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## A New Series of Triazine-core Based Mesogenic Derivatives: Synthesis, Characterization and Mesomorphic Study

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### ABSTRACT

The synthesis, characterization and mesomorphic properties of two new series of triazine-core based liquid crystals have been investigated. The amino triazine derivatives were characterized by elemental analysis, Fourier transforms infrared (FTIR), <sup>1</sup>HNMR and mass spectroscopy. The liquid crystalline properties of these compounds were examined by differential scanning calorimetry (DSC) and polarizing optical microscopy (POM). DSC and POM confirmed nematic (N) and columnar mesophase textures of the materials. The formation of mesomorphic properties was found to be dependent on the number of methylene unit in alkoxy side chains.

### KEYWORDS

Liquid crystals; melamine; malononitrile; heterocyclic; mesophase

## Introduction

The incorporation of heteroatoms such as; nitrogen, oxygen or sulfur into the organic core may cause alteration to the physical and chemical properties of the entire system [1]. The physical and chemical behavior of these compounds will depend on type and number of hetero atoms and ring size that formed [2]. The design and fabrication of heterocyclic-based organic materials is a key factor in the development of new materials with different applications [3], these include; display devices, thermometer, light emitting diodes, photoconductors and semiconductor materials [4–6].

Several interesting applications of these compounds are influenced by their liquid crystalline behavior. Based on this fact, a new two series of five-membered heterocyclic compounds have been designed and prepared in order to improve their liquid crystal behavior [7]. There is a relationship between the molecular structure and mesomorphic properties of molecules that contain heterocyclic compounds [8–10]. It is well documented that the presence of the heteroatom in the cyclic system will have an effect on the molecular dipole as well as dielectric anisotropy of the whole molecules [11].

There are a number of synthesized compounds which includes heterocyclic rings and display the liquid crystalline behavior is reported [12–16]. Depending on the nature of the heterocyclic-segment, a range of pyridyl, piperidine and thiophene derivatives were prepared [17] and compared with that derived from benzene and cyclohexane core, the

introduction of the cyano group into the benzene segment of the piperidine compounds exhibit a large positive dielectric anisotropy. More, the synthesis of heterocyclic units that incorporate two different hetero-atoms that influenced the liquid crystal behavior of the examined compounds are also reported. They aimed to investigate the synergic effect of introducing two different hetero atoms into the system. Therefore, the preparation of heterocyclic-segment that includes two hetero atoms should have an impact on the liquid crystalline behavior.

Limited number of mesogenic 1,3,5- and 1,2,4-triazines derivatives have been reported [18–25]. For example, the preparation of a series of 3-[4-(4'-alkoxybenzoyloxybenzylidene)amino]-1,2,4-triazines, where  $m = 6-14$  have been reported [18]. The aim was to investigate the influence of the alkoxy chain length on the mesomorphic behaviour of these compounds. A Series of novel 1,3,5-triazinebased triphenylene monomer, dimer, and trimer, which were bridged by a novel large  $\pi$ - $\pi$  conjugated polyaromatic core were designed, synthesized and their mesomorphic properties were studied [19]. Two series of discotics derived from unconventional triazine-based dendrimers with C2 symmetry and C3 symmetry have been synthesized and their thermal and photophysical properties are investigated by Lai et al., [20]. A novel columnar liquid crystals whose molecular structures consist of a C3 star-shaped 1,3,5-triazine unit as a central core, and three pendant 2-phenyl-5-(di-, and/or tri-n-alkoxyphenyl)-1,3,4-thiadiazole arms, containing ether connecting groups was reported by Olatea et al., [21] and Jiaoa et al., [22] report the synthesis and characterization of a series of novel C3h-symmetric 2,4,6-tris(2-hydroxyphenyl)-1,3,5-triazine (THPT) derivatives. These compounds possess interesting intramolecular hydrogen bonding pattern, which lead to the planarization between the triazine ring and the pendant aryl groups.

Recently, the two novel triphenylene-Bodipy derivatives with one or two triphenylene units based on 1,3,5-triazine core were designed and synthesized by introducing Bodipy unit and triphenylene units sequentially onto cyanuric chloride [23]. Reith et al., [24] reported the synthesis of phenyl-substituted tris[1, 2, 4]triazolo-[1, 3, 5]triazine, using Huisgen reaction of phenyltetrazole and cyanuric chloride, represents an excellent core for discotic liquid crystals. Nine derivatives of 1,3,5-triazines with linear, branched, and chiral ester chains were synthesized by Taing et al., [25] and their mesomorphism was studied by polarized optical microscopy and differential scanning calorimetry.

