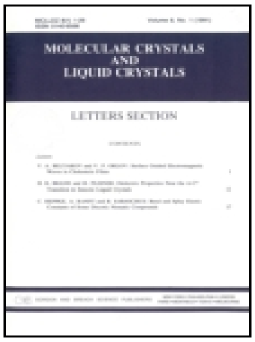

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Synthesis of novel ester series and study of its mesomorphism dependence on terminal end group with lateral –OCH_3 group

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ABSTRACT

Novel liquid crystal (LC) materials of ester derivatives were synthesized and studied with a view to understanding and establishing the effects of molecular structure on LC properties. The novel molecules consist of two phenyl rings bonded through –COO– central group and a laterally substituted methoxy group with $-OC_nH_{2n+1}$ as well as $-COOCH_3$ terminal end groups, and yielded 12 homologous members of an ester series. The C_1 to C_3 members are nonmesomorphic, the C_4 to C_{12} members are enantiotropic nematic only, and the C_{14} to C_{16} members are enantiotropically smectogenic in addition to nematogenic. Transition temperatures and the textures of LC state were observed through an optical polarizing microscope (POM) equipped with a heating stage. The textures of nematic phase are threaded or Schlieren, and that of smectic phase are focal conic of the type A or C. Transition curves of a phase diagram behave in normal manner with the exhibition of an odd-even effect (only N-I). Analytical and spectral data support the molecular structures of the novel ester derivatives. The LC properties of the present series are compared with structurally similar other known series. The average thermal stability of the series is 93°C for smectic and 120.88°C for nematic and the mesogenic phase length ranges between 2°C and 46°C.

KEYWORDS
Enantiotropy; liquid crystal; mesomorphism; nematic; smectic

Introduction

Liquid crystalline (LC) state of a substance is well known for its importance in various fields of applications due to its dual character as to flow as liquid and to possess optical properties of crystals, and was first recognized as a special state of matter in 1888 [1]. A critical survey of literature indicated that many researchers have synthesized novel LC substances through homologous series and binary systems [2–6]. We, being chemists, decided to synthesize novel LC substances and to correlate effects of molecular structure on liquid crystal properties of a substance by varying molecular aromaticity, central groups, terminal groups, positions of same or different functional groups on phenyl ring or rings, geometrical shape, size, polarity and polarizability, etc. Such novel substances may be useful to the researchers who are working with different aims in varying disciplines for the study of biological and pharmaceutical activity as well as in the manufacture of electronic display (LCD) devices like television screen

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and related articles [7–13]. The present investigation is planned to synthesize and characterize novel LC ester substances with two phenyl rings, and aimed to understand and establish the correlation between molecular structure and LC behavior or LC properties of novel substances [14,15] as a consequence of molecular rigidity and flexibility [16–20].

