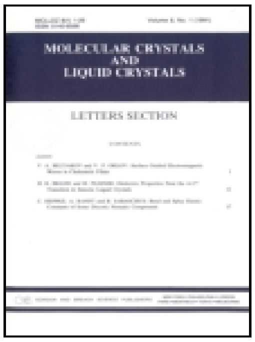

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Mesomorphism dependence on halogenated chalconyl esters in terms of molecular flexibility/rigidity

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ABSTRACT

Vinyl carboxy central group containing a novel chalconyl ester homologous series: $RO.C_6H_4.CH = CH.COO.C_6H_4.CH = CH.CO.C_6H_4.I$ (para) have been synthesized and studied with a view to understand and establish the relation between thermotropic liquid crystalline (LC) behaviors and the molecular structure. Novel chalconyl ester homologous series consists of thirteen homologs (C_1–C_{18}). C_1–C_3 homologs are nonmesogenic, C_4 homolog is enantiotropic nematogenic and the rest of the homologs (C_5–C_{18}) are enantiotropically smectogenic plus nematogenic. Phase transition temperatures and textures of LC phase were determined by an optical polarizing microscopy (POM) equipped with a heating stage. Cr-M/I, Sm-N, N-I transition curves in a phase diagram behaved in normal manner. Sm-N and N-I transition curves exhibited odd-even effect from C_4–C_{10} or nearby C_{10} homolog. Textures of nematic phase are threaded or Schlieren and that of the smectic phase are of the type smectic A or C. Analytical and spectral data confirms the molecular structure of homologs. Thermal stabilities for smectic and nematic mesophases are 155.0 and 180.7, respectively, and their corresponding mesophaselengths are varied from minimum to maximum at 17.0°C–39.0°C as well as 15.0°C–30.0°C. Thus, it is middle ordered melting type series. The group efficiency orders derived on the basis of smectic and nematic thermal stabilities are as under: Sm: $-C_6H_4.I > -C_6H_4.Cl > -C_4H_3S$ and N:- $C_6H_4.I > -C_6H_4.Cl > -C_4H_3S$ from comparative study of structurally similar analogous series

KEYWORDS
Enantiotropy; liquid crystals; mesophaselength; nematic; smectic

Introduction

A liquid crystalline (LC) state [1] has proved its ability and importance in the benefit of mankind in the various fields of scientific and technological applications [2–9]. Present investigation on thermotropic LC state have been planned with an aim to understand and establish the effect of molecular structure on LC properties [10–14] of novel substances. Novel substances will be synthesized through homologous series consisting of two or three phenyl rings, one or two central bridges, and two terminal end groups viz. –OR and –I, the left and right sided polar terminal groups. One of the central groups will be –CH = CH–CO– which will be bioactive and other will be vinyl carboxylate ester group. Thus, thermotropy which may be useful for LC devices as well as bioactivity may be useful for LC devices and bioactivity which

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may be useful for its lyotropic study. Hence dual study of same novel compounds can be facilitated. Present study will include only synthesis, characterization, results and discussion will be interpreted on the basis of molecular rigidity and flexibility [15–17] for thermometric data of novel substances. Number of ester homologous series have been reported till the date [18–25]. Group efficiency order on the basis of thermal stability and the early commencement of mesophase will be derived from comparative study of analogous series.

Experimental

Synthesis

4-*n*-alkoxy cinnamic acids (A) were synthesized by the alkylation of 4-hydroxy benzaldehyde to form 4-alkoxy benzaldehyde and further reaction with Malonic acid in pyridine in presence of few drops of piperidine as a catalyst [26], α-4-Hydroxy phenyl β-4$'$-iodo benzoyl ethylene (m.p 167°C, yield 76%) (B) was prepared by usual established method [27]. Final product were prepared by the esterification of (A) and (B) [28]. Thus, the ester chalconyl 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. Alkyl halides, EtOH, KOH, Acetone, DCM, 4-Iodo acetophenone, 4-Hydroxy benzaldehyde, dicyclohexyl carbodimide, Dimethyl amino pyridine, Malonic acid etc., 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 below as scheme-1

