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## Study of mesomorphism and its relation to molecular structure through molecular rigidity and flexibility

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### ABSTRACT

Novel liquid Crystalline (LC) substances of chalconyl ester derivatives from Vinyl aromatic carboxylic acids are synthesized and studied with a view to understanding and establishing the relation between molecular structure,  $\text{RO-C}_6\text{H}_4\text{-CH=CH-COO-C}_6\text{H}_4\text{-CH=CH-CO-C}_6\text{H}_4\text{-F}$  (para) and LC properties of the thermotropic variety. Homologous series consists of eleven members ( $\text{C}_1$  to  $\text{C}_{16}$ ).  $\text{C}_1$  and  $\text{C}_2$  members of novel series are nonliquid crystals (NLC) and the rest of the homologues ( $\text{C}_3$  to  $\text{C}_{16}$ ) are enantiotropic nematic with absence of smectic property throughout. Transition temperatures and textures of homologues were determined by an optical polarizing microscope (POM) equipped with a heating stage. Textures of nematic phase are threaded or Schlieren. The transition temperatures of novel homologues are relatively lower than the corresponding dimeric trans n-Alkoxy Cinnamic acids from which, novel homologues are synthesized. Thermal, analytical, spectral data confirmed molecular structures of novel homologues. Cr-N/I and N-I transition curves behaved in normal manner except  $\text{C}_8$  to  $\text{C}_{14}$  homologues which deviated from normal descending tendency in N-I transition curve. N-I transition curve exhibited odd-even effect up to  $\text{C}_6$  homologue and odd member's N-I transition curve occupied lower position than even member's N-I transition curve. Thermal stability for nematic is  $118.2^\circ\text{C}$ ; with its total mesophase length ranging from 12.0 to  $50.0^\circ\text{C}$ . Thus, it is a middle ordered melting type series with absence of smectic property. Group efficiency order derived on the basis of thermal stability using comparative study of present novel series with structurally similar analogous series as mentioned below.

Smectic: Series-Y > Series-X = Series-1 and  
Nematic: Series-X > Series-1 > Series-Y

### KEYWORDS

Liquid crystal;  
Mesomorphism; Molecular  
Flexibility; Nematic; Smectic

## Introduction

Liquid Crystal (LC) properties [1] and their behaviors are directly linked to the molecular structure [2–6] through molecular rigidity and flexibility [7–10]. The study of structure-property relationships generates a wealth of knowledge and aids the generation of liquid crystals with specific properties for a range of applications [11–13]. Moreover, chalcones are particularly useful in the pharmaceutical preparations such as antifungal, antibacterial, anticancer agents and in agricultural products due to their bioactivity [14–20]. Thus, the present investigation was planned to synthesize novel chalconyl ester derivatives through

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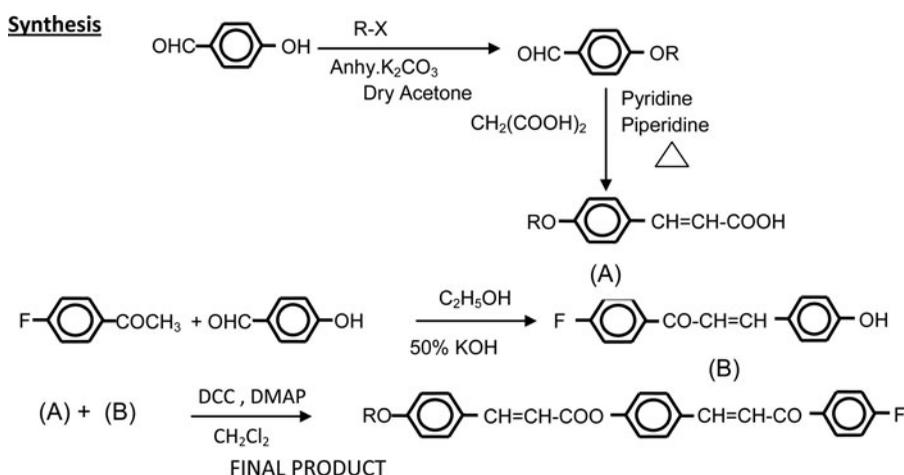
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homologous series containing three phenyl rings bonded through  $-\text{CH}=\text{CH}-\text{COO}-$  and  $-\text{CH}=\text{CH}-\text{CO}-$  central bridges with varying  $-\text{OR}$  and fixed  $-\text{F}$  terminal end groups, with a view to understanding and establishing the effect of molecular structure on LC properties. The present investigation concerns the synthesis and characterization by analytical, thermal and spectral data. The evaluated thermometric data will be interpreted and discussed on the basis of molecular rigidity and flexibility. Several ester homologous series have been reported to date [21–26].

