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Synthesis and mesophase characterisation of a series of new triazine-based disc-shaped molecules

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A series of symmetrical disc-shaped materials with a heteroaromatic 1,3,5-s-triazine core and nine alkoxy chains at the peripheral position has been designed and synthesised in a simplified way. All the compounds show hexagonal columnar mesophase. The mesomorphic properties of the compounds have been characterised with the help of polarising optical microscopy (POM), differential scanning calorimetry (DSC) and temperature controlled low angle XRD-experiments.

Keywords: triazine core; discotic liquid crystal; hexagonal columnar mesophase; POM

1. Introduction

Discotic liquid crystals are attractive due to their versatile application in the devices of the modern era such as one-dimensional conductors, photovoltaic solar cells, optical light emitting devices, etc. [1–5]. The common arrangements observed in case of discotic liquid crystals (DLCs) are hexagonal columnar, rectangular columnar and nematic columnar. The classical molecular structure of DLCs consists of a planar, symmetrical polyaromatic or macrocyclic core surrounded by several saturated aliphatic long chains [6, 7]. Since the discovery of the first discotic liquid crystal in 1977, a number of DLCs based on aromatic cores have been widely studied but those with heteroaromatic nuclei have attracted more attention in recent years due to their wide application in optoelectronics [8–14].

1,3,5-s-Triazine and its derivatives are useful not only in biological science [15], but also have a great importance in material science showing a wide range of applicative behaviour such as liquid crystalline, non-linear optical, polymeric properties [16–20]. Interest in this class of compounds has grown due to its symmetric nature which plays a key role in self-organising ability in forming liquid crystals, polymers, dendrimers, etc. In addition, the planarity of this moiety enhances the electron mobility that is important for light-emitting devices [21]. Liquid crystalline compounds containing a 1,3,5-s-triazine core have been reported by several research groups [22–25].

The high charge carrier mobility is one of the most important factors related to the properties of DLCs [26]. The high charge carrier mobility originates from the organisation of molecules in columns. The parameters responsible for the high charge carrier are the π – π stacking interactions between the two adjacent

aromatic systems [27] and the rotational angle between two adjacent molecules. In case of C_3 -symmetrical molecules, the ideal rotational angles for high charge carrier mobility are 0° , 120° and 240° ; i.e. those orientations in which the side arms of both the molecules are aligned on the top of each other [28]. Thus to obtain the improved charge carrier mobility, the ideal alignment of the discotic molecules should be in such a way that the disc–disc distance is lowered and lateral slippage is reduced. The attractive force between the discogens may be enhanced by introducing linearly joined aromatic cores through linking groups at the side arms which also interact with the adjacent one through π – π stacking.

Considering all the aforesaid aspects and in continuation of our previous experience in the synthesis and characterisation of new liquid crystalline materials [29–34], we undertook a study on the design and synthesis of a series of discotic molecules bearing 1,3,5-s-triazine as the core and trialkoxy chains at the peripheral position. The alkoxy chains are designed to be linked with the core via linearly joined aromatic cores which in turn consist of an imine and ester linkage. Here we report the results of this investigation.

