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Synthesis and Study of Mesomorphic Properties of Liquid Crystalline Compounds Involving Lateral -OCH₃ Group and Vinyl Carboxylate Central Linkage

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ABSTRACT

In the present investigation, a newly synthesized ester homologous series of mesogenic compounds having a vinyl carboxylate group as central linkage with two phenyl rings and a laterally substituted methoxy group as well as -CH=CH-COOC₄H₉ terminal end group were synthesized. Eleven homologues were synthesized. Among these eleven homologues first seven derivatives C₁ to C₇ of the investigated series are nonmesomorphic by nature. Rest of the homologs are smectic by nature, among these C₈, C₁₀ and C₁₂ homologues show monotropically smectic phase, while the last member of the series C₁₄ show smectic phase enantiotropically. Thus, presently investigated series is predominately smectogenic by nature without exhibiting nematic mesophase. Transition temperatures and textures of synthesized compounds were observed through an optical polarizing microscope equipped with a heating stage. The textures of smectogenic mesophase are focal conic fan-shaped. Some representative members were characterized by IR, ¹HNMR, mass spectroscopy and elemental analysis. Transition temperatures of homologues are plotted versus the number of carbon atoms present in n-alkyl chain of left n-alkoxy terminal end group. Isotropic-smectic / smectic-isotropic transition curve behaves in a normal ascending manner. Thermal stability and other mesogenic characteristics are compared with other structurally similar homologous series. The mesogenic phase length ranges between 5 °C and 24 °C and is of middle-order melting type.

Keywords: Liquid crystals, Mesomorphism, Smectogenic, Thermotropic, DSC

1. INTRODUCTION

A state of matter that combines the characteristics of solids and liquids are referred to as a liquid crystal (LC) [1]. It is made up of molecules those are arranged in an order similar to that of a solid, but can flow similarly to a liquid. Normally, they are characterized by long, rod-like molecules, rigidity of the long axis, and strong dipole substituents that align in a specific way to give the material its unique optical features [2, 3]. These characteristics make liquid crystals useful in a wide range of applications, including displays for electronic devices, optical storage, light-emitting diodes, and as detectors [4-10]. The study of liquid crystals began in 1888 when Austrian botanist Friedrich Reinitzer noticed that a chemical known as cholesteryl benzoate changed color depending on the temperature, which was eventually determined to be due to the alignment of the molecules, which were twisted about an axis perpendicular to the molecular layers.

After that numerous homologous series of mesogens were synthesized and reported by using different central linkages, lateral functional groups, and terminal end group [11-16]. Minor changes in molecular structure reveals their different mesomorphic properties. J. J. Travadi et al. have studied the effect of laterally substituted methoxy (-OCH₃) group and -COO- central bridge on mesomorphism and concluded that favorable lamellar packing afforded by the polarity of the lateral -OCH₃ group [17]. Chaudhari et al. synthesized a novel liquid crystal with a -CH=CH-CO- central bridge [18]. R. B. Patel et al. synthesized and reported n-butyl-4-(4'-n-alkoxy- cinnamoyloxy) cinnamates, which contain central linkages -CH=CH-COO- and Terminal substituted functional groups are n-alkoxy and -CH=CH-COOC₄H₉ (n), and possess a nematogenic mesophase with a high melting point order without exhibiting smectogenic character [19].

In the present investigation, we attempt to study the effect of the mesomorphic properties of different homologues involving the latterly substituted polar -OCH₃ group, vinyl carboxylate as the central linkage -CH=CH-COO-, and two terminal end groups named n-alkoxy at one side and normal butyl ester -CH=CH-COOC₄H₉ (n) at the other end. The synthesized series contained Eleven homologues. All homologues differ from each other by an additional methylene group at the n-alkoxy terminal end group. Out of eleven homologous four homologous possess smectogenic mesophase behavior. The synthesized homologues were confirmed using different analytical techniques, and their physical properties were measured by polarizing optical microscopes (POM).

2. MATERIALS AND METHODS

4-hydroxy 3-methoxy cinnamic acid, n-alkyl bromides (R-Br), KOH, 4-hydroxy cinnamic acid, n-butanol, Methanol, HCl, Ethanol, Acetic acid, n-hexane, con. H₂SO₄, Sodium bicarbonate, MgSO₄, dichloromethane (CH₂Cl₂), DMAP (4-Dimethylaminopyridine), Dicyclohexyl Carbodiimide (DCC), and other reagents etc. required for the synthesis were purchased from Spectrochem, Merck, and Sigma-Aldrich. All reagents and solvents were used as received.

Selected homologues of the synthesized series were characterized by elemental analysis (Table 1). Infrared spectroscopy, ¹H NMR spectra, mass spectroscopy, and transition temperatures were confirmed by The Differential Scanning Calorimeter (DSC).

