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Molecular Structure Dependence of Mesomorphism in Chloro Substituted Azoester Series

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ABSTRACT

Azoester novel homologous series RO-CH:CH-COO-C₁₀H₆-N:N-C₆H₄-Cl (ortho) from trans cinnamic acid and α -naphthol using ortho chloro aniline has been synthesized and studied with a view to understand and establish the relations between thermotropic liquid crystalline (LC) behaviours of homologues and their molecular structure as a consequence of molecular rigidity and flexibility with changing number of methylene unit present in left n-alkoxy group (-OR) of a molecule. Novel azoester series consists of thirteen (C₁ to C₁₈) homologues. C₆ to C₁₈ homologues are enantiotropically nematogenic with absence of smectic property. C₁ to C₅ members are nonliquid crystal. Textures of nematic phase and transition temperatures were determined by an optical polarising microscopy (POM) equipped with a heating stage. Textures of nematic phase are threaded or schlieren. Phase transition curves Cr-N/I and N-I behaved in normal manner in a phase diagram. Analytical and spectral data confirmed molecular structures of homologues. Nematic thermal stability is 145.12 and the degree of mesomorphism vary from 26.0 °C to 46.0 °C at the C₁₄ and C₇ homologues respectively. The LC properties of present azoester series are compared with structurally analogous series. Thus, novel series is enantiotropically nematogenic whose mesogenic range is good enough and of middle ordered melting type.

Keywords: Azoester; Liquid Crystal; Smectic; Nematic; Enantiotropy

1. INTRODUCTION

Azoester compounds containing $-N=N-$ linkage undergo $\pi-\pi^*$ transition, which can display a high intensity of light. Therefore the study of light polarization and photochromism can be facilitated by introducing trans azobenzene group in the substances of LC bearing behaviours of azoester derivatives.

Azoester derivatives [1-4], which changes their colours with intensity of exposed light in variety of articles including light emitting articles. Thus, present investigation is planned with a view to understand the effect of molecular structure [5-11], on mesomorphic thermotropic behaviours of a homologous series containing vinyl carboxy and $-N=N-$ central bridges and chloro substituted lateral group.

Thus, present proposed novel azoester series will be synthesized, characterized by analytical, thermometric and spectral data. The results of evaluated data will be discussed and interpreted in terms of molecular rigidity and flexibility [12-15] and will be compared with structurally similar or analogous known series. The group efficiency order will be derived for including mesomorphism.

Numbers of homologous series of esters and azoesters have been reported [16-27] till the date; but an azoester series involving $-CH=CH-COO-$ central group are rarely reported. Therefore effect of vinyl carboxylate unit in combination with $-N=N-$ will be studied by replacing $-COO-$ carboxylate unit; in a novel series.

2. EXPERIMENTAL

2. 1. Synthesis

Trans 4-n-alkoxy cinnamic acids (B) is prepared by modified method Dave and Vora [28], Azo dye (C) 4-hydroxy naphthyl azo-2''-chlorobenzene (m.p. 120 °C, yield 83.38%) was prepared by well-known azotization method [29]. Final azoester products were synthesized by condensation of (B) and (C) [30].

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 polarising microscope equipped with a heating stage. 4-hydroxy benzaldehyde, Alkyl halides, Malonic acid, dicyclohexyl carbodimide, Dimethyl amino pyridine, anhydrous, DCM, MeOH, o-chloroaniline, 1-naphthol, 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.

2. 2. Characterization

Selected members of the novel homologous series were characterized by Elemental Analysis, infra-red spectroscopy, 1H NMR spectra. IR spectra were recorded by Perkin-Elmer spectrum GX, 1H NMR spectra were recorded on Bruker using $CDCl_3$ as solvent. Microanalysis was performed on a Perkin-Elmer PE2400 CHN analyzer. Transition temperature and LC properties (Textures) were determined using an optical polarizing microscope equipped with a heating stage. Textures of nematic phase determined by miscibility method.

