



# Synthesis and comparative studies of phase transition behaviour of new dimeric liquid crystals consisting of dimethyluracil and biphenyl cores



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## ABSTRACT

A new homologous series of mesogens containing 6-amino-1,3-dimethyluracil moiety have been synthesized. The structures of the compounds were characterised by elemental analysis, FT-IR, <sup>1</sup>H and <sup>13</sup>C NMR spectroscopic techniques. Their mesomorphic properties were studied by polarising optical microscopy attached to a heating stage. Microscopy data were supported with transition temperatures and enthalpy change values obtained from the differential scanning calorimetry analysis. The studies have shown that the mesomorphic properties of the compounds are dependent on the lengths of alkoxy-spacers. Compounds **4a–f** with a shorter alkoxy-spacer chain (n = 6) exhibited smectic A phase, while compounds **4g–r** with a alkoxy-spacer chain (n = 8 or 10) displayed nematic phase.

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## 1. Introduction

Liquid crystals play an important role in today's world of science and technology, due to their technological applications, particularly as optical imaging liquid crystal displays, organic light emitting diodes (OLED's), anisotropic networks and semiconductor materials [1–3]. The mesomorphic behaviour of organic compounds depends on the structure of the molecules and can be varied by modifying its molecular structure including linking, terminal and core groups [4].

The simplest molecules consist of two liquid crystalline/non-liquid crystalline units connected by a flexible spacer usually in the sense of linearity, and are known as LC dimers [5,6]. A wide range of low molar mass dimeric molecular architectures are known to exhibit interesting LC properties when compared to the conventional monomer low molar mass LCs [7]. Moreover, the studies directed toward the dimers are enormous due to their LC favorable chemical structure, size/type of the core, connecting groups and terminal/spacer alkyl chains [8,9].

In recent years, many mesomorphic compounds containing differing heterocyclic units have been synthesized, with the interest in such structures growing constantly [10–12]. This is not only because of the greater possibilities with heterocyclics in the design of new mesogenic

molecules, but also due to fact that the insertion of heteroatoms strongly influences the formation of mesomorphic phases. The inclusion effect of heteroatoms (S, O and N) alters the geometric shape of the molecule considerably, thereby influencing the polarity, polarisability and sometime type of mesophase, phase transition temperatures, dielectric- and other properties of the mesogens [13].

Nucleic acids are biologically very important for the sustenance of life on earth, occurring in both RNA and DNA. The heterocyclic base moieties present in nucleic acids are well-known. These bases present in the nucleic acids are expected to be applicable to the design of molecular assemblies with diverse liquid-crystalline properties, since each nucleobase specifically forms a base pair with its predetermined partner. Some attempts to prepare thermotropic liquid crystals of nucleobase derivatives were unsuccessful [14–16]; but there are several reported examples of lyotropic liquid crystals derived from DNA and nucleotides [17,18].

This article reports on our on-going research on the synthesis and characterization of heterocyclic based liquid crystalline materials [19–23]. The present work expands our understanding of the structure-liquid crystalline property relationship of these materials. Specifically we focus on a new series of mesogenic compounds containing 1,3-dimethyluracil core system having alkoxy-spacer [–O–(CH<sub>2</sub>)<sub>n</sub>–O–] with an even number of carbon atoms ranging from C<sub>6</sub>H<sub>10</sub> to C<sub>10</sub>H<sub>24</sub>. A pendant biphenyl moiety is connected to this core by an alkyl chain varying in length from n = 6–16 carbons.

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From the  $^1\text{H}$  NMR data for compound **4a**, twelve aromatic protons of compound **4a** gave rise to six doublets with their resonances observed at  $\delta$  7.01–8.56 ppm. At downfield region of  $^1\text{H}$  NMR spectrum, the resonance of uracil ring proton and azomethine proton showed characteristic singlets at 9.76 and 8.86 ppm, respectively. A triplet at 0.89 ppm is attributed to the methyl protons of the terminal chain. Two triplets attributed to the methoxy protons of the spacer were evident at a slightly higher field at 4.01 and 3.99 ppm. The fact that two triplets were observed instead of one triplet suggests that the molecule is non-symmetric, while a triplet assigned to the ester  $-\text{OCH}_2$  protons could be observed at 4.18 ppm. Two singlets were observed at 3.37 and 3.19 ppm and assigned to two  $\text{CH}_3$  groups in the uracil ring,

### 2.3.1. General methodology for the synthesis of **4a–r**

The title compounds were synthesized according to a general method described by Majumdar et al. [27]. As an example of synthetic procedure for **4a** is given below:

A mixture of 6-amino-1,3-dimethyluracil (0.155 g, 0.001 mol) and hexyl-4'-(6-(4-formylphenoxy)hexyloxy)biphenyl-4-carboxylate, **3a** (0.502 g, 0.001 mol) was refluxed in absolute ethanol in the presence of a catalytic amount of glacial acetic acid for 2 h. The Schiff base **4a** was obtained as a precipitate from the hot reaction mixture. The precipitate was filtered out and repeatedly washed with hot ethanol and dried under vacuum.