In the present study, the synthesis of a new two homologous series of triazine-core compounds that functionalized with 4,5-dihydro-1H-pyrrole and thiazolidin-4-one are reported. Further, the relationship between the lengths of the alkyl chain and the liquid crystal behaviors are also investigated.

## Experimental

### Materials

Chemicals were purchased from Fluka and Aldrich-Sigma and used without further purification. Solvents used in the synthesis were distilled from the proper drying compounds prior to their use.

## Measurements

Uncorrected melting points were determined by using hot-stage Gallen Kamp melting point apparatus. FTIR spectra were recorded as KBr disk by a Shimadzu (IR Affinity-1) FTIR spectrophotometer. Elemental microanalysis (C.H.N) were determined on a Euro EA 3000/Italy analyzer. The  $^1\text{H-NMR}$  spectra were acquired in  $\text{DMSO-d}_6$  solution by a Bruker 400 MHz spectrometer with tetramethylsilane (TMS) as an internal reference. Mass spectra of compounds were measured by Electron Impact (EI) 70eV mass using a MS Model: 5975C VL MSD with Tripe-Axis Detector spectrometer. The textures of the mesophases were studied with a POM model Leica DM2500 M. The transition temperatures and enthalpies were measured using DSC (STA PT-1000 LINSIS) adopting a scanning rate of  $10\text{ }^\circ\text{C}/\text{min}$  under nitrogen atmosphere. Temperature and heat flow calibrated with standard indium of purity  $<99.99\%$ .

## Synthesis

The new compounds were synthesized according to the synthetic route outlined in Scheme 1.

### *N, N',N''-(1,3,5-triazine-2,4,6-triyl)tris(2-chloroacetamide) [I]*

To a mixture of melamine (0.13 g, 1 mmol) and triethylamine (0.5 mL) in *N,N*-dimethylformamide (DMF) (3 mL) was added dropwise chloroacetylchloride (0.34 g, 3 mmol). The reaction mixture was stirred for 3 h and then the resulting mixture was poured into iced water. The product was extracted with ethylacetate (2 x 10) and the combined organic layers were dried over  $\text{MgSO}_4$ . The solvent was evaporated under reduced pressure to yield compound [I].