2. 2. 1. Analytical Data

Table 1. Elemental Analysis for (1) Hexyloxy (2) Octyloxy (3) Decyloxy and (4) Octadecyloxy derivatives.

Sr. No.	Molecular formula	%Elements found			%Elements calculated		
		C	H	N	C	H	N
1	C ₃₁ H ₂₉ O ₃ N ₂ Cl	74.00	6.49	6.50	72.58	5.65	5.46
2	C ₃₃ H ₃₃ O ₃ N ₂ Cl	75.45	7.10	5.45	73.26	6.10	5.18
3	C ₃₅ H ₃₇ O ₃ N ₂ Cl	75.92	7.40	5.38	73.87	6.50	4.92
4	C ₄₃ H ₅₃ O ₃ N ₂ Cl	77.01	8.29	4.66	75.82	7.78	4.11

IR Spectra in cm⁻¹ for Decyloxy & Dodecyloxy Derivatives

Decyloxy: 750 Polymethylene (-CH₂)_n of -OC₁₀H₂₁, 837 (-C-H- def. substituted-Para), 1047 (-C-O-) Str 1201,1253(-C-O str in -(CH₂)_n chain, 1446 and 1507 (-C-H- def. in CH₂), 1571 (-C=C-)str, 1604 (N=N group) and 1710 (-COO- ester group), 2852 and 2926 (-C-H str in CH₃), 1087 (C-Cl) aromatic, 980 trans -CH=CH-.

Dodecyloxy: 721 Polymethylene (-CH₂)_n of -OC₁₂H₂₅, 833 (-C-H- def. m di-substituted-Para), 948 trans -CH=CH-, 1020 and 1114 (-C-O-) Str, 1165,1253 and 1377 (-C-O str in -(CH₂)_n chain, 1465 (-C-H- def. in CH₂), 1502 (-C=C-)str, 1604 (N=N group) and 1660 -CO of -COO- ester group), 2850 and 2922 (-C-H str in CH₃).

¹HNMR spectra in CDCl₃ in δ ppm for Heptoloxo & Hexadecyloxy Derivative:

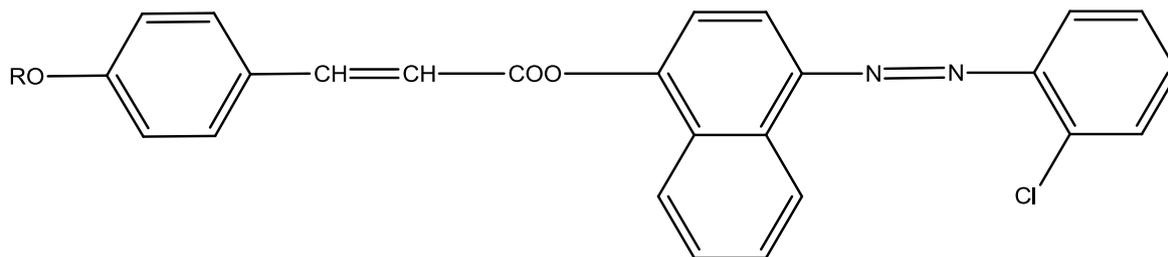
Heptoloxo: 0.86 (t,-CH₃ of -C₇H₁₅), 1.22 (m, n-polymethylene groups of-OC₇H₁₅), 3.7 to 3.9 (s,-OCH₂-CH₂-of -OC₇H₁₅), 4.06 (t, of -OCH₂-), 8.03 (s, naphthalene ring), 7.02 (d, Ar-H), 7.38 (d, Ar-H), 7.7-8.4 (s, p-disubstituted phenyl ring).

Hexadecyloxy: 1.4-1.6 (m, n-poly methylene groups of-OC₁₆H₃₃), 3.3-3.9 (s,-OCH₂-CH₂-of OC₁₆H₃₃), 1.49 (m, 3CH₂), 3.9-4.0 (t,-OCH₂-CH₂-of -OC₁₆H₃₃), 7.3-7.3 (s, naphthalene ring), 7.9-8.1 (s, p-disubstituted phenyl ring).

