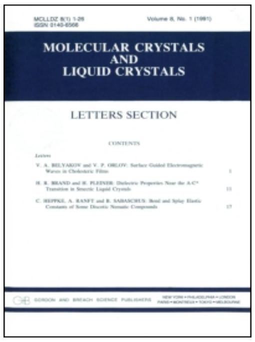

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Dependence of mesomorphism on flexibility of lateral and terminal groups of chalconyl esters

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ABSTRACT

A novel liquid crystalline homologous series of chalconyl vinyl esters with a lateral bromo substituent RO—C_6H_4—CH=CH—COO—C_6H_3—(Br)—CO-CH=CH—C_6H_4—$C_{12}H_{25(n)}$ has been synthesized and studied with a view to understanding and establishing the relation between the molecular structure and liquid crystal (LC) behavior in terms of molecular flexibility of the lateral and terminal groups. The novel homologous series consists of thirteen (C_1–C_{18}) homologues, whose enantiotropic nematic and smectic mesomorphism commences from the C_4, (C_4–C_{18}) and the C_5, (C_5–C_{18}) homologue, respectively. The rest of the homologues (C_1, C_2, C_3) are nonliquid crystals (NLC). Mesomorphism and transition temperatures were examined using an optical polarising microscope (POM) equipped with a heating stage. Textures of nematic phase are threaded or Schlieren and those of the Smectic-A or Smectic-C are focal conic. Analytical, thermal and spectral data supported molecular structures of the novel homologues. Thermal for smectic and nematic are 87.5°C and 101.1°C, respectively, whose, total mesophase length ranges from 17°C to 30°C at C_{14} and C_7 homologue, respectively. The mesomorphic transition temperature ranges are between 70°C and 113°C.

KEYWORDS
Azoester; chalconyl ester; liquid crystals; nematic; smectic

Introduction

The potential dual utility of LC chalconyl ester derivatives [1] to operate LC devices at room temperature and exploitation of their bioactivity as antifungal, antibacterial, antimalarial, antioxidant, anticancer etc. is beneficial through pharmaceutical or agricultural industrial production [2–9] and inspired the present investigation; with a view to understanding and establishing the effects of molecular structure on thermotropic LC properties [10–15]. The object in view is to synthesize novel chalconyl ester derivatives through homologous series, characterization of novel materials through analytical, thermal and spectral methods. The results are discussed and interpreted in terms of molecular rigidity and flexibility [16–19] and finally conclusions are drawn qualitatively including probable utility. Group efficiency order will be derived on the basis of thermal stabilities, early commencement of mesophase and mesophase length from comparative study of present novel series with structurally similar analogous series derived from cis-trans isomeric cinnamic acids. Several homologous series of esters, azoesters, chalconyl esters, benzyl esters have been reported to date [20–25].

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Experimental

Synthesis

4-Hydroxy benzaldehyde was alkylated by using suitable alkyl halides (R-X) to generate 4-n alkoxy benzaldehyde (A) which reacts with malonic acid, in presence of piperidine and pyridine to form trans 4-n alkoxy cinnamic acid (B).

α-3-Bromo-4-hydroxy benzoyl-β-4′-n-dodecyloxy phenyl ethylene (C) was prepared by a usual established method [26]. 4-n-Alkoxy cinnamic acids and α-3-bromo-4-hydroxy benzoyl-β-4′-dodecyloxy phenyl ethylene were condensed in 1, 3-dicyclohexyl carbodiimide (DCC), 4-dimethyl amino pyridine (DMAP) and CH_2Cl_2 (DCM) [27] to obtain α-4-(4′-n-alkoxy Cinnamoyloxy) 3-bromo benzoyl β-4′-dodecyloxy phenyl ethylenes (D) [28]. Final products were filtered, washed, dried and purified until constant transition temperatures obtained. The synthetic route to the novel homologous series of ethylene derivatives is shown in Scheme 1.