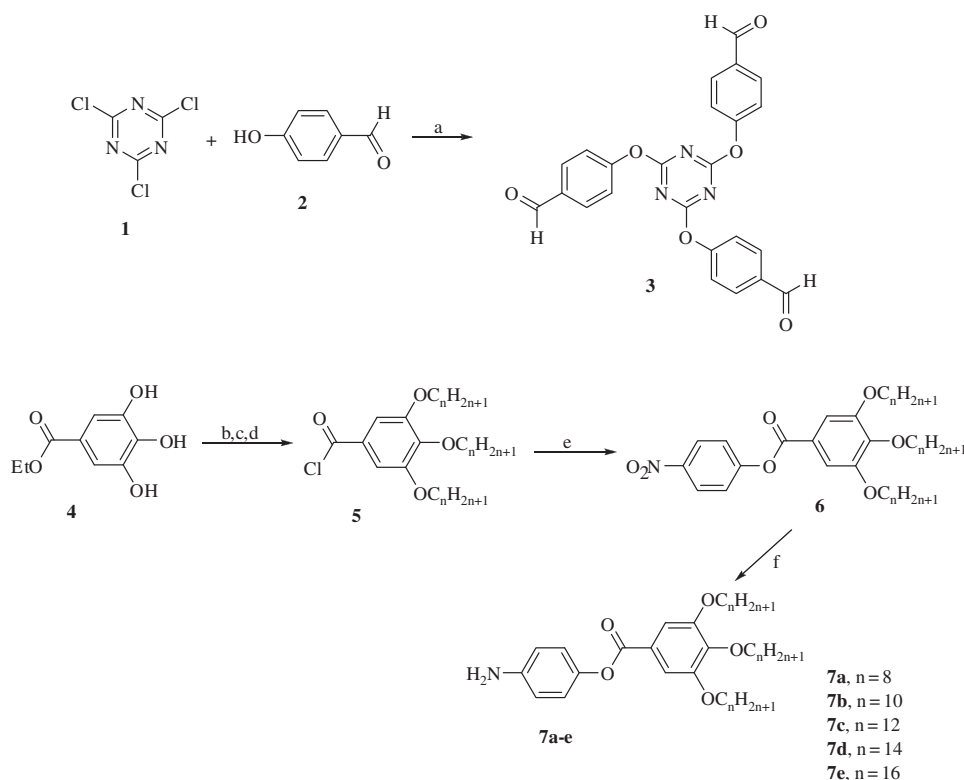
2. Experimental

2.1 Synthetic routes

The synthetic routes employed to obtain the requisite starting materials are outlined in Scheme 1 and those of the target discotic molecules are shown in Scheme 2.

We synthesised the triazine-trialdehyde **3** according to the procedure described by Tahmassebi and Sasaki [35]. The reaction of cyanuric chloride with

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Reagents and conditions: (a) Na_2CO_3 , benzene, reflux, 20h (b) $\text{C}_n\text{H}_{2n+1}\text{Br}$, K_2CO_3 , NaI (cat.), ethyl methyl ketone, reflux, 72h (c) KOH (5.0 eqv.), EtOH, reflux, 4h (d) SOCl_2 , DMF (1drop), reflux, 3h (e) *p*-nitro phenol, DMAP (cat.), Et_3N , CHCl_3 , stirring, r.t., 4h (f) SnCl_2 (4 eqv.), EtOAc, reflux, 12h.

Scheme 1.

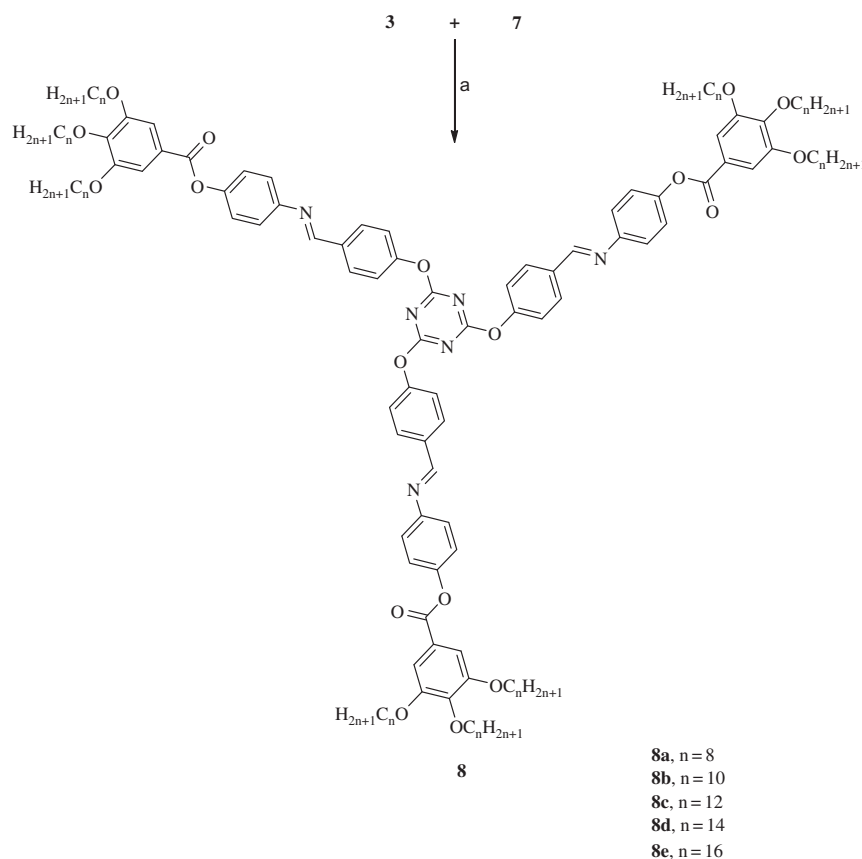
excess *p*-hydroxy benzaldehyde in the presence of Na_2CO_3 in benzene under refluxing conditions gave the trialdehyde **3**. The other precursors, **7a–e**, were synthesised from ethyl gallate esters **4**.