3. EXPERIMENTAL

3. 1. Synthesis of 4-n-alkoxy 3-methoxy Cinnamic acid [A]

As shown in the Reaction Scheme-1. 4-n-alkoxy 3-methoxy cinnamic acid [A] prepared by Dave and Vora's modification method [20], a mixture of 4-hydroxy 3-methoxy cinnamic acid (4.85 g, 0.025 mol), alkyl bromide (0.03 mol), potassium hydroxide (3.2 g, 0.062 mol), and ethanol (50 ml) were refluxed in RBF for 4-5 hours. After cooling, the mixture was poured into a stirred mixture of concentrated hydrochloric acid (25 ml) and ice-cold water (25 ml). The product was then filtered. The compounds were washed with hot water, dried, and recrystallized using ethanol to obtain a constant melting point. The purity of the compounds was checked by TLC and the Melting point. [21]

3. 2. Synthesis of 4-hydroxy n-butyl cinnamate [B]

An Equimolar proportion of 4-hydroxy cinnamic acid and absolute n-butyl alcohol was mixed in a 250 ml single neck round bottom flask in the presence of two drops of H₂SO₄, and the reaction mixture was refluxed for 4-5 hours. The reaction mixture was then decomposed in ice-cold water and allowed to settle. A Crude product, 4-hydroxy n-butyl cinnamate, was obtained. The product was filtered, washed, dried, and purified by crystallization to yield 63.74% and M.P. 126 °C [22, 23].

3. 3. Synthesis of n-butyl-4-(4'-n-alkoxy-m'-methoxy cinnamoyloxy) cinnamates [A+B]

Homologues of the titled homologous series were prepared using the Steglich esterification method to yield n-butyl-4-(4'-n-alkoxy-m'-methoxy cinnamoyloxy) cinnamates. The synthesized compounds (A) and (B) were condensed using N,N'-dicyclohexyl carbodiimide (DCC), 4-dimethyl amino pyridine (DMAP), and dichloromethane (DCM) as the solvent, and the reaction was performed at room temperature. The product was then filtered, washed with water and NaHCO₃, dried, and recrystallized in glacial acetic acid until a constant melting point was obtained. The purity of the compounds was checked by TLC and characterized by elemental analysis and spectral data [24-27] (Scheme 1).

3. 4. Analytical data

3. 4. 1. ¹H NMR

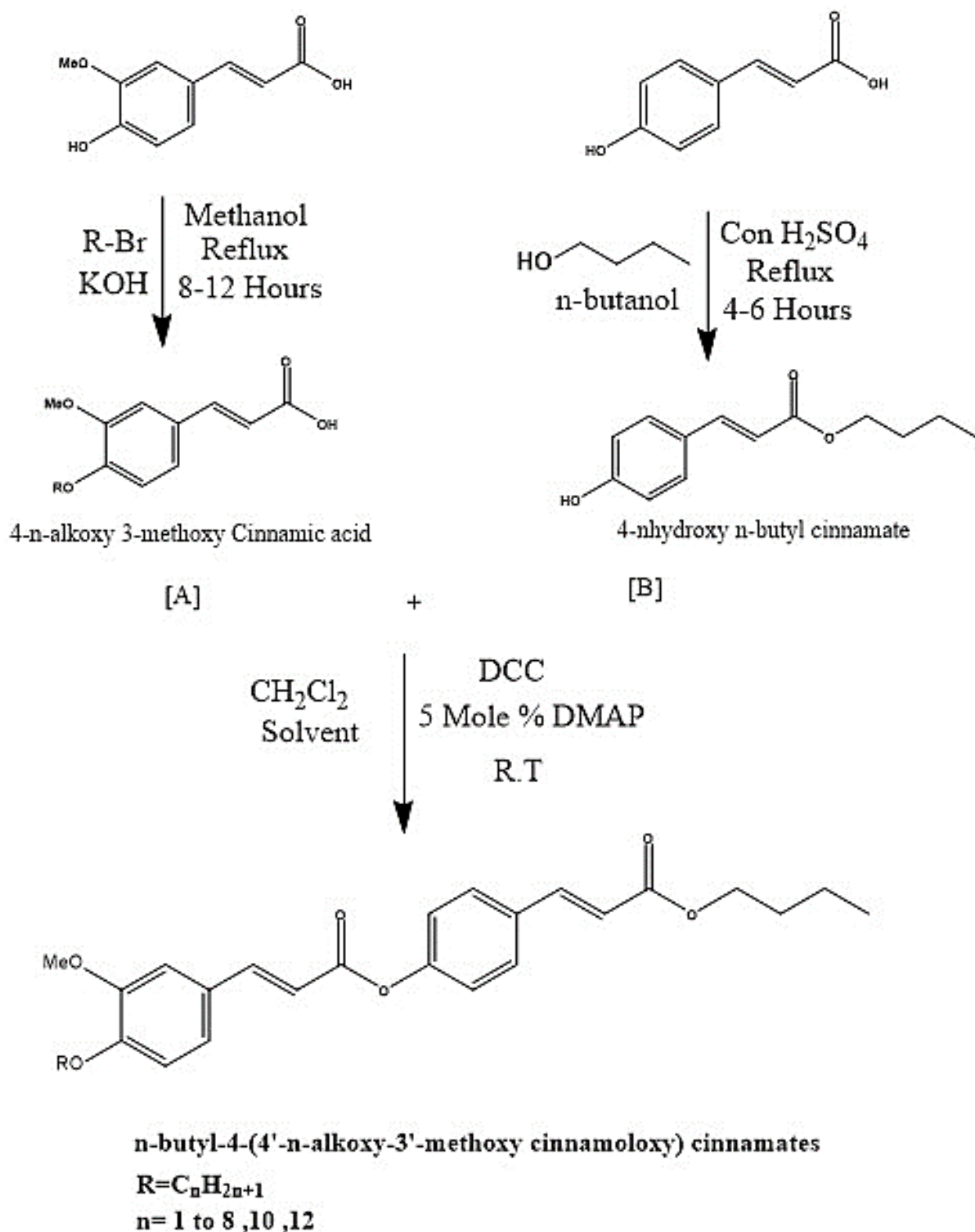
¹H NMR in δppm of Decyloxy derivative (C₁₀)

