# Synthesis and Characterization of Liquid Crystalline Property of a New Thermotropic Liquid Crystalline Molecules with Terminal End Group and DSC Study

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## Abstract

We synthesized and studied a novel homologous series of esters with a lateral methoxy group and a terminal nheptyl benzoate group to study how mesomorphic behavior correlates with structure. The twelve-membered series comprises non-mesomorphic methoxy to heptyloxy derivatives, while the remaining homologs are smectegenically mesomorphic without expressing any nematic characteristics. On a heating-stage-equipped optical polarizing microscope, we observed transition temperatures and textures. IR, <sup>1</sup>H NMR, mass spectra, and elemental analysis characterized some representative members. Analytical data confirm the molecular structures of the homologs. The mesomorphic characteristics of the current novel series are compared with those of recognized structurally comparable series. The average thermal stability of the current series is 72 °C. **Keywords:** Liquid Crystals, Smectogenic, Mesomorphism, Ester, DSC

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## I. INTRODUCTION

The state of matter known as the liquid crystal state possesses characteristics of both liquids and crystals; hence, it is referred to as an anisotropic liquid state, a mesomorphic state, or simply a liquid crystal (LC) state [1]. The combination of flow properties, such as liquids and optical properties associated with crystals, makes it applicable in various fields [2-4]. This study aimed to investigate and establish the correlation between the crystalline properties and molecular structures by synthesizing new liquid crystal substances through homologous series. Will modify molecular structures by altering the central groups that connect the phenyl rings, the terminal end groups, the groups with different polarities, or the position (o, m, p) of the same polar group in the isomeric series [5-8]. Consequently, thermotropic and lyotropic LC materials have great potential to benefit humanity [9-12]. The synthesis of ester homologs, which involves the incorporation of a lateral -OCH<sub>3</sub> group at the ortho position, a -CH=CH-COO- group as a central bridge and right-handed terminal end group of -COOC<sub>7</sub>H<sub>15</sub>(n), was influenced by a review of the literature on thermotropic liquid crystals [13-16]. Following the characterization of the newly synthesized compounds, we analyze the obtained data in terms of the molecular rigidity and flexibility of their molecular structures [17-20].

## II. MATERIAL AND METHODS

Thin layer chromatography was performed on the precoated silica gel 60 F254 (Merck), and the compounds were visualized with UV light at 254 nm and 365 nm or with iodine steam. IR spectrometers were recorded using the ATR technique on the Shimadzu FT-IR spectrometer. The <sup>1</sup>H spectra were recorded with a Bruker AVANCE III spectrometer (400 MHz) in DMSO- $d_6$ . As an internal standard, chemical changes are expressed in ppm from Tetramethylsilane (TMS). The mass spectrometers were recorded using direct input probes on the Shimadzu GCMS QP2010 Ultra mass spectrometer. All reactions were carried out under an ambient atmosphere. All chemicals were purchased from Loba, Molychem, SRL, and CDH and used without further purification. Transition temperatures for synthesized compounds and phases were identified through a polarizing optical microscope attached to a Mettler FP82HT heating plate. Enthalpies and transition temperatures were determined using differential scanning calorimetry (DSC) on the PerkinElmer thermal analyzer at 10 °C min<sup>-1</sup>.

## **III. EXPERIMENTAL**

#### **3.1** General synthesis of (*E*)-3-(4-alkoxy-3-methoxyphenyl) acrylic acid (A):

The mixture of (E)-3-(4-hydroxy-3-methoxyphenyl) acrylic acid (0.1 mmol), corresponding alkyl halide (0.12 mmol), and potassium hydroxide (0.25 mmol) were dissolved in 10 mL of MeOH. The reaction mass was refluxed for 3 to 4 hr. Subsequently, 10% aqueous KOH solution (20 mL) was added, and reflux continued for another 2 hr. The reaction mixture was then cooled and acidified with dil. hydrochloric acid to precipitate the corresponding alkoxy acids. The time for refluxing reaction mass extended with increasing alkyl chain length. The obtained product was recrystallized from ethanol or acetic acid [21, 22].

#### 3.2 Synthesis of n-heptyl-4-hydroxy-benzoate (B):

4-hydroxybenzoic acid was esterified using n-heptyl alcohol with conc.  $H_2SO_4$  as a catalyst using the usual established mixture method. The progress of the reaction was monitored using TLC. The resulting reaction mixture was cooled, filtered, and dried. These solid esters were crystallized using distilled n-hexane to give a pure compound [23, 24].