First the three hydroxyl groups of compounds **4** were alkylated with alkyl bromides in refluxing ethyl methyl ketone using K_2CO_3 in the presence of a catalytic amount of NaI for about 72 h. The ester group was then hydrolysed with KOH and the resulting 3,4,5-trialkoxo benzoic acids were converted to the corresponding acid chlorides **5** with SOCl_2 in refluxing condition. The crude acid chlorides were then esterified with *p*-nitro phenol using the Et_3N -DMAP protocol. The synthon amines **7a–e** were obtained by the reduction of the corresponding nitro compounds **6** with SnCl_2 in ethyl acetate as solvent.

The target compounds **8a–e** were synthesised by Schiff's base formation of triazine-trialdehyde **3** with excess of synthon amines **7a–e** in refluxing ethanol in the presence of a catalytic amount of glacial acetic acid for 12 h. The compounds **8a–e** were purified by repeated precipitation from benzene with ethanol.

2.2 Reagents and techniques

All the chemicals were procured from either Sigma Aldrich Chemicals Pvt. Ltd or Spectrochem, India. Silica gel (60–120 mesh) was used for chromatographic separation. Silica gel G (E-Merck, India) was used for TLC. IR spectra were recorded on a Perkin-Elmer L 120-000A spectrometer (ν_{max} in cm^{-1}) on KBr discs. ^1H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Bruker DPX-500 spectrometer in CDCl_3 (chemical shift in δ) with TMS as internal standard. CHN was recorded on a Perkin Elmer 2400 series II CHN analyser. The liquid crystalline properties were established by thermal microscopy (Nikon polarising microscope LV100POL) attached with an Instec hot and cold stage HCS302, with a STC200 temperature controller configured for HCS302. Differential scanning calorimetry (DSC) was recorded on a Perkin-Elmer Diamond DSC Pyris1 system. Powder X-ray diffraction pattern of the sample was obtained on a D8 Advance Bruker AXS diffractometer (operated at 40 kV voltage and 40 mA current and calibrated with standard silicon



Reagents and conditions: (a) Glacial AcOH (cat.), EtOH, reflux, 12h.

Scheme 2.

sample) attached to a temperature controller using Cu $K\alpha$ ($\lambda = 0.15406$) radiation at IACS, Kolkata.

2.3 Synthetic procedure

2.3.1 Synthesis of compound 3

p-Hydroxy benzaldehyde (1.33 g, 10.88 mmol) and cyanuric chloride (500 mg, 2.71 mmol) were added to a solution of Na_2CO_3 (10 g) in dry benzene (50 ml). The mixture was refluxed for 20 h. The reaction mixture was allowed to cool to room temperature, filtered and the solid was washed with hot ethyl acetate. The filtrate was collected, and then washed with 10% Na_2CO_3 solution and water successively. The organic layer was dried over Na_2SO_4 and evaporated under reduced pressure to afford a white solid. The solid was purified by recrystallisation from ethyl acetate to give compound 3.

Compounds 7a-e were prepared according to the literature procedure [30, 31].

2.3.2 General procedure for the preparation of compounds 8a-e

A mixture of 3 (100 mg, 0.254 mmol) and 7a (500 mg, 0.839 mmol) was refluxed in absolute ethanol (10 ml) in the presence of a catalytic amount of glacial acetic acid for 12 h. The Schiff's base 8a was precipitated out from reaction mixture. It was collected, washed repeatedly with hot ethanol and dried in vacuum. Similarly the other derivatives 8b-e were prepared.