Characterization

Some selected representative homologues of the series were characterized by infrared [IR],^{1}HNMR technique and elemental analysis. IR spectra were recorded on Perkin Elmer spectrum GX.^{1}HNMR spectra were recorded on a Bruker spectrometer using $CDCl_3$ as solvent. Elemental analysis was performed on Perkin Elmer PE 2400 CHN analyzer (Table 1). Table 1 shows elemental analysis of mesomorphs and Table 2 shows textures of different phases. Liquid Crystal properties i.e. transition and melting temperatures of homologues were investigated by an optical polarizing microscopy equipped with heating stage. Textures of the novel homologues were determined by miscibility method.

Analytical data

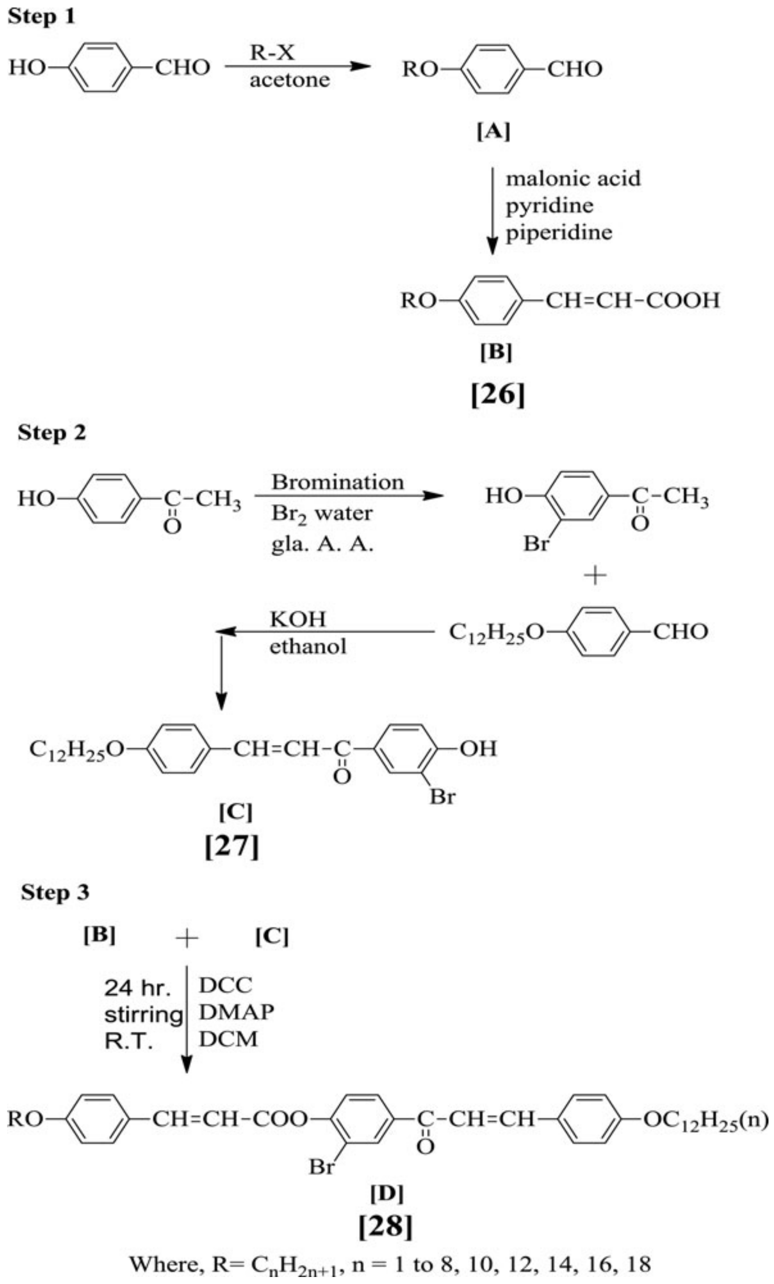
IR Spectra in cm^{-1} for Hexyloxy and Hexadecyloxy Derivatives