Scheme 1. Synthesis of n-heptyl (E)-4-((3-(4-alkoxy-3- methoxyphenyl)acryloyl)oxy)benzoate

## **3.3** General synthesis of n-heptyl(*E*)-4-((3-(4-alkoxy-3-methoxyphenyl)acryloyl)oxy)benzoate (C):

The Steglich esterification method was then used to esterify each molecule of (E)-3-(4-alkoxy-3methoxyphenyl) acrylic acid with n-heptyl-4-hydroxybenzoate (**Scheme 1**). To a stirred solution of compound, **A** (10 mmol) and **B** (8 mmol) in 10 mL dichloromethane, *N*, *N*-di-cyclohexyl carbodiimide (10 mmol), and a catalytic amount of 4-dimethyl amino pyridine were added, and stirred overnight. The organic layer was filtered, washed twice with sodium bicarbonate solution, and concentrated to obtain a residue from which the product was isolated as white crystals by adding hexane [25-27].

Та	ble 1:	Physic	cochen	nical (	charact	teristi	cs
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Sr. No.	Compound	R	Molecular weight	Molecular formula	Yield (%)
1	C1	CH <sub>3</sub>	426.51	C <sub>25</sub> H <sub>30</sub> O <sub>6</sub>	78
2	C2	C <sub>2</sub> H <sub>5</sub>	440.54	C <sub>26</sub> H <sub>32</sub> O <sub>6</sub>	73
3	C3	C <sub>3</sub> H <sub>7</sub>	454.56	C <sub>27</sub> H <sub>34</sub> O <sub>6</sub>	70
4	C4	C <sub>4</sub> H <sub>9</sub>	468.59	C <sub>28</sub> H <sub>36</sub> O <sub>6</sub>	75
5	C5	C5H11	482.62	C <sub>29</sub> H <sub>38</sub> O <sub>6</sub>	71
6	C <sub>6</sub>	C6H13	496.64	C <sub>30</sub> H <sub>40</sub> O <sub>6</sub>	74
7	C <sub>7</sub>	C7H15	510.67	C <sub>31</sub> H <sub>42</sub> O <sub>6</sub>	72
8	C8	C8H17	524.70	C <sub>32</sub> H <sub>44</sub> O <sub>6</sub>	70
9	C10	C10H21	552.75	C <sub>34</sub> H <sub>48</sub> O <sub>6</sub>	71
10	C <sub>12</sub>	C12H25	580.81	$C_{36}H_{52}O_{6}$	70
11	C14	C14H29	608.86	C <sub>38</sub> H <sub>56</sub> O <sub>6</sub>	69

12 C <sub>16</sub>	C <sub>16</sub> H <sub>33</sub>	636.91	C <sub>40</sub> H <sub>60</sub> O <sub>6</sub>	65
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## n-heptyl (E)-4-((3-(4-pentyloxy-3-methoxyphenyl)acryloyl)oxy)benzoate (Cs)

White Crystals, Yield: 71%, FTIR (ATR,  $v_{max}$ , cm<sup>-1</sup>): 617.24 (poly methylene of  $-C_5H_{11}$  and  $-C_7H_{15}$ ), 856.42 (p-sub. Benzene ring), 2894.18 (CH<sub>2</sub> Str.), 1720.56, 1604.83 & 1280.78 (-COO- & -CO-), 1512.24 & 1450.52 (aromatic -HC=CH-), 1118.75 (CO- of alkoxy), 3000.05 (C-H of aromatic Str.); <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>,  $\delta$  *ppm*): 0.889 (m, 6H, -CH<sub>3</sub> of  $-C_7H_{15}$  &  $-C_5H_{11}$ ), 1.363-1.345 (m, 4H, *J* = 7.2, -CH<sub>2</sub> of  $-C_5H_{11}$ ), 1.267 (m, 6H, -CH<sub>2</sub>-CH<sub>2</sub>-), 1.415 (m, 2H, -CH<sub>2</sub>-), 1.750-1.699 (m, 4H, *J* = 6.8 Hz, -CH<sub>2</sub>-), 3.864 (s, 3H, m-substituted -OCH<sub>3</sub>), 4.298-4.281 (m, 2H, *J* = 6.8 Hz, -CH<sub>2</sub> of COOC<sub>7</sub>H<sub>15</sub>), 4.040-4.008 (t, 2H, *J* = 6.4 Hz, -OCH<sub>2</sub>-), 8.090-8.075 (dd, 2H, *J* = 6 Hz, Ar-H), 7.358-7.353 (dd, 2H, *J* = 2 Hz, Ar-H), 7.036, 7.333 & 7.471 (d & s, 3H, Ar - CH=CH-), 6.838 & 7.828 (d, 2H, CH=CH); MS (*m*/*z*): 482 (M<sup>+</sup>); Anal. Calc. For C<sub>29</sub>H<sub>38</sub>O<sub>6</sub>: C, 72.17; H, 7.94; Found: C, 72.23; H, 7.98.