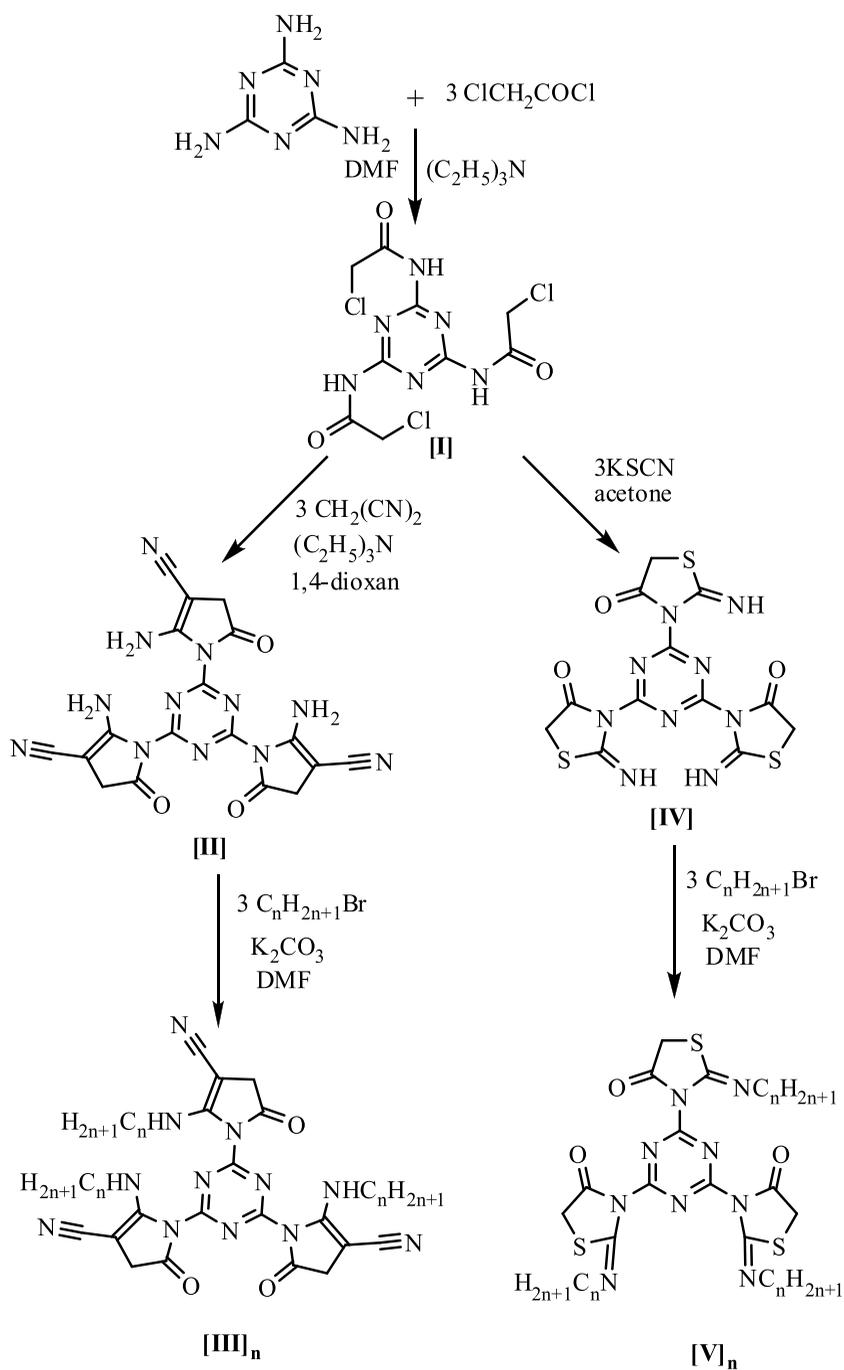
## Analytical data

### *N, N',N''-(1,3,5-triazine-2,4,6-triyl)tris(2-chloroacetamide) [I]*

Yield: 85%; m.p.:  $> 200\text{ }^\circ\text{C}$  dec; FTIR ( $\nu/\text{cm}^{-1}$ ): 3383 (NH), 1678 (C=O), 671 (C-Cl). Anal. Calcd. for  $\text{C}_9\text{H}_9\text{Cl}_3\text{N}_6\text{O}_3$ : C, 30.50; H, 2.54; N, 23.72. Found: C, 30.58; H, 2.70; N, 23.42.

### *1,1',1''-(1,3,5-triazine-2,4,6-triyl)tris(2-amino-5-oxo-4,5-dihydro-1H-pyrrole-3-carbonitrile) [II]*

A solution of malononitrile (0.20 g, 3 mmol) in 5 mL dioxane was added dropwise with stirring to a mixture of compound [I] (0.36 g, 1 mmol) and triethylamine (0.5 mL) in dioxane (30 mL). The reaction mixture was heated at reflux for 6 h and then cooled at room temperature. A solid that formed was filtered off and recrystallized from ethanol to give the required compound.



**Scheme 1.** The synthetic route of compounds [III]<sub>n</sub> and [V]<sub>n</sub>.

## Analytical data

### ***1,1',1''-(1,3,5-triazine-2,4,6-triyl)tris(2-amino-5-oxo-4,5-dihydro-1H-pyrrole-3-carbonitrile) [II]***

Brown powder; yield: 88%; m.p.: 230 °C. FTIR ( $\nu/\text{cm}^{-1}$ ): 3404-3244 (NH<sub>2</sub>), 2212 (C≡N), 1714 (C=O); <sup>1</sup>HNMR (400MHz, DMSO-d<sub>6</sub>)  $\delta$  (ppm): 7.30 (s, 6H, 3NH<sub>2</sub>), 4.11 (s, 6H, 3CH<sub>2</sub> in cyclic); Anal. Calcd. for C<sub>18</sub>H<sub>12</sub>N<sub>12</sub>O<sub>3</sub>: C,48.64; H,2.70; N,37.83. Found: C, 48.70; H, 2.45; N, 38.04.