**4a.** Yield 68%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2982, 2875, 1752 1625, 1581, 1256.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.70 (s, 1H, Uracil-H), 8.90 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.01–8.56 (6d, 12H, Ar–H), 4.18 (t, 2H,  $J = 6.70$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.01 (t, 2H,  $J = 6.30$  Hz,  $-\text{OCH}_2-$ ), 3.99 (t, 2H,  $J = 6.67$  Hz,  $-\text{OCH}_2-$ ), 3.37 (s, 3H,  $-\text{NCH}_3$ ), 3.19 (s, 3H,  $-\text{NCH}_3$ ), 1.78–1.28 (m, 16H, Alkyl  $-\text{CH}_2-$ ), 0.89 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 179.45, 168.33, 164.27 (C=O), 163.45 (C=N), 160.00 (Ar–C–O), 114.30–142.16 (Ar–C), 61.06 (C–O–C), 22.62 ( $\text{CH}_2$ ), 14.33 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{38}\text{H}_{45}\text{N}_3\text{O}_6$  (%): C, 71.18; H, 7.23; N, 6.43. Calc (%), C, 71.34; H, 7.09; N, 6.57.

**4b.** Yield 60%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2980, 2862, 1750 1621, 1577, 1250.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.72 (s, 1H, Uracil-H), 8.91 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.00–8.40 (6d, 12H, Ar–H), 4.19 (t, 2H,  $J = 6.80$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.03 (t, 2H,  $J = 6.55$  Hz,  $-\text{OCH}_2-$ ), 3.98 (t, 2H,  $J = 6.60$  Hz,  $-\text{OCH}_2-$ ), 3.35 (s, 3H,  $-\text{NCH}_3$ ), 3.12 (s, 3H,  $-\text{NCH}_3$ ), 1.77–1.27 (m, 20H, Alkyl  $-\text{CH}_2-$ ), 0.87 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 177.03, 167.89, 162.44 (C=O), 161.89 (C=N), 159.64 (Ar–C–O), 115.00–141.84 (Ar–C), 60.94 (C–O–C), 23.77 ( $\text{CH}_2$ ), 14.77 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{40}\text{H}_{49}\text{N}_3\text{O}_6$  (%): C, 71.80; H, 7.53; N, 7.32. Calc (%), C, 71.94; H, 7.40; N, 7.29;

**4c.** Yield 71%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2988, 2873, 1757 1623, 1580, 1250.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.81 (s, 1H, Uracil-H), 8.87 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.04–8.50 (6d, 12H, Ar–H), 4.20 (t, 2H,  $J = 6.86$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.09 (t, 2H,  $J = 6.67$  Hz,  $-\text{OCH}_2-$ ), 3.93 (t, 2H,  $J = 6.26$  Hz,  $-\text{OCH}_2-$ ), 3.42 (s, 3H,  $-\text{NCH}_3$ ), 3.20 (s, 3H,  $-\text{NCH}_3$ ), 1.78–1.25 (m, 24H, Alkyl  $-\text{CH}_2-$ ), 0.90 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 177.20, 167.14, 163.83 (C=O), 162.09 (C=N), 161.80 (Ar–C–O), 114.90–141.00 (Ar–C), 60.39 (C–O–C), 22.67 ( $\text{CH}_2$ ), 14.88 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{42}\text{H}_{53}\text{N}_3\text{O}_6$  (%): C, 72.60; H, 7.52; N, 6.19. Calc (%), C, 72.49; H, 7.68; N, 6.04.

**4d.** Yield 68%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2990, 2879, 1760 1625, 1586, 1254.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.83 (s, 1H, Uracil-H), 8.84 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.02–8.47 (6d, 12H, Ar–H), 4.18 (t, 2H,  $J = 6.38$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.04 (t, 2H,  $J = 6.80$  Hz,  $-\text{OCH}_2-$ ), 3.91 (t, 2H,  $J = 6.33$  Hz,  $-\text{OCH}_2-$ ), 3.38 (s, 3H,  $-\text{NCH}_3$ ), 3.17 (s, 3H,  $-\text{NCH}_3$ ), 1.79–1.24 (m, 28H, Alkyl  $-\text{CH}_2-$ ), 0.88 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 175.14, 165.60, 163.78 (C=O), 162.80 (C=N), 160.30 (Ar–C–O), 115.09–141.80 (Ar–C), 61.07 (C–O–C), 22.72 ( $\text{CH}_2$ ), 14.70 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{44}\text{H}_{57}\text{N}_3\text{O}_6$  (%): C, 73.18; H, 7.85; N, 5.90. Calc (%), C, 73.00; H, 7.94; N, 5.80.