Characterization

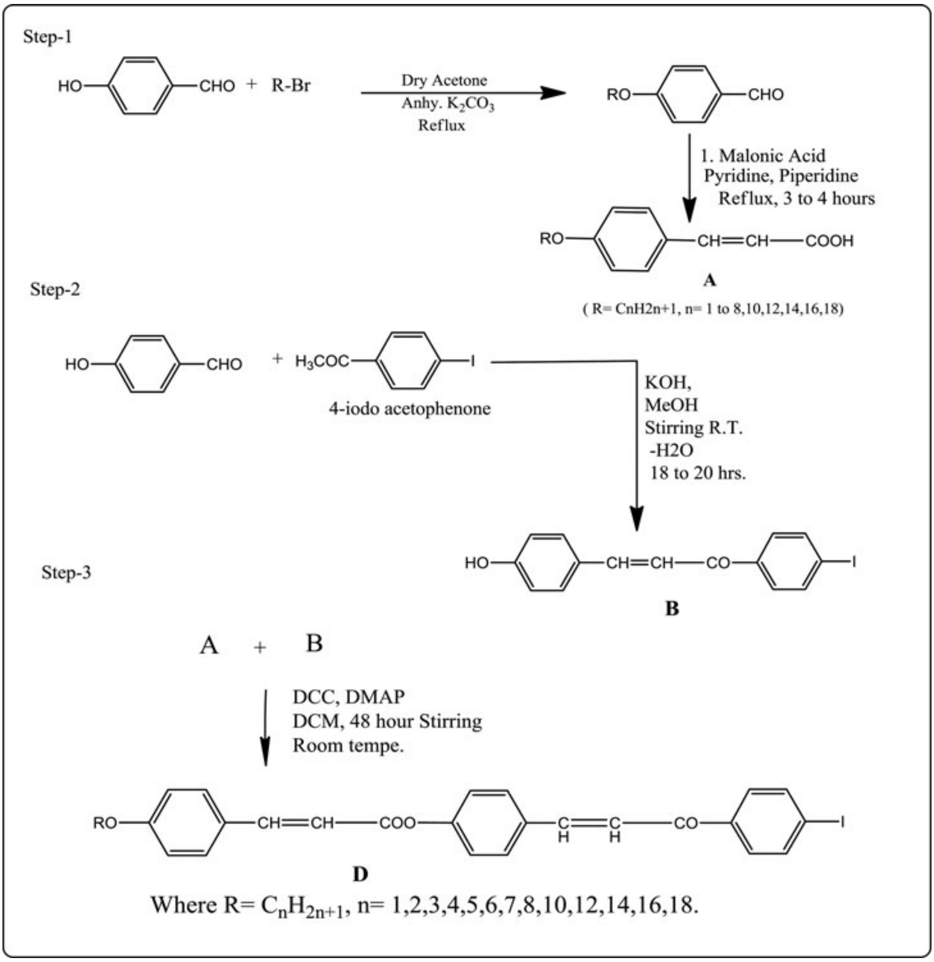
Representative homologs of a series were characterized by elemental analysis, Infrared spectroscopy, ^{1}H NMR spectra, IR spectra were recorded on Perkin-Elmer spectrum GX, ^{1}H NMR spectra were recorded on Bruker using $CDCl_3$ as solvent. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyzer (Table 1). Transition temperature (Table 4) and LC properties (Textures) were determined using an optical polarizing microscopy equipped with heating stage and digital camera (POM). Texture images of nematic phase were characterized by miscibility method (Table 2), Decomposition temperatures (Table 3) were determined using of Shimadzu DSC 60 differential Scanning Calorimeter with a heating rate of 5–10.0°C min^{-1} in air and it was calibrated with indium(156.6°C, 28.45 Jg^{-1}) Holland. Thermodynamic quantities enthalpy (ΔH) and entropy($\Delta S = \Delta H/T$) are qualitatively discussed.

Analytical data

IR spectra in cm^{-1} for hexadecyloxy & octadecyloxy derivatives

Hecadecyloxy: 2924 (C-H str. of alkane), 2850 (C-H str. of $-(CH_2\text{-})n$ group of $-OC_{16}H_{33}$ gorup,1658 (C = O str. of carbonyl carbon of ester), 1600 (C = C str. of alkene), 1448, 1510, 1571 (C = C str. of aromatic ring), 952, 1024 (C-H bending of alkene), 1170 (C-O str. of ether linkage), 1251 (C-O str. of ester group), 1359 (C-H bending of alkene), 648, 570 (C-I str.). IR data confirms the molecular structure.

Octadecyloxy: 2916 (C-H str. of alkane), 2870 (C-H str. of $-(CH_2\text{-})n$ group of $-OC_{18}H_{37}$ group, 1730(C = O str. of carbonyl carbon of ester), 1600(C = C str. of alkene), 1467, 1533,



Scheme 1. Synthesis route to the series.