### ***General procedure for the synthesis of compounds [III]<sub>n</sub>***

In a two-necked round bottom flask, a solution of compound [II] (4.44 g, 10 mmol) was dissolved in 15 mL of (DMF) was added dropwise a mixture of anhydrous potassium carbonate (4.14 g, 30 mmol) and n-alkylbromide (30 mmol). The stirred mixture was heated at reflux for 4 h and then allowed to cool down at room temperature. A cold water (400 mL) was poured into the reaction mixture and the solution was kept overnight at 5 °C. A solid was collected by filtration which allowed to dry in air to give [III]<sub>n</sub>.

n	3	4	5	6	7	8
Yield (%)	79	87	77	72	75	81

## Analytical data

### ***1,1',1''-(1,3,5-triazine-2,4,6-triyl)tris(5-oxo-2-(butylamino)-4,5-dihydro-1H-pyrrole-3-carbonitrile) [III]<sub>4</sub>***

Brown powder; yield: 87%; m.p.: 189 °C; FTIR ( $\nu/\text{cm}^{-1}$ ): 3402 (NH), 2927-2866 (C-H aliph.), 2212 (C≡N), 1728 (C=O); <sup>1</sup>HNMR (400MHz, DMSO-d<sub>6</sub>)  $\delta$  (ppm): 8.32 (s, 3H, 3NH), 3.31 (s, 6H, 3CH<sub>2</sub>), 3.07 (t, 6H, 3NCH<sub>2</sub>-), 2.33-2.08 (m, 6H, 3(-CH<sub>2</sub>)), 1.37-1.23 (m, 6H, 3(-CH<sub>2</sub>)), 0.95(t, 9H, 3CH<sub>3</sub>).

### ***1,1',1''-(1,3,5-triazine-2,4,6-triyl)tris(5-oxo-2-(hexylamino)-4,5-dihydro-1H-pyrrole-3-carbonitrile) [III]<sub>6</sub>***

Brown powder; yield: 72%, m.p.: 128 °C. FTIR ( $\nu/\text{cm}^{-1}$ ): 3334 (NH), 2954-2858 (C-H aliph.), 2206 (C≡N), 1716 (C=O); <sup>1</sup>HNMR (400MHz, DMSO-d<sub>6</sub>)  $\delta$  (ppm): 8.45 (s, 3H, 3 NH), 3.24 (s, 6H, 3CH<sub>2</sub>), 3.06 (t, 6H, 3 NCH<sub>2</sub>-), 2.80-2.75 (m, 6H, 3(-CH<sub>2</sub>)), 1.52-1.43(m, 6H, 3 (-CH<sub>2</sub>)), 1.26-1.06 (m, 12H, 3 (-CH<sub>2</sub>)<sub>2</sub>), 0.70 (t, 9H, 3CH<sub>3</sub>).

### ***3,3',3''-(1,3,5-triazine-2,4,6-triyl)tris(2-iminothiazolidin-4-one) [IV]***

A mixture of compound [I] (1.06 g, 3 mmol) and potassium thiocyanate (0.87 g, 9 mmol) in dry acetone (10 mL) was heated at reflux for 3 h. The brown solid that formed was filtered off and then recrystallized from acetone to give the required compound.

## Analytical data

### ***3,3',3''-(1,3,5-triazine-2,4,6-triyl)tris(2-iminothiazolidin-4-one) [IV]***

Pale brown powder; yield: 89%; m.p.: 190 °C dec. FTIR ( $\nu/\text{cm}^{-1}$ ): 3361 (NH), 1726 (C=O), 1610 (C=N), 769 (C-S);  $^1\text{H NMR}$  (400MHz, DMSO- $d_6$ )  $\delta$  (ppm): 11.15 (s, 3H, 3 NH), 4.44 (s, 6H, 3 CH<sub>2</sub> in cyclic); MS: m/z 421 (M-1)<sup>+</sup>; Anal. Calcd. for C<sub>12</sub>H<sub>9</sub>N<sub>9</sub>O<sub>3</sub>S<sub>3</sub>: C, 34.04; H, 2.12; N, 29.78; Found: C, 33.89; H, 2.38; N, 30.02.