Hexyloxy. 501.49 and 651.94 (—CH-Br str.), 721.38 and 767.67 (polymethylene $(—CH_2)_n$ of $—OC_6H_{13}$), 833.25 (—C—H def. of m, p-disubstituted), 979.84 (—C—H def. of hydrocarbon), 1033.85, 1053.13, 1111.00, and 1172.72 (—C—O str.), 1249.87, 1307.74, 1423.47, and 1469.76 (—C—O str. in $(\text{-}CH_2)_n$ chain), 1512.19 (—C—H def. in —CH), 1573.91 (—C=C- str.), 1600.92 (—C=O group), 1689.64 (—COO— ester group), 2850.79, and 2920.23 (—C—H str. in $—CH_3$). IR confirms the structure.

Hexadecyloxy. 520.78 and 651.94 (—CH—Br str.), 721.38 and 763.81 (polymethylene $(—CH_2)_n$ of $—OC_{16}H_{33}$), 833.25 (—C—H def. of m, p-disubstituted), 979.84 (—C—H def. of hydrocarbon), 1033.85, 1053.13, 1111.00, and 1161.15 (—C—O str.), 1249.87, 1307.74, 1427.32, and 1465.90 (—C—O str. in $(—CH_2)_n$ chain), 1512.19 (—C—H def. in $—CH_2$), 1573.91 (—C=C— str.), 1600.92 (—C=O group), 1689.64 (—COO— ester group), 2850.79 and 2920.23 (—C—H str. in $—CH_3$), IR confirms the structure.

1 HNMR spectra in $CDCl_3$ in δ ppm for Heptyloxy and Octadecyloxy Derivative

Heptyloxy. 0.78 and 1.00 ($—CH_3$ of $—OC_7H_{15}$), 2.74 & 3.27 (—CH=CH—), 3.76 (t, $—OCH_2—CH_2—$ of $—OC_7H_{15}$), 5.02 ($—OCH_2$ of $—OC_7H_{15}$), 6.06 (p-substituted phenyl ring). NMR confirms the structure.



Scheme 1. Synthetic route to the series.

Octadecyloxy. 0.89 (—CH_3 of —$OC_{18}H_{37}$), 1.26 (—$(CH_2)_n$ -polymethylene group of —$OC_{18}H_{37}$), 3.82 (t, —OCH_2—CH_2— of —$OC_{18}H_{37}$), 4.98 (—OCH_2 of —$OC_{18}H_{37}$), 8.1 (p-substituted phenyl ring), 3.03 (—CH═CH—). NMR confirms the structure.

Results and Discussion

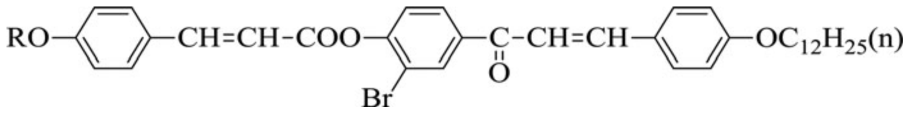
α-3-Bromo-4-hydroxy benzoyl-β-4′-n-dodecyloxy phenyl ethylene (m.p.: 87°C) was condensed with trans 4-n-Alkoxy cinnamic acids to yield novel chalconyl ester derivatives C_1

Table 1. Elemental analysis for butyloxy, pentyloxy, octyloxy, decyloxy.

	Element % found			Element % calculated		
Molecular formula	C	H	Br	C	H	Br
$C_{40}H_{49}O_5Br$	68.54%	6.54%	11.10%	69.66%	7.11%	11.61%
$C_{41}H_{52}O_5Br$	69.34%	6.99%	10.76%	69.88%	7.38%	11.36%
$C_{44}H_{58}O_5Br$	69.75%	7.65%%	10.05%	70.77%	7.77%	10.72%
$C_{46}H_{62}O_5Br$	70.98%	7.84%	9.64%	71.31%	8.01%	10.33%

Table 2. Textures of nematic phase of C_4, C_6, C_{12}, and C_{14} by miscibility method.

