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## Synthetic pathway for a new calamitic series of liquid crystal: Comparison with corresponding linkage group

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#### ABSTRACT

Two homologous series based on three linking groups have been synthesized and characterized by elemental analyses and spectroscopic techniques such as Fourier transform infrared [FT-IR] and protonmagnetic resonance magnetic resonance [<sup>1</sup>H-NMR] spectroscopy. The mesomorphic properties of these compounds were observed by optical polarized light microscopy (POM) and differential scanning calorimetry (DSC). In this present investigation, we have synthesized two homologous series viz. 4-(3-oxo-3-(4-((-3-phenylallylidene) amino) phenyl) prop-1-en-1-yl) phenyl 4-n-alkoxy benzoate) (series-1) and 4-(3-oxo-3-(4-((-3-phenylallylidene) amino) phenyl) prop-1-en-1-yl) phenyl 3-(4-n-alkoxy phenyl) acrylate (series-2). Both of the series are differing with respect to the first linking group. All the homologous in the following series exhibits LC phase on heating as well as cooling condition except first two homologue (C<sub>1</sub>, C<sub>2</sub>) in series-1 and three homologue (C<sub>1</sub>, C<sub>2</sub>, C<sub>3</sub>) in series-2.

#### **GRAPHICAL ABSTRACT**



#### KEYWORDS

Calamitic; liquid crystals; nematic; rod type; smectic

### 1. Introduction

The arrangement of molecules in liquid crystals (LCs) takes place in a number of ways leading to various phases of different order and symmetry between crystalline solid and isotropic liquid [1–4]. Study on LC state is planned with a view to understand and establish the effect

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of molecular structure on LC properties [5] as a consequence of molecular rigidity and flexibility [6–8]. In order to avail novel LC materials to the various groups of researchers working with different aims, objects, and views with different angles to continue and extend their research. Liquid crystals can be divided into thermotropic, lyotropic and metallotropic phases. Thermotropic and lyotropic liquid crystals consist mostly of organic molecules although few minerals are also known [9–10]. Liquid crystals in the nematic group are most widely used in production of liquid crystal displays (LCD) mainly due to their unique physical property such as a wide temperature range. In addition, the nematic phase in which liquid crystal molecules are oriented on average along to a particular direction. By applying an electric or magnetic field, the orientation of the molecules can be derived in a probable approach [11–12]. Literature studies revealed that the schiff base has been widely employed as a linking group for the synthesis of different forms of liquid crystals [13–15].

Different chalcone derivatives have exhibited broad spectrum of anti-bacterial, antifungal, antiulcer, antimalarial, antitumor, anticancer, anti-inflammatory and antituberculor activity [16–20]. It is believed that the presence of  $\alpha$ ,  $\beta$ -unsaturated functional group in chalcone (-CH=CH-CO-) is responsible for the anti-microbial activity, which can be altered depending upon the type of substituent present on the aromatic rings [21-22]. To the best of our knowledge, liquid crystalline materials based on chalconyl central linking group are rarely reported in the literature. It has been observed that -CO-CH=CH- linkage is less conducive to mesomorphism as compared to -CH=N-, -COO-, -N=N-, -CH=CH-COO- linkages group because of the presence of non-linearity and angle strain arising from the presence of keto (-C=O) group [23]. Surprisingly, when -CO-CH=CH- linkage is linked with another linking group and it becomes conductive to exhibits mesophase. In the literature studies, there are several reports of mesogenic compounds having chalcone linkage. Vora et al. [24] reported a homologous series of polymers containing a chalcone linkage. Soon afterword Chudghar and his co-workers reported a homologous series containing on ester-chalcone linkages [25]. Yeap et al. have also synthesized mesomorphic compounds containing on ester-chalcone linkage [26]. Guan et al. reported phloroglucinol based star shaped symmetrical trimeric liquid crystalline compounds containing three rod like schiff base arms [27]. The mesomorphic properties of aromatic Schiff base esters arising from substituents varying in their polarities have been reported by Ha et al. [28]. Many mesogenic homologous series contain two central linkages, one of which may be ester and the other azomethine [29-33]. In 2007, Yellamaggad et al. have developed salicylaldimine based banana-shaped mesogens derived from laterally substituted resorcinol [34]. Recently, Doshi et al. have reported chalconyl ester and chalconyl vinyl ester linkage group inbuilt between three phenyl rings and studied the effect on mesomorphism by substituted groups at terminal and lateral side [35-39].