Experimental

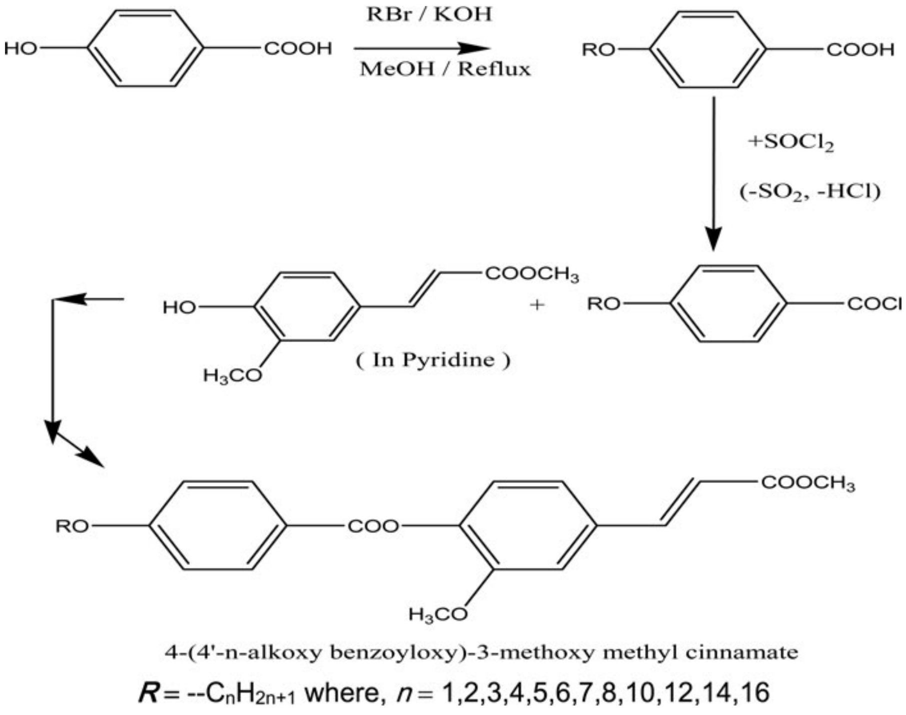
Synthesis

4-Hydroxybenzoic acid was alkylated by suitable alkylating agent (R-X) to convert it into dimeric 4-*n*-alkoxy benzoic acid by the modified method of Dave and Vora [21].

4-Hydroxy 3-methoxy cinnamic acid (m.p. 64°C yield 67%) was esterified by methanol by using conc. H_2SO_4 by a usual established method [22]. The solid ester was crystallized from distilled *n*-hexane. The melting point is 62–63°C. The yield was 75.6%. Dimeric 4-*n*-alkoxy benzoic acids were individually condensed with 4-hydroxy, 3-methoxy methyl cinnamate in dry cold pyridine through corresponding acid chlorides [23, 24]. Final products were individually decomposed, filtered, washed, dried, and purified until constant transition temperatures were obtained.

The synthetic route to the series is shown in Scheme 1.

The chemicals required were 4-hydroxy benzoic acid, MeOH, KOH, alkyl halide (R-X), thionyl chloride, 4-hydroxy,3-methoxy, cinnamic acid (ferulic acid), conc. H_2SO_4, pyridine, 1:1 HCl, $NaHCO_3$, NaOH, anhydrous $CaCl_2$, silicagel, petrolium ether, methyl acetate, and *n*-hexane, and were used as received except solvents that were dried and purified prior to use.



Scheme 1. Synthetic route to the series. $R = -C_nH_{2n+1}$ where, $n = 1,2,3,4,5,6,7,8,10,12,14,16$.

Characterization

Representative homologues of the series were characterized by elemental analysis, IR spectra, and ^{1}H NMR spectra. Microanalysis was performed on Perkin-Elmer PE CHN analyzer. IR spectra were recorded on Perkin-Elmer spectrum GX and ^{1}H NMR spectra were recorded on Bruker using $CDCl_3$ solvent. Mesomorphic properties were investigated using hot stage polarizing microscope. Thermodynamic quantity enthalpy (ΔH) and entropy $(\boldsymbol{\Delta S = \Delta}H/T)$ were discussed qualitatively instead of DSC scan.

Analytical data

Elemental Analysis for Hexyloxy, Octyloxy, Decyloxy, and Dodecyloxy Derivative

IR Spectra in cm^{-1} for Propyloxy and Pentyloxy Homologoue Derivatives

Propyloxy. *The IR spectrum of the compound showed 677.33 cm^{-1}polymethylene of –C_6H_{13} group; 873.34 cm^{-1} and 828.46 cm^{-1} as p-sub. benzene ring; 1152.50 cm^{-1} as –CO– of alkoxy group; 1240 cm^{-1}, 1508 cm^{-1}, 1600 cm^{-1}, and 1701.78 cm^{-1} confirms –COO– of ester group; 971.58 cm^{-1} confirms trans –CH=CH– group; and 3065.2 cm^{-1} confirms alkyl group* IR supports the structure.

Pentyloxy. *The IR spectrum of the compound showed 689.26 cm^{-1}polymethylene of –C_6H_{13} group; 842.91 cm^{-1} as p-sub. benzene ring; 1156.90 cm^{-1} as –CO– of alkoxy group; 1250.22 cm^{-1}, 1604.22 cm^{-1}, 1703.28 cm^{-1}, and 1736.76 cm^{-1} conforms –COO– of ester group; 977.27 cm^{-1} confirms trans –CH=CH– group; and 3192.95 cm^{-1} confirms alkyl group* IR supports the structure.