Table 1. Elemental analysis for (1) hexyloxy (2) heptyloxy (3) decyloxy (4) octadecyloxy derivatives.

		%Elements found			%Elements calculated		
Sr. No.	Molecular formula	C	H	I	C	H	I
1	$C_{30}H_{29}O_4I$	62.41	4.98	21.80	62.50	5.03	21.87
2	$C_{31}H_{31}O_4I$	62.66	5.18	21.20	62.73	5.22	21.24
3	$C_{34}H_{39}O_4I$	64.00	6.08	19.72	64.05	6.12	19.78
4	$C_{42}H_{53}O_4I$	67.39	8.03	16.82	67.46	7.09	16.86

Table 2. Texture of nematic phase of C_5, C_6, C_{12}, C_{14} by miscibility method.

Sr. No.	Homolog	Texture
1	C_5	Threaded
2	C_6	Threaded
3	C_{12}	Schlieren
4	C_{14}	Schlieren

Table 3. Transition temperature (°C) and enthalpy (J g^{-1}) and entropy change (J $g^{-1}k^{-1}$) by DSC measurement.

Homologs or Comp.	Transition	Heating scan(°C)	Cooling scan(°C)	ΔH (-Jg^{-1})	ΔH (Jg^{-1})	ΔS(J $g^{-1}k^{-1}$)	ΔS(J $g^{-1}k^{-1}$)
C_4	**Cr-SmC**	–	–	–	–	–	–
	SmC-N	175	168	8.32	7.21	0.0185	0.0163
	N-I	>180	–	–	–	–	–
C_7	**Cr-SmC**	122	121	30.30	33.01	0.0767	0.0837
	SmC-N	159	160	6.28	5.97	0.0145	0.0137
	N-I	>170	–	–	–	–	–
C_{10}	**Cr-SmC**	134	132	38.54	40.08	0.0946	0.0989
	SmC-N	166	162	7.84	6.62	0.0178	0.0152
	N-I	>180	–	–	–	–	–
C_{14}	**Cr-SmC**	116	104	33.56	34.06	0.0862	0.0903
	SmC-N	137	131	5.56	5.40	0.0135	0.0133
	N-I	>145	–	–	–	–	–

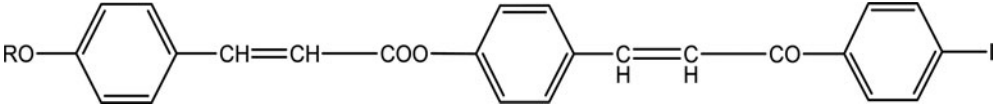
1579 (C = C str. of aromatic ring), 1004, 1062 (C-H bending of alkene), 1143, 1199 (C-O str. of ether linkage), 1251 (C-O str. of ester group), 1309 (C-H bending of alkene), 690, 570 (C-I str.). IR data confirms the molecular structure.

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

Heptyloxy: 0.82 (t, 3H, -CH_3 of –C_7H_{15}), 1.29 (t, 4H, CH_3-CH_2-CH_2-CH_2-CH_2- OC_7H_{15}), 1.43 (p, 5H –CH_2-CH_2-CH_2- of –OC_7H_{15}), 1.31 (q, 4H, -CH_2-CH_3), 1.73(t, 2H,-CH_2-CH_2-of -OC_7H_{15}), 4.06(t, 3H, -OCH_2-CH_2-of –OC_7H_{15}), 6.51 & 7.59 (d, 2H, -CH = CH-), 7.2 & 7.74 (4.01 H, middle phenyl ring), 7.65 & 7.97 (4H, third phenyl ring), 6.93 & 7.61 (4H, phenyl ring with alkoxy chain). NMR data confirms the molecular structure.

Octyloxy: 0.82 (t, 3H, -CH_3 of –C_8H_{17}), 1.75(t, 4H, CH_3-CH_2-CH_2-CH_2-CH_2-CH_2 -OC_8H_{17}), 1.29 (p, 5H –CH_2-CH_2-CH_2- of –OC_8H_{17}), 1.31 (q, 4H, -CH_2-CH_3), 1.73(t, 2H,-CH_2-CH_2-of -OC_8H_{17}), 3.93 (t, 3H, -OCH_2-CH_2-of –OC_8H_{17}), 6.51 & 7.59 (d, 2H, -CH =

Table 4. Transition temperatures in°C for series 1. α-4-(4′-*n*-alkoxycinnamoyloxy) phenyl-β-4″-iodo benzoyl ethylenes.