Thus, the main object of this present work to synthesized and studied the effect of chalconyl-ester and allylidene amino linkage group on mesomorphism. Many series of liquid crystalline compounds containing heterocyclic groups have been synthesized due to their usefulness in wide application such as electrical, optical, biological and medical field [40]. In our recent reports, we have developed some homologous series based on *Cis* and *Trans* cinnamate and benzoate linkage group [41–45]. Our research group also reported some linear and nonlinear homologous series which consisted three phenyl rings bonded through vinyl ester (–CH=CH–COO–) and ester (–COO–) linkage group connected with chalconyl central group having alkoxy terminal group at left end and different terminal group or lateral substitution on right side [46–48]. We have also reported the single chalconyl linkage group between two phenyl rings exhibited monotropy or enantiotropy mesophase with low

thermal stability [49–51]. In continuation of our efforts to synthesized newly liquid crystalline materials, we have introduce some new calamitic liquid crystal based on ester (–COO–) or vinyl ester (–CH=CH–COO–) with chalcone (–CH=CH–CO–) and allylidene amino (–N=CH–CH=CH–) group. We have compared the thermal stability and mesophase properties of series-1 and series-2 as well as studied the effect of extra part of ethylene (–CH=CH–) on mesomorphism in series-2.

#### 2. Experimental

#### 2.1. General

For the synthesis of target compounds in homologous series-1 and -2, the following materials were used: 4-hydroxy benzoic acid, 4-hydroxy benzaldehyde, Alkyl halides (Lancaster, England), Anhydrous K<sub>2</sub>CO<sub>3</sub>, KOH, Malonic acid, 4-amino acetophenone, cinnamaldehyde were purchased from (SRL, Mumbai, India), DCC (Dicyclohexyl carbodimide) was purchased from Fluka Chemie (Switzerland), Dimethyl amino pyridine DMAP (4,4'-dimethylamino pyridine) was purchased from Finar chemicals (India), Dichloro methane, Ethanol, Pyridine, Acetone required for synthesis were used as received except solvents which were dried and distilled prior to use. The molecular structures of the target products and the intermediates were confirmed by spectroscopic analysis and elemental analysis. Representative homologous series were characterised by elemental analysis, Infrared spectroscopy, <sup>1</sup>H NMR spectra. IR spectra were recorded by Perkin-Elmer spectrum GX, <sup>1</sup>HNMR spectra were recorded on Bruker (400 MHz) using CDCl<sub>3</sub> as a solvent. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyser. Texture image of nematic phase were determined by using miscibility method. Transition temperature and LC properties (Textures) were determined using an optical polarising microscopy (POM) equipped with heating stage attached to the camera. Decomposition temperatures were determined using of Shimadzu (DSC) differential Scanning Calorimeter with a heating rate of 5°C to 10°C min<sup>-1</sup>. Thermodynamic quantities enthalpy ( $\Delta$ H) and entropy ( $\Delta$ S= $\Delta$ H/T) are mentioned in Table-4.

#### 2.2. Synthesis of Series-1 and Series-2 compounds

**2.2.1.** Synthesis of 4-n-alkoxy benzoic acid (1a) and Trans 4-n-alkoxy cinnamic acid (2b) 4-n-alkoxy benzoic acid and 4-n-alkoxy cinnamic acid were prepared by method reported by Dave and Vora [52].

#### 2.2.2. Synthesis of 4-n-alkoxy benzaldehyde (2a)

4-*n*-Alkoxybenzaldehydes were synthesized by refluxing 4-hydroxybenzaldehyde (1 equiv.) with corresponding *n*-alkyl bromides (1 equiv.) in the presence of anhydrous  $K_2CO_3$  (1 equiv.) and dry acetone as a solvent [53].

#### 2.2.3. Synthesis of 1-(4-((3-phenyl allylidene) amino) phenyl) ethanone (3a)

Schiff base of 4-amino acetophenone and cinnamaldehyde was prepared by reported method [54]. (Brown colour, Yield: 74%), IR (KBr):  $\nu_{max}/cm^{-1}$  3077 (C–H str.), 1597 (CH=N), 961 (CH=CH–), 1650 (C=O str.). elemental analysis: calculated for C<sub>17</sub>H<sub>15</sub>ON: C, 81.92%; H, 6.02%; O, 6.42%; N, 5.62%; found: C, 81.18%; H, 5.94%; O, 6.37%; N, 5.58%.

# 2.2.4. Synthesis of 3-(4-hydroxy phenyl)-1-4-((3-phenyl allylidene) amino) phenyl) prop-2-en-1-one (4a)

Chalcone (4a) was prepared by usual reported method [55]. (brownish yellow, Yield: 67%), IR (KBr):  $v_{max}/cm^{-1}$  3460 (-OH str.-bonded), 3079–3020 (-C–H str. aliphatic alkyl group), 2932, 1598 (CH=N), 1667 (-C=O, group), 1584–1610 (-C=C–) aromatic, 1280 (-O–CH<sub>2</sub>– ether linkage), 962 (Trans alkene). elemental analysis: calculated for C<sub>24</sub>H<sub>19</sub>O<sub>2</sub>N: C, 81.58%; H, 5.38%; O, 9.06%; N, 3.96%; found: C, 81.48%; H, 5.28%; O, 8.77%; N, 3.89%.