Table 2. Texture of Nematic Phase of C₅, C₇, C₈, C₁₂ by miscibility method.

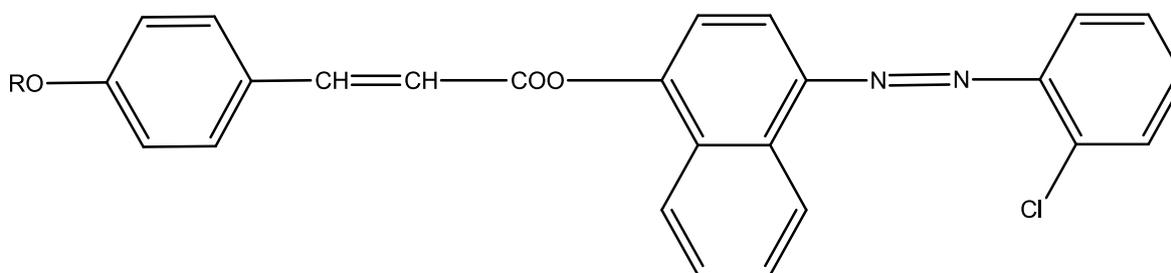
Sr. No.	Homologue	Texture
1	C ₅	Schlieren
2	C ₇	Threaded
3	C ₈	Threaded
4	C ₁₂	Nematic droplets

Table 3. Transition Temperatures in °C for Series - 1.



Trans-4-(4'-n-alkoxycinnamoyloxy)naphthylazo-2''-chlorobenzenes

Sr.no	R= n-alkyl group	Transition temperatures in °C		
		Smectic	Nematic	Isotropic
1	C ₁	-	-	178.0
2	C ₂	-	-	182.0
3	C ₃	-	-	179.0
4	C ₄	-	-	169.0
5	C ₅	-	-	157.0
6	C ₆	-	129.0	168.0
7	C ₇	-	114.0	160.0
8	C ₈	-	119.0	156.0
9	C ₁₀	-	124.0	154.0
10	C ₁₂	-	110.0	138.0
11	C ₁₄	-	102.0	128.0
12	C ₁₆	-	96.0	130.0
13	C ₁₈	-	94.0	127.0