Sr.no	R = *n*-alkyl group	Transition temperatures in ^{0}C		
		Smectic	Nematic	Isotropic
1	C_1	–	–	210.0
2	C_2	–	–	208.0
3	C_3	–	–	218.0
4	C_4	–	176.0	206.0
5	C_5	137.0	164.0	194.0
6	C_6	142.0	159.0	189.0
7	C_7	128.0	156.0	184.0
8	C_8	130.0	169.0	188.0
9	C_{10}	147.0	177.0	198.0
10	C_{12}	139.0	178.0	193.0
11	C_{14}	128.0	152.0	171.0
12	C_{16}	99.0	122.0	145.0
13	C_{18}	96.0	118.0	139.0

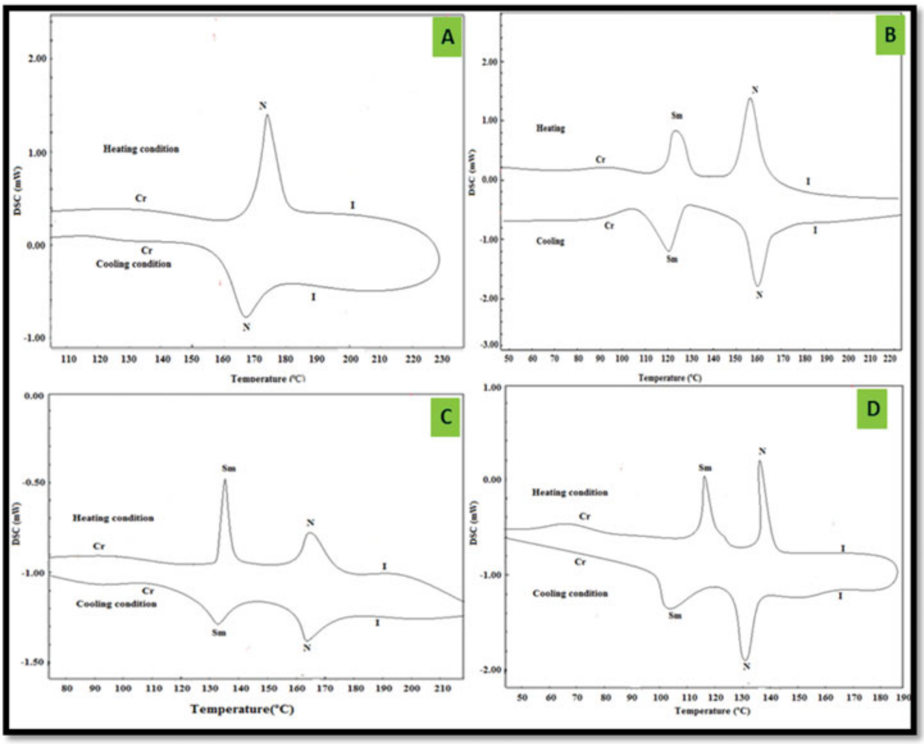
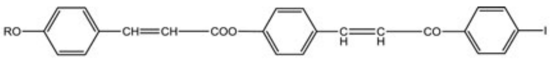


Figure 1. DSC measurement **(A)** C_4 homolog at heating condition endothermic peak at 175°C, while at cooled condition peak observed at 168°C. **(B)** C_7 homolog, first endothermic peak at 122°C and second peak at 159°C during heating stage, while in cooling stage two peak trace at 160°C and 121°C enantiotropically. **(C)** C_{10} homolog, heating condition first endothermic peak observed at 134°C and second endothermic at 166°C and at cooled condition two peaks are obtained at 162°C and 132°C. **(D)** C_{14} homolog, two endothermic peak observed at 116°C and 137°C, while at cooling stage trace two peaks at 131°C and 104°C.

CH-),7.21 & 7.74 (4.01 H, middle phenyl ring), 7.56 & 7.97 (4H, third phenyl ring), 6.93 & 7.61 (4H, phenyl ring with alkoxy chain). NMR data confirms the molecular structure.