#### 2.2.5. General procedure for the synthesis of (Series-1)

The compound has been prepared by esterification of the appropriate 4-*n*-alkoxy benzoic acid (1a) (2.02 mmol) and chalcone (4a) (0.246g, 2.02 mmol), dicyclohexyl carbodiimide (DCC) (0.457g, 2.22 mmol) and dimethylaminopyridine (DMAP) in catalytic amount (0.002g, 0.2 mmol) in dry  $CH_2Cl_2$  (DCM) (30 mL) was stirred at room temperature for 48h. The slightly white precipitate of DCU is obtained which was isolated by filtration and remove, while the filtrate was evaporated to dryness. The resultant crude residue was purified by column chromatography on silica gel eluting with methanol: chloroform as eluent (1:4) [56]. The synthesis route of derivatives for series-1 and series-2 were mention in scheme -1 and -2.

**C**<sub>1</sub> (series-1): IR (KBr):  $v_{max}/cm^{-1}$  2966 (C–H str. of alkane), 1650 (C=O str. of carbonyl carbon of chalconyl group), 1601 (C=C str. of alkene in chalcone), 1730 (–COO–group), 992 (C–H bending of alkene), 1170 (C–O str. of ether linkage), 1231 (C–O str. of carbonyl (>CO) group, 960 (*trans*, –CH=CH– of –CH=CH–CO–) group. <sup>1</sup>H-NMR: 3.81 (t, 3H, –CH<sub>3</sub> of polymethylene –OCH<sub>3</sub>), 7.81 & 7.52 (d, 2H, –CH=CH–CO–), 7.44 (d, 1H, –N=CH–CH=C–), 6.83 & 7.20 (d, 2H, –N=CH–CH=CH–), 8.07 & 7.10 (4H, first phenyl ring), 7.32 & 7.72 (4H, second phenyl ring), 7.87 & 7.38 (4H, third phenyl ring), 7.61, 7.40, 7.31 (5H, fourth phenyl ring). Elemental analysis: calculated for  $C_{32}H_{25}O_4N$ : C, 78.85%; H, 5.13%; O, 13.14%; N, 2.87%; found: C, 78.45%; H, 5.01%; O, 13.07%; N, 2.81%.

**C**<sub>2</sub> (series-1): IR (KBr):  $v_{max}/cm^{-1}$  2960 (C–H str. of alkane), 1660 (C=O str. of carbonyl carbon of chalconyl group), 1608 (C=C str. of alkene in chalcone), 1760 (–COO– group), 992 (C–H bending of alkene), 1180 (C–O str. of ether linkage), 1241 (C–O str. of carbonyl (>CO) group, 960 (*trans*, –CH=CH– of –CH=CH–CO–) group. <sup>1</sup>H-NMR: 1.32 (t, 3H, –CH<sub>3</sub> of polymethylene –OC<sub>2</sub>H<sub>5</sub>), 4.06 (t, 2H, –OCH<sub>2</sub>–CH<sub>2</sub>– of –OC<sub>2</sub>H<sub>5</sub>), 7.82 & 7.52 (d, 2H, –CH=CH–CO–), 7.46 (d, 1H, –N=CH–CH=C–), 6.83 & 7.21 (d, 2H, –N=CH–CH=CH–), 8.07 & 7.10 (4H, first phenyl ring), 7.32 & 7.71 (4H, second phenyl ring), 7.87 & 7.38 (4H, third phenyl ring), 7.61, 7.40, 7.32 (5H, fourth phenyl ring). Elemental analysis: calculated for C<sub>33</sub>H<sub>27</sub>O<sub>4</sub>N: C, 79.04%; H, 5.38%; O, 12.77%; N, 2.79%; found: C, 78.88%; H, 5.31%; O, 12.71%; N, 2.72%.

**C**<sub>4</sub> (series-1): IR (KBr):  $v_{max}/cm^{-1}$  2930 (C−H str. of alkane), 2820 (C−H str. of −(CH<sub>2</sub>−)n group of −OC<sub>4</sub>H<sub>9</sub> alkyl chain, 1660 (C=O str. of carbonyl carbon of chalconyl group), 1612 (C=C str. of alkene in chalcone), 1750 (−COO− group), 1520 (−C=C str. of aromatic ring), 984 (C−H bending of alkene), 1188 (C−O str. of ether linkage), 1240 (C−O str. of carbonyl (>CO) group, 962 (*trans*, −CH=CH− of −CH=CH−CO−) group, 781 polymethylene (−CH<sub>2</sub>−) of −OC<sub>4</sub>H<sub>9</sub>. <sup>1</sup>H-NMR: 0.92 (t, 3H, −CH<sub>3</sub> of polymethylene −OC<sub>4</sub>H<sub>9</sub>), 1.42−1.76 (m, 4H, −OC<sub>4</sub>H<sub>9</sub>), 4.06 (t, 2H, −OCH<sub>2</sub>−CH<sub>2</sub>− of −OC<sub>4</sub>H<sub>9</sub>), 7.87 & 7.51 (d, 2H, −CH=CH−CO−), 7.48 (d, 1H, −N=CH−CH=C−), 6.84 & 7.22 (d, 2H, −N=CH−CH=CH−), 8.06 & 7.12 (4H, first phenyl ring), 7.30 & 7.72 (4H, second phenyl ring), 7.86 & 7.38 (4H, third phenyl ring), 7.61, 7.40, 7.31 (5H, fourth phenyl ring). Elemental analysis: calculated for C<sub>35</sub>H<sub>31</sub>O<sub>4</sub>N: C, 79.39%; H, 5.86%; O, 12.09%; N, 2.64%; found: C, 79.31%; H, 5.78%; O, 11.97%; N, 2.58%.