^{1}H NMR Spectra in ppm for Hexyloxy and Octyloxy Derivatives

Hexyloxy. *H^1 NMR data in δ ppm (n-octyloxy derivative, 400 MHz, $CDCl_3$, standard TMS): -0.828 (-CH_3 of -C_6H_{13}), 1.288 and 1.18 (-CH_2 of - C_6H_{13}), 1.7365 (-OCH_2-CH_2 of -C_6H_{13}), 3.7755 and 3.8569 (-O-CH_3), 3.9518 (-O-CH_2- of -C_6H_{13}), 6.91–7.19 (-O-C_6H_4-CH=CH-CO–), 7.96–8.08 (p-sub. benzene ring)*

The data supports the molecular structure.

Octyloxy. *H^1 NMR data in δ ppm (n-octyloxy derivative, 400 MHz, $CDCl_3$, standard TMS): -0.817 (-CH_3 of -C_8H_{17}), 1.255 (-CH_2 of -C_8H_{17}), 1.753 (-OCH_2-CH_2 of -C_8H_{17}), 3.749 and 3.798(-O-CH_3), 3.965 (-O-CH_2- of -C_8H_{17}), 6.36 and 7.189 (-O-C_6H_4-CH=CH-CO–), 6.88 and 8.05 (p-sub. benzene ring)*

The data supports the molecular structure.

Textures by Miscibility Method.

- C_4 ——→ Threaded nematic
- C_7 ——→ Schlieren nematic
- C_{16} ——→ Smectic–C

Result and discussion

4-Hydroxy-3 methoxy methyl cinnamate is a nonliquid crystal substance. However, on linking it with dimerized 4-*n*-alkoxy benzoic acids yielded C_4 to C_{16} derivatives as mesomorphic and C_1 to C_3 nonmesomorphic. C_4 to C_{12} homologues are only enantiotropic nematic

Table 1. Elemental analysis for hexyloxy, octyloxy, decyloxy and dodecyloxy derivatives.

Compound no.	Molecular Molecular formula	Mol.% of C experimental (theoretical)	Mol.% of H experimental (theoretical)
C_6	$C_{24}H_{28}O_6$	69.90 (69.63)	6.80 (7.08)
C_8	$C_{26}H_{32}O_6$	70.91 (70.48)	7.27 (7.08)
C_{10}	$C_{28}H_{36}O_6$	71.79 (71.37)	7.69 (7.26)
C_{12}	$C_{30}H_{40}O_6$	72.58 (73.00)	8.07 (8.31)

Table 2. Transition temperatures in °C.

Compound no.	*n*-Alkyl group $R = \text{-}C_nH_{2n+1}$	Sm	N	Isotropic
1	C_1	—	—	145.0
2	C_2	—	—	154.0
3	C_3	—	—	119.0
4	C_4	—	96.0	123.0
5	C_5	—	115.0	117.0
6	C_6	—	101.0	145.0
7	C_7	—	86.0	92.0
8	C_8	—	97.0	137.0
9	C_{10}	—	86.0	118.0
10	C_{12}	—	91.0	113.0
11	C_{14}	70.0	88.0	116.0
12	C_{16}	76.0	92.0	121.0

Note: Sm = Smectic; N = Nematic.

