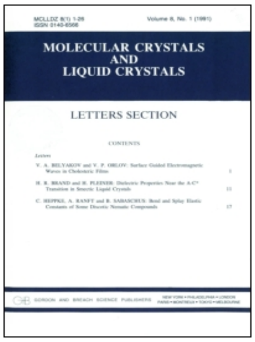

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The effect of molecular rigidity and flexibility on the mesomorphism of azoesters

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ASBTRACT

An Azoester novel homologous series RO-C_6H_4-COO-C_6H_4-N=N-C_6H_4-$OC_4H_{9(n)}$ (para) of liquid crystalline (LC) materials are synthesized and studied with a view to understanding and establishing the effects of molecular structure on liquid crystal behavior with reference to lateral, terminal, or central group or groups on the basis of molecular rigidity and/or flexibility. The novel homologous series consists of thirteen (C_1 to C_{18}) homologues. C_1 to C_5 homologues are nonmesogenic and the rest of the homologues are enantiotropically mesogenic. C_8 to C_{18} homologues are smectogenic plus nematogenic and the remaining two C_6 and C_7 homologues are only nematogenic without exhibition of smectic property. Analytical, thermal and spectral data confirms the molecular structures. Textures and transition temperatures of homologues were determined by an optical polarizing microscope (POM) equipped with a heating stage. Textures of a nematic phases are threaded or Schlieren and that of a smectic phase are of the type of A or C. The average thermal stabilities for smectic and nematic are 111.3°C and 124.7°C, respectively. The smectic and nematicmesophase lengths from 10.0°C to 28.0°C and 7.0°C to 16.0°C, respectively. The transition temperatures are compared with known series.

KEYWORDS
Azoester; liquid crystal; smectic; nematic; monotropy

Introduction

Azoester LC materials [1] are useful for thermographic and LC devices, optical imaging, light emitting diods, semiconductor materials and equally important as dye materials [2–6]. Moreover the central ester group is biologically active, which is useful for mankind [7–10]. The aim is to understand and establish the relation between mesomorphism and molecular structure [11–14] on the basis of molecular rigidity and flexibility [15–18] by changing single phenyl or nepthyl ring as well as fixed tail end group and varying left –OR group for the same series but differ tailed group from series to series for the same homologue. Thus, present investigation is planed with a view to synthesize and then to characterize, through analytical, spectral and thermotropic data. The evaluated data will be discussed and interprited with reference to molecular rigidity and flexibility. Number of ester or azoester series are reported till the date [19–24].

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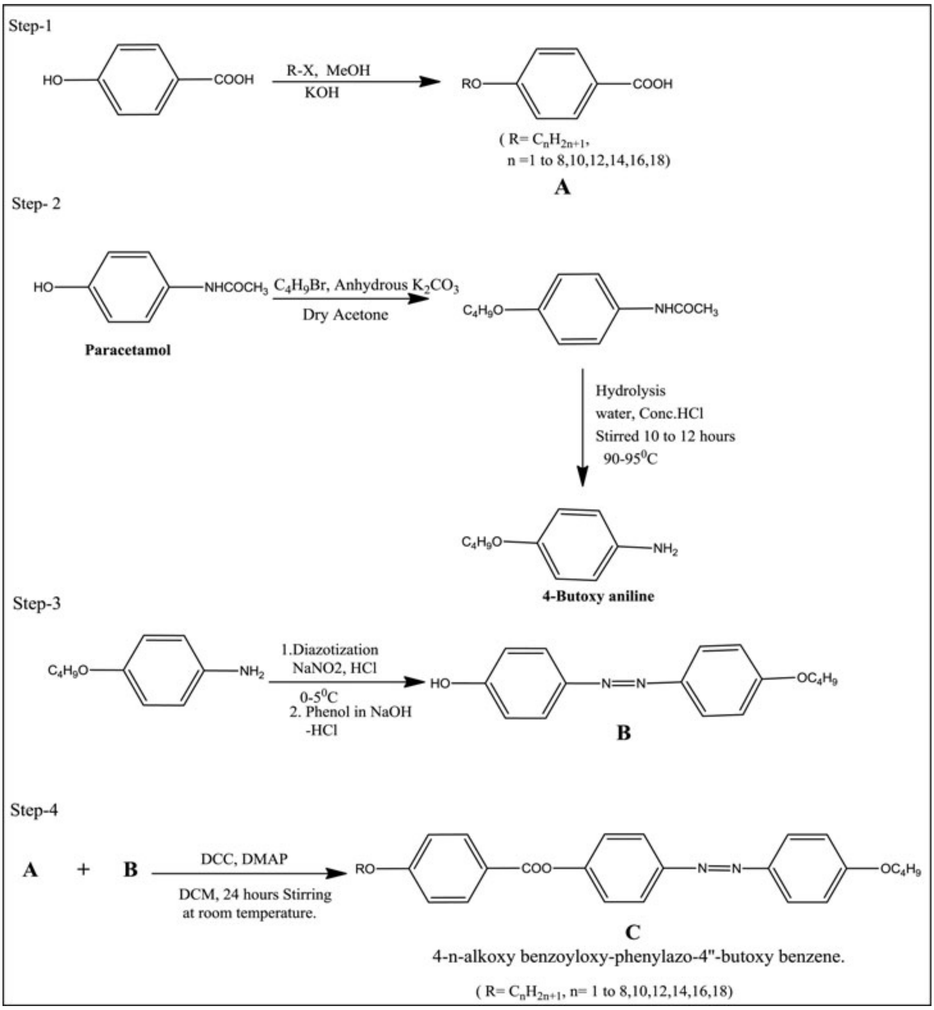