i. R-Br, MeOH, KOH, Reflux, (R = CnH2n+1, n = 1 to 8, 10, 12, 14, 16, 18); ii. R-Br, Anhydrous K<sub>2</sub>CO<sub>3</sub>, dry Acetone, Reflux; iii. Malonic acid; piperidine, pyridine, 3 to 4 hours reflux; iv. ethanol, few drops of glacial acetic acid; v. KOH, ethanol, 48 hour stirring at room temperature.

**C**<sub>5</sub> (series-1): IR (KBr):  $v_{max}/cm^{-1}$  2940 (C−H str. of alkane), 2830 (C−H str. of −(CH<sub>2</sub>−)n group of −OC<sub>5</sub>H<sub>11</sub> alkyl chain, 1610 (C=O str. of carbonyl carbon of chalconyl group), 1602 (C=C str. of alkene in chalcone), 1730 (−COO− group), 1540 (−C=C str. of aromatic ring), 994 (C−H bending of alkene), 1188 (C−O str. of ether linkage), 1246 (C−O str. of carbonyl (>CO) group, 960 (*trans*, −CH=CH− of −CH=CH−CO−) group, 789 polymethylene (−CH<sub>2</sub>−) of −OC<sub>5</sub>H<sub>11</sub>. <sup>1</sup>H-NMR: 0.90 (t, 3H, −CH<sub>3</sub> of polymethylene −OC<sub>5</sub>H<sub>11</sub>), 1.31−1.76 (m, 6H, −OC<sub>5</sub>H<sub>11</sub>), 4.06 (t, 2H, −OCH<sub>2</sub>−CH<sub>2</sub>− of −OC<sub>5</sub>H<sub>11</sub>), 7.88 & 7.52 (d, 2H, −CH=CH−CO−), 7.48 (d, 1H, −N=CH−CH=C−), 6.84 & 7.22 (d, 2H, −N=CH−CH=CH−), 8.08 & 7.10 (4H, first phenyl ring), 7.31 & 7.74 (4H, second phenyl ring), 7.87 & 7.38 (4H, third phenyl ring), 7.61, 7.40, 7.32 (5H, fourth phenyl ring). Elemental analysis: calculated for C<sub>36</sub>H<sub>33</sub>O<sub>4</sub>N: C, 79.55%; H, 6.07%; O, 11.78%; N, 2.57%; found: C, 79.49%; H, 5.91%; O, 11.73%; N, 2.49%.

Scheme 1. Synthetic route of derivatives.

Step-4



v and vi. DCC, DMAP, DCM, 48 hour stirring.

Scheme 2. Synthetic route of series-1 and series-2.

#### 2.2.6. General procedure for the synthesis of (Series-2)

The compound has been prepared by esterification of the appropriate Trans 4-*n*-alkoxy cinnamic acid (A) (2.02 mmol) and chalcone (C) (0.246g, 2.02 mmol), dicyclohexylcarbodiimide (DCC) (0.457g, 2.22 mmol) and dimethylaminopyridine (DMAP) in catalytic amount (0.002g, 0.2 mmol) in dry  $CH_2Cl_2$  (DCM) (30 mL) was stirred at room temperature for 48h. The slightly white precipitate of DCU is obtained which was isolated by filtration and discarded, while the filtrate was evaporated to dryness. The resultant crude residue was purified by column chromatography on silica gel eluting methanol: chloroform (1:4) [56].