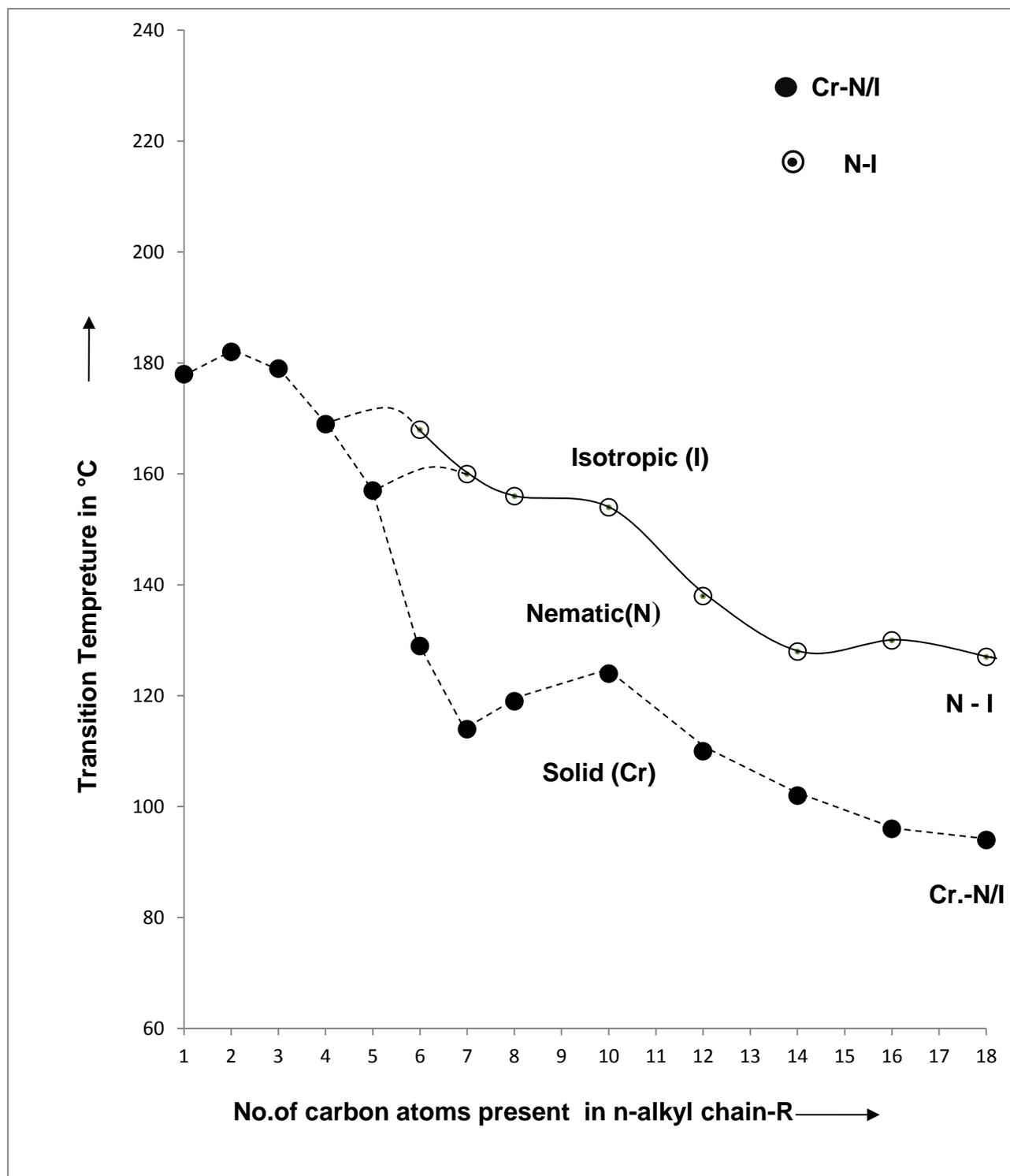
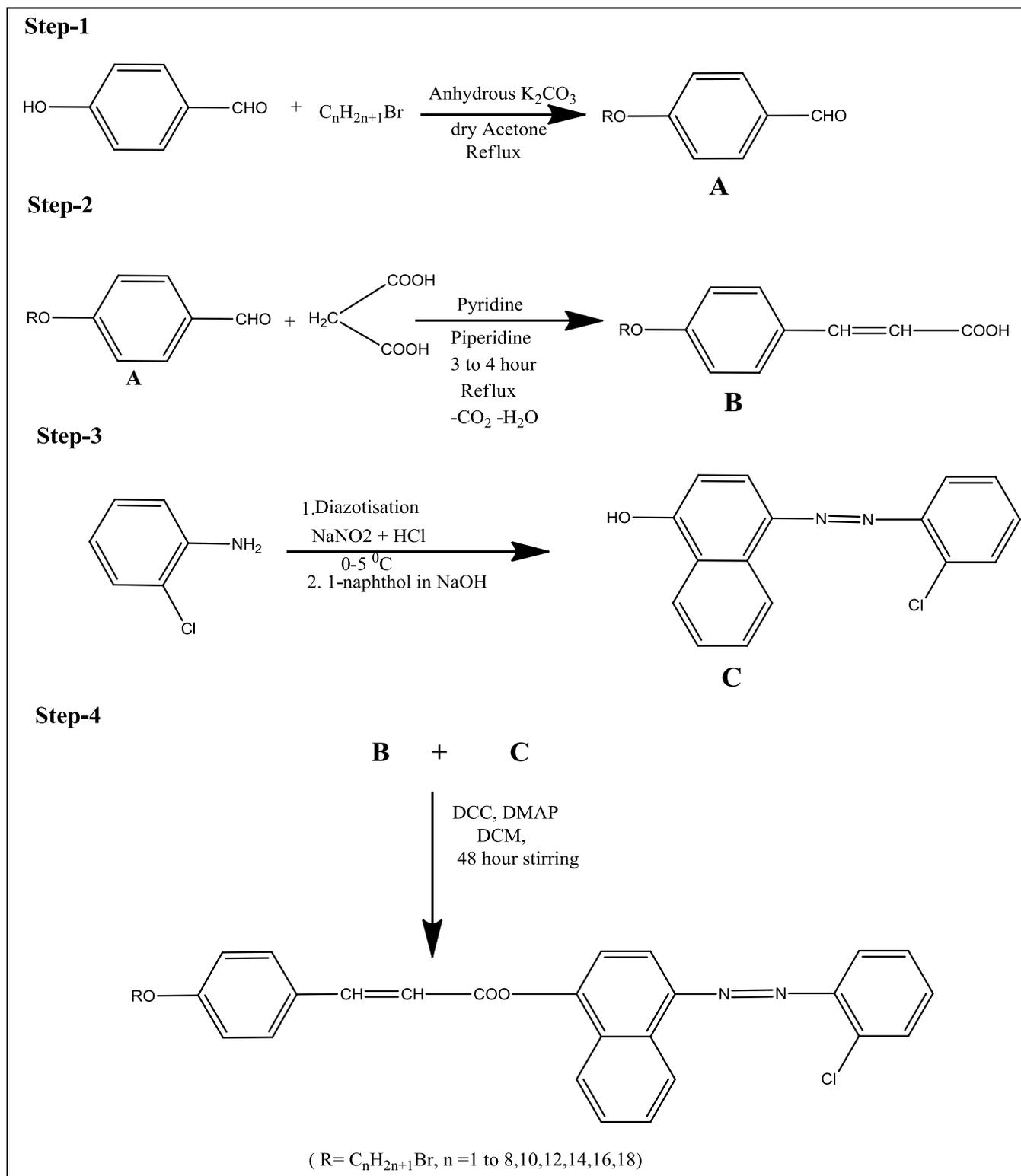