Scheme 1. Synthetic route of Series.

Experimental

Synthesis

4-Hydroxy benzoic acid was alkylated using the appropriate alkylating agent(R-X) to convert it into 4-n-alkoxy benzoic acids(A) by a modified method of Dave and Vora [25]. Alkylation of paracetamol using alkylating agent n-C_4H_9Br was carried out to form 4-n-butyloxy acetanilide, which on hydrolysis was converted to 4-n-butyloxy aniline by a usual establish method. Azodye (B) 4-hydroxy phenyl azo 4′-butyloxy benzene (m.p. 126°C, yield 73%) was prepared by well-known azotization method [26], Final azoester products were synthesized by condensation of (A) and (B) [27]. Thus, the azo-ester homologue derivatives were filtered, washed with sodium bicarbonate solution followed by distilled water, dried and purified until constant transition temperatures obtained, using an optical polarizing microscope equipped with a heating stage. 4-Hydroxy benzoic acid, alkyl halides, paracetamol, phenol, $NaNO_2$, dicyclohexylcarbodimide (DCC), dimethyl amino pyridine (DMAP), K_2CO_3, 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 in Scheme 1.

Table 1. Elemental analysis for propyloxy, hexyloxy, decyloxy, dodecyloxyderivatives.

		%Elements found			%Elements calculated		
Sr. No.	Molecular formula	C	H	N	C	H	N
1	$C_{26}H_{28}O_4N_2$	72.18%	6.46%	6.45%	72.22%	6.48%	6.48%
2	$C_{29}H_{34}O_4N_2$	73.38%	7.14%	5.82%	73.41%	7.17%	5.90%
3	$C_{33}H_{42}O_4N_2$	74.66%	7.84%	5.24%	74.71%	7.92%	5.28%
4	$C_{35}H_{46}O_4N_2$	75.21%	8.20%	4.92%	75.26%	8.24%	5.01%

Characterization

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

Analytical data

IR spectra (KBr) in cm^{-1} for hexyloxy, hexadecyloxy derivatives

Hexyloxy: 761 Poly methylene $(-CH_2-)n$ of $-OC_6H_{13}$, 885(-C-H- def. di-substituted-Para), 690 Polymethylene $(-CH_2-)$ of $-OC_6H_{13}$, 989 (-C-H- def. hydrocarbon), 1060 and 1105 (-C-O-) Str, 1296 and 1321 and 1423, 1494 (-C-O str in $-(CH_2)n$ chain), 1579 (-C-H- def. in CH_2), 1390 (-N=N-)str, 1641 (-C = O group), 1735 (-COO- ester group), 2870 and 3072 and (-C-H str in CH_3). IR confirms the molecular structure.