Sr. No.	Homologue	Texture
1	C_4	Threaded nematic
2	C_6	Smectic A
3	C_{12}	Smectic A
4	C_{14}	Smectic C

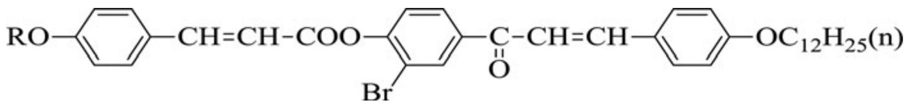


Homologous series: α-4 [4′-n-alkoxy cinnamoyloxy] 3-bromo benzoyl β-4′ dodecyloxy phenyl ethylenes.

to C_{18} homologues. The C_1 to C_3 homologues are nonliquid crystals but the rest of the homologues C_4 to C_{18} are enantiotropically liquid crystals. All the mesomorphic homologues are smectogenic plus nematogenic except C_4 homologue, which exhibited only nematic property without exhibition of smectic property. Transition temperatures of novel homologues are lower than corresponding n-alkoxy cinnamic acids. Alternation of transition temperatures are observed from C_4 to C_8. Transition and melting temperatures (Table 3) as determined from POM equipped with a heating stage were plotted against the number of carbon atoms present in n-alkyl chain 'R' of –OR group and then, transition curves Cr-M/I, Sm-N and N-I are obtained on linking like or related points, as shown in Fig. 1 of a phase diagram. Sm-N

Table 3. Transition temperature in°C.

		Transition temperatures in°C		
Compound no.	Homologue (n-alkyl chain)	Smectic	Nematic	Isotropic
1	C_1	—	—	130.0
2	C_2	—	—	132.0
3	C_3	—	—	134.0
4	C_4	—	79.0	108.0
5	C_5	86.0	98.0	113.0
6	C_6	84.0	92.0	106.0
7	C_7	80.0	90.0	110.0
8	C_8	81.0	91.0	104.0
9	C_{10}	78.0	84.0	98.0
10	C_{12}	76.0	86.0	94.0
11	C_{14}	72.0	79.0	89.0
12	C_{16}	73.0	88.0	101.0
13	C_{18}	70.0	80.0	88.0



Homologous series: α-4 [4′-n-alkoxy cinnamoyloxy] 3-bromo benzoyl β-4′ dodecyloxy phenyl ethylenes.

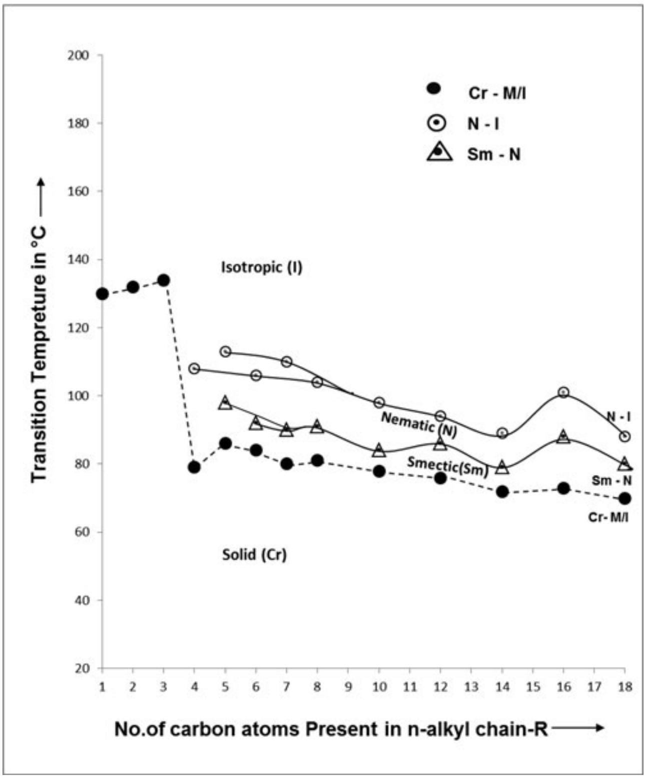


Figure 1. Phase behavior of series.