## Experimental

### Synthesis

4-Hydroxy benzaldehydes were treated with malonic acid to convert them into trans 4-n-alkoxy cinnamic acids (A) [27]. n-Alkoxy cinnamic acids (A) were condensed with  $\alpha$ -4-flouro benzoyl  $-\beta$ -4'-hexyloxy phenyl ethylene [28] (M.P.-112 °C)[B] by a usual established method [29] to give final products. The synthetic route to series is shown below in Scheme 1. Final products were individually, filtered, washed, dried, and purified until the constant transition temperatures obtained.



**Scheme 1.** Synthetic route to the series-1.

### Characterization

Some selected members of the titled series were characterized by elemental analysis,  $^1\text{H}$ NMR spectra and IR spectra, Textures of mesophases were characterized by miscibility method. Microanalysis for C,H,N elements were performed on Perkin Elmer PE 2400 analyzer (Table 1).  $^1\text{H}$ NMR spectra were obtained on Bruker spectrometer using  $\text{CDCl}_3$  as solvent. IR spectra were recorded on a Perkin- Elmer spectrum GX. Transition temperatures and liquid crystal properties were investigated using optical polarizing microscope (POM) with heating stage.

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

Molecular formula	Elemental % found (Compared with % calculated)	
	C	H
C <sub>32</sub> H <sub>33</sub> O <sub>4</sub> F	76.76 (76.80)	6.33 (6.60)
C <sub>34</sub> H <sub>37</sub> O <sub>4</sub> F	77.39 (77.27)	7.32 (7.00)
C <sub>36</sub> H <sub>41</sub> O <sub>4</sub> F	77.50 (77.69)	7.45 (7.37)

### Analytical data

**H<sup>1</sup>NMR: in ppm.** For octyloxy homologue.

Ethylenes: (200 MHZ)  $\delta$  (CDCl<sub>3</sub>) (ppm) 0.98 (-CH<sub>3</sub>-CH<sub>2</sub> of -C<sub>8</sub>H<sub>17</sub>), 1.40 (long-CH<sub>2</sub>-chain), 3.4 -OCH<sub>2</sub> of (-OCH<sub>2</sub> of C<sub>8</sub>H<sub>17</sub>), 6.35 & 6.45, 8.45, and 8.24 (p-sub. benzene rings)

**H<sup>1</sup>NMR: in ppm.** For decyloxy homologue.

Ethylenes: (200 MHZ)  $\delta$  (CDCl<sub>3</sub>) (ppm) 1.05 (-CH<sub>3</sub>-CH<sub>2</sub> of -C<sub>8</sub>H<sub>17</sub>), 1.52 (long-CH<sub>2</sub>-chain), 3.25 -OCH<sub>2</sub> of (-OCH<sub>2</sub> of C<sub>8</sub>H<sub>17</sub>), 6.52 & 6.29, 8.41, and 8.25 (p-sub. benzene rings)

**IR in Cm<sup>-1</sup>,**

**IR Spectrum.** For octyloxy homologue

Ethylenes: (vmax/cm-1): 2958, 2858, 1482, 1345 (-C-H, aliphatic), 1735, 1266 (ester group), 17 (>C=O group), 1558 (-C=C-, aromatic), 1031, 1235 (ether group), 845.0 (p- sub. benzene ring), 1599, 1510, 1494 (aromatic ring).