**4e.** Yield 72%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2984, 2860, 1764 1623, 1575, 1250.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.80 (s, 1H, Uracil-H), 8.82 (s, 1H,

$-\text{N}=\text{CH}$ ), 7.01–8.57 (6d, 12H, Ar–H), 4.16 (t, 2H,  $J = 6.83$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.07 (t, 2H,  $J = 6.33$  Hz,  $-\text{OCH}_2-$ ), 3.93 (t, 2H,  $J = 6.79$  Hz,  $-\text{OCH}_2-$ ), 3.36 (s, 3H,  $-\text{NCH}_3$ ), 3.12 (s, 3H,  $-\text{NCH}_3$ ), 1.77–1.20 (m, 32H, Alkyl  $-\text{CH}_2-$ ), 0.85 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 173.79, 164.00, 162.59 (C=O), 161.08 (C=N), 159.65 (Ar–C–O), 114.07–139.87 (Ar–C), 60.58 (C–O–C), 21.06 ( $\text{CH}_2$ ), 14.18 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{46}\text{H}_{61}\text{N}_3\text{O}_6$  (%): C, 73.35; H, 8.27; N, 5.46. Calc (%), C, 73.47; H, 8.18; N, 5.59.

**4f.** Yield 74%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2988, 2869, 1767 1620, 1572, 1254.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.83 (s, 1H, Uracil-H), 8.82 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.03–8.58 (6d, 12H, Ar–H), 4.20 (t, 2H,  $J = 6.10$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.09 (t, 2H,  $J = 6.38$  Hz,  $-\text{OCH}_2-$ ), 3.90 (t, 2H,  $J = 6.89$  Hz,  $-\text{OCH}_2-$ ), 3.30 (s, 3H,  $-\text{NCH}_3$ ), 3.10 (s, 3H,  $-\text{NCH}_3$ ), 1.79–1.26 (m, 36H, Alkyl  $-\text{CH}_2-$ ), 0.86 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 176.06, 165.40, 163.64 (C=O), 162.29 (C=N), 161.75 (Ar–C–O), 114.88–140.29 (Ar–C), 60.44 (C–O–C), 22.30 ( $\text{CH}_2$ ), 14.69 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{48}\text{H}_{65}\text{N}_3\text{O}_6$  (%): C, 73.35; H, 8.27; N, 5.46. Calc (%), C, 73.91; H, 8.40; N, 5.39.

**4g.** Yield 60%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2990, 2872, 1752 1622, 1580, 1252.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.72 (s, 1H, Uracil-H), 8.83 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.05–8.60 (6d, 12H, Ar–H), 4.19 (t, 2H,  $J = 6.58$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.06 (t, 2H,  $J = 6.67$  Hz,  $-\text{OCH}_2-$ ), 3.96 (t, 2H,  $J = 6.47$  Hz,  $-\text{OCH}_2-$ ), 3.35 (s, 3H,  $-\text{NCH}_3$ ), 3.14 (s, 3H,  $-\text{NCH}_3$ ), 1.79–1.28 (m, 20H, Alkyl  $-\text{CH}_2-$ ), 0.86 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 177.06, 168.20, 165.17 (C=O), 163.79 (C=N), 160.65 (Ar–C–O), 114.98–141.08 (Ar–C), 61.53 (C–O–C), 22.03 ( $\text{CH}_2$ ), 14.85 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{40}\text{H}_{49}\text{N}_3\text{O}_6$  (%): C, 71.80; H, 7.51; N, 6.19. Calc (%), C, 71.94; H, 7.40; N, 6.29.

**4h.** Yield 66%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2986, 2867, 1750 1620, 1582, 1254.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.81 (s, 1H, Uracil-H), 8.80 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.03–8.57 (6d, 12H, Ar–H), 4.18 (t, 2H,  $J = 6.77$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.01 (t, 2H,  $J = 6.08$  Hz,  $-\text{OCH}_2-$ ), 3.93 (t, 2H,  $J = 6.70$  Hz,  $-\text{OCH}_2-$ ), 3.38 (s, 3H,  $-\text{NCH}_3$ ), 3.15 (s, 3H,  $-\text{NCH}_3$ ), 1.73–1.20 (m, 24H, Alkyl  $-\text{CH}_2-$ ), 0.89 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 176.60, 167.69, 165.50 (C=O), 162.68 (C=N), 161.08 (Ar–C–O), 114.40–140.38 (Ar–C), 61.08 (C–O–C), 22.62 ( $\text{CH}_2$ ), 14.74 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{42}\text{H}_{53}\text{N}_3\text{O}_6$  (%): C, 72.53; H, 7.79; N, 6.20. Calc (%), C, 72.49; H, 7.68; N, 6.04.