**C**<sub>1</sub> (series-2): IR (KBr):  $v_{max}/cm^{-1}$  2956 (C–H str. of alkane), 1660 (C=O str. of carbonyl carbon of chalconyl group), 1608 (C=C str. of alkene in chalcone), 1740 (–COO– group), 1510, 1543 (C=C str. of aromatic ring), 996 (C–H bending of alkene), 1178 (C–O str. of ether linkage), 1288, 1246 (C–O str. of carbonyl (>CO) group, 971 (*trans*, –CH=CH–) group. <sup>1</sup>H-NMR: 3.83 (t, 3H, –CH<sub>3</sub> of polymethylene –OCH<sub>3</sub>), 7.62 & 6.32 (d, 2H, –CH=CH–COO–), 7.46 (d, 1H, –N=CH–CH=C–), 6.82 & 7.21 (d, 2H, –N=CH–CH=CH–), 6.86 & 7.61 (4H, first phenyl ring), 7.20 & 7.71 (4H, second phenyl ring), 7.87 & 7.39 (4H, third phenyl ring), 7.57, 7.32, 7.31 (5H, fourth phenyl ring). Elemental analysis: calculated for  $C_{34}H_{27}O_4N$ : C, 79.53%; H, 5.26%; O, 12.47%; N, 2.72%; found: C, 79.48%; H, 5.21%; O, 12.37%; N, 2.68%.

C<sub>2</sub> (series-2): IR (KBr):  $v_{max}/cm^{-1}$  2952 (C–H str. of alkane), 1660 (C=O str. of carbonyl carbon of chalconyl group), 1604 (C=C str. of alkene in chalcone), 1750 (–COO– group), 1510, 1541 (C=C str. of aromatic ring), 997 (C–H bending of alkene), 1172 (C–O str. of ether linkage), 1286, 1243 (C–O str. of carbonyl (>CO) group, 972 (*trans*, –CH=CH–) group. <sup>1</sup>H-NMR: 1.32 (t, 3H, –CH<sub>3</sub> of polymethylene –OC<sub>2</sub>H<sub>5</sub>), 4.06 (t, 2H, –OCH<sub>2</sub>–CH<sub>2</sub>– of –OC<sub>2</sub>H<sub>5</sub>),

7.61 & 6.31 (d, 2H, -CH=CH-COO-), 7.47 (d, 1H, -N=CH-CH=C-), 6.83 & 7.20 (d, 2H, -N=CH-CH=CH-), 6.85 & 7.61 (4H, first phenyl ring), 7.21 & 7.70 (4H, second phenyl ring), 7.86 & 7.39 (4H, third phenyl ring), 7.57, 7.32, 7.31 (5H, fourth phenyl ring). Elemental analysis: calculated for  $C_{35}H_{29}O_4N$ : C, 79.69%; H, 5.50%; O, 12.14%; N, 2.65%; found: C, 79.61%; H, 5.42%; O, 12.07%; N, 2.59%.

**C**<sub>4</sub> (series-2): IR (KBr):  $v_{max}/cm^{-1}$  2951 (C−H str. of alkane), 1650 (C=O str. of carbonyl carbon of chalconyl group), 1608 (C=C str. of alkene in chalcone), 1760 (−COO− group), 1510, 1543 (−C=C str. of aromatic ring), 996 (C−H bending of alkene), 1178 (C−O str. of ether linkage), 1288, 1246 (C−O str. of carbonyl (>CO) group, 972 (*trans*, −CH=CH−) group, 770 polymethylene (−CH<sub>2</sub>−) of −OC<sub>4</sub>H<sub>9</sub>. <sup>1</sup>H-NMR: 0.90 (t, 3H, −CH<sub>3</sub> of polymethylene  $-OC_4H_9$ ), 1.45–1.76 (m, 4H,  $-OC_4H_9$ ), 4.06 (t, 2H,  $-OCH_2-CH_2-$  of  $-OC_4H_9$ ), 7.61 & 6.31 (d, 2H, -CH=CH-COO-), 7.48 (d, 1H, -N=CH-CH=C-), 6.84 & 7.21 (d, 2H, -N=CH-CH=CH-), 6.85 & 7.61 (4H, first phenyl ring), 7.20 & 7.70 (4H, second phenyl ring), 7.86 & 7.39 (4H, third phenyl ring), 7.56, 7.32, 7.31 (5H, fourth phenyl ring). Elemental analysis: calculated for C<sub>37</sub>H<sub>33</sub>O<sub>4</sub>N: C, 80.0%; H, 5.94%; O, 11.53%; N, 2.52%; found: C, 79.92%; H, 5.89%; O, 11.49%; N, 2.47%.