¹H NMR in δppm MHz, (400 MHz, DMSO-*d*₆) δ 0.853-0.886 (t, 3H, -CH₃ of -C₁₀H₂₂), 0.917-0.954 (t, 3H, -CH₃ of -C₄H₁₀), 1.267(m, -CH₂-CH₂-), 1.427 (m, 2H, -CH₂-), 1.697-1.7764 (m, 2H, -CH₂-), 3.838 (s, 3H, m-substituted -OCH₃), 4.347-4.311 (m, 2H, -CH₂ of COOC₄H₁₀), 4.002-4.034 (t, 2H, -OCH₂-), 7.260-7.281 (dd, 2H, Ar-H), 7.351-7.336 (dd, 2H, Ar-H), 7.036, 7.260 & 7.461 (d & s, 3H, Ar -CH=CH-), 6.787 & 7.709 (d, 2H, -CH=CH-);

¹H NMR in δppm of Tetradecyloxy derivative (C₁₄)

¹H NMR in δppm MHz, (400 MHz, DMSO-*d*₆) δ 0.837-0.871 (t, 3H, -CH₃ of -C₁₄H₃₀), 0.956-0.939 (t, 3H, -CH₃ of -C₄H₁₀), 1.242 (m, -CH₂-CH₂-), 1.403 (m, 2H, -CH₂-), 1.929-1.996 (m, 2H, -CH₂-), 3.828 (s, 3H, m-substituted -OCH₃), 3.948-3.965 (m, 2H, *J* = -CH₂ of

COOC₄H₁₀), 3.993-4.025 (t, 2H, -OCH₂-), 7.253-7.274 (dd, 2H, Ar-H), 7.308-7.328(dd, 2H, Ar-H), 7.036, 7.253 & 7.451 (d & s, 3H, Ar -CH=CH-), 6.780 & 7.712 (d, 2H, -CH=CH-)



Scheme 1. Synthesis of n-butyl-4-(4'-n-alkoxy-3'-methoxy cinnamoyloxy) cinnamates.

3. 4. 2. FTIR Spectrum

FTIR Spectrum in Cm^{-1} of Decyloxy derivative (C_{10})

FTIR (ATR, ν_{max} , cm^{-1}): 682.41 (poly methylene of $-\text{C}_{10}\text{H}_{22}$ and $-\text{CH}_3$), 841.20 (p-sub. Benzene ring), 2949.62 & 2930.33 (CH_2 Str.), 1717.16, 1627.82 & 1277.42 ($-\text{COO}$ & $-\text{CO}$), 1511.20 & 1422.63 (aromatic $-\text{HC}=\text{CH}-$), 1161.77 ($-\text{CO}$ of alkoxy), 923.03 ($-\text{CH}=\text{CH}-$), 3003.95 (CH of aromatic Str), 802 (p-substitution aromatic ring)

FTIR Spectrum in Cm^{-1} of Tetradecyloxy derivative (C_{14})

FTIR (ATR, ν_{max} , cm^{-1}): 682.41 (poly methylene of $-\text{C}_{14}\text{H}_{30}$ and $-\text{CH}_3$), 841.20 (p-sub. Benzene ring), 2949.62 & 2930.33 (CH_2 Str.), 1717.16, 1627.82 & 1277.42 ($-\text{COO}$ & $-\text{CO}$), 1511.20 & 1422.63 (aromatic $-\text{HC}=\text{CH}-$), 1161.77 ($-\text{CO}$ of alkoxy), 923.03 ($-\text{CH}=\text{CH}-$), 3003.95 (CH of aromatic Str), 802 (p-substitution aromatic ring)

3. 4. 3. Mass Spectra

Mass Spectra of Decyloxy derivative (C_{10})

m/z (int of %) 536 (M^+), 317 (100% base peak ester linkage break)

Mass Spectra of Tetradecyloxy derivative (C_{14})

m/z (int of %) 592 (M^+), 373 (100% base peak ester linkage break)

Table 1. Elemental analysis of (1) Hexyloxy (2) Octyloxy (3) Tetradecyloxy derivatives.

Sr. No.	R=n-alkyl chain	Molecular formula	Element % found			Element % calculated		
			C	H	O	C	H	O
1	C_6	$\text{C}_{29}\text{H}_{36}\text{O}_6$	72.48	7.55	19.97	72.60	7.50	19.90
2	C_8	$\text{C}_{31}\text{H}_{40}\text{O}_6$	73.20	7.93	18.87	73.50	7.90	18.60
3	C_{14}	$\text{C}_{37}\text{H}_{52}\text{O}_6$	74.97	8.84	16.19	74.91	8.88	16.21

4. RESULT AND DISCUSSIONS

Here, the synthesized homologous series “n-butyl-4-(4'-n-alkoxy-3'-methoxy cinnamloxy) cinnamates” is discussed in a brief, comparative study, and some important conclusions were drawn.