Figure 1. Phase behaviours of Series - 1

2. 3. Reaction Scheme



Scheme 1. Synthetic route to the series.

3. RESULT AND DISCUSSION

4-Hydroxy naphthyl azo-2'-chloro benzene (M.P. 120 °C, yield 81.38%) on linking with trans 4-n-Alkoxy cinnamic acids yielded LC dyes (C₆ to C₁₈) and non LC dyes (C₁ to C₅) in present homologous series of azoesters. The LC homologues are enantiotropic nematic with absence of smectogenic and monotropic nematic properties. The transition temperatures of novel homologues are lower than the corresponding dimerised n-alkoxy cinnamic acids. Transition temperatures as determined from an optical polarising microscopy (POM) equipped with a heating stage are plotted against the number of carbon atoms present in n-alkyl chain (R), bonded to first phenyl ring through oxygen atom and the transition curves, Cr-N/I and N-I are obtained on linking like or related points, which shows, phase behaviours of series. A Cr-N/I transition curve follows a zigzag path of rising and falling with overall descending tendency and behaves in normal manner. A N-I transition curve is descended as series is ascended in normal manner with absence of odd-even effect. The N-I transition curve is extrapolated to nonmesomorphic C₄ and C₅ homologues from C₇ homologue, keeping in view of the trends of a curve to show hypothetically probable odd-even effect. Probable extrapolated curves for odd-even effect merges into each other at the C₇ homologue and then prolonged as a single curve. Textures of nematic phase are threaded or schilieren. Thermal stability for nematic is 145.12 and mesophase length or the degree of mesomorphism vary from 26.0 °C to 46.0 °C at the C₁₄ and C₇ homologues respectively.