Hexadecyloxy: 759 and796 Polymethylene $(-CH_2-)n$ of $-OC_{16}H_{33}$, 883(-C-H- def. m di-substituted-Para), 989 (-C-H- def. hydrocarbon), 1008, 1056, (-C-O-) Str, of $-OC_5H_{11}$,1276 and 1392 and 1373, 1246(-C-O str in $-(CH_2)n$ chain, 1496(-C-H- def. in CH_2),1512 and 1392 (-N=N-)str, 1598 (-C = O group), 1737 (-COO- ester group), 2733 and 2916 and 3084 (-C-H str in CH_3).IR confirms the molecular structure.

^{1}HNMR spectra in $CDCl_3$ in δ ppm for tetradecyloxy & octyloxy derivative

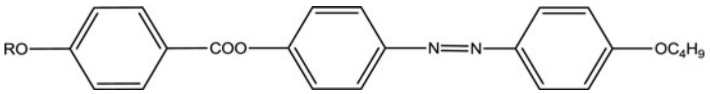
Tetradecyloxy: 0.89(t, $-CH_2-CH_3$, 4H), 1.26 (m, n-poly methylene groups of $-OC_{14}H_{29}$), 1.31–1.76(q, $-CH_2-CH_3$, 6H), 1.76(m, 8H of poly methylene group),3.98(t,$-OCH_2-CH_2-$,4H), 7.19 and 7.83(s, Ar-H, p-disubstituted),8.07–8.19(s, ArH, p-di substituted phenyl ring). NMR confirms the molecular structure.

Octyloxy: 0.88–0.89(t, $-CH_2-CH_3$, 4H, of $-C_8H_{17}$), 1.29(m, n-poly methylene groups of $-OC_8H_{17}$), 1.76(m, 8H of polymethylene), 4.06 (t, 4H, $-OCH_2-CH_2-$of $-OC_6H_{13}$ and $-OC_8H_{17}$), 7.18–7.57 (s, Ar-H, substituted benzene), 8.07–8.11 (s, Ar-H, p-substituted phenyl ring).NMR confirms the molecular structure.

Table 2. Texture of nematic and smectic phase of C_6, C_8, C_{16}, C_{18} by miscibility method.

Sr. No.	Homologue	Texture
1	C_6	Threaded Nematic
2	C_8	Smectic-A
3	C_{16}	Schlieren Nematic
4	C_{18}	Smectic-C

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



4-n-Alkoxy benzoyloxy phenylazo-4"-butoxy benzene.

Compound No	n-alkyl chain C_nH_{2n+1}	Transition Temperatures in(°C)		
		Smectic	Nematic	Isotropic
1	C_1	—	—	172.0
2	C_2	—	—	157.0
3	C_3	—	—	148.0
4	C_4	—	—	140.0
5	C_5	—	—	136.0
6	C_6	—	123.0	130.0
7	C_7	—	108.0	124.0
8	C_8	112.0	122.0	136.0
9	C_{10}	104.0	118.0	128.0
10	C_{12}	94.0	120.0	132.0
11	C_{14}	84.0	112.0	124.0
12	C_{16}	82.0	104.0	116.0
13	C_{18}	72.0	92.0	108.0

Results and discussion

Novel azoester homologous series of LC substances is synthesized by extending molecular length by linking dimeric 4-n-alkoxy benzoic acids and an azodye (m.p.: 126°C, yield 73%) with its nonmesomorphicity which yielded C_6 to C_{18} homologues as mesogenic and the rest of the homologues as non-mesogenic. Mesogenic homologues are smectogenic plus nematogenic except C_6 to C_7 which are only nematogenic without exhibition of smectic property. Thus, mesomorphism commences from C_6 homologue and their transition temperatures are lowered as compared to corresponding dimerisedn-alkoxy benzoic acids on esterification process. Transition temperatures (Table 3) as determined from POM were plotted versus the number of carbon atoms present in n-alkyl chain "R" of –OR group, and then on linking like or related points, the Cr-M / I, Sm-N and N-I transition curves are obtained as shown in a phase diagram (Fig. 1); Cr-M / I transition curve follows a zigzag path of rising and falling with overall descending tendency and behaved in normal manner. Sm-N transition curve rises, passes through maxima at C_{12} homologue of a series and then descended with absence

Table 4. Relative thermal stabilities in°C.