#### n-heptyl (E)-4-((3-(4-octyloxy-3-methoxyphenyl)acryloyl)oxy)benzoate (C<sub>8</sub>)

White Crystals, Yield: 70%, FTIR (ATR,  $v_{max}$ , cm<sup>-1</sup>): 617.21 (poly methylene of -C<sub>8</sub>H<sub>17</sub> and -C<sub>7</sub>H<sub>15</sub>), 857.58 (p-sub. Benzene ring), 2920.09 (CH<sub>2</sub> Str.), 1720.45, 1603.76 & 1279.92 (-COO- & -CO-), 1511.88 & 1449.68 (aromatic -HC=CH-), 1118.21 (CO- of alkoxy), 3001.23 (C-H of aromatic Str).; <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>,  $\delta$  *ppm*): 0.891 (m, 6H, -CH<sub>3</sub> of -C<sub>7</sub>H<sub>15</sub> & -C<sub>8</sub>H<sub>17</sub>), 1.365-1.347 (m, 4H, *J* = 7.2, -CH<sub>2</sub> of -C<sub>8</sub>H<sub>17</sub>), 1.269 (m, 10H, -CH<sub>2</sub>-CH<sub>2</sub>-), 1.417 (m, 4H, -CH<sub>2</sub>-), 1.749-1.698 (m, 4H, *J* = 6.8 Hz, -CH<sub>2</sub>-), 3.862 (s, 3H, m-substituted -OCH<sub>3</sub>), 4.299-4.283 (m, 2H, *J* = 6.4 Hz, -CH<sub>2</sub> of COOC<sub>7</sub>H<sub>15</sub>), 4.046-4.009 (t, 2H, *J* = 6.4 Hz, -OCH<sub>2</sub>-), 8.092-8.076 (dd, 2H, *J* = 6.4 Hz, Ar-H), 7.359-7.354 (dd, 2H, *J* = 2 Hz, Ar-H), 7.037, 7.339 & 7.473 (d & s, 3H, Ar -CH=CH-), 6.839 & 7.830 (d, 2H, CH=CH); MS (*m*/*z*): 524 (M<sup>+</sup>); Anal. Calc. For C<sub>32</sub>H<sub>44</sub>O<sub>6</sub>: C, 73.25; H, 8.45; Found: C, 73.77; H, 8.49.

Transition temperatures for synthesized compounds and phases were identified through a polarizing optical microscope attached to a Mettler FP82HT heating plate.

### 4.1 Mesomorphic Behaviour

## IV. RESULT AND DISCUSSION

The mesomorphic characteristics of novel synthesized homologs series are observed by the polarizing optical microscopy method (POM). All observations of phase transition temperatures (°C) are given in **table** 2, and the mesophase textures of the representative compounds observed by POM are shown in **fig. 1**.

Compound	n-Alkyl group R= -CnH <sub>2n+1</sub>	Smectic	Isotropic
C1	CH <sub>3</sub>	-	81
C2	C <sub>2</sub> H <sub>5</sub>	-	98
C3	C <sub>3</sub> H <sub>7</sub>	-	127
C4	C4H9	-	117
C5	C5H11	-	113
C6	C <sub>6</sub> H <sub>13</sub>	-	106
C7	C7H15	-	82
C8	C8H17	71	93
C10	C10H21	76	97
C12	C12H25	69	88
C14	C14H29	63	72
C16	C16H33	57	62

Table 2: Transition te	mperatures of	f homologous	series in °C
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Fig. 1. Mesophase texture observed by POM (a) Smectic phase of compound C<sub>8</sub> at 56°C during the cooling cycle; (b) Smectic phase of compound C<sub>12</sub> at 59°C during the cooling cycle; (c) Smectic phase of compound C<sub>14</sub> at 61°C during the heating cycle

Phase Homologous series n-heptyl(E)-4-((3-(4-alkoxy-3-methoxyphenyl)acryloyl)oxy)benzoates were synthesized from 4-n-alkoxy ferulic acid and a non-mesomorphic component, 4- hydroxybenzoate. The homologs C<sub>1</sub> to C<sub>7</sub> are not mesomorphic, but the mesomorphism starts with the homolog C<sub>8</sub> and appears to last until the C<sub>16</sub> homologs as an enantiotropic smectic phase. The nematic mesophase exhibition is absent for the homologs of the entire series of the present investigation. The homologs ' transition temperatures (table 2) are plotted versus the number of carbon atoms in the n-alkyl chain (-CnH2n + 1).