Eleven homologues were synthesized by coupling (E)-4-n-alkoxy 3-methoxy Cinnamic acid with the non-mesogenic compound 4-hydroxy n-butyl cinnamate. And the reaction scheme of the synthesized series was shown in Scheme-1 and a detailed experimental procedure was described in the experimental procedure. The homologues were confirmed by different analytical data, such as ^1H NMR, IR, and Mass spectrometry.

The synthesized homologs of C₁ to C₇ are not mesomorphic 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 the inability to resist exposed thermal vibrations to cause mesomorphism, and hence possess a high crystallizing tendency, but from the homolog of C₈ onward, mesomorphism commences in a monotropic manner as smectic type and continues until the C₁₂ homolog of series, while C₁₄ homolog of series possess enantiotropic smectic phase. And generate a lamellar packing of molecular with a layered structure. Thus, only smectogenic mesophase formation is observed within a definite temperature range. Nematic type of mesophase is not exhibited by any of the series showing mesogenic character. Mesomorphic properties vary from homolog to homolog in synthesised present series because of the change of number in methylene units present at left side of n-alkoxy chain. which changes molecular length, polarizability and polarity, length-to-breadth ratio etc. which causes variation in molecular rigidity and flexibility for emerging suitable or unsuitable magnitudes of anisotropic forces of intermolecular attraction to induce mesomorphism [25]. 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 in Figure 2. Careful observation of the phase diagram indicate that the solid-isotropic or solid-smectic transition curve show a falling tendency as series is ascended up to hexyl derivative of the series without following zig-zag path of rising and falling up to heptyl derivative of series from hexyl and then transition curve rises from Heptyl to octyl and from then on the curve is falls to the last homologous of the series. The Crystallin-Smectic or Crystallin-Isotropic and Smectic-isotropic transition curves behaved in a normal manner. The average Thermal stability of smectic is 89.25 °C, and the mesomorphic phase length ranges from 5 °C to 24 °C in tetradecyl and octyl homologue respectively. Transition and melting temperatures are relatively low. Thus, the series is of low melting type with a low range of smectogenic character and without exhibiting nematogenic character.

4. 1. Mesomorphic Behavior.

The mesomorphic properties of synthesized compounds were recorded in a polarizing optical microscope (POM) equipped with a Mettler FP82HT heating plate. The sample was prepared as follows, ~1 mg compound was sandwiched in between a glass plate and coverslip with the help of heating up to its mesomorphic/isotropic temperature; The prepared slip with a thin section of the compound was observed under POM using heating and cooling rate for the transition temperature is 10 °C min⁻¹. According to measurements of phase transition temperature (°C) are tabulated in Table 2, and the mesophase texture observed by POM is shown in Figure 1.

Table 2. Transition temperature in °C on heating rate

Compound No.	R = n-alkyl group: C _n H _{2n+1}	Commencement temperature of Smectic phase during heating	Isotropic
1	C ₁	-	128 °

2	C ₂	-	125 °
3	C ₃	-	120 °
4	C ₄	-	118 °
5	C ₅	-	114 °
6	C ₆	-	80 °
7	C ₇	-	84 °
8	C ₈	(72) °	96 °
9	C ₁₀	(68) °	91 °
10	C ₁₂	(71) °	88 °
11	C ₁₄	77 °	79 °

Values in parenthesis indicate monotropic transition

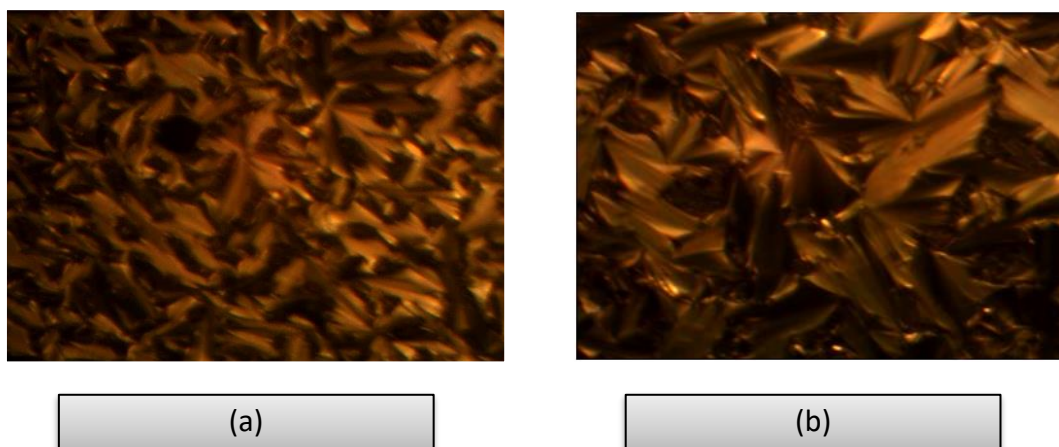


Figure 1. Mesophase texture observed by POM (a) Smectic A shape phase of compound C₈ at 72 °C; (b) Smectic A phase of compound C₁₄ at 77 °C during the cooling cycle.

4. 2. DSC Analysis

DSC is used for the measurement of changes in physical properties by the influence of temperature and detection of the change in the physical properties of the compound at a particular temperature. The Mesogens (C₈ and C₁₄ of series-1) Were screened using the DSC Method. To confirm the phase transition temperature of the synthesized ester-based homologues, analysis was performed on both heating and cooling cycles at a rate of 10 °C /min. The two specific thermograms are presented in Figure 3 and 4; their phase lengths are listed in

Table 3, and Figures 3 and 4 show the DSC spectra of the octyloxy and dodecyloxy derivatives, respectively.