### ***General procedure for the synthesis of compounds [V]<sub>n</sub>***

To a 100 mL round bottom flask charged with a solution of compound [IV] (4.23 g, 10 mmol) was dissolved in 15 mL of DMF was added dropwise a mixture of anhydrous potassium carbonate (4.14 g, 30 mmol) and n-alkylbromide (30 mmol). The stirred mixture was heated at reflux for 4 h and then allowed to cool down at room temperature. A cold water (400 mL) was poured into the reaction mixture and the solution was kept overnight at 5 °C. A solid was collected by filtration which allowed drying in air to give [V]<sub>n</sub>.

n	3	4	5	6	7	8
Yield (%)	72	79	75	81	70	73

## Analytical data

### ***3,3',3''-(1,3,5-triazine-2,4,6-triyl)tris(2-(pentylimino)thiazolidin-4-one) [V]<sub>5</sub>***

Brown color; yield: 75%; m.p.: 193 °C; FTIR ( $\nu/\text{cm}^{-1}$ ): 2929-2867 (C-H aliph.), 1727 (C=O), 1618 (C=N), 769 (C-S);  $^1\text{H NMR}$  (400MHz, DMSO- $d_6$ )  $\delta$  (ppm): 3.36 (s, 6H, 3CH<sub>2</sub>), 3.05 (t, 6H, 3 NCH<sub>2</sub>-), 2.35- 2.11 (m, 6H, 3 (-CH<sub>2</sub>)), 1.36- 1.23 (m, 12H, 3 (-CH<sub>2</sub>)<sub>2</sub>), 0.94 (t, 9H, 3CH<sub>3</sub>).

### ***3,3',3''-(1,3,5-triazine-2,4,6-triyl)tris(2-(heptylimino)thiazolidin-4-one) [V]<sub>7</sub>***

Brown color; yield: 70%; m.p.: 187 °C; FTIR ( $\nu/\text{cm}^{-1}$ ): 2953-2856 (C-H aliph.), 1714 (C=O), 1610 (C=N), 767 (C-S);  $^1\text{H NMR}$  (400MHz, DMSO- $d_6$ )  $\delta$  (ppm): 3.28 (s, 6H, 3CH<sub>2</sub>), 3.03 (t, 6H, 3 NCH<sub>2</sub>-), 2.81- 2.73 (m, 6H, 3 (-CH<sub>2</sub>)), 1.50- 1.42 (m, 12H, 3 (-CH<sub>2</sub>)<sub>2</sub>), 1.18-1.08 (m, 12H, 3 (-CH<sub>2</sub>)<sub>2</sub>), 0.70 (t, 9H, 3CH<sub>3</sub>).

## Results and discussion

### ***Characterization***

The synthetic routes of the two series derived from triazines segment [III]<sub>n</sub> and [V]<sub>n</sub> is outlined in Scheme 1. Several synthetic procedures were used to isolate the target compounds in high yields. The reaction of melamine with chloroacetyl chloride in a 1:3 mole ratio resulted in the formation of N-chloroacetyl compound [I]. Subsequently,

the reaction of compound [I] with malononitrile in a dioxane medium with triethylamine as hydrochloride acceptor gave compound [II]; pyrrole, 1,1',1''-(1,3,5-triazine-2,4,6-triyl)tris(2-amino-5-oxo-4,5-dihydro-1H-pyrrole-3-carbonitrile). The synthesis of N-alkyl derivatives [III]<sub>n</sub> was achieved by heating at reflux compound [II] with a primary alkylbromide and K<sub>2</sub>CO<sub>3</sub> in DMF medium [26]. The formation of compound [IV] was based on the cyclization reaction. The reaction of chloroacetamide compound [I] with potassium thiocyanate in dry acetone yielded the required compound. The new thiazolidinone of N-alkyl derivatives [V]<sub>n</sub> were obtained by heating at reflux compound [IV] with a primary alkylbromide and K<sub>2</sub>CO<sub>3</sub> in DMF medium.

A range of physico-chemical techniques was used to characterize the entity of compounds including their liquid crystalline behavior. The observed values of the elemental analyses of the synthesized compounds are in well agreement with theoretical values indicating structure of respective compounds, Table 1.

The FTIR spectrum of N-chloroacetyl compound [I] indicated the disappearance of the absorption band related to the NH<sub>2</sub> group of the starting material. Bands observed at 1678 and 671 cm<sup>-1</sup> assigned to ν<sub>C=O</sub> amide and ν<sub>C-Cl</sub> group, respectively. The FTIR spectrum of compound [II] showed characteristic absorption bands of (NH<sub>2</sub>), (C≡N) and (C=O) functional groups at 3404 and 3244, 2212 and 1714 cm<sup>-1</sup>, respectively.