Fig. 2. Phase behavior of homologous series

The phase behaviors of a series are represented in a phase diagram (**Fig. 2**) by transition curves (Cr-I or Cr-Sm and Sm-I). The Cr-I or Cr-Sm transition curve adopts a zigzag path of rising and falling values with an overall descending tendency as the series ascends. The Sm-I transition curve adopts a continuous rising and falling pattern. The average thermal stability for smectic is 72 °C, and the mesomorphic (Sm) phase length is between 3°C and 15°C. Mesomorphic characteristics vary from homolog to homolog as a function of molecular length. Therefore, the ester series under investigation with lateral substitution by the -OCH<sub>3</sub> group is smectogenic without exhibiting nematic properties. The C<sub>1</sub> to C<sub>7</sub> ester homologs do not exhibit any mesomorphism because the magnitudes of the anisotropic forces of the intermolecular end-to-end and/or lateral attractions occurred as a result of inappropriate molecular rigidity and flexibility, which result in an inability to resist exposed thermal vibrations to cause mesomorphism and therefore have a high crystalizing tendency. A lamellar packing of molecules with a layered structure is produced by the molecules of the homolog derivatives C<sub>8</sub> to C<sub>16</sub>. Thus, a specific temperature range seems to be where smectogenic mesophase formation is seen. The smectogenic mesophase generation reappears from and below the isotropic temperature in a solid crystalline state in the same sample when it is cooled from an isotropic state at the correct cooling rate. As a result, the

development of enantiotropic smectic mesophase without nematic phase manifestation was observed for homologs  $C_8$  to  $C_{16}$  homologs of the current new series. In the current series, mesomorphic properties vary from homolog to homolog due to changes in the number of methylene units, which affect the molecular length, polarity, polarizability, length-to-breadth ratio, and other properties. These differences in molecular stiffness and flexibility caused by changes in molecular characteristics allow for the formation of mesomorphism-inducing anisotropic forces of appropriate or inappropriate magnitudes [28].

## 4.2 DSC analysis

We have performed an additional study utilizing DSC analysis on heating and cooling cycles to confirm the phase transition temperature of the synthesized ester-based materials. Two selected homolog thermograms are shown in fig. 3. A DSC thermogram is traced under heating and cooling circumstances to determine the precise temperature of the phase transition. Compound C<sub>8</sub> with the octyloxy group showed two endothermic peaks, a mesomorphic phase transition at 65°C, and a smectic mesophase behavior up to 68 °C. The isotropic point is the temperature at which the compound forms the smectic phase, melts, and retains its liquid crystal mesophase behavior. Additionally, at temperatures above 73°C, the isotropic phase change is noticeable. The enthalpy was found to be attained at 105 and 5.9 J/g. These two transition curves were also observed during the cooling or exothermic cycle in the opposite cycle. At 59°C, the substance exhibited isotropic behavior while cooling, and below this range, the smectic transition occurred. The notable difference shows the sharp smectic phase within the molecule during the exothermic cycle. This cycle, which displays the mesophase LC, results in a collapse of the isotropic behavior and a transition with an enthalpy change from -6.5 J / g in the isotropic phase to -77.89 J/g in the smectic phase. At a temperature of  $42^{\circ}$ C, the compound reverts to the crystalline state. Like compound  $C_{12}$ , which has two endothermic peaks at 60 °C and 75 °C, respectively, the first is for smectic, and the other is for isotropic. The system's enthalpy was found to be 187 J/g and 1.03 J/g. The compound also showed smectic mesophase behavior while cooling at 59 °C and reverted to the crystalline form at 40°C. During this cycle, enthalpy went from -5.9 to -52.2 J/g.



Fig. 3. DSC thermograms of compounds C<sub>8</sub> & C<sub>12</sub> during heating and cooling cycles (10°C min<sup>-1</sup>)

These transition temperatures agree well with the POM transition temperatures. The enthalpies and transition temperatures were calculated using a DSC and a PerkinElmer Thermal analyzer at 10°C min-1. The DSC investigation further confirmed the enantiotropic mesophase behavior of the current series.

## Comparative study of the present series

The mesomorphic properties of the present series 1 are compared with the structurally similar homologous series-X [29], as shown in **fig. 4**.



