibility. Thus, changing trends in mesogenic properties and the degree of mesomorphism will depend upon the magnitudes of changing features among the series 1, X, and Y. Thus, the degree of mesomorphism will depend upon the magnitudes of changing features among the series 1, X, and Y under comparative study as a consequence of effective molecular rigidity, flexibility, and thermal resistivity against exposed thermal vibrations. Following Table 3 represents some thermometric data of series-1, X, and Y in comparative manner as mentioned below.

Table 3 involving comparative data indicates that,

- A homologous series 1 of present investigation is smectogenic plus nematogenic whereas homologous series X and Y chosen for comparative study are nematogenic only with absence of smectic property.
- Commencement of mesophase takes place from C_6 homolog of a present series 1, whereas it commences earliest from very first member C_1 of the series X and Y under comparison.
- Thermal stability for smectic of present novel series 1 is 115.5, but it does not stabilize till the last homolog of the series X and Y.

Table 4. Relative thermal stabilities in°C.

Series →	Series-1($-OC_4H_9$)	Series-X($-CH_3$)	Series-Y(–H)
Sm-N Or Sm-I	115.5		
Commencement of Smectic phase	(C_{12}–C_{18}) C_{12}	—	—
N-I	138.5	128.63	140.45
Commencement of nematic phase	(C_6–C_{18}) C_4	(C_1–C_{14}) C_1	(C_1–C_{14}) C_1
Total mesophase length in°C	13.0–36.0 C_7 C_{14}	10.0–41.0 C_{14} C_8	17.0–62.0 C_3 C_8

- Thermal stability for nematic and the total lower mesophase length is in increasing order from series X to series 1 to series Y.
- Upper total mesophase lengths are in increasing order from series 1 to series X to series Y under comparison.

The lack of smectogenic property by homologous series X and Y indicates absence of lamellar packing of molecules. This effect may be linked to decreasing order of polarity and polarizability of tail-ended terminal groups $-OCH_2-CH_2-CH_2-CH_3$, $-CH_3$, $-H$, respectively, for series 1, X and Y. Late commencement of mesophase formation of series-1 from C_6 homolog as compared to series X and Y in which mesophase commences from very first member (C_1) of the series is attributed to the extent of molecular noncoplanarity caused by lateral or terminal or central groups and their magnitudes of polarity as well as polarizability. The tail groups of series 1 is $-CH_2-CH2-CH_2-CH_3$ which is longer by three (Series X) and four (Series Y) $-CH_2$- groups in X and Y homologous series respectively under comparison; which causes difference of the extent of molecular noncoplanarity and magnitudes of molecular linearity or nonlinearity, resulting into differing magnitudes of intermolecular cohesion and closeness. Now regarding thermal stability which is related with the energy stored in a molecule as heat of formation (ΔH) due to its mass and characteristics, which is higher for present novel series as compared to series X and Y, whose thermal resistivity toward exposed thermal vibrations relates with upper mesophase length, as consequence of differing molecular rigidity and flexibility of individual series under comparison. Thus, an azoester series containing napthyl unit under comparison is generally or predominantly nematogenic, but introduction of *n*-butyloxy group as tail-end group partly induces smectogenic property.

4. Conclusions

- Novel azoester homologous series of thermotropic liquid crystal is predominantly nematogenic and partly smectogenic of considerable phase length and it is of middle ordered melting type.
- The group efficiency order derived for smectic and nematic on the basis of (i) thermal stability (ii) Early commencement of mesophase and (iii) total mesophase lengths are as under.
  - (i) **Smectic**
    $-O-CH_2-CH_2-CH_2-CH_3 > -CH_3 = -H$
    Nematic
    $-H > -O-CH_2-CH_2-CH_2-CH_3 > -CH_3$
  - (ii) **Smectic**
    $-O-CH_2-CH_2-CH_2-CH_3 > -CH_3 = -H$
    Nematic
    $-CH_3 = -H > -O-CH_2-CH_2-CH_2-CH_3$
  - (iii) **Total mesophaselengths**
    Lower: $-H > -O-CH_2-CH_2-CH_2-CH_3 > -CH_3$
    Upper: $-H > -CH_3 > -O-CH_2-CH_2-CH_2-CH_3$
- A homologous series containing napthyl unit are generally nematogenic, but introduction of *n*-alkyl terminal tail group may induce smectic property.
- Mesomorphism is very sensitive and susceptible to the molecular structure.
- Combined effects of effective molecular rigidity and flexibility of suitable magnitudes can induce mesomorphism.

- Present investigation may be useful for the manufacture of LC devices, the study of light polarization and in the ester needed vegetable plants and trees.
- Present investigation supported and raised the credibility to the conclusions drawn earlier.

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