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Dependence of mesomorphism on tail group in combination with a lateral methoxy group

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ABSTRACT

A novel homologous series of esters with a lateral methoxy group and terminal n-heptyl cinnamate group was synthesized and studied to investigate mesomorphic behavior in relation to structure. The twelve membered series consists of methoxy to pentyloxy derivatives that are nonmesomorphic and the rest of the homologs as smectegenically mesomorphic, including C_6 and C_7 monotropic smectic and C_8 to C_{16} enantiotropic smectic without exhibition of any nematic property. The textures of smectogenic mesophases are focal conic fan shaped or batonates of the type smectic-A or Schlieren-C. An odd-even effect is exhibited by the Sm–I transition curve in the phase diagram. The Cr–Sm and Sm-I transition curves behave in a normal manner. Transition temperatures and textures were determined on an optical polarizing microscope, equipped with a heating stage. Some representative members were characterized by IR, H^1NMR, mass spectra, and elemental analysis. Analytical data support the molecular structures of the homologs. Mesomorphic properties of the present novel series are compared with the structurally similar known series. The present series is smectogenic only, whose thermal average stability is 89.8°C and of a middle-ordered melting type.

KEYWORDS

Liquid crystal; mesogenic ester; mesomorphism; nematic; smectic

Introduction

The liquid crystal state has flow properties like a liquid and optical properties like crystals, hence it is termed a liquid crystal (LC) state or an anisotropic liquid state or a mesomorphic state [1]. The dual character of flow and anisotropy brings a usefulness in various fields of applications [2–5]. The present study is planned with a view to synthesize novel LC substances through homologous series by varying molecular structures by changing number of phenyl rings or central groups linking phenyl rings or terminal end group or groups of different polarity or by changing position (o, m, p) or positions of same polar group in isomeric series, etc. to understand and establish the relations between LC property and the molecular structures [6–8]. Thus, novel substances can be useful to physics, biology, pharmacy, agriculture, engineering, and technology including chemistry, because LC materials either of the thermotropic type or Lyotropic type are useful to mankind [9–12]. A literature survey on thermotropic LCs

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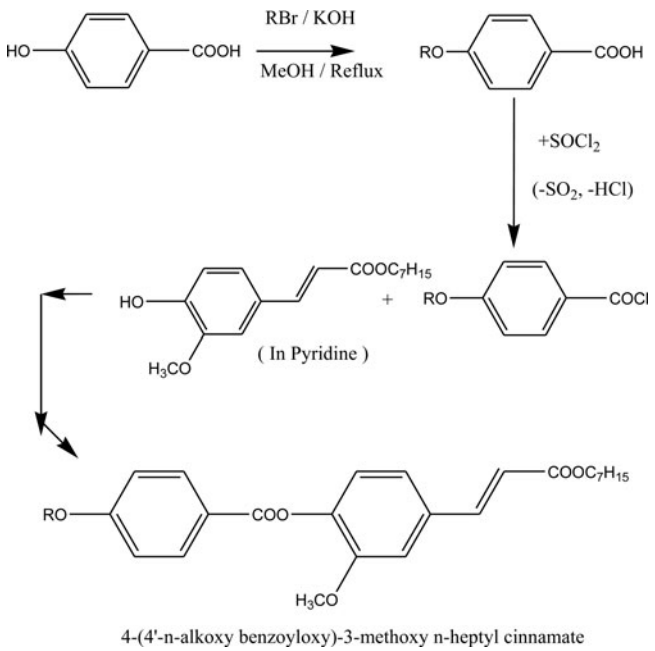
[13–16] prompted the proposed investigation of the synthesis of ester homologs with a lateral $-OCH_3$ group at the ortho position to central –COO– group and varying right handed terminal end groups of $-CH{=}CH\text{-}COOC_7H_{15}$ (n) in combination with left $-OC_nH_{2n+1}$ end group. Then the novel data are interpreted on the basis of molecular rigidity and flexibility [17–20] of the molecular structure after characterization of novel substances.