**4i.** Yield 70%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2980, 2860, 1755 1624, 1580, 1250.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.86 (s, 1H, Uracil-H), 8.77 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.06–8.61 (6d, 12H, Ar–H), 4.17 (t, 2H,  $J = 6.80$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.07 (t, 2H,  $J = 6.77$  Hz,  $-\text{OCH}_2-$ ), 3.96 (t, 2H,  $J = 6.19$  Hz,  $-\text{OCH}_2-$ ), 3.32 (s, 3H,  $-\text{NCH}_3$ ), 3.10 (s, 3H,  $-\text{NCH}_3$ ), 1.74–1.28 (m, 28H, Alkyl  $-\text{CH}_2-$ ), 0.90 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 177.20, 167.19, 165.88 (C=O), 161.16 (C=N), 160.50 (Ar–C–O), 114.00–140.06 (Ar–C), 61.38 (C–O–C), 21.59 ( $\text{CH}_2$ ), 15.36 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{44}\text{H}_{57}\text{N}_3\text{O}_6$  (%): C, 73.12; H, 7.83; N, 5.95. Calc (%), C, 73.00; H, 7.94; N, 5.80.

**4j.** Yield 70%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2988, 2874, 1760 1626, 1584, 1253.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.83 (s, 1H, Uracil-H), 8.80 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.03–8.58 (6d, 12H, Ar–H), 4.14 (t, 2H,  $J = 6.94$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.02 (t, 2H,  $J = 6.18$  Hz,  $-\text{OCH}_2-$ ), 3.94 (t, 2H,  $J = 6.70$  Hz,  $-\text{OCH}_2-$ ), 3.37 (s, 3H,  $-\text{NCH}_3$ ), 3.16 (s, 3H,  $-\text{NCH}_3$ ), 1.79–1.23 (m, 32H, Alkyl  $-\text{CH}_2-$ ), 0.88 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 176.60, 168.05, 166.49 (C=O), 162.59 (C=N), 161.40 (Ar–C–O), 114.89–140.76 (Ar–C), 61.94 (C–O–C), 22.67 ( $\text{CH}_2$ ), 14.80 ( $\text{CH}_3$ ). Elemental analysis found for  $\text{C}_{46}\text{H}_{61}\text{N}_3\text{O}_6$  (%): C, 73.28; H, 8.29; N, 5.37. Calc (%), C, 73.47; H, 8.18; N, 5.59.

**4k.** Yield 67%. IR:  $\nu_{\text{max}}$  (KBr,  $\text{cm}^{-1}$ ): 2979, 2870, 1756 1622, 1580, 1251.  $^1\text{H}$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.85 (s, 1H, Uracil-H), 8.72 (s, 1H,  $-\text{N}=\text{CH}$ ), 7.00–8.40 (6d, 12H, Ar–H), 4.18 (t, 2H,  $J = 6.64$  Hz,  $-\text{COO}-\text{CH}_2-$ ), 4.08 (t, 2H,  $J = 6.59$  Hz,  $-\text{OCH}_2-$ ), 3.90 (t, 2H,  $J = 6.19$  Hz,  $-\text{OCH}_2-$ ), 3.33 (s, 3H,  $-\text{NCH}_3$ ), 3.18 (s, 3H,  $-\text{NCH}_3$ ), 1.75–1.20 (m, 36H, Alkyl  $-\text{CH}_2-$ ), 0.86 (t, 3H, alkyl  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR  $\delta$  (ppm): 177.20, 168.56, 165.03 (C=O), 162.48 (C=N), 161.00 (Ar–C–O), 114.12–140.07 (Ar–C), 61.60 (C–O–C), 22.15 ( $\text{CH}_2$ ), 14.27 ( $\text{CH}_3$ ). Elemental analysis

found for  $C_{48}H_{65}N_3O_6$  (%): C, 73.82; H, 8.56; N, 5.46. Calc (%), C, 73.91; H, 8.40; N, 5.39.