The variation in mesomorphism behaviours from homologue to homologue in the same series are observed. The lowering of transition temperatures of novel homologues and the disappearance of dimerization of corresponding trans n-alkoxy aromatic acids are attributed to the breaking of hydrogen bonding between them by the process of esterification. The exhibition of nematic property by C₆ to C₁₈ homologues is due to the disalignment of their molecules of an angle less than ninety degree with the plane of a floating surface and to resist exposed thermal vibrations for definite range of temperature, during which the stastically parallel orientational ordering of molecules is maintained by suitable magnitudes of dispersion forces and dipole-dipole interactions as a consequence of favourable molecular rigidity and flexibility to cause nematic mesophase formation.

However the magnitudes of intermolecular dispersion forces of cohesions and closeness as well as dipole-dipole interactions are unsuitable for C₁ to C₅ as well as C₆ to C₁₈ homologues to either induce any sort of mesomorphism or fails to induce lamellar packing of molecules in preoccupied crystal lattices of rigid crystals to cause nonmesomorphicity in the C₁ to C₅ homologues and absence of smectogenic character in nematogenic homologues. Mesomorphic homologues or non mesomorphic homologues, on continued heating transform into isotropic state, with or without formation of LC state and then molecules are randomly oriented in all possible directions with high order of disorder or high entropy or randomness in uncontrolled manner.

But, on cooling the same from and below isotropic temperature, the mesophase (nematic) reappears for mesogenic homologues (C₆ to C₁₈) exactly at a temperature at which mesophase was appeared in reversible manner. Therefore all transition temperatures for C₆ to C₁₈ homologues are enantiotropic transitions. Nonmesogenic homologues (C₁ to C₅) sharply and directly transform into isotropic state and on cooling the same solidifies in rigid crystalline state due to their high crystallising tendency arising from low dispersion forces and low dipole-dipole interactions as a results of unfavourable combined effect of molecular

rigidity and flexibility. Absence of odd-even effect in N-I curve is attributed to the absence of any sort of mesomorphism in C₁, C₃, C₅ homologues of shorter n-alkyl chain. Hypothetical odd-even effect is attributed to the probable extrapolation [31,32,33] of N-I transition curve which matches with the isotropic point of nonmesogenic C₄ and C₅ homologue from C₇ homologue. Which is actually realizable. Average middle ordered thermal stability 145.12 of nematogenic mesophase is due to the lowering of N-I transition temperatures by breaking hydrogen bonding from dimerized trans n-alkoxy aromatic acids. The variations in mesomorphic properties among the homologues of presently investigated azoester series is attributed to the changing number of methylene unit or units in left -OR flexible group, keeping remaining molecular part unchanged molecular rigidity unaltered throughout the series. Thus, variations in molecular flexibility by 'R' of -OR group operates the mesogenic (LC) behaviours of a series consisted of azoester. The LC properties of present novel series-1 are compared with structurally similar or analogous series X [34] and Y [35] as mentioned below in Figure - 2.