Results and discussion

A novel chalconyl ester homologous series of LC (thermotropic) property determined from condensation of dimeric trans cinnamic acids and α-4-Hydroxy phenyl β-4′-Iodo benzoyl ethylene (m.p 167°C & yield 76%) is synthesized and evaluated their some thermometric data to correlate mesomorphic behaviors and the molecular structure through molecular rigidity and flexibility. Mesomorphism commences from C_4 homolog. All the mesogenic homologs are enantiotropically smectogenic plus nematogenic except C_4 homolog which missed smectogenic character and exhibited only nematic character in enantiotropic manner. Transition temperatures (Table 4) as determined using an optical polarizing microscopy (POM) equipped with heating stage are plotted versus the number of carbon atoms present in n-alkyl chain ‘R’ of –OR group. DSC thermogram of C_4, C_7, C_{10}, C_{14} homologue is shown in Figure 1 during heating and cooling condition. Then, on linking like or related transition points; Cr-M/I, Sm-N and N-I transition curves are obtained, showing phase behaviors of series as shown in Fig. 2. Cr-M/I transition curve adopts a zigzag path of rising and falling with overall descending tendency and behaved in normal manner. Sm-N transition curve initially rises



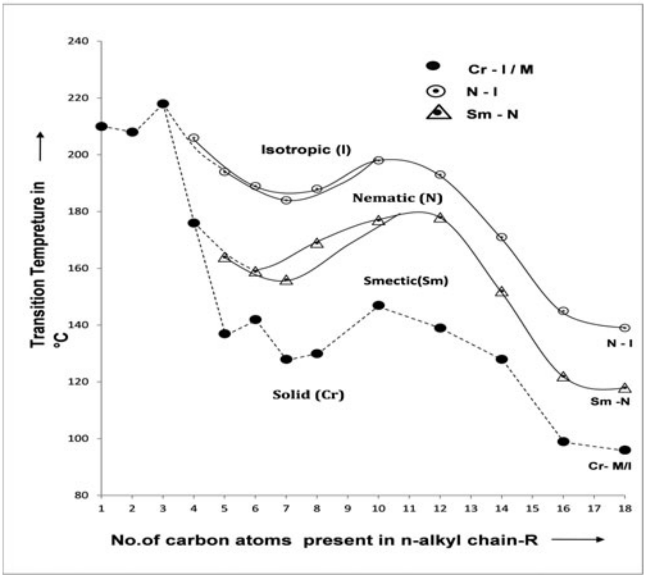


Figure 2. Phase behaviors of series.

and then passes through maxima at C_{12} homolog and descended as series is ascended with exhibition of odd-even effect and behaved in normal manner. N-I transition curve initially descended up to C_7 homolog, ascended up to C_{10} homolog and then descended up to C_{18} homolog with exhibition of odd-even effect. Thus, it behaved in normal established manner. Odd-even effect in Sm-N and N-I transition curves appeared up to nearby C_{10} or exactly at C_{10} homolog respectively, and then it disappeared for higher homologs of longer *n*-alkyl chain 'R' of –OR group. Thus, alternation and lowering of transition temperature as compared to the corresponding trans *n*-alkoxy cinnamic acids are observed for present novel homologous series. Transition curves for smectic and nematic are extrapolated to nonsmectogen C_4 and nonmesogen C_3 homolog, respectively, to predict [29,30,31,32] their probable latent transition temperature (LTT) and to magnify the odd-even effects.

The changing of mesogenic behavior from homolog to homolog in the same novel series is observed by varying number of carbon atom or atoms in *n*-alkyl chain 'R' of -OR group, keeping the rest of the molecular part unchanged throughout a series. Textures of nematic phase are threaded or schlieren and that of the smectic phase are of the type smectic A or C. Analytical, thermal and the spectral data supported molecular structures of homologs. Thermal stabilities for smectic and nematic are 155°C and 180.7°C, respectively, whose total mesophaselength (Sm + N) are from 30°C to 58°C. The POM image and phase sequence at cooled condition shown inFig. 3(i) and Fig. 3(ii), respectively.

The disappearance of dimerization of trans-4-*n*-alkoxy cinnamic acids and the lowering of transition temperatures of corresponding novel homologs of present investigation is attributed to the breaking of hydrogen bonding of trans vinyl carboxylic acids by esterification process. The inexhibition of mesogenic behaviors by C_1, C_2 and C_3 homolog is due to the low magnitudes of dispersion forces and low magnitudes of dipole-dipole interactions which induces unsuitable magnitudes of intermolecular anisotropic forces of cohesion and closeness. Instead, it induces high crystallizing tendency and abruptly breaks the crystal lattices under