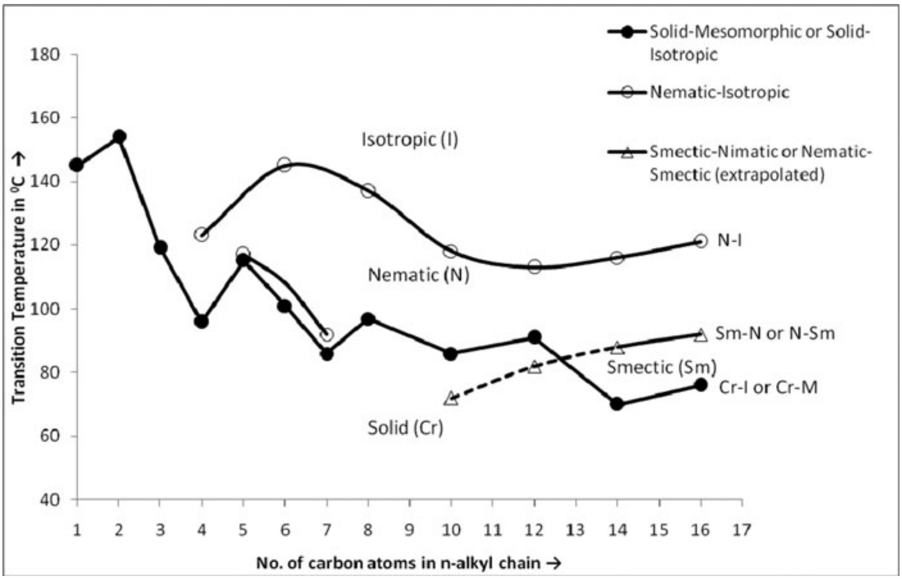


Figure 1. Phase behavior of the series.

and remaining two mesomorphic derivatives are enantiotropically smectogenic in addition to nematogenic. Dimerization of aromatic acids disappears on esterification of process. Transition temperatures of homologues (Table 2) are plotted versus the number of carbon atoms present in *n*-alkyl chain of left *n*-alkoxy terminal end group. Transition curves showing phase behaviors are obtained by linking like or related transition points. Thus, a phase diagram is constructed (Fig. 1) to depict the phase behavior of the novel series. Cr-I or Cr-M transition curve adopt a zigzag path of rising and falling as series is ascended with overall falling tendency. Sm-N transition curve shows rising tendency in usual manner. However Sm-N transition curve is extrapolated [25–27] toward left side of a curve to predict and determine the latent ability of nonsmectogenic homologues C_{10} and C_{12} keeping in view of the trend of Sm-N transition curve. The predicted latent transition temperatures (LTT) for C_{10} and C_{12} are hypothetically 72.5°C and 82.0°C to exhibit smectogenic character. However, these values are not realizable, due to their high crystallizing tendency. N-I transition curve initially rises and then descends as series is ascended with exhibition of odd-even effect. N-I transition curve for odd members occupies relatively at lower position than the position of a N-I transition curve for even members of series. Thus, all the transition curves of a phase diagram behaved in normal manner with negligible abnormality (N-I) at C_{14} and C_{16} homologues. N-I transition curve for odd members below even membered curve do not merge into each other but the effect discontinued from and beyond heptyloxy (C_7) derivative and then, single transition curve for higher homologues prolonged. Variations in mesomorphic behavior and LC properties from homologue to homologue in present series appear by sequential addition of methylene unit or units. Thermal stability as related to enthalpy change (ΔH) in average for a series is 93.0°C for smectic and 120.88°C for nematic mesophase. The mesogenic phase length ranges from 2.0°C to 46.0°C.

Thus, a novel ester series under discussion is predominantly nematogenic and partly smectogenic with middle-ordered melting type.

The dimerization of *n*-alkoxy aromatic acids disappears by breaking of hydrogen bonding between two acid molecules by esterification process. The molecular length, polarizability, aromaticity, etc. undergo variations in suitable or unsuitable magnitudes of anisotropic forces of intermolecular cohesion on linking acid chlorides of 4-*n*-alkoxy benzoic acid with 4-hydroxy-3-methoxy methyl cinnamate. The homologues C_1 to C_3 molecules are nonliquid crystals, because their unsuitability in magnitudes of anisotropic forces of intermolecular attractions as a consequence of molecular rigidity and flexibility arising due to their permanent dipole moment across the long molecular axis, dipole–dipole interaction, dispersion forces, etc., which are insufficient and unsuitable to resist, exposed thermal vibrations. Thus, their crystal lattices are abruptly breaking and sharply transform directly into isotropic liquid without passing through an intermediate state (LC state) of physical existence. At this stage molecules of C_1 to C_3 homologues are randomly oriented in all possible directions without any ordered arrangement to acquire LC state.