Fig. 4. Structurally similar series

The primary similarity lies in their two phenyl rings in both Series 7 and the selected Series X. However, Series 7 exhibits notable differences from Series X due to substituting a polar group,  $-OCH_3$ , in a lateral position. This modification results in the relocation of the terminal alkoxy group (methoxy) from the center to the end position, bringing it closer to the terminal end group alkoxy group present in Series 7. Furthermore, another potential alteration in Series 7 involves repositioning the unsaturated ester group to the central position, with the unsaturated double bond positioned adjacent to the ester bond (-CH=CH-COO-). On the other hand, Series X contains an unsaturated double bond near the ester group at the terminal position (-CH=CH-COOC<sub>7</sub>H<sub>15</sub>(n)). These two significant modifications significantly impact the structure's polarization and the length-to-breadth ratio of the homologues and even introduce distinct steric obstacles.

Table 3 represents some mesomorphic properties like average thermal stability, the beginning of mesophase, the number of homologs and their type, mesophase length range, odd even effect, etc., in comparison.

	<u> </u>	
Series: →	Series-1	Series-X
Smectic-isotropic	$72^{\circ}C(C_8-C_{16})$	89.8°C (C <sub>8</sub> -C <sub>16</sub> )
Commencement of Smectic phase	$C_8$	$C_6$
Mesophase length in °C	3°C to 15°C	11°C to 35°C

Table 3: Average thermal stability in °C

Table 3 indicates that,

- Homologous series-1 of the present investigation is only smectogenic, whereas a series X chosen for comparison is also smectogenic.
- The smectic mesophase begins from the  $C_8$  homolog in series-1 and  $C_6$  in series- X.
- For series-X, the total mesophase length range is between 11°C to 35°C which is higher than the series-1, in which the total mesophase length range lies between 3 °C and 15 °C.
- The average thermal stability of the present series-1 for mesophase (Sm) is relatively lower than a series-X.

When a highly polar methoxy group is laterally substituted into homologous series 1, the resulting broadening of the molecule reduces intermolecular attraction while simultaneously increasing molecular polarizability and enhancing intermolecular attraction. Therefore, two opposing effects of molecular cohesion or intermolecular forces can be attributed to the exact cause. Thus, the superiority of the two opposing effects operating simultaneously for the same reason determines the net impact of intermolecular attractions. Also, series 1 contained two phenyl rings, whereas series X had two. As a result, the aromaticity and molecular stiffness of series X are nearly identical to those of series I. Series-1 and X have completely different constitutions, whereas their left n-alkoxy terminal end groups are similar for the identical series homolog. i.e.,

the right-handed terminal end group bonded to  $-COO-C_7H_{15}(n)$  group and -CH=CH-COO- is the central bridge in series-1, whereas  $-CH=CH-C_7H_{15}(n)$  is the terminal end group and -COO- is the central bridge in series-X. The broadening of the molecular width effect, which dominated between two opposing effects and was carried out by the lateral substitution of the  $-OCH_3$  group, is responsible for reducing the smectic thermal stability of the current novel series-1. With distinct smectic phase initiations from the homolog C<sub>8</sub> and C<sub>6</sub> homolog for series-1 and series-X, respectively, transition temperatures (Cr-Sm and Sm-I) and thermal stabilities are lower for series-1 compared to series-X. However, neither series exhibits nematic mesophase. Hence, the molecular stiffness and flexibility that result from a substance's molecular structure determine its mesomorphic properties.

## **V. CONCLUSIONS**

- Homologous series of present investigation with two phenyl rings,  $-OCH_3$  lateral group and  $-COO-C_7H_{15}$  (n) terminal end group, is smectogenic without exhibition of nematic property whose smectic property commences from  $C_8$ .
- The group efficiency order derived for the smectic based on (i) average thermal stability, (ii) commencement of the mesophase, and (iii) total mesophase length range is as follows.
  - (i) Smectic average thermal stability: (Series X > Series-1)
  - (ii) Early commencement of the Smectic mesophase: (Series-X > Series-1)
  - (iii) Total mesophase length (temperature range): (Series-X > Series-1)
- Mesomorphic properties (LC) and degree of mesomorphism are sensitive and susceptible to molecular structure.
- The molecular rigidity and flexibility, depending on the molecular structure, operate the mesomorphic behaviors of a substance.

DSC and POM techniques confirmed the mesomorphic thermal and optical characteristics. The findings show that the alkyl chain length significantly influences the mesomorphic properties and thermal stabilities of the various mesophases. The evaluation results of the compounds under investigation show that the molecules are sensitive to their lateral substituents, which affects the mesophase's thermal properties and stability.

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