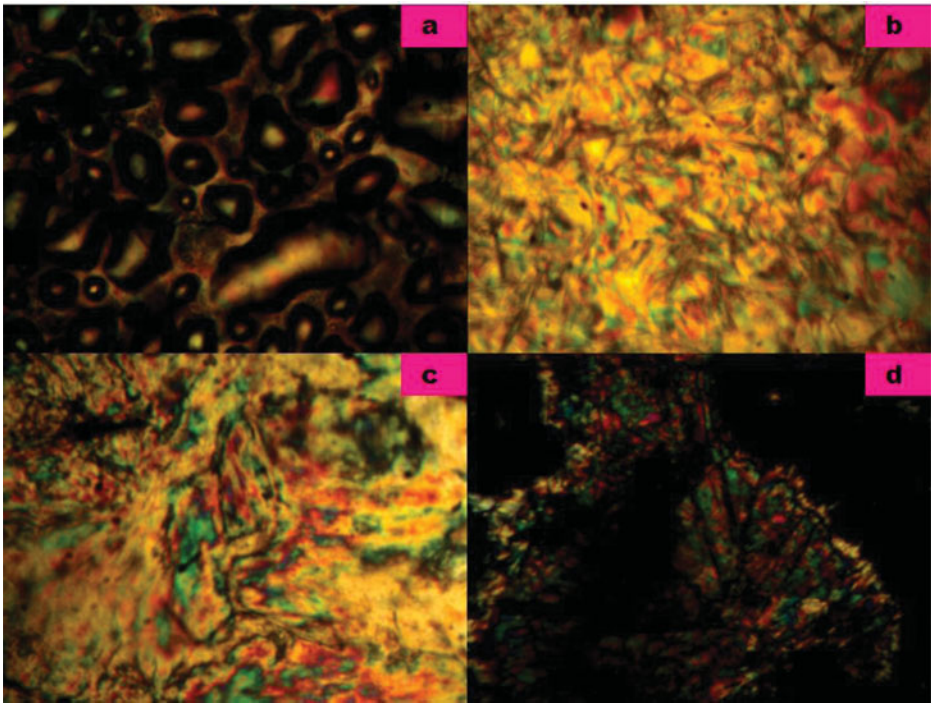
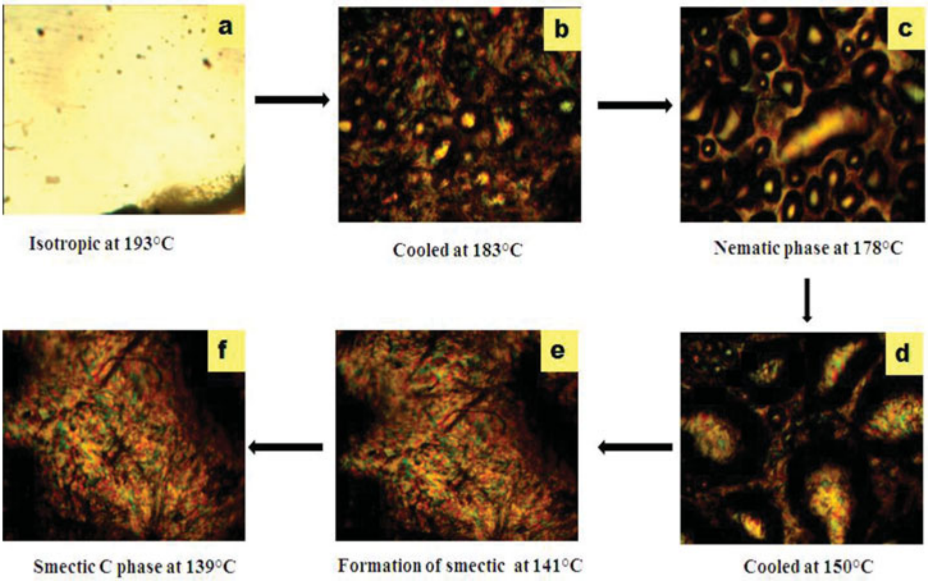


Figure 3. **(i). (a)**. Schlieren type texture of nematic phase of comp. C_{12} at 178°C. (**b**). Sm C phase of comp.C_{14} at 128°C. (c). nematic phase of comp. C_{16} at 122°C. (**d**). smectic phase of comp. C_{18} at 96°C.

thermometric condition and sharply transform into isotropic liquid from crystalline state (or vice versa) without passing through an intermediate state of existence LC. The exhibition of smectic nematic mesophase formation is attributed to the suitable magnitudes of anisotropic forces of intermolecular lateral and then end-to-end cohesion and closeness as a consequence



Isotropic at 193°C

Cooled at 183°C

Nematic phase at 178°C

Cooled at 150°C

Formation of smectic at 141°C

Smectic C phase at 139°C

Figure 3. (ii). Phase sequence of C12 homolog on cooling condition.