Compound 8a: yield = 73% off-white powder; IR (KBr): $\nu = 2925, 2855, 1732, 1628 \text{ cm}^{-1}$; ^1H NMR (CDCl_3 , 300 MHz): $\delta_{\text{H}} = 8.47$ (s, 3H, CH=N), 7.94 (d, 6H, $J = 8.1$ Hz, ArH), 7.40 (s, 6H, ArH), 7.20–7.36 (m, 18H, ArH), 4.01–4.07 (m, 18H, OCH_2), 0.89–1.85 (m, 135H, aliphatic hydrogens are overlapped); ^{13}C NMR (CDCl_3 , 100 MHz): 173.6, 165.1, 158.9, 153.7, 153.0, 152.9, 149.3, 143.0, 134.4, 131.3, 130.1, 123.8, 122.5, 122.3, 121.8, 115.7, 108.5, 73.6, 69.2, 31.9, 30.4, 29.5, 29.4, 29.3, 26.1, 22.7, 14.1; Analysis calculated for $\text{C}_{135}\text{H}_{186}\text{N}_6\text{O}_{18}$: C = 74.35, H = 8.60, N = 3.85%. Found: C = 74.63, H = 8.71, N = 3.77%.

Compound 8b: yield = 75% off-white powder; IR (KBr): $\nu = 2924, 2854, 1732, 1628 \text{ cm}^{-1}$; $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.48$ (s, 3H, CH=N), 7.96 (d, 6H, $J = 8.8$ Hz, ArH), 7.39 (s, 6H, ArH), 7.20–7.30 (m, 18H, ArH), 4.01–4.07 (m, 18H, OCH_2), 0.81–1.86 (m, 171H, aliphatic hydrogens are overlapped); $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): 173.6, 165.6, 158.9, 153.6, 153.0, 149.3, 144.2, 143.2, 134.4, 123.8, 122.3, 121.8, 115.7, 108.5, 73.6, 69.2, 31.9, 30.4, 29.8, 29.7, 29.6, 29.4, 29.3, 26.1, 22.7, 14.1; Analysis calculated for $\text{C}_{153}\text{H}_{222}\text{N}_6\text{O}_{18}$: C = 75.52, H = 9.20, N = 3.45%. Found: C = 75.29, H = 9.43, N = 3.51%.

Compound 8c: yield = 66% off-white powder; IR (KBr): $\nu = 2924, 2853, 1731, 1627 \text{ cm}^{-1}$; $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.48$ (s, 3H, CH=N), 7.96 (d, 6H, $J = 8.8$ Hz, ArH), 7.40 (s, 6H, ArH), 7.20–7.31 (m, 18H, ArH), 4.01–4.07 (m, 18H, OCH_2), 0.86–1.84 (m, 207H, aliphatic hydrogens are overlapped); $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): 173.5, 165.1, 158.9, 153.7, 152.9, 149.3, 143.0, 130.2, 123.8, 122.5, 121.9, 121.8, 108.5, 73.6, 69.2, 31.9, 30.4, 29.8, 29.7, 29.6, 29.4, 29.3, 26.1, 22.7, 14.1; Analysis calculated for $\text{C}_{171}\text{H}_{258}\text{N}_6\text{O}_{18}$: C = 76.47, H = 9.68, N = 3.13%. Found: C = 76.62, H = 9.41, N = 3.27%.

Compound 8d: yield = 70% off-white powder; IR (KBr): $\nu = 2923, 2853, 1731, 1627 \text{ cm}^{-1}$; $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.47$ (s, 3H, CH=N), 7.95 (d, 6H, $J = 8.8$ Hz, ArH), 7.39 (s, 6H, ArH), 7.20–7.31 (m, 18H, ArH), 4.01–4.07 (m, 18H, OCH_2), 0.86–1.84 (m, 243H, aliphatic hydrogens are overlapped); $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): 173.5, 165.1, 158.9, 153.7, 152.9, 149.3, 143.0, 130.2, 123.8, 122.5, 121.9, 121.8, 108.5, 73.6, 69.2, 31.9, 30.4, 29.8, 29.7, 29.6, 29.4, 29.3, 26.1, 22.7, 14.1; Analysis calculated for $\text{C}_{189}\text{H}_{294}\text{N}_6\text{O}_{18}$: C = 77.25, H = 10.08, N = 2.86%. Found: C = 77.52, H = 10.26, N = 2.85%.