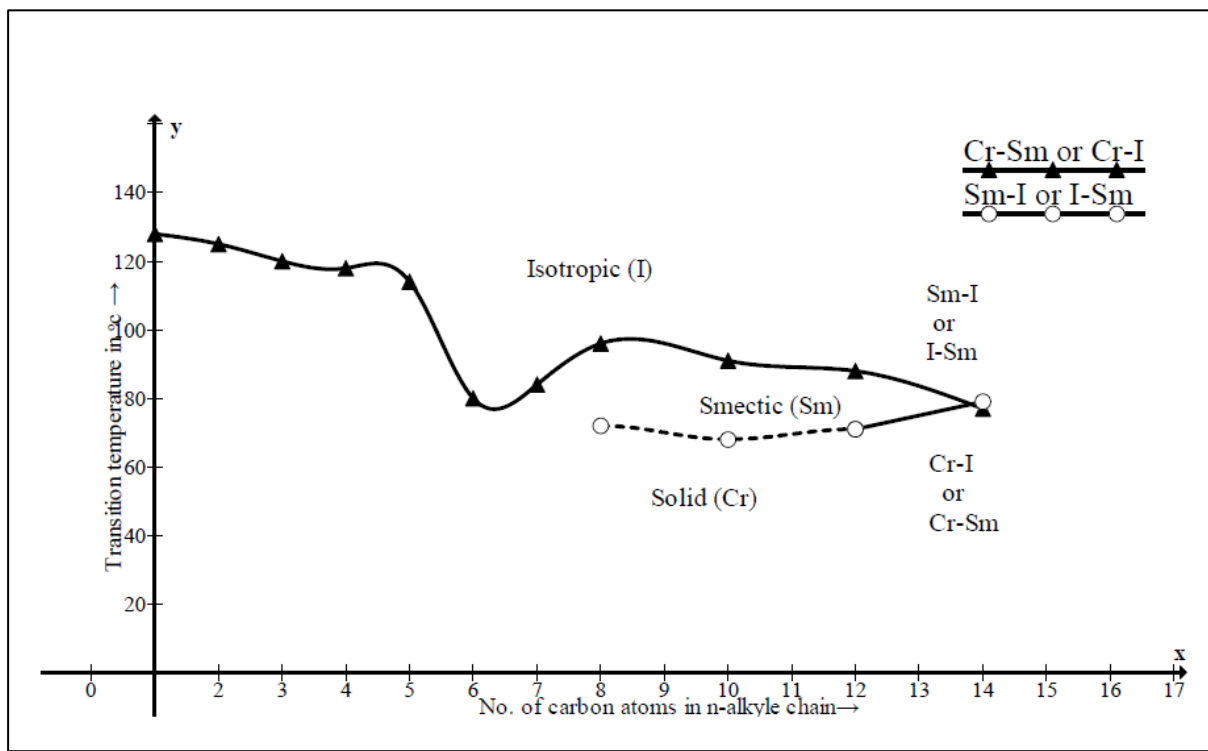


Figure 2. Phase behavior of homologues series.

From Figures 3 and 4, we observed that the presence of two endothermic peaks in higher alkyl chain substituted compounds indicates the presence of solid-smectic and smectic-isotropic phases. The tetradecyloxy derivative containing compound C_{14} had two endothermic peaks, followed by a mesomorphic phase change at 77 °C, and displayed smectic mesophase behavior up to 80 °C.

At the temperature at which the smectic phase develops, the compounds melt, and their liquid crystal mesophase behavior persists through the isotropic point. Additionally, at temperatures above 82 °C, an isotropic phase change was noticeable. And the system's enthalpy was found to be 78.7 J/g and 1.1 J/g, which is similar to a transition state in the endothermic phase. During the cooling or exothermic cycles, these two-phase transition curves were also observed in reverse order.

The material displayed isotropic behavior throughout the cooling process at 82 °C, whereas the smectic transition commenced below this temperature range at 75 °C. This large difference indicates that the chemical has a considerable smectic phase preference during the exothermic cycle.

During this cycle, the isotropic behavior collapses. This indicates that the LC mesophase and a transition with an enthalpy change, starting at -20.17 J/g, occurs. The molecule reverted to its crystalline form at temperatures below 50 °C.