Exhibition of mesogenic behaviors of C_4 to C_{16} members of a series is attributed to the disalignment of molecules at an angle 90° or less than 90° with the floating surface, depending upon the suitable magnitudes of inter-molecular cohesive forces and closeness and its resistivity against the intensity of exposed thermal vibrations for some degree of temperature difference to cause either only nematic or only smectic or smectic and nematic mesophase formation. The molecules of C_4 to C_{12} homologue derivatives are floated on the surface under the influence of exposed thermal vibrations with only statistically parallel orientational order of molecules, which exhibited only nematogenic mesophase formation prior to isotropic temperature in reversible manner. However, the homologue derivatives C_{14} and C_{16} are floated on the surface with higher-ordered sliding layered arrangement of molecules for some temperature difference prior to less-ordered statistically parallel orientational order of molecules for another some degree of temperature difference in reversible manner till isotropic temperature. Thus, C_{14} and C_{16} derivatives exhibited smectogenic mesophase formation prior to nematogenic mesophase formation with reversible manner, i.e., the molecules of C_{14} and C_{16} as well as C_4 to C_{12} are randomly oriented in all possible directions from and beyond isotropic

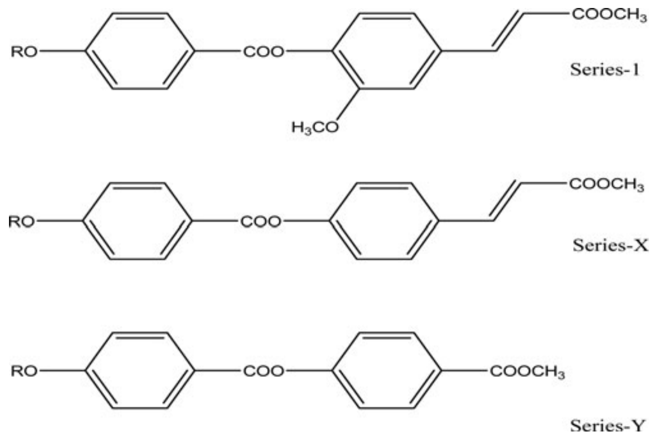


Figure 2. Structurally similar series.

temperature, but on cooling the same from and below isotropic temperature exhibit respective mesophase (smectic and/or nematic) formation at their respective transition temperatures. The magnitudes of intermolecular attractions as a consequence of molecular rigidity and flexibility (in present series) depended upon intermolecular distance, i.e., intermolecular attractions decreases by increasing intermolecular width by laterally substituted $-OCH_3$ group. But at the same time increasing molecular width by $-OCH_3$ increases intermolecular attractions due to increase into molecular polarizability. Thus, two opposing forces of intermolecular attraction causes two opposite effect operated at a same time for the same reason. The extrapolation of Sm-N transition curve for C_{10} and C_{12} homologues, whose missing of smectogenic character is attributed to their high crystallizing tendency, hinders lamellar packing of molecules in their crystal lattices. The suitable magnitudes of intermolecular cohesive forces occurred in C_{14} and C_{16} homologue molecules by their permanent dipole moment across the long molecular axes, dipole–dipole interactions, dispersion forces, etc. as a consequence of favorable molecular rigidity and flexibility, which causes the formation of lamellar packing of molecules in their crystal lattices to build up three-dimensional network in their crystalline state and occupy sliding layered arrangement of molecular network of two-dimensional molecular order, under floating condition to facilitate smectic mesophase formation under the influence of thermal vibrations for some degree temperature difference. The exhibition of odd–even effect in N-I transition curve is attributed to the sequentially and progressively added methylene unit to the *n*-alkoxy terminal end group that varies LC property from homologue to homologue in present novel series, keeping rest of the molecular part unchanged for all the homologue molecules of a series. Thus, variations in mesomorphic properties of a substance depend upon the molecular structure of respective molecules.

Some mesomorphic properties of presently investigated series-1 are compared with the structurally similar other known series-X [28] and Y [29] as shown in Fig. 2.