Compound 8e: yield = 72% off-white powder; IR (KBr): $\nu = 2920, 2851, 1731, 1627 \text{ cm}^{-1}$; $^1\text{H NMR}$

(CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.48$ (s, 3H, CH=N), 7.96 (d, 6H, $J = 8.8$ Hz, ArH), 7.39 (s, 6H, ArH), 7.22–7.31 (m, 18H, ArH), 4.01–4.07 (m, 18H, OCH_2), 0.86–1.84 (m, 279H, aliphatic hydrogens are overlapped); $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): 173.5, 165.1, 158.9, 153.7, 152.9, 149.3, 143.0, 134.3, 130.2, 123.8, 122.5, 121.9, 121.8, 108.5, 73.6, 69.2, 31.9, 30.4, 29.7, 29.6, 29.4, 29.3, 26.1, 22.7, 14.1; Analysis calculated for $\text{C}_{207}\text{H}_{330}\text{N}_6\text{O}_{18}$: C = 77.92, H = 10.42, N = 2.63%. Found: C = 77.69, H = 10.59, N = 2.44%.

3. Results and discussion

3.1 Mesomorphic behaviour

The mesomorphic behaviour of compounds **8a–e** was initially characterised by optical polarising microscopy (POM). Some of the micrographs obtained on cooling the sample sandwiched between a glass slide and a cover slip are shown in Figure 1. It was observed that a homeotropic dendritic domain gradually grows up to build a beautiful fan-shaped focal conic texture in all the cases when the samples are allowed to cool slowly from the isotropic melts. These textures are typical for columnar mesophases.

DSC experiments were performed for all the compounds at a rate of $5^\circ\text{C}/\text{min}$ to correlate the mesophase behaviour observed under the microscope and also to obtain the associated transition temperatures. The results are summarised in Table 1. Two peaks were observed in the DSC thermogram for both the heating and cooling cycles of all the compounds. These indicate the presence of a mesophase; the first peak in the heating cycle is due to crystalline-to-columnar phase transition and the second one is for isotropic phase transition. Wax-like solid (i.e. with a lower order nature of the molecules in the solid phase) may perhaps account for the low enthalpy values for crystal-to-columnar phase transitions [8]. The

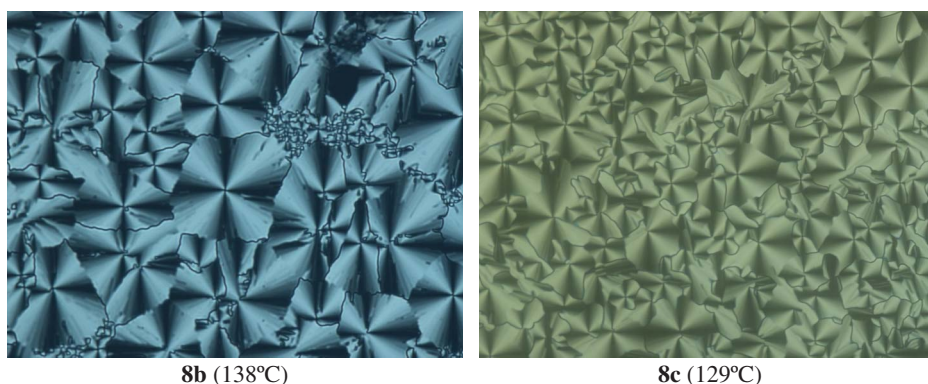


Figure 1. Optical textures of hexagonal columnar mesophase obtained during cooling the samples from isotropic to liquid crystalline phase of compounds **8b** and **8c** in POM study.

Table 1. Phase transition temperatures and associated enthalpies of compounds **8a–e** in the heating and cooling cycles.