Series→	Series-1	Series-X	Series-Y
Sm-N or Sm-I	111.3	115.5	137.0
Commencement of smectic phase	(C_8-C_{18}). C_8	(C_{12}-C_{18}) C_{12}	(C_{14}-C_{16}) C_{14}
N-I	124.7	138.5	251.7
Commencement of nematic phase	(C_6-C_{18}) C_6	(C_6-C_{18}) C_6	(C_1-C_{14}) C_1
Total (Sm+N) mesophase lengths (from upper to lower)	07.0 to 40.0 C_6 C_{14}	13.0 to 36.0 C_7 C_{14}	70.0 to 170.0 C_3 C_4

Above table-4 indicate that,

- All the homologous series-1, X and Y under comparative study are smectogenic plus nematogenic.
- The commencement of smectic phase is in decreasing order of early commencement of mesophase. i.e., earliest for series-1 and the latest for series-Y.
- Smectic, nematic thermal stabilities and lower mesophase lengths are in increasing order of their sequential order of series under comparison.
- Upper mesophase lengths is the highest for series-Y and the lowest for a series-1 among the series-1, X and Y.

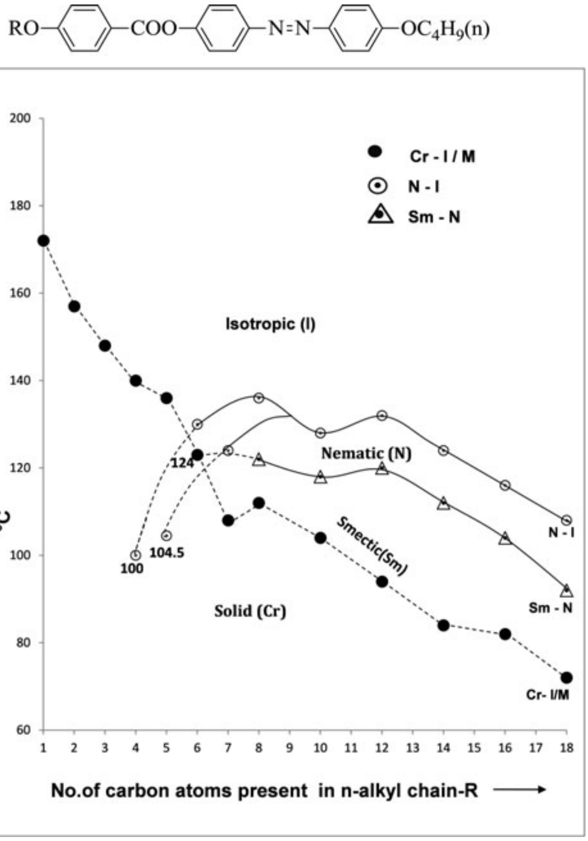


Figure 1. Phase behaviors of Series.

of exhibiting odd–even effect. Thus it behaved in normal established manner. N-I transition curve initially ascended and then descended with negligible deviation at C_{10} homologue with exhibition of odd–even effect from C_6 to C_9 members of a series. Thus, it behaved in normal manner. N-I transition curves for odd and even homologues are merging into each other at C_9 and then odd–even effect disappears for higher homologues of longer n-alkyl chain "R" of –OR group. N-I transition curve for odd members occupy lower position as compared to even members of a series. Textures of a nematic phase are threaded or schlieren and that of a smectic phase are of the type A or C. Thermal stabilities for smectic and nematic are 111.3°C and 124.7°C, whose total mesophase length (Sm+N) vary from 7.0 to 40.0 at the C_6 and C_{14} homologues respectively. The mesogenic properties are varied from homologue to homologue in the same series. Thus, it is predominantly nematogenic and partly smectogenic series of middle ordered melting type series. Sm-N and N-I transition curves are extrapolated to C_6 and C_4 as well as C_5 homologues for predicting and determining [28–31] their latent ability to exhibit smectic or nematicmesophase formation keeping in mind the trend of the transition curves, respectively.