Experimental

Synthesis

4-Hydroxy benzoic acid was alkylated by suitable alkylating agents (R-X) to convert it into dimeric 4-n-alkoxy benzoic acids by the modified method of Dave and Vora [21]. 4-Hydroxy 3-methoxy cinnamic acid (m.p. 64°C yield 67%) was esterified by n-heptanol by using conc. H_2SO_4 by a usual established method [22]. The ester was a very viscous and dense brown liquid, which was purified from distilled n-hexane. The yield was 62.5%. Dimeric 4-n-alkoxy benzoic acids were individually condensed with 4-hydroxy, 3-methoxy n-heptyl cinnamate in dry cold pyridine through corresponding acid chlorides [23]. Final products were individually decomposed, filtered, washed, dried, and purified until constant transition temperatures were obtained.

The synthetic route to the series is shown below in Scheme 1. The chemicals required, 4-hydroxy benzoic acid, MeOH, KOH, alkyl halides (R-X), thionyl chloride, 4-hydroxy, 3-methoxy, cinnamic acid (Ferulic acid), n-heptanol, Conc. H_2SO_4, pyridine, 1:1 HCl, $NaHCO_3$, NaOH, anhydrous $CaCl_2$, silica gel, petroleum ether, methyl acetate, and n-hexane were used as received, except solvents which were dried and purified prior to use.



Scheme 1. Synthetic route to the series

Table 1. Elemental analysis for ethyloxy, butyloxy, octyloxy and dodecyloxy derivatives.

Compound no.	Molecular formula	Mol.% of C Experimental (Theoretical)	Mol.% of H Experimental (Theoretical)
C_2	$C_{26}H_{32}O_6$	70.42 (70.89)	6.76 (7.32)
C_4	$C_{28}H_{36}O_6$	71.22 (71.77)	7.36 (7.74)
C_8	$C_{32}H_{44}O_6$	72.70 (73.25)	8.11 (8.45)
C_{12}	$C_{36}H_{52}O_6$	73.75 (74.45)	8.55 (9.02)

Characterization

Representative homologs of the series were selected for characterization of structure by elemental analysis (Table 1), IR spectra, mass spectra and H^1 NMR spectroscopy. Microanalysis was performed on Perkin Elmer PE 2400 CHN analyzer. IR spectra were recorded on Perkin Elmer spectra GX. ^{1}H NMR spectroscopy performed were recorded on Bruker instrument using $CDCl_3$ as solvent.

Mesomorphic properties were investigated using hot stage polarizing microscope.

Analytical data

Elemental analysis for ethyloxy, butyloxy, octyloxy, and dodecyloxy derivatives:

IR spectra in cm^{-1} for methyloxy and ethyloxy homologue derivatives

Methyloxy: The IR spectrum of the compound showed,
841.66 cm^{-1} and 758.39 cm^{-1} confirms p-sub. benzene ring,
688.72 cm^{-1} confirms polymethylene of –C7H15 group,
2956.00 cm^{-1} and 2917.90 cm^{-1} & 2854.00 cm^{-1} confirms alkyl group,
1249.92 cm^{-1}, 1604.08 cm^{-1} and 1709.64 cm^{-1}, confirms –COO– & –CO– group,
1511.33 & 1459.05 cm^{-1} confirms aromatic –C=C–,
1164.49 cm^{-1} as .CO- of alkoxy group,
1026.20 cm^{-1} and 980.78 cm^{-1} confirms trans –CH=CH– group,
IR data supports the structure.