Homologous series-1 of present investigation and series-X and Y chosen for comparison are similar with respect to two phenyl rings, –COO– central bridge and varying flexible $-OC_nH_{2n+1}$ end group for the same homologue from series to series. However they differ with respect to terminal end groups $-CH{=}CH-COOCH_3$, including and excluding laterally substituted $-OCH_3$ group respectively and $-COOCH_3$. Homologous series-1 and X are identical with respect to two phenyl rings, central bridge –COO–, and both terminal end groups, but they differ with respect to the presence of laterally substituted $-OCH_3$ group that replaces –H from second phenyl ring in series-1 and it is absent in series-X. Homologous series-1 and

Table 3. Average thermal stability in °C.

Series:→	1	X	Y
Smectic-isotropic or Smectic-nematic	93.0 (C_{14}-C_{16})	—	111.75 (C_6-C_{16})
Commencement of Smectic phase	C_{14}	—	C_5
Nematic-isotropic	120.88(C_4-C_{16})	105.0 (C_2-C_{16})	—
Commencement of nematic phase	C_4	C_2	—
Mesophase length range in °C(Sm +N)	2.0–46.0	11.0–40.0	21.0°C *to 58.0°C*

Y are identical in all respect except terminal end group at the other end of second phenyl ring, i.e., –CH=CH-COOCH$_3$ is replaced by –COOCH$_3$ with absence of lateral substituted –OCH$_3$ group in series-Y.

Therefore, the liquid crystal properties or behavior and the degree of liquid crystallinity will depend upon the differing molecular part as a consequence of differing magnitudes of molecular rigidity, flexibility, and the magnitudes of thermal resistivity toward externally exposed thermal vibrations.

Following Table 3 represents some LC properties of presently investigated homologous series-1 and the series-X and Y selected for comparative study.

Table 3 indicates that:

- Homologous series-1 of present investigation is smectogenic in addition to nematogenic in character, whereas series-X and Y chosen for comparison are only nematogenic and only smectogenic, respectively.
- Smectic and nematic mesophases commences from C_{14} and C_4 homologue in series-1 whereas the same commences (nematic only) from C_2 homologue in series-X and from C_5 homologue as smectic only in case of series-Y.
- Average thermal stability of series-X and Y are 105.0 (N) and 111.75 (Sm), whereas that of series-1 are 93.0 and 120.88 for smectic and nematic, respectively.
- Smectic thermal stability of present series-1 is lower than a smectogenic series-Y, while nematic thermal stability of present series-1 is higher than a nematogenic series-X.
- Total mesophase length of present series-1 is the highest and that of series-X is the lowest among the series under comparative study.

Homologous series-X and Y under comparison with series-1 do not contain any functional group at lateral position on any phenyl ring. Homologous series-1 bears mesogenic ortho-substituted highly polar functional group –OCH$_3$, which is substituted on lateral position to central group –COO– on second phenyl ring to which –CH=CH–COOCH$_3$ terminal end group is substituted. Laterally substituted –OCH$_3$ functional group increases intermolecular width as compared to series-X and Y. Therefore, two opposing effects are operated at a time as: (i) increase in the intermolecular distance reduces suitable magnitudes of anisotropic forces of intermolecular attractions, which induce or affect mesomorphism; and (ii) presence of lateral –OCH$_3$ group increases molecular polarizability, which can enhance intermolecular anisotropic forces of attractions to induce and affect mesomorphism. Thus, two opposing forces or effects are operated at a time for the same reason. Therefore, the resultant net effect of intermolecular end-to-end and lateral attractions depends upon the predominancy of the effects (i) or (ii) as mentioned above. Second effect (ii) due to polarizability is relatively less operative in case of series-X and Y as compared to series-1 as a consequence of effective molecular rigidity and flexibility inducing LC property in the homologue substances.