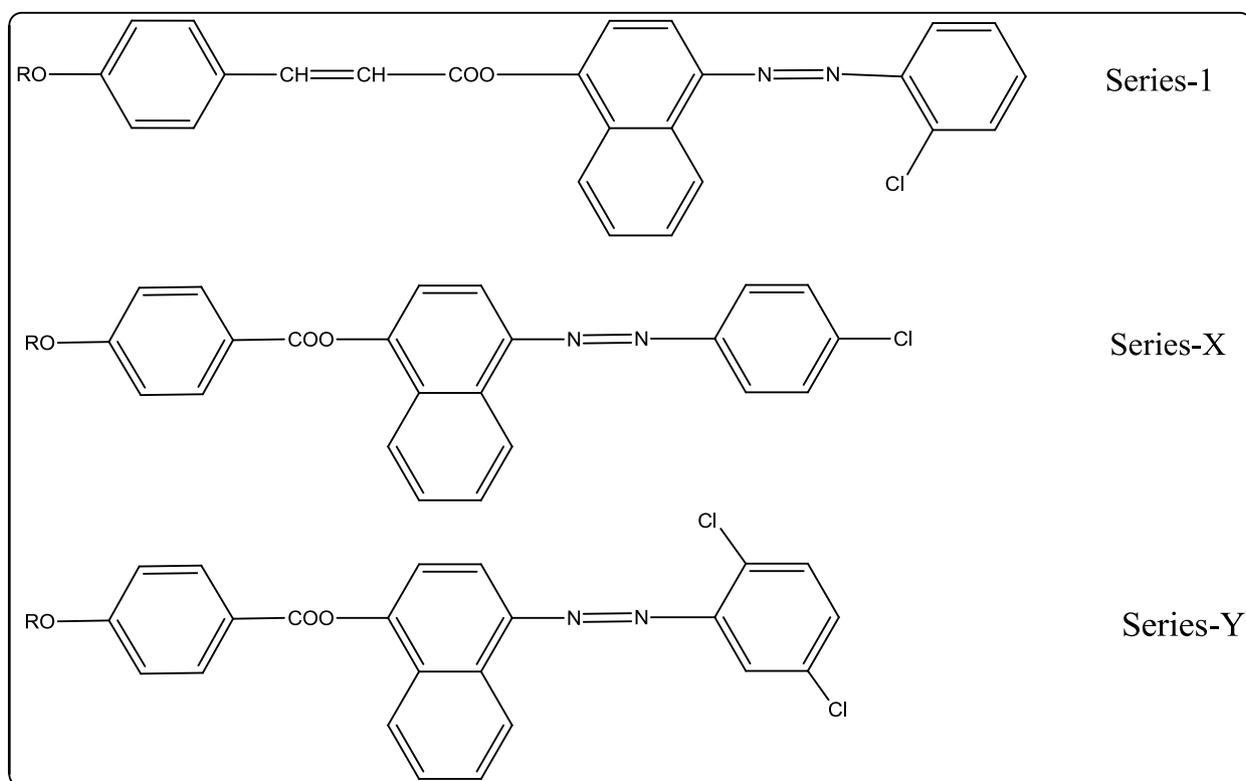


Figure 2. Structurally similar series

Homologues novel azoester series of LC and the analogous series-X and Y selected for comparative study are identical with respect to two phenyl and one naphthyl aromatic rings, which partly contributes equally to total molecular rigidity and an identical flexible left -OR terminally situated end group for the same homologue from series to series which equally and partly contribute to total molecular flexibility.

The central bridges –N=N- is commonly present in all the series 1, X, and Y linking naphthyl ring and a third phenyl ring, contributing partly to the molecular rigidity. However, a central bridges –CH=CH-COO- (Series - 1) and –COO- linking first and middle naphthyl ring contributing partly to total molecular rigidity and substitutional position of chloro group or groups on third phenyl ring, contributing partly to the total molecular flexibility differs for the same homologue and from homologue to homologue in the same series. Thus, variations in the LC properties and the degree of mesomorphism among the series 1, X, and Y will depend upon the differing features of azoester series under present comparative study as shown in Figure - 2. Table 3 represents some LC properties of series 1, X and Y under comparison as mentioned below.

Table 3. Thermal stability in °C.

Series →	1	X	Y
Sm-I or Sm-N commencement of smectic phase	—	—	—
N-I commencement of nematic phase	145.12 (C ₆ - C ₁₈) C ₆	121.9 (C ₃ - C ₁₆) C ₃	129.5 (C ₅ - C ₁₆) C ₅
Total Degree of mesomorphism (Sm+N) in °C	26.0 to 46.0 C ₁₄ C ₇	31.0 to 50.0 C ₁₆ C ₁₂	21.0 to 47.0 C ₅ C ₈

From Table - 3 it is clear that,

- All the homologues series under comparative study are nematogenic only without exhibition of smectogenic mesophase formation.
- Commencement of nematogenic mesophase formation is earliest from C₃ homologue in series-X, than from C₅ homologue in series-Y and then latest from C₆ homologue of series-1.
- Thermal stability for nematic is in decreasing order from series-1 to series-Y to series-X.
- Upper degree of mesomorphism is in decreasing order from series-X to series-Y to series-1 and the lower degree of mesomorphism is in decreasing order from series-X to series-1 to series-Y.