**4l.** Yield 61%. IR:  $\nu_{\max}$  (KBr,  $cm^{-1}$ ): 2983, 2868, 1752 1625, 1583, 1255.  $^1H$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.87 (s, 1H, Uracil-H), 8.78 (s, 1H,  $-N=CH$ ), 7.03–8.54 (6d, 12H, Ar–H), 4.12 (t, 2H,  $J = 6.09$  Hz,  $-COO-CH_2-$ ), 4.07 (t, 2H,  $J = 6.48$  Hz,  $-OCH_2-$ ), 3.96 (t, 2H,  $J = 6.53$  Hz,  $-OCH_2-$ ), 3.32 (s, 3H,  $-NCH_3$ ), 3.15 (s, 3H,  $-NCH_3$ ), 1.76–1.22 (m, 40H, Alkyl  $-CH_2-$ ), 0.89 (t, 3H, alkyl  $CH_3$ ).  $^{13}C$  NMR  $\delta$  (ppm): 176.56, 167.49, 164.69 (C=O), 161.10 (C=N), 160.78 (Ar–C–O), 114.63–141.26 (Ar–C), 61.47 (C–O–C), 22.00 (CH<sub>2</sub>), 15.08 (CH<sub>3</sub>). Elemental analysis found for  $C_{50}H_{69}N_3O_6$  (%): C, 74.23; H, 8.72; N, 5.11. Calc (%), C, 74.31; H, 8.61; N, 5.20.

**4m.** Yield 74%. IR:  $\nu_{\max}$  (KBr,  $cm^{-1}$ ): 2989, 2873, 1756 1624, 1580, 1253.  $^1H$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.86 (s, 1H, Uracil-H), 8.74 (s, 1H,  $-N=CH$ ), 7.00–8.60 (6d, 12H, Ar–H), 4.19 (t, 2H,  $J = 6.41$  Hz,  $-COO-CH_2-$ ), 4.04 (t, 2H,  $J = 6.39$  Hz,  $-OCH_2-$ ), 3.90 (t, 2H,  $J = 6.07$  Hz,  $-OCH_2-$ ), 3.39 (s, 3H,  $-NCH_3$ ), 3.16 (s, 3H,  $-NCH_3$ ), 1.77–1.26 (m, 24H, Alkyl  $-CH_2-$ ), 0.90 (t, 3H, alkyl  $CH_3$ ).  $^{13}C$  NMR  $\delta$  (ppm): 177.07, 166.27, 164.06 (C=O), 162.97 (C=N), 161.43 (Ar–C–O), 115.39–140.44 (Ar–C), 62.04 (C–O–C), 22.60 (CH<sub>2</sub>), 14.76 (CH<sub>3</sub>). Elemental analysis found for  $C_{42}H_{53}N_3O_6$  (%): C, 72.30; H, 7.54; N, 6.21. Calc (%), C, 72.49; H, 7.68; N, 6.04.

**4n.** Yield 70%. IR:  $\nu_{\max}$  (KBr,  $cm^{-1}$ ): 2984, 2870, 1752 1622, 1586, 1250.  $^1H$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.89 (s, 1H, Uracil-H), 8.80 (s, 1H,  $-N=CH$ ), 7.08–8.66 (6d, 12H, Ar–H), 4.11 (t, 2H,  $J = 6.73$  Hz,  $-COO-CH_2-$ ), 4.08 (t, 2H,  $J = 6.04$  Hz,  $-OCH_2-$ ), 3.97 (t, 2H,  $J = 6.29$  Hz,  $-OCH_2-$ ), 3.32 (s, 3H,  $-NCH_3$ ), 3.11 (s, 3H,  $-NCH_3$ ), 1.79–1.28 (m, 28H, Alkyl  $-CH_2-$ ), 0.88 (t, 3H, alkyl  $CH_3$ ).  $^{13}C$  NMR  $\delta$  (ppm): 176.83, 165.49, 163.67 (C=O), 162.18 (C=N), 161.20 (Ar–C–O), 114.89–140.62 (Ar–C), 61.78 (C–O–C), 22.37 (CH<sub>2</sub>), 14.08 (CH<sub>3</sub>). Elemental analysis found for  $C_{44}H_{57}N_3O_6$  (%): C, 73.18; H, 7.83; N, 5.61. Calc (%), C, 73.00; H, 7.94; N, 5.80.