Compound	Phase transition	Temperature (°C)	Enthalpy (kJmol ⁻¹)	Phase transition	Temperature (°C)	Enthalpy (kJmol ⁻¹)
	Heating scan (5°C/min)			Cooling scan (5°C/min)		
8a	Cr → Col _h	114.5	1.73	I → Col _h	158.6	0.62
	Col _h → I	159.5	0.51	Col _h → Cr	109.1	1.02
8b	Cr → Col _h	133.6	1.65	I → Col _h	157.8	0.57
	Col _h → I	158.5	0.64	Col _h → Cr	131.7	1.15
8c	Cr → Col _h	127.8	0.98	I → Col _h	130.0	0.56
	Col _h → I	131.6	0.43	Col _h → Cr	125.3	0.89
8d	Cr → Col _h	90.6	1.64	I → Col _h	117.3	0.41
	Col _h → I	119.3	2.19	Col _h → Cr	88.4	0.82
8e	Cr → Col _h	89.2	1.72	I → Col _h	105.7	1.97
	Col _h → I	108.9	1.76	Col _h → Cr	82.3	0.60

Notes: Cr = Crystal; Col_h = Hexagonal columnar; I = Isotropic

associated isotropic transitions are also weak, indicating that the molecule may possess some extra fluidity in its liquid crystalline state.

Combining the observations of POM and the DSC experiments it is found that the thermal range of the liquid crystalline phase as well as the clearing temperature gradually decreases as the peripheral chain length increases.

3.2 X-ray diffraction study

We carried out a low angle XRD-experiment of the unoriented sample of **8b** in its liquid crystalline state (134°C) to confirm the mesogenic behaviour. The diffraction pattern is shown in Figure 2. In the small angle region, there are three peaks at $\theta = 1.09^\circ$, 1.84° , and 2.18° which correspond to the layer spacing 40.5 \AA (d_1), 23.8 \AA (d_2) and 20.21 \AA (d_3). The ratio $d_1:d_2:d_3$ ($1:1/\sqrt{3}:1/2$) clearly indicates the presence of a hexag-

onal columnar (Col_h) mesophase. In the wide angle region (not shown in the figure), the halo at 4.76 \AA corresponds to liquid-like packing of the aliphatic chains and the peak at 3.7 \AA is due to core–core separation.

In 1998 Gibson *et al.* [36] synthesised a series of liquid crystalline compounds containing a 1,3,5-triazine core linked with the mono alkyl/alkoxy chains at the terminal positions. Afterwards, 1,3,5-triazine based mesogenic compounds with azobenzene at the peripheral arms were also synthesised [37]. In those cases, smectic A mesophase was observed. To interpret this mesomorphic behaviour, it is assumed that the terminal side arm behaves rod-like, i.e. a fork-type arrangement in which the core moiety acts as the linking unit [38]. But in our case the hexagonal columnar arrangement was observed. It is assumed that the molecules are stacked over each other one after one, forming a single column and that the columns

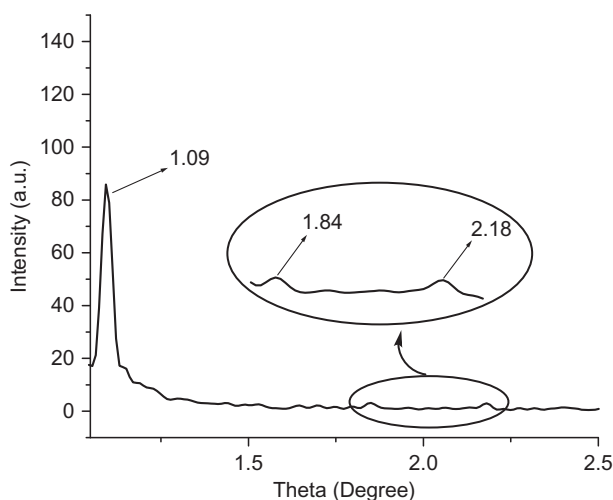


Figure 2. XRD pattern of compound **8b** in its liquid crystalline state (134°C).

are positioned symmetrically in a hexagonal fashion [27, 39].

This work is interesting in view of a recent report by Sandeep Kumar's group [8] who reported a series of C₃-symmetric dendrimers that are non-mesogenic. Those non-mesogenic compounds when mixed with electron deficient molecule TNF (2,4,7-trinitrofluoren-9-one) showed columnar mesophase. Our synthesised symmetric dendrimers show columnar mesophase as such without mixing with any other compounds.

In conclusion, a simple and straightforward synthesis of a novel class of heteroaromatic core-based mesogenic materials has been achieved. All the compounds show good liquid crystalline properties, exhibiting hexagonal columnar mesophase. These particular compounds may be interesting and useful for various multifunctional materials.

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