IR Spectra in cm^{-1} for ethyloxy homolog derivatives

Ethyloxy:- The IR spectrum of the compound showed,
845.19 and 769.25 cm^{-1}confirms p-sub. benzene ring,
647.18 cm^{-1} and 695.28 cm^{-1} confirms polymethylene of –C_7H_{15} group,
2931.19 and 2860.06 cm^{-1} confirms alkyl group,
1248.21, 1605.48 cm^{-1} and 1711.3 cm^{-1} confirms –COO– & –CO of ester group,
1510.78 cm^{-1} confirms aromatic –C=C–,
1165.83 cm^{-1} as –CO– of alkoxy group,
1042.36 and 1099.61 cm^{-1} confirms trans –CH=CH– group.
IR data supports the molecular structure.

^{1}H NMR spectra in ppm for heptyloxy and decyloxy derivatives heptyloxy

H^1 NMR Data in δ ppm (n-hexyloxy derivative, 400 MHz, CDCl$_3$, Standard TMS)

0.808 (−CH_3 of −C_7H_{15}), 1.2376 (−CH_2 of —−C_7H_{15}), 1.7629 (−OCH_2−CH_2 of −C_7H_{15}), 3.7696 (−O−CH_3), 3.947 (−O−CH_2− of −C_7H_{15}), 7.0883 and 7.0723 (−O−C_6H_3−CH=CH−CO−), 8.059 and 8.0812 (p-sub. Benzene ring).

The data support the molecular structure.

Decyloxy

H^1 NMR data in δ ppm (n-Decyloxy Derivative, 400 MHz CDCl$_3$, Standard TMS)

0.8128 (−CH_3 of −$C_{10}H_{21}$), 1.2229 (−CH_2 of −$C_{10}H_{21}$), 1.7308 and 1.7481 (−OCH_2−CH_2 of −$C_{10}H_{21}$), 3.7547 (−O−CH_3), 3.9458 (−O−CH_2− of −$C_{10}H_{21}$), 7.0764 and 7.0616 (−O−C_6H_3−CH=CH−CO−), 8.0493 and 8.0714 (p-sub. Benzene ring).

The data supports the molecular structure.

Texture determination by miscibility method

- Octyloxy homolog ——→ Smectic – A
- Decyloxy homolog ——→ Smectic – A
- Tetradecyloxy homolog ——→ Smectic –C

Mass spectra

Homolog:	Theoretical	Experimental
C_2	440.53	440.0
C_4	568.58	568.25

Results and discussion

Homologous series 4-(4'-n-alkoxy benzoyloxy)-3-methoxy-n-heptyl cinnamates was synthesized from dimeric 4-n-akoxy benzoic acid through their corresponding acid chlorides and a nonmesomophic component 3-methoxy–4-hydroxy-n-heptyl cinnamate (very viscous and dense brown liquid, yield 62.5%). The C_1 to C_5 homologs are nonmesomorphic, but mesomorphism commences from the C_6 homolog in a monotropic manner as a smectic type and continues until the last C_{16} member of the series as an enantiotropic smectic phase; however, the C_7 homolog behaves in monotropic (Sm) manner. Nematic mesophase exhibition is missing for the homologs of entire series of the present investigation. Transition temperatures (Table 2) of the homologs are plotted versus the number of carbon atoms present in n-alkyl (-C_nH_{2n+1}) chain. Transition curves (Cr-I or Sm and Sm-I or I-Sm) are formed showing phase behaviors of a series in a phase diagram (Fig. 1). Cr-I or Sm transition curve adopt a zigzag path of rising and falling values with overall descending tendency as series is ascended. Sm-I (or I-Sm) transition curve initially rises for odd members and falls for even members up to the decyloxy (C_{10}) homolog. Both transition curves for odd and even members are merge into each other between C_9 and C_{10} homologs and then from and beyond, (Sm-I) transition curve rises to maxima at dodecyloxy (C_{12}) homolog and descended as series is ascended in usual