**C**<sub>12</sub> (series-2): IR (KBr):  $v_{max}/cm^{-1}$  2954 (C−H str. of alkane), 2842 (C−H str. of −(CH<sub>2</sub>−)n group of −OC<sub>12</sub>H<sub>25</sub> alkyl chain, 1640 (C=O str. of carbonyl carbon of chalconyl group), 1604 (C=C str. of alkene in chalcone), 1730 (−COO− group), 1510, 1543 (C=C str. of aromatic ring), 996 (C−H bending of alkene), 1178 (C−O str. of ether linkage), 1288, 1246 (C−O str. of carbonyl (>CO) group, 972 (*trans*, −CH=CH−) group, 770 polymethylene (−CH<sub>2</sub>−) of −OC<sub>12</sub>H<sub>25</sub>. <sup>1</sup>H-NMR: 0.90 (t, 3H, −CH<sub>3</sub> of polymethylene −OC<sub>12</sub>H<sub>25</sub>), 1.29−1.76 (m, 20H, −OC<sub>12</sub>H<sub>25</sub>), 4.06 (t, 2H, −OCH<sub>2</sub>−CH<sub>2</sub>− of −OC<sub>12</sub>H<sub>25</sub>), 7.63 & 6.31 (d, 2H, −CH=CH−COO−), 7.48 (d, 1H, −N=CH−CH=C−), 6.84 & 7.21 (d, 2H, −N=CH−CH=CH−), 6.86 & 7.61 (4H, first phenyl ring), 7.21 & 7.70 (4H, second phenyl ring), 7.87 & 7.39 (4H, third phenyl ring), 7.57, 7.32, 7.31 (5H, fourth phenyl ring). Elemental analysis: calculated for C<sub>45</sub>H<sub>49</sub>O<sub>4</sub>N: C, 80.95%; H, 7.34%; O, 9.59%; N, 2.09%; found: C, 80.89%; H, 7.18%; O, 9.47%; N, 2.01%.

#### 3. Result and discussion

In the present study, we have synthesized 13 homologues of two series such as 4-(3-oxo-3-(4-((3-phenylallylidene) amino) phenyl) prop-1-en-1-yl) phenyl 4-alkoxy benzoate (series-1) and 4-(3-oxo-3-(4-((3-phenylallylidene) amino) phenyl) prop-1-en-1-yl) phenyl-3-(4-alkoxy phenyl) acrylate (series-2) and their mesomorphic properties were studied by using differential scanning calorimetry (DSC) and polarizing optical microscopy (POM).

#### 3.1. POM investigation

We have synthesized and studied the effect of a linking group as well as the effect of varying aliphatic alkyl chain length from small spacer ( $-OCH_3$ ) to the high spacer ( $-OC_{18}H_{37}$ ) with presence of four benzene rings as a central rigid core in present homologous series-1 and series-2. In the present investigation, we have synthesized thirteen homologous ( $C_1$ to  $C_8$ ,  $C_{12}$ ,  $C_{14}$ ,  $C_{16}$ ,  $C_{18}$ ) in both series. All the compounds of both the series exhibit only nematic mesophase on heating as well as cooling condition. In series-1, mesophase commences from  $C_3$  homologue while in series-2, it commences from  $C_4$  homologue respectively. Texture images of nematic phase were determined by using miscibility method (Table–1). The transition temperature of series-1 and series-2 are shown in Table–2 and Table–3.

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Sr. No.	Homologue	Texture (Series-1)	Texture (Series-2)
1	C <sub>10</sub>	Threaded	Threaded
2	C <sub>12</sub>	Rod-like	Schlieren
3	C <sub>14</sub>	Rod-like	Threaded
4	C <sub>16</sub>	Schlieren	Schlieren

Table 1. Texture of nematic phase of  $C_{10}$ ,  $C_{12}$ ,  $C_{14}$ ,  $C_{16}$  by miscibility method.

#### Table 2. Transition temperature in°C by POM (Series-1).

		Transition temperatures in°C						
Sr. No.	R = n-alkyl group	Cr	Smectic		Nematic		lsotropic	
1	C,		_	_	_	_	188.0	
2	C,	•	_	_	_	_	186.0	
3	C,	•	_	_	152.0	_	182.0	
4	$C_{A}^{J}$		—	_	148.0	_	177.0	
5	$C_5$		—	_	140.0		170.0	
6	C <sub>6</sub>		—		146.0		171.0	
7	$C_7$		—		141.0		172.0	
8	C <sub>8</sub>		—		136.0		168.0	
9	C <sub>10</sub>		—		131.0		165.0	
10	C <sub>12</sub>		—		128.0		163.0	
11	C <sup>12</sup>		—		118.0		148.0	
12	C <sub>16</sub>		—	-	116.0	•	142.0	
13	C <sub>18</sub>	•	—	•	108.0	•	138.0	•

#### Table 3. Transition temperature in°C by POM (Series-2).

		Transition temperatures in°C						
Sr. No.	R = n-alkyl group	Cr Smectic		Nematic		lsotropic		
1	C,		_	_	_	_	218.0	
2	C,		_	_	_	_	212.0	
3	C,		—	_	_	—	208.0	
4	$C_{4}$		—	_	163.0	—	204.0	
5	C <sub>5</sub>		—	_	161.0		201.0	
6	C <sub>6</sub>		—		154.0		197.0	
7	C <sub>7</sub>		—		152.0		194.0	
8	C <sub>8</sub>		—		143.0		189.0	
9	C <sub>10</sub>		—		138.0		186.0	
10	C <sub>12</sub>		—		132.0		183.0	
11	C <sub>14</sub>		—		120.0		182.0	
12	C <sub>16</sub>		—		112.0		172.0	
13	C <sub>18</sub>	•	—	•	110.0	•	161.0	•