The FTIR spectra for N-alkyl derivatives [III]<sub>n</sub> indicated the disappearance band related to the NH<sub>2</sub> group in the compound [II] and the existence of bands belong to N-H and C-H aliphatic groups in the range 3327-3444 and 2966-2831 cm<sup>-1</sup>, respectively. The FTIR spectrum of compound [IV] revealed band assigned to NH group at 3361 cm<sup>-1</sup>. Bands recorded at 1726, 1610 and 769 cm<sup>-1</sup> attributed to ν<sub>C=O</sub>, ν<sub>C=N</sub> and ν<sub>C-S</sub> group, respectively. The FTIR spectra of the thiazolidinone derivatives with N-alkyl substitution [V]<sub>n</sub> showed the disappearance of the absorption bands of NH groups for compound [IV] and the appearance of the absorption bands of C-H aliphatic for alkyl groups at range 2986-2856 cm<sup>-1</sup>.

<sup>1</sup>HNMR spectrum for the compound [II] showed peak at δ 7.30 ppm for protons of NH<sub>2</sub> group and peak at δ 4.11 ppm for protons of CH<sub>2</sub> group in cyclic. In <sup>1</sup>HNMR spectra, the synthesized [III]<sub>n</sub> compounds showed peaks of the terminal alkyl spacer (-CH<sub>2</sub>(CH<sub>2</sub>)<sub>n</sub>CH<sub>3</sub>) at ≈ δ 3.07-0.70 ppm. The <sup>1</sup>HNMR spectrum for compound [IV] display, peak at δ 11.15 ppm due to the proton of NH groups and peak at δ 4.44 ppm

**Table 1.** Elemental analysis data for the series [III]<sub>n</sub> and [V]<sub>n</sub>

Comp. No.	Molecular formula	Molecular weight (g/mol)	Analysis % found (calcd.)			
			C	H	N	S
[III] <sub>3</sub>	C <sub>27</sub> H <sub>30</sub> N <sub>12</sub> O <sub>3</sub>	570	57.15(56.84)	5.18(5.26)	29.73(29.47)	
[III] <sub>4</sub>	C <sub>30</sub> H <sub>36</sub> N <sub>12</sub> O <sub>3</sub>	612	58.94(58.82)	6.05(5.88)	27.67(27.45)	
[III] <sub>5</sub>	C <sub>33</sub> H <sub>42</sub> N <sub>12</sub> O <sub>3</sub>	654	60.19(60.55)	6.73 (6.42)	25.18(25.68)	
[III] <sub>6</sub>	C <sub>36</sub> H <sub>48</sub> N <sub>12</sub> O <sub>3</sub>	696	61.57(62.06)	7.03(6.89)	23.92(24.13)	
[III] <sub>7</sub>	C <sub>39</sub> H <sub>54</sub> N <sub>12</sub> O <sub>3</sub>	738	62.97(63.41)	7.58(7.31)	23.09(22.76)	
[III] <sub>8</sub>	C <sub>42</sub> H <sub>60</sub> N <sub>12</sub> O <sub>3</sub>	780	64.89(64.61)	7.92(7.69)	21.18(21.53)	
[V] <sub>3</sub>	C <sub>21</sub> H <sub>27</sub> N <sub>9</sub> O <sub>3</sub> S <sub>3</sub>	549	46.12(45.90)	5.18(4.91)	22.64(22.95)	16.71(17.49)
[V] <sub>4</sub>	C <sub>24</sub> H <sub>33</sub> N <sub>9</sub> O <sub>3</sub> S <sub>3</sub>	591	48.32(48.73)	5.25(5.58)	21.48(21.31)	15.57(16.24)
[V] <sub>5</sub>	C <sub>27</sub> H <sub>39</sub> N <sub>9</sub> O <sub>3</sub> S <sub>3</sub>	633	50.91(51.18)	6.39(6.16)	20.12(19.90)	14.43(15.17)
[V] <sub>6</sub>	C <sub>30</sub> H <sub>45</sub> N <sub>9</sub> O <sub>3</sub> S <sub>3</sub>	675	52.98(53.33)	6.91(6.66)	18.20(18.66)	14.58(14.22)
[V] <sub>7</sub>	C <sub>33</sub> H <sub>51</sub> N <sub>9</sub> O <sub>3</sub> S <sub>3</sub>	717	54.89(55.23)	7.35(7.11)	17.31(17.57)	13.08(13.39)
[V] <sub>8</sub>	C <sub>36</sub> H <sub>57</sub> N <sub>9</sub> O <sub>3</sub> S <sub>3</sub>	759	57.02(56.91)	7.25(7.50)	16.46(16.60)	12.38(12.65)