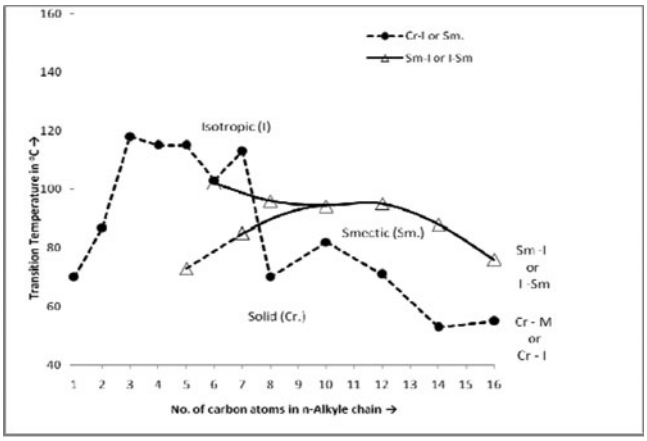


Figure 1. Phase behaviors of series.

established manner. Thus, odd-even effect is exhibited by Sm-I transition curve and both transition curves i.e. Cr-Sm or Cr-I and Sm-I transition curves behaved in normal manner. The Sm-I transition curve for odd members is extrapolated to pentyloxy ($C_{5)}$ homolog to predict and determine its latent transition temperature (L.T.T.). Sm-I transition curve follows as single curve from and beyond decyloxy (C_{10}) homolog, i.e., odd-even effect disappears from and beyond C_{10} homolog for higher homologs of longer n-alkyl chain. The average thermal stability for smectic is 89.8°C and the mesomorphic (Sm) phase length is varied between 11°C and 35°C. The mesomorphic properties from homolog to homolog are changing with molecular length. Thus, presently investigated ester series with lateral substitution by $-OCH_3$ group is smectogenic without exhibition of smectic property.

The ester homologs from C_1 to C_5 do not exhibit any type of mesomorphism, because the magnitudes of anisotropic forces of intermolecular end to end and/or lateral attractions occurred as a consequence of unsuitable molecular rigidity and flexibility which induces inability to resist exposed thermal vibrations to cause mesomorphism, and hence have a high crystallizing tendency. The molecules of homolog derivatives C_6 to C_{16} generate a lamellar packing of molecules with layered structure. Thus, smectogenic mesophase formation is observed within a definite temperature range. The same sample on cooling from isotropic state by proper rate of cooling, the smectogenic mesophase formation reappears from and below

Table 2. Transition temperatures in °C.

Compound No.	n-Alkyl group R=-C_nH_{2n+1}	Sm	N	Isotropic
	C_1	–	–	70.0
2	C_2	–	–	87.0
3	C_3	–	–	118.0
4	C_4	–	–	115.0
5	C_5	–	–	115.0
6	C_6	(102.0)	–	103.0
7	C_7	(85.0)	–	113.0
8	C_8	70.0	–	97.0
9	C_{10}	82.0	–	93.0
10	C_{12}	71.0	–	95.0
11	C_{14}	53.0	–	88.0
12	C_{16}	55.0	–	76.0

() indicates monotropy.

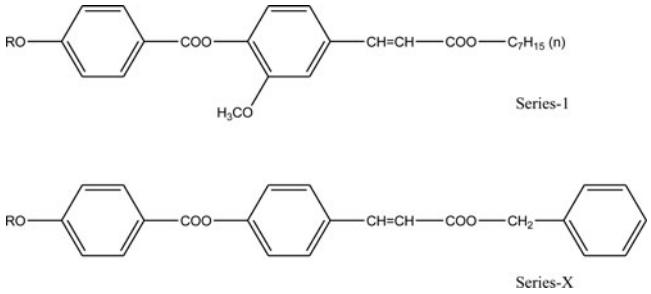


Figure 2. Structurally similar series.