and N-I transition curves exhibited odd-even effect up to C_8 and C_9 homologues, respectively, beyond which odd-even effects disappears for higher homologues of longer n-alkyl chain 'R' of —OR group. The LC property commenced from C_4 homologue and it varies from homologue to homologue in the same novel series. Thermal stabilities for smectic and nematic are 87.5 and 101.1, respectively, whose Sm-N mesophase lengths vary from 7.0°C to 15.0°C and N-I mesophase length vary between 8.0°C and 29.0°C. Analytical, thermal and spectral data confirmed the molecular structures of homologues.

Condensation of vinyl carboxy acids with chalcone, increases the molecular length, permanent dipole moment across the long molecular axis, suitable magnitudes of anisotropic forces of intermolecular end to end and lateral attractions as a consequence of effective molecular rigidity and flexibility, which facilitate mesophase formation from C_4 member of a novel series. The lowering of transition temperatures of novel homologues as compared to the corresponding n-alkoxy trans cinnamic acid is attributed to the breaking of hydrogen bonding between their two molecules by esterification process. The combined effect of molecular structural rigidity and flexibility operating the suitable magnitudes of anisotropic forces of end to end and lateral intermolecular attractions and favorable closeness do not induce either nematic or smectic mesophase from C_1 to C_3 homologues due to their high crystallizing tendency as a result of respective low magnitudes of dispersion forces and the low magnitudes of dipole–dipole interactions. However, favorable and suitable magnitudes of anisotropic forces of intermolecular end to end and lateral attractions due to the effective polarizability contributed by —Br polar flexible lateral group and two terminally situated flexible polar end groups, initiated from C_4 and ended to C_{18} homologue, which, disalign the molecules at an angle ninety degree or/and less than ninety degree with the plane of floating surface; to

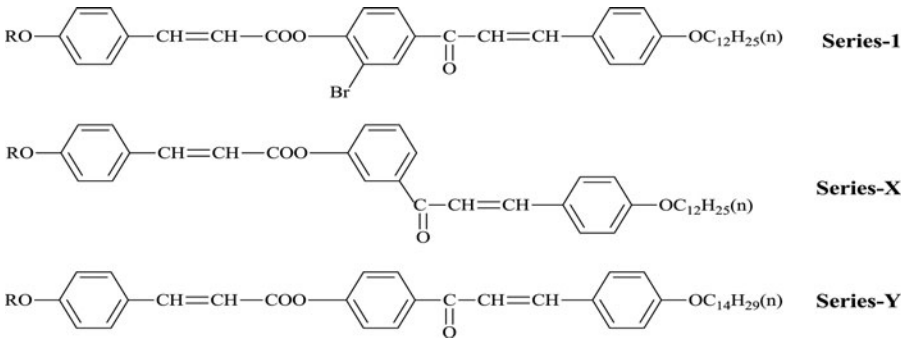


Figure 2. Structurally similar analogous series.

arrange the molecules in sliding layered molecular organization and/or then in statically parallel orientational ordered organization under the influence of exposed thermal vibrations for different range of temperature from homologue to homologue in the same series. Thus, smectic plus nematic (C_5–C_{18}) or only nematic mesophase (C_4) facilitated. None of the non-mesogenic homologues (C_1, C_2, C_3) did show monotropic mesophase formation on cooling below isotropic temperature in irreversible manner due to their high crystallising tendancy. The appearance of odd-even effect in the Sm-N or N-I transition curves of a phase diagram up to C_8 or C_9 homologue is attributed to the gradual and sequential addition of a methylene unit for the shorter n-alkyl. The odd-even effect disappears from and beyond higher homologues of longer than C_8 or C_9. The low thermal stabilities for smectic and nematic in a series containing —CH=CH— units in the both central bridges is attributed to the presence of laterally substituted polar and polarizable Bromo flexible group which broadens a molecule and increases intermolecular distance. The variations in mesomorphic properties from homologue to homologue in the present novel series is attributed to the changing number of carbon atoms present in n-alkyl chain 'R' of —OR group, keeping the rest of the molecular part unaltered; which causes variation in molecular flexibility, keeping molecular rigidity unchanged. The mesomorphic properties or behaviors of present novel series-1, are compared with the structurally similar analogous series-X [29] and Y [30] as in Fig. 2.