**4o.** Yield 63%. IR:  $\nu_{\max}$  (KBr,  $cm^{-1}$ ): 2989, 2876, 1750 1624, 1580, 1253.  $^1H$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.85 (s, 1H, Uracil-H), 8.84 (s, 1H,  $-N=CH$ ), 7.01–8.60 (6d, 12H, Ar–H), 4.20 (t, 2H,  $J = 6.95$  Hz,  $-COO-CH_2-$ ), 4.01 (t, 2H,  $J = 6.88$  Hz,  $-OCH_2-$ ), 3.96 (t, 2H,  $J = 6.73$  Hz,  $-OCH_2-$ ), 3.39 (s, 3H,  $-NCH_3$ ), 3.14 (s, 3H,  $-NCH_3$ ), 1.81–1.29 (m, 32H, Alkyl  $-CH_2-$ ), 0.86 (t, 3H, alkyl  $CH_3$ ).  $^{13}C$  NMR  $\delta$  (ppm): 178.30, 165.19, 164.69 (C=O), 162.68 (C=N), 161.20 (Ar–C–O), 114.30–141.59 (Ar–C), 62.20 (C–O–C), 22.06 (CH<sub>2</sub>), 15.11 (CH<sub>3</sub>). Elemental analysis found for  $C_{46}H_{61}N_3O_6$  (%): C, 73.58; H, 8.30; N, 5.70. Calc (%), C, 73.47; H, 8.18; N, 5.59.

**4p.** Yield 75%. IR:  $\nu_{\max}$  (KBr,  $cm^{-1}$ ): 2980, 2884, 1760 1623, 1582, 1251.  $^1H$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.70 (s, 1H, Uracil-H), 8.81 (s, 1H,  $-N=CH$ ), 7.12–8.30 (6d, 12H, Ar–H), 4.12 (t, 2H,  $J = 6.40$  Hz,  $-COO-$

$CH_2-$ ), 4.07 (t, 2H,  $J = 6.20$  Hz,  $-OCH_2-$ ), 3.92 (t, 2H,  $J = 6.07$  Hz,  $-OCH_2-$ ), 3.32 (s, 3H,  $-NCH_3$ ), 3.11 (s, 3H,  $-NCH_3$ ), 1.82–1.24 (m, 36H, Alkyl  $-CH_2-$ ), 0.85 (t, 3H, alkyl  $CH_3$ ).  $^{13}C$  NMR  $\delta$  (ppm): 176.70, 164.42, 163.17 (C=O), 162.06 (C=N), 160.11 (Ar–C–O), 114.27–141.60 (Ar–C), 61.59 (C–O–C), 22.30 (CH<sub>2</sub>), 14.89 (CH<sub>3</sub>). Elemental analysis found for  $C_{48}H_{65}N_3O_6$  (%): C, 73.76; H, 8.53; N, 5.22. Calc (%), C, 73.91; H, 8.40; N, 5.39.

**4q.** Yield 63%. IR:  $\nu_{\max}$  (KBr,  $cm^{-1}$ ): 2983, 2873, 1753 1620, 1577, 1253.  $^1H$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.76 (s, 1H, Uracil-H), 8.86 (s, 1H,  $-N=CH$ ), 7.10–8.42 (6d, 12H, Ar–H), 4.13 (t, 2H,  $J = 6.63$  Hz,  $-COO-CH_2-$ ), 4.09 (t, 2H,  $J = 6.86$  Hz,  $-OCH_2-$ ), 3.90 (t, 2H,  $J = 6.69$  Hz,  $-OCH_2-$ ), 3.37 (s, 3H,  $-NCH_3$ ), 3.10 (s, 3H,  $-NCH_3$ ), 1.79–1.22 (m, 40H, Alkyl  $-CH_2-$ ), 0.89 (t, 3H, alkyl  $CH_3$ ).  $^{13}C$  NMR  $\delta$  (ppm): 178.05, 166.10, 164.89 (C=O), 162.58 (C=N), 161.28 (Ar–C–O), 115.00–140.29 (Ar–C), 62.72 (C–O–C), 22.52 (CH<sub>2</sub>), 15.08 (CH<sub>3</sub>). Elemental analysis found for  $C_{50}H_{69}N_3O_6$  (%): C, 74.16; H, 8.77; N, 5.09. Calc (%), C, 74.31; H, 8.61; N, 5.20.