**Spectrum.** For Decyloxy homologue

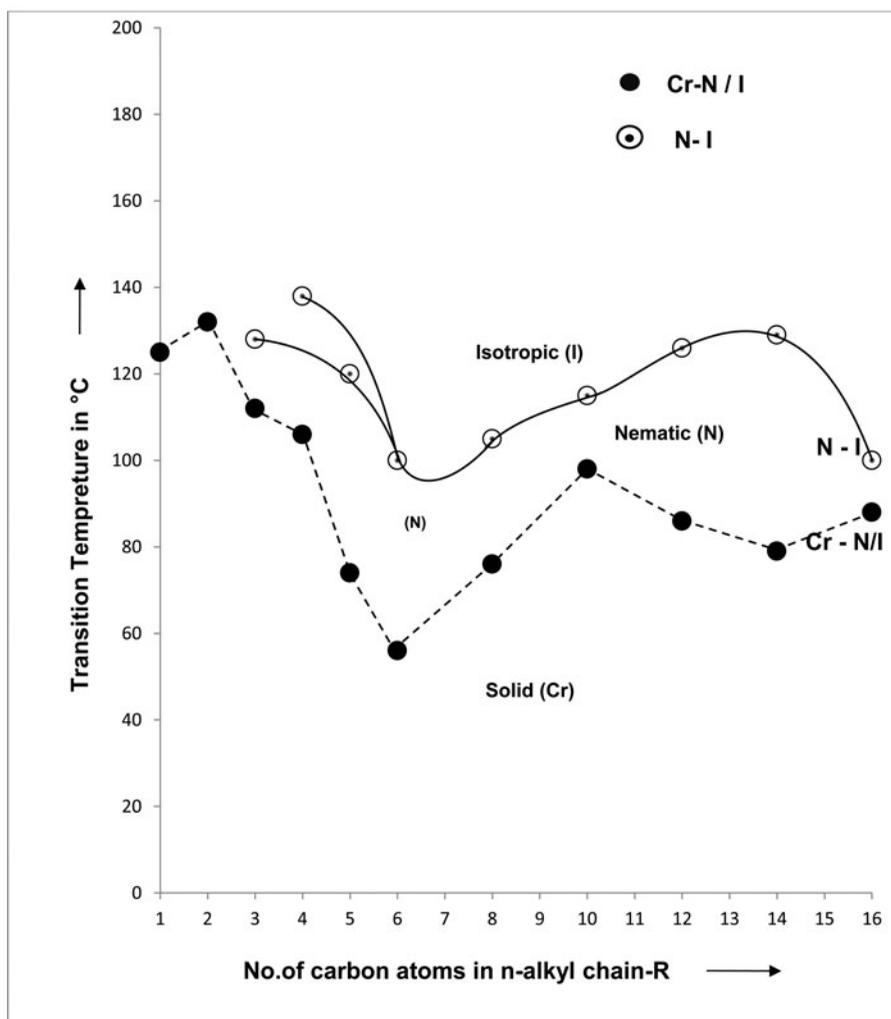
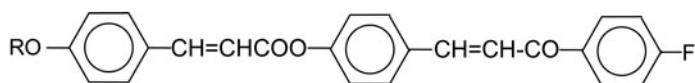
Ethylenes: (vmax/cm-1): 2998, 2825, 1466, 1382 (-C-H, aliphatic), 1713, 1245 (ester group), 1736 (>C=O group), 1566 (-C=C-, aromatic), 1036, 1266 (ether group), 845.2 (p- sub. benzene ring), 1642, 1475, 1480 (Aromatic ring).

### Result and discussion

A novel chalconyl ester homologous series of thermotropic LC derivatives were synthesized by condensing trans-4-n-alkoxy cinnamic acids individually with  $\alpha$ -4-hydroxy phenyl  $\beta$ -4'-fluorobenzoyl ethylene. Lowering of transition temperature of novel compounds as compared to corresponding dimeric trans-n-alkoxy cinnamic acids take place by esterification process. Enantiotropic nematogenic mesomorphism commences from the C<sub>3</sub> homologue and continued up to the C<sub>16</sub> homologue. The C<sub>1</sub> and C<sub>2</sub> homologues are not liquid crystals. Transition temperatures (Table 2) as determined by POM equipped with a heating stage were plotted against the number of carbon atoms present in n-alkyl chain 'R' of -OR group and Cr-N/I

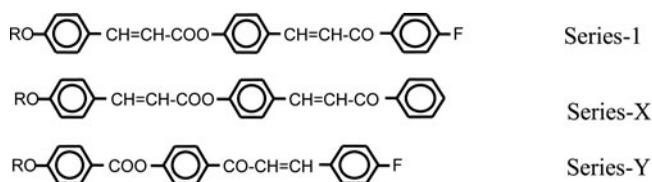
**Table 2.** Transition temperatures in °C.