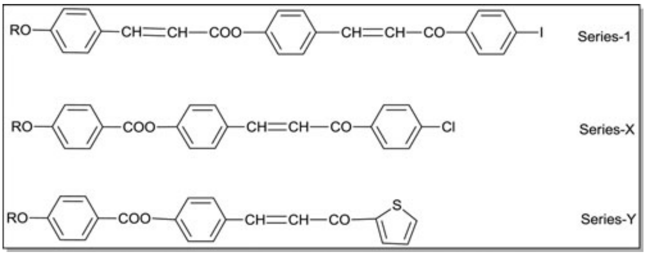


Figure 4. Structurally similar analog series.

of favorable molecular rigidity and flexibility, which maintain the floating of a sample substance with sliding layered molecular arrangement due to preoccupied lamellar packing of molecules for definite range of temperatures and then continue floating with residual end to end attractions which is suitable to facilitate floating of the same smectogenic molecular state with statistically parallel orientational order for another higher ranged temperature to show nematic mesophase formation under the influence of externally exposed thermal vibrations. The exhibition of odd-even effect is due to added methylene unit to *n*-alkyl chain 'R' of –OR group up to C_{10} or nearby C_{10} homolog. However, alternation of transition temperatures diminishes or disappears for higher homologs of longer *n*-alkyl chain '*R*' from and beyond C_{10} homolog, because longer *n*-alkyl chain may coil or bend or flex or couple with major axis of core structure of molecules. Thus, unexpected status of *n*-alkyl chain eliminates odd-even effect.

The variations in mesomorphic properties and their magnitudes from homolog to homolog in the same novel series of present investigation is attributed to the sequentially and progressively added methylene unit or units at the *n*-alkyl chain '*R*' which alters the magnitudes of length to breadth ratio, permanent dipole moment across the long molecules axis, dispersive forces, molecular polarity, polarizability, rigidity, flexibility, etc. affecting intermolecular cohesion and closeness. The extrapolation of Sm-N and the N-I transition curves to C_4 and C_3 homologs for smectic and nematic, respectively, which suggests the possibility of missing the corresponding mesophase formation because extrapolated curves matches with Cr-N and Cr-I points of respective homolog. The changing trends in mesogenic behaviors of present novel series 1 are compared with structurally similar analogs series X [33] and Y [34] as mentioned in below Fig. 4.

Homologous series 1, X, and Y are identical with respect to two phenyl rings and one of the central bridge (-CH = CH-CO-) commonly present linking middle and last phenyl rings either homocyclic or heterocyclic which contributes partly to the total molecular rigidity, as well as left *n*-alkoxy terminal end group –OR for the same analog or homolog from series to series which partly contributes to total molecular flexibility. However, homologous series 1, X, and Y differ with respect to central bridge –CH = CH-COO- linking first and middle phenyl ring of series 1 and –COO- of series X and Y which contributes partly to the total molecular rigidity. Moreover last phenyl ring bonded through –CH = CH-CO- common central bridge with para substituted iodo or chloro group, i.e., ($-C_6H_4.I$ and $-C_6H_4.Cl$) in series 1 and X, as well as a heterocyclic ring ($-C_4H_3S$), which partly contribute to the total molecular rigidity and partly to the total molecular flexibility whose magnitudes differ from series to series for the same homolog and from homolog to homolog in the same series. Thus, variations in mesogenic properties and the degree of mesomorphism for the same homolog from series to series or from homolog to homolog in the same series will depend upon the differing magnitudes of differing features like molecular rigidity and/or flexibility as well as differing

Table 5. Thermal stability in°C.

Series→	$-C_6H_4I$ (series 1)	$-C_6H_4Cl$(series-X)	$-C_4H_3S$ (series-Y)
Sm-N or Sm-I Commencement of Smectic phase	155.0	142.6	115.66
	$(C_5–C_{18})$ C_5	$(C_4–C_{16})$ C_4	$(C_{12}–C_{16})$ C_8
N-I Commencement of Nematic phase	180.7	154.3	126.83
	$(C_4–C_{18})$ C_4	$(C_4–C_{14})$ C_3	$(C_7–C_{16})$ C_7
Total mesophase lengths from minimum to maximum from t1°C to t2°C	30°C–58°C	19.7°C–72.2°C	6.9°C–36.8°C
	C_4 C_8	C_4 C_{14}	C_7 C_{14}

molecular polarity and polarizability etc. Following Table 5 represents some thermal data for the series under comparative study.