**Table 2.** Phase transition temperatures ( $^{\circ}\text{C}$ ) and transition enthalpies  $\Delta H(\text{kJ/mol})$  of series  $[\text{III}]_n$  and  $[\text{V}]_n$  determined by DSC ( $10^{\circ}\text{C}/\text{min}$ ) during first heating

Compound No.	Transition temperatures ( $^{\circ}\text{C}$ ) ( $\Delta H$ , kJ/mol)
$[\text{III}]_3$	Cr 168 (23.56) N 193 (6.43) I
$[\text{III}]_4$	Cr 183 (16.78) N 215 (4.80) I
$[\text{III}]_5$	Cr 150 I
$[\text{III}]_6$	Cr 120 (22.04) $\text{Col}_x$ 160 (0.76) I
$[\text{III}]_7$	Cr 134 (10.08) $\text{Col}_x$ 197 (33.62) N 211 (0.84) I
$[\text{III}]_8$	Cr 82 Cr 102 (21.42) $\text{Col}_x$ 121 (1.16) N 151 (0.78) I
$[\text{V}]_3$	Cr 139 (25.22) N 175 (5.76) I
$[\text{V}]_4$	Cr 221 I
$[\text{V}]_5$	Cr 180 (0.63) N 198 (0.52) I
$[\text{V}]_6$	Cr 250 (19.60) $\text{Col}_x$ 273 (3.08) I
$[\text{V}]_7$	Cr 201 (11.78) $\text{Col}_x$ 216 (0.87) I
$[\text{V}]_8$	Cr 185 (16.79) $\text{Col}_x$ 217 (0.68) I

for the protons of methylene group in cyclic. The  $^1\text{H}$ NMR spectra for the synthesized  $[\text{V}]_n$  compounds showed peaks of the terminal alkyl spacer at  $\approx \delta$  3.03–0.70 ppm.

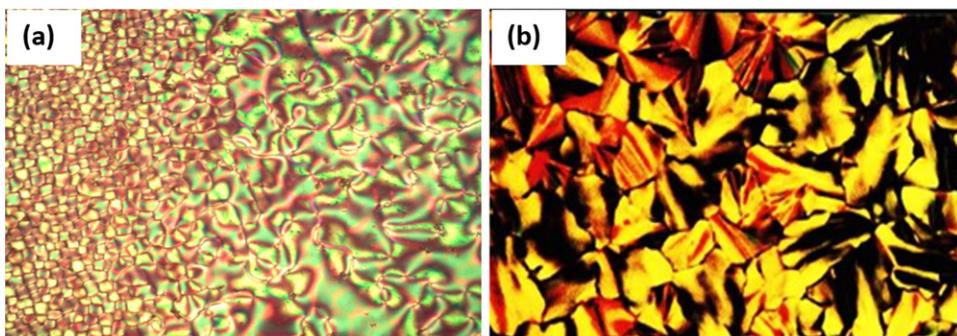
### Liquid crystalline properties

The phase transition temperatures and mesophase type (texture identity) of all compounds were investigated by using DSC and POM. Generally, the thermal behavior obtained by DSC is consistent with the data observed by POM, where it was possible to identify the mesomorphic character of the mesophases. The phase transition temperatures and thermodynamic data of the two series  $[\text{III}]_n$  and  $[\text{V}]_n$  are listed in Table 2. On heating, the first endothermic peak is accompanied with high enthalpy indicates a transition from solid crystalline state to a liquid crystalline mesophase. The second endothermic transition at higher temperature presented lower associated enthalpy, which indicates transitions between distinct mesophases and to the isotropic state.