Compound no.	n-Alkyl (C <sub>n</sub> H <sub>2n+1</sub> ) group	Smectic	Nematic	Isotropic
1	C <sub>1</sub>	—	—	125.0
2	C <sub>2</sub>	—	—	132.0
3	C <sub>3</sub>	—	112.0	128.0
4	C <sub>4</sub>	—	106.0	138.0
5	C <sub>5</sub>	—	74.0	120.0
6	C <sub>6</sub>	—	90.0	100.0
7	C <sub>8</sub>	—	75.0	108.0
8	C <sub>10</sub>	—	98.0	115.0
9	C <sub>12</sub>	—	86.0	126.0
10	C <sub>14</sub>	—	79.0	129.0
11	C <sub>16</sub>	—	88.0	100.0



**Figure 1.** Homologous series:  $\alpha$ -4-[4'-n-alkoxy cinnamoyloxy] phenyl-  $\beta$ -4''-floro benzoyl ethylenes.

as well as N-I transition curves are obtained on linking like or related points as depicted in a phase diagram as shown in Fig 1. The Cr-N/I transition curve follows a zigzag path of rising and falling values with an overall descending tendency and behaves in normal manner. The N-I transition curve initially descended and then ascended from C<sub>8</sub> to C<sub>14</sub> in an unexpected deviating manner and then finally descended in the C<sub>16</sub> homologue in a normal manner with exhibition of the odd-even effect from C<sub>3</sub> to C<sub>6</sub> homologue. The smectic phase is totally absent either in a monotropic or an enantiotropic manner. The odd-even effect disappears from the C<sub>6</sub> member of the series and continues as a single transition curve for higher homologues of longer n-alkyl chain 'R' of -OR group. Odd member's N-I transition curve occupied lower position as compared to even member's N-I transition curve. Textures of the nematic phase are threaded or Schlieren. Analytical, thermal and spectral data confirmed the molecular structures of homologues. Thermal stability in average for nematic phase is



**Figure 2.** Structurally similar analogous series.

118.2 and mesophase lengths ranges between 12.0 and 50.0°C. Thus, novel chalconyl ester homologous series is nematogenic without exhibition of smectic property. The LC properties of series from homologue to homologue shows variations depending upon the suitable magnitudes of anisotropic forces of intermolecular attractions and closeness as a consequence of individual molecular structural rigidity and flexibility. Lowering of transition temperatures of novel homologues as compared to corresponding to trans dimeric n-Alkoxy Cinnamic acids is attributed to the breaking of hydrogen bonding between two same molecules of vinyl aromatic acid through esterification process. The nonmesomorphicity of methoxy and ethoxy derivatives of present novel series is attributed to their high crystallizing tendency. Thus, the molecules of C<sub>1</sub> and C<sub>2</sub> homologues sharply and directly transform into isotropic state. The exhibition of a nematogenic mesophase commences from the C<sub>3</sub> homologue and continues up to last C<sub>16</sub> homologue of the series in an enantiotropic manner. The molecular arrangement required to facilitate smectic mesophase formation is hindered among the molecules of entire series due to absence of lamellar packing of molecules in their respective crystal lattices. The appearance of odd-even effect up to C<sub>6</sub> homologue is attributed to the sequentially added methylene unit in n-alkyl chain 'R' of -OR group which maintained expected status of n-alkyl chain 'R' and the rest of the molecular part including tailed ended monoatomic fluoro (F) group. The disappearance or diminishing of odd-even effect from and beyond merging of odd and even N-I transition curves for higher homologues of longer n-alkyl chain 'R' is attributed to coiling or bending or flexing or coupling of n-alkyl chain 'R' with core structure of a molecule which modifies the magnitudes of molecular rigidity and flexibility. The changing trend in mesogenic (LC) behavior or properties with changing molecular structure of present novel series-1 are compared with the structurally similar analogous series : X [30] and Y [31] as mentioned below in Fig. 2.