All the three series under comparative study are

- Smectogenic plus nematogenic in character.
- Smectogenic thermal stabilities of series 1, X, and Y are in decreasing order.
- Nematogenic thermal stabilities of series 1, X, and Y are in decreasing order.
- Smectogenic and nematogenic mesophase commences the earliest from (C_4 and C_3) in series X and then a little bit later from (C_5 and C_4) in case of present series 1 and the latest from (C_8 and C_7) in case of series Y involving heterocyclic ring.
- Lower mesophaselength are in decreasing order from series 1 to X to Y; but it is in decreasing order from series X to 1 to Y.

The exhibition of smectogenic property from C_4, C_5, or C_8 homolog by the series 1, X, and Y, respectively, is attributed to the presence of lamellar packing of molecules in their preoccupied crystal lattices in solid crystalline state due to the difference occurred in intermolecular attractions by different central bridges linking first and middle phenyl rings viz. $-CH = CH-COO-$ and $-COO-$ in which $-CH = CH-COO-$ is longer and stronger than $-COO-$ containing $-CH = CH-$ vinyl group, involving conjugated double bond, which increases molecular rigidity more than $-COO-$ central bridge. Therefore, the extent of molecular noncoplanarity maintained favorably to facilitated smectic and nematic phase formation earlier than the series X and Y. Simultaneously homologous series X and Y contain identically $-COO-$ central bridge linking first and middle phenyl rings differs in magnitudes of flexibility by $-Cl$ and $-H$ of $-C_6H_4.Cl$ and $-C_4H_3S$, respectively, in which $-C_4H_3S$ does not contain any flexible group (except $-H$) like $-Cl$. Thus, late commencement of smectic and nematic phase as compared to series 1 and X is facilitated; by flexibility difference. Thus, suitable magnitudes of anisotropic forces of intermolecular end-to-end and lateral cohesion and closeness which maintain and facilitate either sliding layered molecular arrangement or/and then statistically parallel orientational order of molecules in floating condition to cause smectic and/or nematic mesophase formation for definite range of temperature, depending upon individual thermal resistivity generated from constitutionally energy stored in each molecule of same homolog from series to series and from homolog to homolog in the same series. Decreasing order of smectic and nematic thermal stabilities is attributed to the decreasing order of mainly molecular flexibility by tails ends and partly due to differences of vinyl carboxy and carboxy central bridges which contributes to molecular rigidity. $-I$ is more polarizable than $-Cl$ and $-H$ due to size difference, the effectiveness of size difference reflects in the order of the thermal stabilities and the lower and upper degree of mesomorphism of series 1 and Y. The exceptionally highest value of upper degree of mesomorphism (72.2°C) of series X is attributed to the high valued thermal resistivity towards exposed thermal vibrations induced by Vander waals forces of higher magnitude causing intermolecular attractions for its (series-X) C_{14} homolog.

Conclusions

- Novel chalconyl ester derivatives of thermotropic LC behavior is predominantly nematogenic and partly smectogenic whose smectogenic and nematogenic mesophaselengths vary from 17°C to 39°C and 15°C to 30°C, respectively, and it is of middle ordered melting type.
- Group efficiency order derived on the basis of (i) thermal stability (ii) early commencement of smectic phase and (iii) total mesophaselengths for smectic and nematic are as under.
  (i). Smectic: $-C_6H_4.I > -C_6H_4.Cl > -C_4H_3S$
  (ii). Nematic: $-C_6H_4.I > -C_6H_4.Cl > -C_4H_3S$
  (iii). Smectic: $-C_6H_4.Cl > -C_6H_4.I > -C_4H_3S$
  (iv). Nematic: $-C_6H_4.Cl > -C_6H_4.I > -C_4H_3S$
  (v) (Smectic ± Nematic)
  (vi) Lower: $-C_6H_4.I > -C_6H_4.Cl > -C_4H_3S$
  (vii) Upper: $-C_6H_4.Cl > -C_6H_4.I > -C_4H_3S$
- A phenomena of mesomorphism is very sensitive and susceptible to molecular structure as a consequence of changing magnitudes of molecular rigidity and/or flexibility.
- Present investigation is useful for the study of binary systems and can be useful for pharmaceutical preparations due to their bioactivity.
- Present study supports and raises the credibility to the conclusions drawn earlier.

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