**4r.** Yield 69%. IR:  $\nu_{\max}$  (KBr,  $cm^{-1}$ ): 2990, 2887, 1760 1623, 1582, 1255.  $^1H$  NMR  $\delta$  (ppm, DMSO- $d_6$ ): 9.84 (s, 1H, Uracil-H), 8.89 (s, 1H,  $-N=CH$ ), 7.14–8.64 (6d, 12H, Ar–H), 4.20 (t, 2H,  $J = 6.06$  Hz,  $-COO-CH_2-$ ), 4.10 (t, 2H,  $J = 6.59$  Hz,  $-OCH_2-$ ), 3.95 (t, 2H,  $J = 6.20$  Hz,  $-OCH_2-$ ), 3.32 (s, 3H,  $-NCH_3$ ), 3.19 (s, 3H,  $-NCH_3$ ), 1.80–1.20 (m, 44H, Alkyl  $-CH_2-$ ), 0.90 (t, 3H, alkyl  $CH_3$ ).  $^{13}C$  NMR  $\delta$  (ppm): 176.30, 165.70, 163.04 (C=O), 162.12 (C=N), 160.86 (Ar–C–O), 114.30–141.35 (Ar–C), 61.00 (C–O–C), 22.73 (CH<sub>2</sub>), 14.29 (CH<sub>3</sub>). Elemental analysis found for  $C_{52}H_{73}N_3O_6$  (%): C, 74.80; H, 8.66; N, 5.20. Calc (%), C, 74.69; H, 8.80; N, 5.03.

### 3. Results and discussion

#### 3.1. Phase transition and mesomorphic behaviour

The second heating and cooling transition temperatures and enthalpies for the title compounds were determined by DSC. The mesophases are identified according to the textures observed through POM and the classification system reported by Dierking [28] and Gray/Goodby et al. [29]. The phase transitions and associated enthalpy value changes data of the title compounds **4a–r** are summarized in Table 1. All the compounds **4a–r** formed liquid crystalline phases regardless of the length of the alkoxy spacer or the terminal alkyl chain. However, we find that the type of mesophase is dependent on the length of the spacer chain. The melting points of the all compounds increased as the length of the alkoxy spacer chains and terminal chains increased.

**Table 1**  
Transition temperature  $^{\circ}C$  (corresponding enthalpy changes in  $KJ/mol^{-1}$ ) on second heating/cooling scans for the final compounds **4a–r**.

| No        | n/R   | Heating scan                        | Cooling scan                          |
|-----------|-------|-------------------------------------|---------------------------------------|
| <b>4a</b> | 6/6   | Cr 106.2 (6.45) SmA 124.7 (60.12) I | I 120.1 (–7.33) SmA 101.2 (–59.67) Cr |
| <b>4b</b> | 6/8   | Cr 114.6 (3.78) SmA 132.4 (47.37) I | I 121.2 (–5.77) SmA 98.5 (–30.78) Cr  |
| <b>4c</b> | 6/10  | Cr 116 (6.23) SmA 135.6 (80.19) I   | I 129.3 (–2.89) SmA 101.1 (–44.15) Cr |
| <b>4d</b> | 6/12  | Cr 128.1 (4.30) SmA 150.1 (63.46) I | I 144.1 (–5.58) SmA110.3 (–65.20) Cr  |
| <b>4e</b> | 6/14  | Cr 132.9 (2.11) SmA 158 (80.72) I   | I 154.1 (–7.20) SmA 119.5 (–50.29) Cr |
| <b>4f</b> | 6/16  | Cr 144.3 (3.70) SmA 166.5 (50.27) I | I 160.2 (–4.09) SmA 128.1 (–20.57) Cr |
| <b>4g</b> | 8/6   | Cr 114.2 (9.03) N 130.9 (33.41) I   | I 123.8 (–1.69) N 102.7 (–24.48) Cr   |
| <b>4h</b> | 8/8   | Cr 118.3 (1.07) N 142.7 (31.30) I   | I 136 (–2.31) N 111.3 (–18.06) Cr     |
| <b>4i</b> | 8/10  | Cr 127 (1.15) N 149.7 (19.55) I     | I 140 (–1.60) N 117.3 (–30.21) Cr     |
| <b>4j</b> | 8/12  | Cr 130.9 (2.20) N 155.3 (14.87) I   | I 148.1 (–1.39) N 123.6 (–21.00) Cr   |
| <b>4k</b> | 8/14  | Cr 133.9 (3.60) N 164 (18.30) I     | I 158.1 (–2.66) N 130.2 (–29.11) Cr   |
| <b>4l</b> | 8/16  | Cr 140.6 (1.30) N 170.3 (28.09) I   | I 164.4 (–1.06) N 131.2 (–14.20) Cr   |
| <b>4m</b> | 10/6  | Cr 125.1 (3.37) N 150.4 (19.90) I   | I 143.1 (–4.39) N 122.1 (–19.47) Cr   |
| <b>4n</b> | 10/8  | Cr 129.4 (6.70) N 158.8 (24.67) I   | I 146.6 (–3.59) N 124.3 (–27.03) Cr   |
| <b>4o</b> | 10/10 | Cr 136.8 (1.56) N 171.4 (16.30) I   | I 166.2 (–7.23) N 142.1 (–20.55) Cr   |
| <b>4p</b> | 10/12 | Cr 140 (1.39) N 187 (31.15) I       | I 172.3 (–4.30) N 137.1 (–17.93) Cr   |
| <b>4q</b> | 10/14 | Cr 154.3 (2.47) N 184.6 (22.50) I   | I 177.6 (–1.76) N 142.6 (–23.18) Cr   |
| <b>4r</b> | 10/16 | Cr 161.3 (1.50) N 196.1 (15.60) I   | I 190 (–2.04) N 158 (–14.67) Cr       |

Note: Cr = crystal; SmA = smectic A phase; N = nematic phase; I = isotropic phase.