Series 1 and X are identical with respect to three phenyl rings and two central bridges linking them, which contributes to the total molecular rigidity. Their left flexible n-alkoxy group -OR of series 1 and X are identical for the same homologues contributing partly to the total flexibility, but their right handed tailed ended -F and -H groups differ, which partly differs with respect to respective total molecular flexibility. Homologous series 1 and Y are identical with respect to three phenyl rings and one of the central groups linking middle and third phenyl rings which contributes partly to the total molecular rigidity, but, differs with respect to -CH=CH- unit for a central bridges linking first and middle phenyl rings and causes difference in total molecular rigidity offered by -CH=CH- conjugated unit through molecular rigidity offered double bond and the negligible positional difference of -CO- in central group, nearest and away from third phenyl ring respectively. The total molecular flexibility of a novel series-1 of present investigation and a series-Y chosen for comparative study are the same for the same homologue, because the left ended terminal group -OR and the right ended tailed group-F are same for the same homologue from series-1 and Y. Therefore, the variations in LC properties, behaviors and the degree of mesomorphism for the same homologue from series to series will depend upon the magnitudes of differing features of series-1, X and Y

**Table 3.** Relative thermal stabilities in °C.

Series →	Series-1	Series-2	Series-3
Sm-N or Sm-I Commencement of smectic phase	—	—	158.55 (C <sub>1</sub> –C <sub>16</sub> ) C <sub>1</sub>
N-I Commencement of nematic phase	118.2 (C <sub>3</sub> –C <sub>16</sub> ) C <sub>3</sub>	144.5 (C <sub>6</sub> –C <sub>16</sub> ) C <sub>6</sub>	—
Total mesophase lengths from t <sub>1</sub> °C to t <sub>2</sub> °C (Sm+N)	12.0–50.0 C <sub>16</sub> C <sub>14</sub>	06.0–47.0 C <sub>8</sub> C <sub>12</sub>	5.1–74.5 C <sub>12</sub> C <sub>14</sub>

under comparative study. However variations in mesomorphic properties from homologue to homologue in the (any) same series will depend upon the magnitudes of added methylene unit and its related magnitudes of flexibility, keeping rest of the molecular part and its molecular rigidity plus flexibility due to tail end group remain unaltered, throughout the same series. Following Table 3 represents comparative thermometric data for series-1, X and Y as under.

Thermometric data of Table 3 suggest that,

- Homologous series -1 and X are nematogenic, whereas series-Y is smectogenic.
- The smectogenic mesomorphism commences from very first member of a series-Y, but, it does not commence till the last member of series 1 and X.
- The nematogenic mesomorphism commences earliest from C<sub>3</sub> homologue of present novel series-1 and late from C<sub>6</sub> homologue of series-X, but, it does not commence till the last C<sub>16</sub> homologue of series-Y.
- Thermal stability for smectic is the highest for series-Y but it does not stabilize for series X and 1.
- Thermal stability for nematic of present series-1 is lower than series-X, but it does not stabilize for series-Y till the last homologue.
- Lower total mesophase length decreases from series-1 to X to Y and upper mesophase lengths increases from series-X to 1 to Y.

Homologous series 1 and Y identical with respect to molecular flexibility for the same homologue but differs with respect to molecular rigidity due to difference of presence and absence of –CH=CH– unit of first central bridge linking first and a middle phenyl ring including difference in the place of Carbonyl group of direct attachment to middle (series-Y) or third phenyl ring (series-1) longing to chalconyl central bridge. The same homologue from series to series. Therefore, molecules of series-1 being more rigid but, facing unsuitable magnitudes of anisotropic forces of intermolecular cohesive forces of attraction to build up layered molecular arrangement in preoccupied crystal lattices and subsequently unable to maintain sliding layered molecular organization under floating condition to facilitate smectic mesophase formation. However intermolecular end to end forces of cohesions are suitable and sufficient enough to arrange and maintain the molecules in statically parallel orientational order in floating condition to facilitate nematic mesophase only with neglecting formation of smectic phase as a result of unfavourable magnitudes of dispersion forces, dipole–dipole interactions, length to breadth ratio and the ratio of the molecular polarity to polarizability and absence of lamellar packing of molecules of presently investigated series-1. On the other hand the molecules bearing –COO– central bridge instead of –CH=CH–COO– and –CO group of chalconyl central bridge being directly attached to middle phenyl ring linking middle and third phenyl ring through –CH=CH– unit facilitate suitable magnitudes of anisotropic forces of intermolecular attractions which builds up lamellar packing of molecules in the molecules of crystal lattices with the positional status of –CO group attached to central phenyl ring; right from the very first member of a series-Y and subsequently focal conic sliding layered molecular arrangement is maintained under floating condition to facilitate only smectic phase, but, residual end to end intermolecular dispersion forces are insufficient to induce nematic phase. Thus,