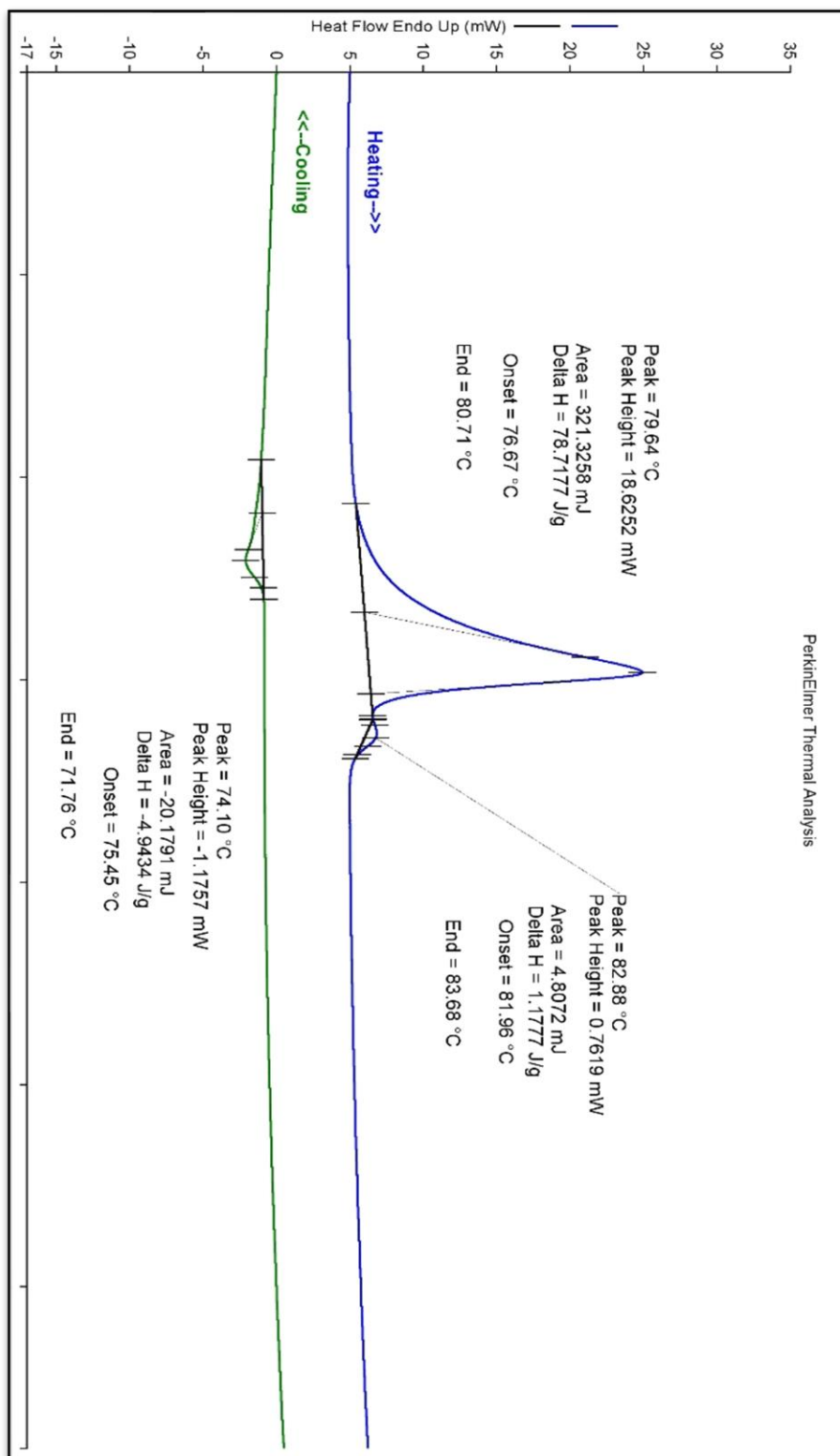


Figure 3. Representative DSC thermograms of the C₁₄ derivative during the heating and cooling cycles (10 °C /min).