Homologous  $C_1$  and  $C_2$  from series-1 and  $C_1$  to  $C_3$  from series-2 directly passing through isotropic condition without display mesophase due to the presence of short alkyl spacer on left side chain causes high crystallinity tendency. The nonmesomorphicity of  $C_1$  to  $C_2$  from

Table 4.	Relative	thermal	stability	/ in°C.
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Series	Series-1	Series-2		
N-I Commencement of nematic phase Total temperature range of mesophase	163.27°C $C_3$ 30.0 to 35.0 $C_3$ $C_{12}$	186.90°C C <sub>4</sub> 41.0 to 60.0 C <sub>5</sub> C <sub>16</sub>		



Figure 1. Phase diagram of series-1.

series-1 and  $C_1$  to  $C_3$  from series-2 is mainly attributed to the low magnitudes of intermolecular dispersion forces and low magnitudes of dipole-dipole interactions leading to its high crystallising tendency which causes abrupt breaking of crystal lattices due to unsuitable and unfavourable magnitudes of anisotropic forces of intermolecular attractions. Thus, an irregular breaking of crystal lattices and suddenly takes place at relatively higher temperature. Therefore, the crystalline solid state directly converted into isotropic phase without showing LC phase. The phase diagram of both the series are given in Figure-1 and Figure-2.

A phase diagram (Figure-1) of a newly series-1 consisted of Cr–N/I and N–I transition curves indicating the phase behaviours of series-1. The Cr–N/I transition curve initially falling up to  $C_5$  homologue and then continued with overall descending tendency up to last  $C_{18}$  homologue. The Odd-even effect is present only in  $C_5$  to  $C_7$  homologues of series-1. It can be noted that the Cr–N/I transition curve decrease with increment of alkoxy group in



Figure 2. Phase diagram of series-2.



**Figure 3.** Photomicrograph of optical texture obtained for the nematic image of  $C_8$  at 136°C (series-1); (b) Photomicrograph of optical texture obtained for the nematic image of  $C_{10}$  at 138°C (series-2).

the side chain except  $C_6$  homologue. Whereas in N–I transition curve start decrease at  $C_5$  homologue and then rises at  $C_7$  homologue due to the presence of odd-even effect and then continued decrease up to last  $C_{18}$  homologue of series-1. The plot of transition temperature against the number of carbon atoms in the alkoxy group (Figure–2) shows a smooth falling tendency for Cr–N/I and N–I transition curve throughout the series. No odd-even effect is observed in Cr–N/I and N–I transition curve. It has observed that sematic mesophase is not found in both the series. The exhibition of nematic phase in  $C_3$  to  $C_{18}$  (series-1) and  $C_4$  to  $C_{18}$  (series-2) is attributed to the suitable magnitudes of end to end intermolecular anisotropic forces of attractions and closeness as a consequence of favourable molecular rigidity, linearity due to the presence linking group and flexibility caused by the presence of left side chain generate permanent dipole moment across the long molecular axis, suitable magnitudes of dispersion forces and dipole-dipole interactions, molecular polarizability and polarity which facilitate the molecules under microscopic study to float on the surface with statically parallel orientational order within certain range of temperature as nematic phase [57].

The solid samples held between an untreated glass slide and a coverslip, which were heated to their isotropic phase and cooled slowly. As shown in Figure 3a, a schlieren type nematic texture pattern of compound  $C_8$  (series-1) seen at 136°C, while in compound  $C_{10}$  (series-2) schlieren type nematic pattern seen at 138°C on cooling condition.

The changing trend in mesomorphic behaviours of series-1 is compared with structurally similar analogous series-2, as mention in Figure-4. Both the series are identical with respect to two linking group and four phenyl ring. The first linkage group in both the series are different. Presence of vinyl ester (-CH=CH-COO-) group in series-2 increases the molecular length as compare to ester (-COO-) group of series-1. Thus, changing in molecular length, permanent dipole moment across the long molecular axis, molecular rigidity or flexibility due to presence of side alkoxy tail group, which may vary from changing to lower member



Figure 4. Structurally similar analogous series.



Figure 5. Space filling diagram of (a) series-1; (b) series-2.

to higher member altering the magnitudes of mesomorphism. The presence of ester linkage group between two phenyl rings plays a major role in the emergence of nematic mesophase. It can be noted that the ester group increases the mobility and flexibility of particular parts of liquid crystal molecule and hence affects the type of mesophase and its mesophase length. It is observed that the presence of vinyl ester (-CH=CH-COO-) part of series-2 increases the mesophase thermal stability as compare to ester (-COO-) part of series-1.