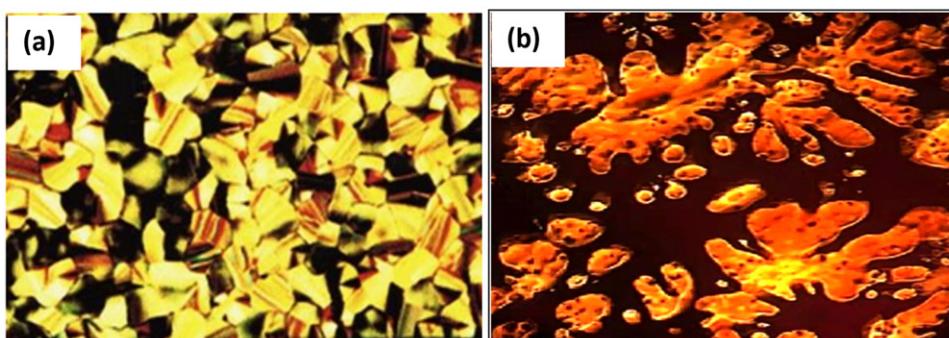
The mesophases that exhibited by compounds of series  $[\text{III}]_n$  and  $[\text{V}]_n$  were identified according to their optical textures, which were observed by POM, using the classification systems reported by Sackmann and Demus and Richter [27, 28] and Gray and Goodby [29].

The homologues  $[\text{III}]_3$ ,  $[\text{III}]_4$ ,  $[\text{V}]_3$  and  $[\text{V}]_5$  showed pure enantiotropic nematic mesophase. Upon cooling the isotropic liquid of each of this compound, nematic droplets appeared at transition point which eventually coalesces to form the typical Schlieren texture with characteristic two- and four-brush singularities (Fig. 1(a)). The homologues  $[\text{III}]_6$  and  $[\text{III}]_7$  exhibit an enantiotropic dimorphism nematic (N) and columnar ( $\text{Col}_x$ ) phases. As indicted above, upon further cooling of the nematic phase, the emergence of the  $\text{Col}_x$  phase was observed in its mosaic texture (Fig. 1(b)).

Compounds  $[\text{III}]_5$  and  $[\text{V}]_4$  did not show any mesophase and showed a crystal to isotropic transition at 150 and  $221^{\circ}\text{C}$ , respectively. The non mesomorphism of those two compounds is basically due to their high crystallizing tendency and the lack of flexibility that cause mesophase formation. The non mesomorphicity of those homologues is attributed to the low magnitudes of intermolecular dispersion forces and low magnitudes of dipole–dipole interfaces leading to the high crystallizing tendency. As a result, it causes the hasty breaking of crystal lattices because of unsuitable magnitudes of anisotropic forces of intermolecular desirability. Therefore, the improper breaking of crystal lattices will result in direct melts without displaying the liquid crystalline phase [30–32].



**Figure 1.** Photomicrographs of textures as seen by POM for the (a) droplet and thread-like nematic phase of compound  $[\text{III}]_3$  at  $181\text{ }^\circ\text{C}$  and (b)  $\text{Col}_x$  phase of compound  $[\text{III}]_6$  at  $146\text{ }^\circ\text{C}$  ( $200\times$  magnification).



**Figure 2.** Photomicrographs of textures as seen by POM for the  $\text{Col}_x$  phase of (a) compound  $[\text{V}]_6$  at  $261\text{ }^\circ\text{C}$  and (b) compound  $[\text{V}]_8$  at  $198\text{ }^\circ\text{C}$  ( $200\times$  magnification).

Compounds  $[\text{III}]_6$ ,  $[\text{V}]_6$ ,  $[\text{V}]_7$  and  $[\text{V}]_8$  display enantiotropic mesomorphism. The texture observed by POM on cooling from the isotropic liquid are consistent with the presence of columnar mesomorphism, with a typical mosaic texture and dendritic shape [33] as shown in Figs. 2(a) and 2(b), respectively. The demonstration of the columnar mesophase from the hexyl to octyl derivatives of the reported series is attributed to the packing of the molecules, which increases the suitable magnitude of anisotropic forces of attractions to cause the sliding layers (a two-dimensional array of molecules) [34].

## Conclusions

In this study, a new two series of disc-shaped liquid crystal molecules were synthesized based on 1,3,5-triazine central core. Increase in the polyoxymethylene spacer length influence the thermal and phase stability of these compounds. The shorter length of the alkoxy group exhibited the nematic liquid crystalline behavior. The compounds with  $n \geq 6$ , exhibited an anantiotropic columnar phase; however, compounds  $[\text{III}]_5$ , and  $[\text{V}]_4$  formed a crystalline phase. The homologous series showed mesogenic behavior with good thermal stability.

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