the role of carbonyl unit near or away from middle phenyl ring by chalconyl central bridge as well as presence or absence of  $-\text{CH}=\text{CH}-$  unit in ester central bridge are very important for a molecule made up of three phenyl rings bonded through two central bridges and identically similar terminal ends of similar or same polarity and polarizability for the same homologue. The molecular polarity and polarizability of series-1 for the same homologue leading to intermolecular end to end attractions as compared to series-X arising out of combined effects of molecular rigidity and flexibility as a consequence of favorable length to breadth ratio is relatively lower than a series-X and Y which facilitate nematic mesophase in absence of smectogenic character of lowest thermal stability. The increasing order of smectic or nematic thermal stabilities from series-1 to X to Y indicates the increasing proportions of internal thermal resistivity or internal energy stored ( $\Delta H$ ) due to mass and characteristics of a thermodynamic system of structurally similar but of differing molecular structure to some extent against externally exposed thermal vibrations under identical condition or intensity of heat which causes stabilization of mesophase to more or less extent of differing period of time or differing ranges of temperature as mesophase lengths from  $t_1$  °C to  $t_2$  °C. The early or late commencement of mesophase depends upon the extent of molecular noncoplanarity which decides suitable (not more or not less) magnitudes of intermolecular attractions to which victim molecules start to float with either statistically parallel orientational ordered molecular arrangement or with sliding layered molecule arrangement under floating condition which is independent of energy stored as  $\Delta H$ . Thus, mesophase commences earliest from  $C_1$  homologue in series-Y, then from  $C_3$  homologue of series-1 and the latest from  $C_6$  homologue of series X.

Hence, the variations in mesomorphic properties and the degree of mesomorphism for the same homologue from series to series or from homologue to homologue in the same series depended upon the varying proportions of suitable magnitudes of intermolecular anisotropic forces of effective cohesion and closeness as a consequence of effective dipole–dipole interactions, electronic interactions, dispersion forces, effective length to breadth ratio, the ratio of the molecular polarity to polarizability which results into favorable molecular rigidity and/or flexibility, needed for phenomena of mesomorphism.

## Conclusions

- Novel homologous series is nematogenic without exhibition of smectic property and of middle ordered melting type.
- Carboxylate ( $-\text{COO}-$ ) ester central group containing homologous series including, carbonyl group of chalcone directly linked to middle phenyl ring ( $-\text{Ph}-\text{CO}-\text{CH}=\text{CH}-$ ) are smectogenic, but, vinyl carboxylate ( $-\text{CH}=\text{CH}-\text{COO}-$ ) ester central group containing homologous series including, carbonyl group of chalcone linked to third phenyl ring ( $-\text{CH}=\text{CH}-\text{CO}-\text{Ph}$ ) are only nematogenic or partly nematogenic and partly smectogenic.
- The group efficiency order derived for smectic and nematic on the basis of (1) thermal stabilities (2) early commencement of mesophase and (3) total mesophase lengths are as under.

### (1) Smectic

Series-Y > Series-X = Series-1

### Nematic

Series-X > Series-1 > Series-Y

## (2) Smectic

Series-Y > Series-1 = Series-X

## Nematic

Series-1 > Series-X > Series-Y

## (3) Total mesophase lengths ( $S_m \pm N$ )

**Lower:** Series-1 > Series-X > Series-Y

**Upper:** Series-Y > Series-1 > Series-X

- Mesomorphism is very sensitive and susceptible to a molecular structure.
- Carboxylate or vinyl carboxylate group containing chalconyl derivatives are bioactive which may be exploited and useful to the pharmaceutical, agricultural or medicinal preparation and mesomorphic property may be exploited for thermographic LC devices to be operated at desired temperature.
- Present investigation very well supports and raises the credibility to the conclusions drawn earlier.

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