Figure 2 represents that, present novel homologous series-1, X and Y are identical with respect to three phenyl rings and two central bridges —CH=CH—COO— and —CO—CH=CH— which constitutes total molecular rigidity depending upon shape, size and induced flexibility of terminal and lateral groups as well as their polarity and polarizability. The left n-alkoxy group –OR and tailed ended $—OC_{12}H_{25(n)}$ of series-1 and X are same for the same homologue from series to series, but their geometrical shapes as well as presence of lateral group —Br and —H are different; whose polarity and polarizabilities or the ratio of the molecular polarity and polarizability causes differences in the magnitudes of molecular flexibility. Homologous series-1 and Y are identical with respect to geometrical shapes, for the same homologue from series to series, but, they differ with respect to tailed ended groups $—OC_{12}H_{25(n)}$ and $—OC_{14}H_{29(n)}$ including effect due to cis-trans isomerism of vinyl carboxylic acids as well as polarizability or the ratio of the polarity to polarizability affecting magnitudes of molecular flexibility for the same homologue from series to series. Thus, variations in LC properties, behaviors and the degree of mesomorphism are depended upon the magnitudes of differing features amongst the series-1, X and Y for the same homologue from series to series or from homologue to homologue in the same series, affecting the combined effect of molecular rigidity plus flexibility which operates, the suitable or unsuitable magnitudes of anisotropic

Table 4. Relative thermal stabilities in°C.

Series→	Series-1	Series-X	Series-Y
Sm-N or Sm-I	87.5	110.88	
Commencement of smectic phase	$(C_5{-}C_{18})\ C_5$	$(C_5{-}C_{18})\ C_5$	—
N-I	101.1	136.2	99.75
Commencement of nematic phase	$(C_4{-}C_{18})\ C_4$	$(C_4{-}C_{18})\ C_4$	$(C_6{-}C_{18})\ C_6$
Total mesophase length (Sm + N) in°C from minimum to maximum	17.0 to 30.0 C_{14} C_7	06.0 to 54.0 C_4 C_{12}	19.0 to 34.0 C_6 C_8

forces of intermolecular cohesion and closeness to facilitate or to not facilitate a phenomena of mesomorphism. Following Table 4 represents some thermometrically evaluated data of the analogous series-1, X and Y under comparative study.

Above Table 4 indicates that,

- Homologous series-1 and X are smectogenic plus nematogenic but, series Y is only nematogenic without exhibition of smectogenic property.
- Smectogenic property commences from C_5 homologue for present series-1 and a series-X chosen for comparative study but, it does not commence till the last C_{18} homologue of a series-Y.
- The nematogenic mesophase commences from C_4 homologue for series-1 and X, but it commences late from C_6 homologue of a series-Y.
- Smectic thermal stability increases from series-1 to X but it does not stabilise at all for series-Y.
- The nematogenic thermal stabilities of series-1 and Y are lower than a series-X, i.e., Series-X > Series-Y > Series-1.
- Total lower and upper mesophase lengths follow increasing order as under:

Lower: series-Y > series-1 > series-X

Upper: series-X > series-Y > series-1

Homologous series-1 and X derived from condensation of trans n-alkoxy cinnamic acids and corresponding Bromo substituted 4-hydroxy or 3-hydroxy derivatives of 4′-dodecyloxy chalcones. Therefore, Bromo substituted lateral group of series-1 and nonlinear shape of series-X increases intermolecular attractions and distance due to enhancing polarizability. But the enhancement in molecular polarizability in competition with intermolecular distance which causes opposing effects i.e. reduction in intermolecular adhesive forces which predominates in case of series-X as compared to series-1. Hence the lateral Bromo group of series-1 is less effective as compared to effectivity induced by polarizability factor through nonlinear shape of a series-X. Thus, both series-1 and X commences smectic and nematic mesophases from C_5 and C_4 homologues, respectively, in equal manner, but, the resulted magnitudes of intermolecular attractions differs due to differing magnitudes of induced polarizability as a consequence of combined effect of molecular rigidity and flexibility of series-1 and X, respectively. Thus, resistivity toward exposed thermal vibrations and the internal energy stored (ΔH) by molecules for the same homologue from series to series and from homologue to homologue in the same series differs, irrespective of identical commencement of smectic and nematic phase; because the commencement of mesophase depends upon only suitable magnitudes (not more or less) of anisotropic forces of intermolecular end to end or / and lateral attractions. Homologous series-Y is synthesized from cis isomer of 4-n-alkoxy cinnamic acid and 4-hydroxy derivative of 4′-tetradecyloxy chalcone. Therefore, carbonyl unit of chalcone central bridge, though linked directly with middle phenyl ring like molecules of series-1 and X, but, a lamellar packing of molecules in the crystal lattices of series-Y are not facilitated due to cis configuration of cinnamic acid, whereas, it facilitated in case of molecules of series-1 and X which are formed from trans configurated cinnamic acid. Thus, smectogenic character is absent in the molecules of series-Y or only nematic property is exhibited by the molecules of series-Y and smectic plus nematic property are exhibited by the molecules of series-1 and X. Hence, cis-trans isomerism of a molecule can affect the extent of molecular noncoplanarity, resistivity against exposed thermal vibrations, the degree of mesomorphism, type and extent of disalignment of molecules on floating surface, thermal stability for smectic and nematic and respective transition temperature as well as texture, magnitudes of energy stored (ΔH), magnitudes of dispersion forces, dipolarity of bond between carbon of phenyl ring and tailed end group and dipole–dipole interactions, etc. Thus, presently investigated series-1 is predominantly nematogenic and partly smectogenic.

Conclusions

- Novel chalconyl series with Bromo substituted lateral group ester series of liquid crystal is predominantly nematogenic and partly smectogenic is of lower middle ordered melting type series.
- Trans configurated cinnamic acid derivatives of LC chalcone which links middle and third phenyl ring by $-\mathrm{CO}-\mathrm{CH}=\mathrm{CH}-$ group facilitate the formation of smectic phase, but, isomeric cis configurated cinnamic acid under identical condition, is missing facilitation of smectogenic mesophase formation.
- Group efficiency order derived on the basis of (i) thermal stability, (ii) early commencement of mesophase, and (iii) mesophase lengths for smectic and nematic are as under.
    1. Smectic Nonlinear > Linear series-1 > Linear series-X with –Br lateral series-Y group Nematic Series-X > Series-1 > Series-Y
    2. Smectic Series-1 = Series-X > Series-Y Nematic Series-1 = Series-X > Series-Y
    3. Total mesophaselength (Sm ± N)

Lower: Series-Y > Series-1 > Series-X
Upper: Series-X > Series-Y > Series-1

- Cis-trans isomerism of a molecular structure affects a phenomena of mesomorphism, the degree of mesomorphism and the type of texture of mesophase.
- Mesomorphism is very sensitive and susceptible to a molecular structure as a consequence of effective molecular rigidity and flexibility.
- Study of biological activity and application of thermotropic LC at desired temperature can be useful to the mankind.
- Present novel investigation supports and raises the conclusions drawn earlier and contribute more to the existing conclusions.

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