isotropic temperature till solid crystalline state. Thus, enantiotropic smectic mesophase formation without exhibition of nematic phase observed for C_6 to C_{16} homologs of the present novel series. The exhibition of an odd-even effect and alternation of transition temperatures of ester derivatives are due to the changing number of methylene unit at the n-alkyl chain of left n-alkoxy (-OR) terminal end group. Disappearance of odd-even effect from and beyond C_{10} homologs for higher homologs of longer n-alkyl chain is attributed to the coiling, bending, flexing, or coupling of n-alky chain with major axis of the core structure of molecules. The extrapolation [24–27] of the Sm-I transition curve for C_5 nonmesomorphic homolog to determine its probable L.T.T. which is at 74° C. Mesomorphic properties vary from homolog to homolog in present series is attributed to the changing number of methylene units which changes molecular length, polarity and polarizability, length to breadth ratio, etc. which causes variations in molecular rigidity and flexibility for emerging suitable or unsuitable magnitudes of anisotropic forces of intermolecular attractions to induce mesomorphism. The mesomorphic properties of present series-1 are compared with the structurally similar homologous series-X [28] as shown below in Fig. 2.

The homologous series of the present investigation (series-1) and a known homologous series-X chosen for comparison are identical with respect to two phenyl rings, central bridge –COO–, left n-alkoxy ($-OC_nH_{2n+1}$) terminal end group for the same homolog from series to series and the part of terminal end group of series-1 and a central group of series-X, i.e., –CH=CH–COO–. The differences between the structures of the two series (1 and X) are clearly seen in Fig. 2. Table 3 represents some mesomorphic properties like average thermal stability, commencement of mesophase, number of homologs and their type, mesophase length range, odd even effect, etc. under comparison.

Table 3 indicates that

- homologous series-1 of present investigation is only smectogenic, whereas a series-X chosen for comparison is smectogenic in addition to nematoganic.
- Smectic mesophase commences from C_6 homolog in series-1 and X, i.e., both series are equi-smectogenic.

Table 3. Average thermal stability in °C.

Series:→	1	X
Smectic-Isotropic or Smectic-nematic	89.8 (C_8-C_{16})	144.1 (C_6-C_{16})
Commencement of Smectic phase	C_6	C_6
Nematic- Isotropic	–	17.4 (C_5-C_{16})
Commencement of nematic phase	–	C_5
Total mesophase length range in °C (Sm + N)	11.0 to 35.0 (C_{10}) (C_{14})	33.0 to 59.0 (C_6) (C_{12})

- Nematic mesophase commences from C_5 member of a series-X, but it does not commence till the last C_{16} homolog member of a present series-1.
- The mesophase length range difference between minimum and maximum in °C are (35 – 11 = 24) twenty four for series-1 and (59 – 33 = 25) twenty five for a series-X, i.e., series-1 and X are equally resistive and efficient against exposed thermal vibrations to cause mesophase stabilization, but the mesophase stabilization temperatures of series-X are higher for the same homolog from series to series.
- Thermal stability in average of present series-1 for mesophase (Sm and N) is relatively lower than a series-X under comparison.