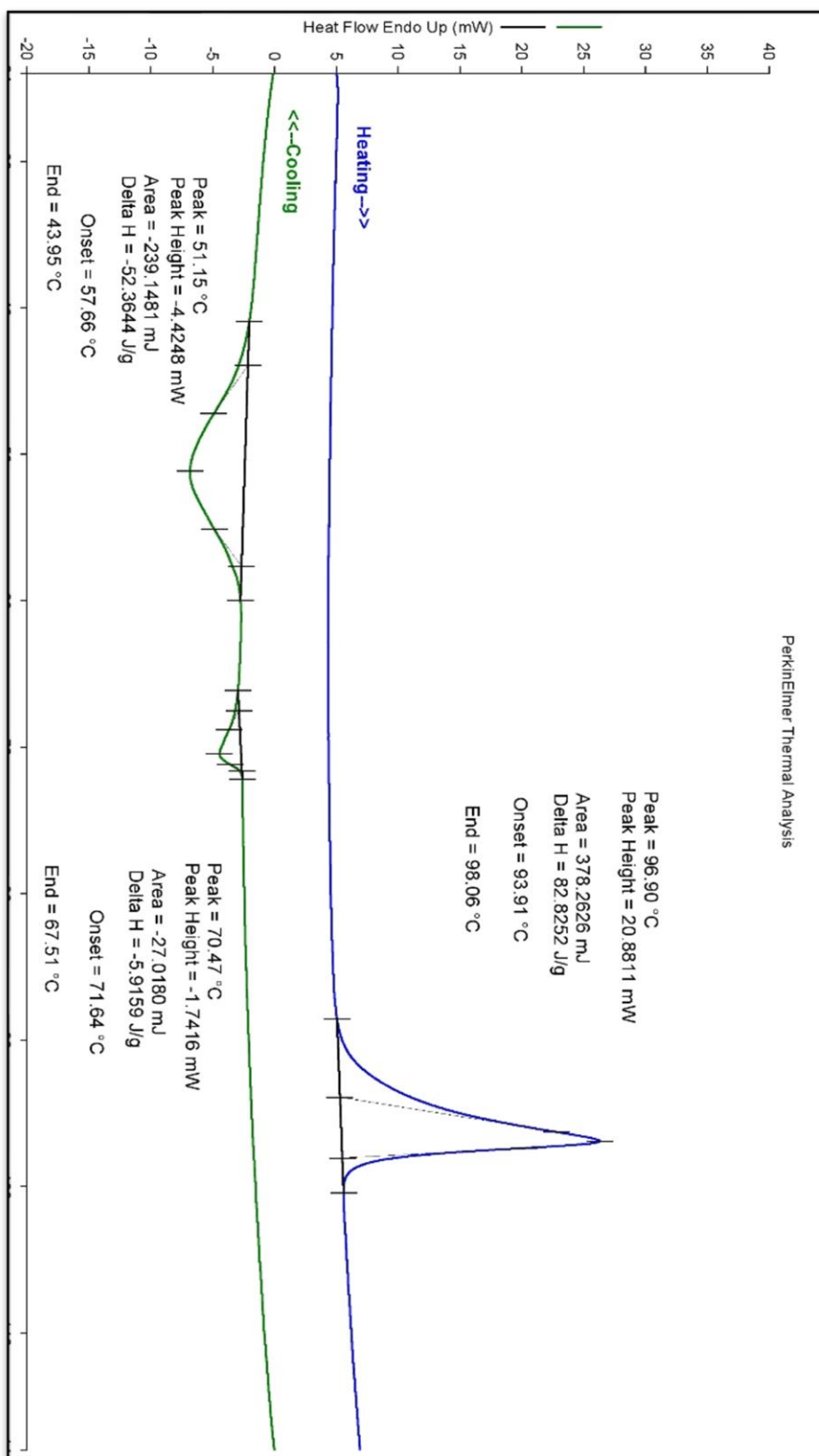


Figure 4. Representative DSC thermograms of the C₈ derivative during the heating and cooling cycles (10 °C /min).

The octyloxy containing compound C₈ had only one endothermic peak observed at 96 °C for the isotropic phase. The enthalpy of the system was found to be 84 J/g and did not provide any phase length during the heating cycle. Under cooling conditions, the compound displayed mesophase behavior at 71.6 °C and returned to the crystalline state at 51.1 °C. The enthalpy change in this cycle is from -5.91 to -52.36 J/g. These transition temperatures and POM transition temperatures are satisfactorily correlated. DSC analysis supported the monotropic and enantiotropic mesophase behavior of the synthesized ester-based homologues.

Table 3. Transition enthalpy and entropy changes of C₈ and C₁₄ homologues.

Homologue	Transition	Peak Temp. in °C	$\Delta H/ \text{Jg}^{-1}$	$\Delta S/ \text{Jg}^{-1}\text{K}^{-1}$	Phase length
C ₈	I-Sm	70.46	5.91	0.0172	Sm-27.59
C ₁₄	Cr-Sm	79.64	78.71	0.2232	Sm-4.04

4. 3. Comparative study of Series A with Series X and Series Y

The mesomorphic properties of the investigated series-A were compared with two structurally similar known homologous series-X [27] and series-Y [19], as shown in Figure 5.

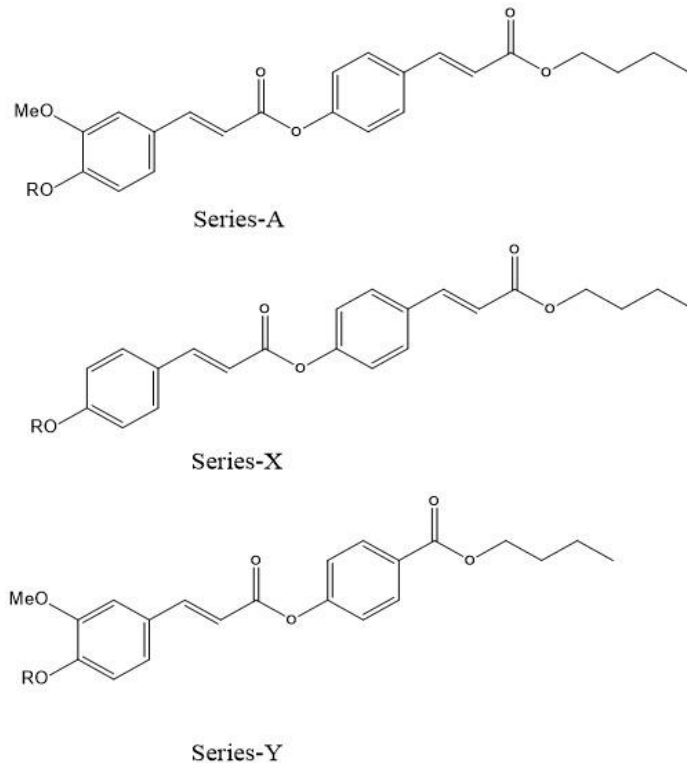


Figure 5. Structurally similar series

The Homologous series of the present investigation (series-A) and known homologous series-X and series-Y were chosen for comparison and were identical for the two phenyl rings. The central bridge was the same in all three series: $-\text{CH}=\text{CH}-\text{COO}-$. Homologous series-A differed significantly from series-X because of the laterally substituted $-\text{OCH}_3$ polar group. Series-Y differs from the terminal substituted functional group $-\text{COOC}_4\text{H}_9$ (n) and the unsaturated double bond is absent in the terminal substitution of the ester linkage, while an unsaturated double bond is present in the ester group at the terminal position $-\text{CH}=\text{CH}-\text{COOC}_4\text{H}_9$ (n) in series-X. These two major changes alter the polarity and polarizability of the structures. Thus, variations in the mesomorphic properties and degree of mesomorphism vary according to variations in the molecular structure. The intermolecular forces of attraction arising from polarity, polarizability, length-to-breadth ratio, π electron density, varying structural arrangement of the butyl end group, and its special steric effect cause variation in the magnitude of intermolecular forces of attraction among series A, X, and Y for the different homologues of the same series and same homologue from series to series. In Table 4. The structural similarities and differences between the series under comparison are shown in tabular form.