The disappearance of dimerization of n- alkoxy benzoic acids is attributed to the breaking of the hydrogen bonding between the two molecules of aromatic acids by way of esterification process. The exhibition of smectic and/or nematic phase are attributed to extended molecular length which increases length to breath ratio, ratio of the polarity to polarizability, permanent dipole moment across the long molecular axis, dipole–dipole interactions, intermolecular dispersion forces and suitable magnitudes of anisotropic forces of cohesions and closeness as a consequence of favorable molecular rigidity and / or flexibility, which causes the disalignment of molecules at an angle perpendicular to the plane of a floating surface and then at the less than ninety degree with the plane of the floating surface, resisting exposed thermal vibrations containing lamellar packing of molecules in their crystal lattices (C_8-C_{18}) to float with sliding layered molecular arrangement in addition to maintaining statistically parallel orientational order of molecules as facilitated by C_8 to C_{18} homologues and maintaining only statistically parallel orientational order of molecules like C_6 and C_7 homologues to induce either only nematic phase orsmectic plus nematicmesophase or, only nematic phase for definite ranges or range of temperatures. The exhibition of odd–even effect by N-I transition curve is due to the expected status of n-alkyl chain ‘R’ of –OR in which $-CH_2$- group is added in sequential manner. Absence of odd- even effect for Sm-N transition curve is due to the presence of smectogenic character in only even members of homologues C_8, C_{10}, C_{12}, C_{14}, C_{16}, and C18 or due to the absence of odd members. The disappearance of odd-even effect for higher homologues from and beyond C_9 homologue, of longer n-alkyl chain for N-I transition curve, which, may coil or bend or flex or couple to lie with major axis of core structure and adopt unexpected status of n-alkyl chain and modify the magnitudes of intermolecular cohesions, rigidity, flexibility or parameters related to exhibition of mesomorphism. Thus, uncertainity in the status of n-alkyl chain ‘R’, the intermolecular cohesive forces for odd and even members of homologues beyond merging of transition curves do not alternate. The variations in mesogenic properties from homologue to homologue in the same novel series is attributed to the sequentially and progressively added methylene unit or units to the n-alkyl chain ‘R’ which alters the molecular flexibility and the suitable magnitudes of permanent dipole moment and intermolecular dispersion forces, responsible to induce mesomorphism. The absence of mesomorphic property in case of C_1 to C_5 homologues is attributed to their low magnitudes of dipole-dipole interactions and low magnitudes of dispersion forces which induces low thermal resistivity towards exposed thermal vibrations. Therefore, they possess inability to resist thermal break down of their crystal lattices and sharply transform into isotropic liquid from crystalline solid state without passing through LC state and adopt high crystallizing tendency on cooling the same. All the homologues of a series further heating from and beyond isotropic temperature, their molecules are randomly oriented in all possible directions with high order of disorder or randomness or high entropy ($\Delta S = \Delta H/T$) or with uncontrolled irregular motion. But on cooling the same with proper rate of cooling, the mesogenic homologues reversibly exhibited (or mesophase reappeared) their respective type of mesophase or mesophases at the same temperature at which the mesophases were appear on heating (C_6 to C_{18}). Moreover under identical condition, the none of the non-mesogenic (C_1 to C_5) homologues did exhibited any sort of mesomorphism in irreversible manner in monotropic condition. Thus, all transition temperatures of mesogenic homologues are enantiotropic transitions. Therefore N-I and Sm-N transition curves extrapolated to C_4, C_5 and C_6 to predict their latent transition temperature (LTT) which are hypothetical and not real or they are unrealizable due to their respective position, much below to their respective isotropic temperature. Thus, no sooner homologues C_4, C_5 and C_6 reach to their targeted mesophase formation; than prior to that, sample homologue either undergo or acquire crystallization state or matches with isotropic state or acquire untargeted mesophase formation (C_6). The mesogenic behaviors of present novel series-1 are compared with the structurally similar analogous series X [32] and Y[33] as mentioned below in Fig. 2.

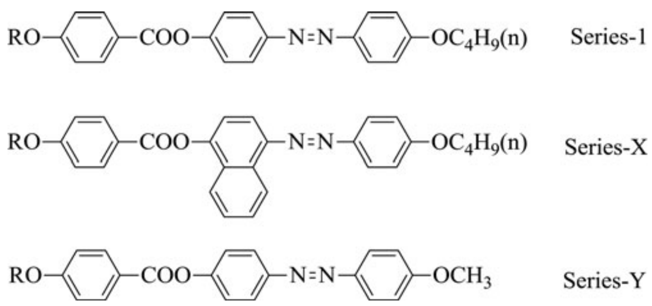


Figure 2. Structurally similar analogous series.