The smectic thermal stability of present series-1 is lower (93.0) than a smectogenic series-Y (111.75), because homologous series-Y contain terminal and central group –COO–, i.e., smectogenic –COO– functional groups, whereas a homologous series-1 of present investigation (and series-X) contain terminal nematogenic –CH=CH–COO– group that involve conjugated double bond affecting molecular rigidity, flexibility, and end-to-end attractions. Therefore, such effect hinders more the lamellar packing of molecules in series-1 (and X) and favors the lamellar packing of molecules in crystal lattices of series-Y. The suitable magnitudes of anisotropic forces of intermolecular end-to-end attractions are disturbed for a series-Y and favored for series-1 and X to induce nematogenic character with accordingly more or less degree of mesomorphism and early commencement of mesophase formation. Absence of lateral substitution $–OCH_3$ group and presence of vinyl carboxy group $–CH{=}CH–COOCH_3$ hinders lamellar packing of molecules in the crystal lattices of homologue substances belonging to series-X and leads to float molecules on floating surface with only statistically parallel orientational two-dimensional order, till isotropic temperature of mesogenic homologues giving rise to exhibit only nematic mesophase formation, neglecting exhibition of smectic character. Homologous series-1, X, and Y contain –COO– central group as common identical part, but difference in lateral substitution and end groups viz. –CH=CH–COO– (series-1 and X) and –COO– (series-Y) causes difference in the magnitudes of molecular flexibility and rigidity for the same homologue from series to series. Thus, in all other respect except the end groups, which is $–CH{=}CH–COO–CH_3$ in case of homologous series-1 and X and carboxylate $–COOCH_3$ in case of series-Y under comparative study, these three series are closely similar to each other. Both these end groups are comfortable, though the vinyl carboxylate –CH=CH–COO– has greater length and 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 methyl group and a phenyl ring. On account of these differences, the smectic thermal stability of series-1 is though lower than a series-Y but nematic thermal stability is higher than the series-X and Y. The difference of the extent of molecular noncoplanarity induces smectic character in C_{14} and C_{16} homologues of series-1, whereas it induces smectic character earlier from C_5 homologue to C_{16} homologue of series-Y at the cost of nematic mesophase formation. Similarly, nematic mesophase commences earlier from C_2 homologue (to C_{16}) in series-X at the cost of smectic mesophase formation as compared to series-1, in which nematic mesophase commences late from C_4 homologue (to C_{16}). Thus, mesomorphic properties and the degree of mesomorphism for the same homologue from series to series and homologue to homologue in the same series depend upon molecular structure as a consequence of molecular rigidity and flexibility as emerged from permanent dipole moment across the long molecular axis, dipole–dipole interactions, dispersion forces, electron–electron interaction, etc.

Conclusions

- Novel homologous ester series consisting of two phenyl rings of 12 homologues is predominantly nematogenic (C_4 to C_{16}) and partly (C_{14} to C_{16}) smectogenic with three homologues (C_1 to C_3) as nonliquid crystals.
- Transition temperatures of mesomorphic homologues range between 70°C and 145°C.
- The group efficiency order derived for smectic and nematic mesophase formation on the basis of (i) thermal stability, (ii) early commencement of mesophase, and (iii) mesophase length are as under.

(i) *Smectic:*
$-COOCH_3 > -CH{=}CH-COOCH_3 > -CH{=}CH-COOCH_3$
With lateral $-OCH_3$ group without lateral $-OCH_3$ group
Nematic:
$-CH{=}CH-COOCH_3 > -CH{=}CH-COOCH_3 > -COOCH_3$
With lateral $-OCH_3$ group without lateral $-OCH_3$ group
Smectic:
$-COOCH_3 > -CH{=}CH-COOCH_3$ with and without $-OCH_3$ group
(ii) *Nematic:*
$-CH{=}CH-COOCH_3 > -CH{=}CH-COOCH_3 > -COOCH_3$
Without $-OCH_3$ group with $-OCH_3$ group
(iii) *Mesophase (Sm ± N):*
$-CH{=}CH-COOCH_3 > -COOCH_3 > -CH{=}CH-COOCH_3$
With $-OCH_3$ group without $-OCH_3$ group

- Phenomena of mesomorphism is very sensitive and susceptible to molecular structure of a substance.
- Present investigation supports the views and conclusions drawn earlier.

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