The space-filling diagram is mention in Figure 5, which indicate energy minimise in both series-1 and series-2 respectively. The molecular length of series-2 is higher than series-1 due to the presence of ethylene (-CH=CH-) group in cinnamate linkage. Therefore they differ with respect to each other with combined effects of molecular rigidity and flexibility, including intermolecular distance and molecular polarizability, which operates LC behaviours of series. Addition of double bond (-CH=CH-) in the system increases the polarizability and length of the molecule which was disclosed by the Gray et al. [58].

#### 3.2. Thermal stability and temperature range of mesophase

Table 4 represents some thermometric data of series-1 and series-2 in a comparative manner. Thermal stability of series-1 is lesser than series-2, while nematic mesophase commences early in series-1 as compare to series-2. Temperature range of mesophase is in increasing order of lower to a higher homologue of series-1 and series-2. The early or late commencement of mesophase depends upon the extent of molecular non-co-planarity caused by central linkage group and their magnitudes of polarity as well as polarizability.

#### 3.3. Propose mechanism

Figure–6 indicates the proposed mechanism of arrangement of molecules to display nematic phase in both the series-1 and series-2, which contains three linkage group and left side alkoxy tail group. Thus, on heating condition, the molecules are randomly oriented in all possible directions with the high order of disorder or high entropy in a reversible manner.

#### 3.4. DSC analysis

The thermal behaviour of newly synthesized homologues of series-1 and series-2 were examined by using DSC measurement. In Figure–7, homologue C<sub>7</sub> shows one endothermic peak at



Figure 6. Propose model of molecular arrangement of nematic phase.

146.2°C in the heating cycle, which indicates the presence of nematic phase. That was further confirmed by POM analysis. While on cooling condition, the exothermic peak observed at 142.1°C. Compound  $C_{18}$  shows first endothermic peak at 101.2°C during the heating cycle, while in cooling cycle, it appears at 108.2°C, which was further confirmed by POM analysis which indicates the nematic mesophase appeared in an enantiotropically manner. Compound  $C_{12}$  exhibits an endothermic peak at 126.1°C on heating cycle which reveals with POM study. On the cooling cycle, exothermic peak observed at 125.2°C. Compound  $C_6$  shows endothermic peak at 156.2°C that indicates the presence of crystal to the nematic phase transition,



**Figure 7.** DSC thermograms of compound (a)  $C_7$  (series-2); (b)  $C_{18}$  (series-1); (c)  $C_{12}$  (series-1); (d)  $C_6$  (series-2).

Homologues or Comp.	Transition	Heating scan(°C)	Cooling scan(°C)	$\Delta$ H (-Jg <sup>-1</sup> )	$\Delta$ H (Jg <sup>-1</sup> )	$\Delta$ S(J g <sup>-1</sup> k <sup>-1</sup> )	$\Delta S(J g^{-1}k^{-1})$
C <sub>7</sub>	Cr–N	146.2	142.1	5.34	3.18	0.0127	0.0076
(series-2)	N-I	>190	_	_		_	_
C <sub>18</sub>	Cr–N	101.2	108.4	4.28	2.91	0.0114	0.0076
(series-1)	N-I	>130	_	_		_	_
C <sub>12</sub>	Cr–N	126.1	125.3	7.84	6.40	0.0196	0.0160
(series-1)	N-I	>160	_	_	_	_	_
C <sub>6</sub>	Cr–N	156.2	154.2	6.45	3.35	0.0150	0.0078
(series-2)	N–I	>195	—	—	_	—	—

**Table 5.** Transition temperature (°C) and enthalpy (J  $g^{-1}$ ) and entropy change (J  $g^{-1}k^{-1}$ ) by DSC measurement.

it traces again on cooling condition, and the single peak was traced at 154.3°C, which was further confirmed by POM analysis.

The phase transition temperatures and the value of enthalpy and entropy were measured by carrying out DSC thermal analysis. The phase transition temperatures, enthalpy and entropy values for the present synthesized series-1 and series-2 are summarized in Table–5. Molecules of every homologue randomly oriented in all possible directions with a high order of disorder or entropy ( $\Delta S = \Delta H/T$ ) beyond isotropic temperature and the enthalpy value ( $\Delta H$ ). But, at cooled condition, the same from and below isotropic temperature, the mesophase is persisted to appear reversibly at the lower temperature at observed during heating condition. The mesophase obtained in this present series is enantiotropically nematogenic.

#### 4. Conclusion

A two new homologous series of ester (series-1) and vinyl ester (series-2) containing chalcone and allylidene amino derivatives having terminal left alkoxy chains (n = 1 to 8, 10, 12, 14, 16, 18) has been synthesized. The lower member C<sub>1</sub> to C<sub>2</sub> (series-1) and C<sub>1</sub> to C<sub>3</sub> (series-2) shows nonmesomorphic behavior while higher members of both the series exhibited enantiotropic nematic mesophase transition with good thermal stabilities.

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