Homologous series-1 is laterally substituted by highly polar methoxy group, which increases molecular width and reduces intermolecular attractions and at the same time, intermolecular attractions are enhanced by increased molecular polarizability through increase in molecular width. Thus, two opposing effects of intermolecular attractions or molecular cohesion occur for the same reason. Therefore, net effect of intermolecular attractions depends upon the dominance of the two opposing effects operated at a time for the same reason. Moreover, series-1, consisted of two phenyl rings whereas series-X under comparison consists of three phenyl rings. Therefore, aromaticity, and the molecular rigidity of series-X is more than a series-1. The left n-alkoxy terminal end group of series-1 and X are identical for the same homolog from series to series, but the right terminal end group though, consisted of seven carbon for both the series, they differ in length, type of hybridization and aromaticity, i.e., right-handed terminal end group bonded to -COO- group in series-1 is an aliphatic open hydrocarbon chain in which sp^3 hybridized carbons are involved, whereas in case of series-X, the six carbons are aromatic hydrocarbons bearing cyclic ring in which sp^2 hybridized carbons are bonded with -COO- group through $-CH_2-$ methylene unit. Thus, molecules of series-1 bears more flexible open n-alkyl chain of unexpected status of flexibility, than, a series-X, in which six sp^2 carbons occupy their restricted position in an aromatic phenyl ring and increases molecular rigidity. Thus, the same homolog molecules from series to series are under competition of the extent of molecular rigidity and flexibility; and the combine effect of which in suitable magnitudes operates induction of mesophase formation, the degree of mesomorphism and the type or types of mesomorphism. The lowering of smectic thermal stability of present novel series-1 is attributed to the broadening of molecular width effect, which predominated between two opposing effects, resulted from laterally substituted $-OCH_3$ group. Thus, transition temperatures (Cr-Sm and Sm-I) and consequently, thermal stabilities are lowered for series-1 as compared to series-X with (equal) commencement of smectic phase from the same C_6 homolog. However, nematic mesophase in present novel series-1 is observed to be missed, while it appeared in case of series-X above its smectic transition temperature (Sm-N), i.e., aromatic ring system being relatively more stable and rigid, bears stronger intermolecular end to end attractions, facilitated, statistically parallel orientational molecular order from and beyond Sm-N, transition temperature to induce nematic mesophase formation from C_5 in series-X as observed against flexibility effect caused by n-C_7H_{15} alkyl chain which hinders nematic mesophase formation till the last C_{16} homolog of a series-1 of present investigation. Thus, mesomorphic properties of a substance depend upon the molecular rigidity and flexibility as emerged from the molecular structure of a substance.

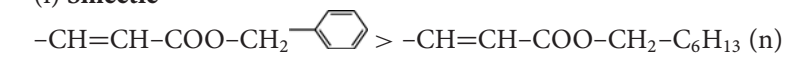
Conclusions

- Homologous series of present investigation with two phenyl rings, $-OCH_3$ lateral group and $-CH{=}CH-COO-C_7H_{15}$ (n) end group is smectogenic without exhibition

of nematic property whose, smectic property commences from C_6 homolog and intermolecular cohesive forces are more conducive to molecular width instead of molecular polarizability.

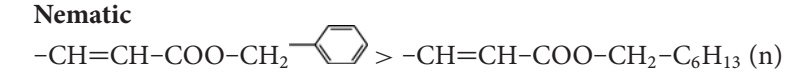
- Group efficiency order derived for smectic and nematic on the basis of (i) thermal stability, (ii) commencement of mesophase, and (iii) total mesophaselength range are as under.

(i) **Smectic**



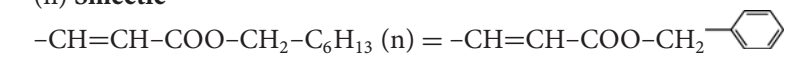
with $-OCH_3$ lateral group

Nematic



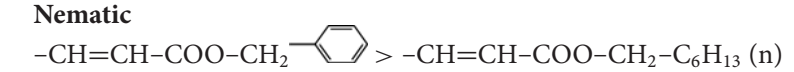
with $-OCH_3$ lateral group

(ii) **Smectic**

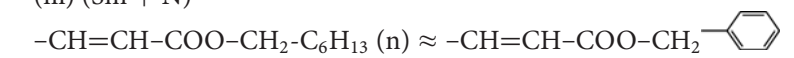


with $-OCH_3$ lateral group

Nematic



(iii) (Sm + N)



- Molecular rigidity and flexibility depending on molecular structure are operating the mesomorphic behaviors of a substance.
- The mesomorphic properties from homolog to homolog in the same series depends upon the sequentially added methylene unit at the left n-alkoxy terminal end group.
- The mesomorphic properties for the same homolog from series to series depends upon the changing part (lateral or terminal or o, m, p position) of the series under comparison.
- Mesomorphism and degree of mesomorphism are sensitive and susceptible to molecular structure.
- Present investigation may be useful for the study of mixed mesomorphism to operate LC devices at desired temperature. Biological activity of the novel compounds may be useful in pharmaceutical, agricultural and medicinal preparations.

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