Homologous series-1 of present investigation and a series-Y chosen for comparison are identical with respect to three phenyl rings and two central bridges -COO- and –N=N- contributing to the total molecular rigidity. Moreover left n-alkoxy (-OR) terminal end groups are identically same, contributing partly to the molecular flexibility for the same homologue from series to series, but differs with respect to the tailed end groups $-OC_4H_{9(n)}$ and $-OCH_3$, which contributes partly to the total molecular flexibility. Homologous series-1 and series-X are identical with respect to two central bridges contributing partly to the molecular rigidity but are identically same for the same terminal groups –OR and $-OC_4H_{9(n)}$ for the same homologue from series to series, contributing total molecular flexibility. However, series-1 and X differ with respect to total molecular rigidity due to presence of middle phenyl ring (series-1) and a middle nepthyl ring (series-X) affecting molecular rigidity. Thus, a homologous series-X differs from series-1 with respect to identical molecular flexibility, but differing with respect to total molecular rigidity for the same homologue and vice versa for the series-1 and Y, i.e. series-1 and Y are identical with respect to molecular rigidity but, differing with respect to molecular flexibility for the same homologue. Thus, difference in mesogenic behaviors, properties and the degree of mesomorphism etc. depended upon the differing features among the analogous series-1, X and Y for the same homologue. Following table-4 represents some thermometric properties of series-1, X and Y in comparative manner as under.

Homologous series-1, X and Y are bearing high polar n-alkoxy tailed ends $-OC_4H_{9(n)}$ or $-OCH_3$ and left –OR groups, whose combined effects of molecular rigidity and flexibility are suitable to induce only nematic or smectic plus nematicmesophase or mesophases. Presence of $-OCH_3$ tailed end group being highly polar and polarizable as compared to tail ended $-OC_4H_{9(n)}$ group which maintain end to end attractions strongly and intermolecular closeness which is more favorable of series-Y than series-1 and X. Moreover, the extent of molecular non co-planarity is less favored by –N=N- central bridge in trans condition which causes variations in the magnitudes of intermolecular cohesions, molecular polarity and polarizability or molecular rigidity and flexibility, resulting into early or late commencement of mesophase, thermal resistivity towards exposed thermal vibrations and the magnitudes of lamellar packing of molecules in their crystal lattices. Thus, the variation and stabilization of a mesophase or mesophases are facilitated. Now as regards to the increasing order observed for smectic and nematic thermal stabilities which are related with the energy stored (ΔH) in a molecule for the same homologue from series to series which varied with the flexibility of shorter ($-OCH_3$) and longer ($-OC_4H_{9(n)}$ or $-OCH_2{-}CH_2{-}CH_2{-}CH_3$) tailed ended groups; differing into thermal resistivity or acceptability of thermal vibrations as related to maintenance of magnitudes of ordered molecular arrangement under floating condition on the surface. Thus, magnitudes of stabilization of mesophase or mesophases or degree of mesomorphism, commencement of nematic phase, and thermal stabilities of series-Y are highest and of series-1 is the lowest or lower.

Conclusions

Novel azoester series of LC substances is predominantly nematogenic and partly smectogenic, whose total mesophase lengths vary from 7.0 to 40°C and is of the middle ordered melting type.

The group efficiency order derived for smectic and nematic on the basis of (a) thermal stabilities (b) early commencement of mesophase and (c) upper mesophase lengths are as under.

a) **Smectic**

$$\text{Series} - \text{Y} > \text{Series} - \text{X} > \text{Series} - 1$$

Nematic

$$\text{Series} - \text{Y} > \text{Series} - \text{X} > \text{Series} - 1$$

b) **Smectic**

$$\text{Series} - 1 > \text{Series} - \text{X} > \text{Series} - \text{Y}$$

Nematic

$$\text{Series} - \text{Y} > \text{Series} - \text{X} = \text{Series} - \text{Y}$$

c) **Total mesophase lengths(Sm = +N)**

$$\textbf{Upper}: \text{Series} - \text{Y} > \text{Series} - 1 > \text{Series} - \text{X}$$

$$\textbf{Lower}: \text{Series} - \text{Y} > \text{Series} - \text{X} > \text{Sereis} - 1$$

- Phenomena of mesomorphism is very sensitive and susceptible to a molecular structure of a substance.
- LC materials of present investigation may be useful for optical imaging and as useful as other dying material.
- Present investigation supports and raises credibility to the conclusions drawn earlier.

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