The exhibition of nematogenic character only by all the three azoester series is attributed to the presence of Naphthyl unit in the molecules concerned irrespective of varying central bridges –CH=CH-COO- or –COO- linking first and naphthyl rings ring. However, a central bridge –CH=CH-COO- in case of novel series-1 which is longer than a central bridge -COO-of series X and Y by –CH=CH- unit, causes more noncoplanarity due to a twist

obtained as the oxygen atoms of the vinyl carboxy group bump into the nonbonded adjacent hydrogen atoms of the aromatic rings. On account of this difference the nematic-isotropic thermal stability of series-1 is higher than the N-I thermal stabilities of series X and Y. Moreover, added an unit $-\text{CH}=\text{CH}-$ of conjugated double bond, which, increases molecular rigidity more than corresponding $-\text{COO}-$ central bridge, which favours the suitable magnitude of anisotropic forces of end to end attraction irrespective of ortho or para mono chloro or dichloro group substituted on the last tailed phenyl ring.

Thus, reduction in intermolecular end to end attraction by the replacement of $-\text{CH}=\text{CH}-\text{COO}-$ central bridge by $-\text{COO}-$ and increasing intermolecular distance reflecting polarizability factor by added $-\text{Cl}$ group on 5'' position of series-Y lowers the thermal stability and causes early commencement of mesophase in series-X and Y from C_3 and C_5 homologue respectively as compared to series 1 of present investigation in which nematic mesophase commence late from C_7 homologue, Thus, suitable magnitude of rigidity and flexibility commences late mesophase formation in case of homologous series containing $-\text{CH}=\text{CH}-$ unit. The upper and lower mesophase lengths or the degree of mesomorphism depends upon thermometric resistivity of a molecular structure and rigidity towards exposed thermal vibration, Hence, it varies accordingly with thermal resistivity of a molecule.

4. CONCLUSIONS

- Homologous series of novel azoester derivative is nematogenic and of middle ordered melting type whose thermal stability the highest and the upper and lower degree of nematogenic mesophase lengths are the lowest as well as commencement of nematic phase is the latest; among the series-1, X and Y.
- The group efficiency order derived on the basis of (i) thermal stability (ii) early commencement of mesophase and (iii) upper mesophase lengths for nematic are as under.

(i) Nematic

2''-chloro with $>$ 2'', 5''-dichloro with $>$ 4''-dichloro with
 $-\text{CH}=\text{CH}-\text{COO}-$ $-\text{COO}-$ $-\text{COO}-$

(ii) Nematic

4''-chloro with $>$ 2'', 5''-dichloro with $>$ 2''-chloro with
 $-\text{COO}-$ $-\text{COO}-$ $-\text{CH}=\text{CH}-\text{COO}-$

(iii) Nematic

4''-chloro with $>$ 2'', 5''-dichloro with $>$ 2''-chloro with
 $-\text{COO}-$ $-\text{COO}-$ $-\text{CH}=\text{CH}-\text{COO}-$

- Intermolecular end to end cohesions of a molecular structure, inducing mesomorphism is operated by suitable magnitudes of combined effect of molecular rigidity and flexibility.
- A phenomena of mesomorphism is very sensitive and susceptible to a molecular structure.
- “Naphthyl unit containing homologous series are generally nematogenic.” This statement is very well supported by present investigation.
- Present investigation may be useful for light polarization.
- Present study supports and raises the credibility to the conclusions drawn earlier.

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