Table 4. A structural overview of the homologues series-A, series-X and series-Y.

Homologues Series	Noticeable change in molecular substitution and polarity and polarizability		
Series-A	Laterally substituted $-\text{OCH}_3$ group present at the left hand	Central bridge is $-\text{CH}=\text{CH}-\text{COO}-$	Terminal substituted function groups are n-alkoxy and $-\text{CH}=\text{CH}-\text{COOC}_4\text{H}_9$ (n)
Series-X	Laterally substituted $-\text{OCH}_3$ group is absent at left hand	Central bridge is $-\text{CH}=\text{CH}-\text{COO}-$	Terminal substituted function groups are n-alkoxy and $-\text{CH}=\text{CH}-\text{COOC}_4\text{H}_9$ (n)
Series-Y	Laterally substituted $-\text{OCH}_3$ group present at the left hand	Central bridge is $-\text{CH}=\text{CH}-\text{COO}-$	Terminal substituted function groups are n-alkoxy and $-\text{COOC}_4\text{H}_9$ (n)

Table 5. Relative thermal stability in $^{\circ}\text{C}$.

Series:	Series-A	Series-X	Series-Y
Smectic-isotropic or Smectic-nematic	89.25 $^{\circ}\text{C}$ (C_8 to C_{14})	-	111.1 $^{\circ}\text{C}$ (C_6 to C_{14})
Commencement of smectic phase	C_8	-	C_6

Nematic-Isotropic or Isotropic-Nematic	-	194.6 °C (C ₆ to C ₁₆)	-
Commencement of nematic phase	-	C ₆	-
Mesophase length (Sm+N) in °C	5 °C to 24 °C	12 °C to 87 °C	21 °C to 33 °C

Table 4 provides a structural overview of the homologues series-A, series-X, and series-Y to investigate and compare “the position of the laterally bulky and polar methoxy -OCH₃ group and change in the terminal substituted function group” on the types of mesomorphism and thermal stabilities of these compounds.

Table 5 reveals the following comparative facts.

- Homologous Series-A and Series-Y were smectogenic, whereas Series-x was the only nematogenic mesophase formation.
- Smectogenic property in series-A commences from octyloxy homologue, while in series-Y it commences from hexyloxy homologue.
- Nematic mesophase only appeared in Series-X and the commencement of nematogenic properties are from the hexyloxy Homologue.
- The mesomorphic phase length range of Series A lies in a comparatively short temperature range (5 °C to 24 °C), whereas the mesomorphic phase length ranges of Series X and Series Y were (12 °C to 87 °C) and (21 °C to 33 °C) respectively.
- Smectic thermal stability increases from series-A to Series-Y,
- Nematic thermal stability is zero for series-A and Series-Y, and up to 194.6 °C for series-X.
- In series-A and Series-Y, changing the laterally substituted -OCH₃ group present on the left hand lowers the transition temperature as compared to series-X. And removed nematogenic mesomorphic properties.
- Series-X possesses middle-order melting temperatures, whereas series-A and series-Y exhibit lower transition temperatures.

5. CONCLUSIONS

- Homologous series of the present investigation with two phenyl rings, the-OCH₃ lateral group at the left-side phenyl ring, -CH=CH-COO- as the Central bridge, and -CH=CH-COO-C₄H₉(n) terminal end group is smectogenic without exhibiting nematic properties, whose smectic property commences from the C₈ homolog, and intermolecular cohesive forces are more conducive to molecular width instead of molecular polarizability.
- The group efficiency order derived for smectic and nematic mesophase formation based on (i) thermal stability, (ii) commencement of mesophase, and (iii) total mesophase temperature length range are as follows.
 - (i-a) Nematic thermal stability: Series-X > Series-A = Series-Y
 - (i-b) Smectic thermal stability: Series-Y > Series-A > Series-X
 - (ii-a) Early commencement of Smectic mesophase: Series-Y > Series-A > Series-X

(ii-b) Early commencement of Nematic mesophase: Series-X > Series-A = Series-Y

(iii) Total Mesophase length range (Sm + N): Series-X > Series-Y > Series-A

- The present investigation supports earlier views that the large size of the laterally substituted -OCH₃ group hinders parallel arrangements of molecules that reduce end-to-end intermolecular attraction, and that the dipole moment associated with this group should operate at an angle across the long molecular axis and could enhance smectic properties.
- The present novel homolog series possesses Sm-I transition temperatures ranging from 68 to 77 °C. Such mesomorphs are useful for studying binary systems for devices that can operate at room temperature.
- The sequentially added methylene unit at the left side of the n-alkoxy terminal end group affects the change in mesomorphic characteristics of the homolog to the homolog in the same synthesis series containing the same central bridge and terminal group.
- The degree of mesomorphism depends on the molecular structure and the rigidity of the molecules.
- The present investigation supports earlier views that the central bridge has less of an effect on mesogenic properties than the terminal end group.

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