Compounds **4a–f** with spacer chain ( $n = 6$ ) with varied terminal alkyl chains ( $n = 6, 8, 10, 12, 14$  and  $16$ ) show enantiotropic liquid crystalline SmA phase. The DSC thermograms of compounds **4a–f** exhibited Cr-to-SmA and SmA-to-I transitions typically in heating and cooling cycles. The liquid crystalline texture was observed under crossed polarizers and demonstrated the formation of batonnets initially. After a few minutes, this enlarged and eventually merged to yield a classic focal conic fan shaped texture of SmA phase on cooling for compounds **4a–f**.

A typical example is illustrated for the compound **4a**, which melts from crystal to smectic A mesophase at  $106.2\text{ }^{\circ}\text{C}$  ( $\Delta H = 6.45$ ). With followed heating, it went to isotropic state at  $124.7\text{ }^{\circ}\text{C}$  ( $\Delta H = 60.12$ ). Again, when it was cooled down, it changed from the isotropic state and started to appear with batonnets texture. It became completely focal-conic texture at  $120.1\text{ }^{\circ}\text{C}$  ( $\Delta H = -7.33$ ), and finally transformed back to crystalline state at  $101.2\text{ }^{\circ}\text{C}$  ( $\Delta H = -59.67$ ). Fig. 1(a) shows a representative optical photomicrograph of compound **4a** exhibiting focal conic fan-shaped texture during heating cycle.

Similar transition behaviors with existence of fan-shaped textures were observed for the remaining compounds **4b–f**. All the observed liquid crystalline textures are typical according to reports [25,26].

Compounds **4g–l** with spacer ( $n = 8$ ) with varied terminal alkyl chains exhibit enantiotropic nematogenic properties. The DSC thermogram of **4g–l** reveals the Cr-N-I transitions on heating, and I-N-Cr transitions on cooling scan. The nematic to isotropic transitions are increased with increase in the terminal chains. The temperature ranges of mesophase were found to be 16.75, 24.39, 22.86, 24.3, 30.13 and  $29.72\text{ }^{\circ}\text{C}$  for **4g–l** respectively. The clearing temperatures of the compounds **4g–l**, increased smoothly as the chain length increases. The POM also reveals the N-I phase transition upon the heating run for compounds **4g–l**, whereby the POM shows only a Schlieren texture nematic phase.

Compounds **4m–r** possess a little longer spacer chain ( $n = 10$ ) with terminal chains of even parity numbers from 6 to 18. The representative DSC thermogram of compound **4p** is given in Fig. 2. It shows phase transitions of Cr-N-I and I-N-Cr on heating and cooling scans. The texture observed for **4p** compound upon the heating scan shown in Fig. 1(b) with Schlieren texture for nematic phase. Similar phase transition and texture observations were found for the whole set of compounds **4m–r** suggesting that the mesophase is same for the homologous series of compounds **4g–l** and **4m–r**. Moreover, compounds **4m–r** exhibited a nematic phase on cooling from the isotropic state, whereby nematic droplets are seen to form and gradually fuse together to form the Schlieren texture. The nematic phase appears to have high mobility with flashes when subjected to mechanical stress [25].

It may be noted here that generally in dimeric compounds, transition temperatures follows pronounced odd-even effect. Odd number spacer groups have low melting transition temperatures and even number

spacer groups have high melting transition temperatures. This behaviour is most commonly attributed to the pronounced dependence on the molecular shape, length and parity of the spacer. Such behaviour has been accounted by theory, by means of the different shapes of the conformers having odd or even membered spacers and their associated conformational distributions [5–7].

In our work and the present series of dimmers, the odd-even effect is eliminated and the values of the Cr-N and Iso-N entropies are irregular for some compounds. This is presumably due to the rather bulky shape of the dimethyluracil group and the increased molecular biaxiality, which has been used to account for irregular entropies. The phase behaviour of compounds **4g–r** is unusual. The conventional behaviour in liquid crystal dimers is smectic behaviour to be favoured on increasing the spacer length. Since in the present series of compounds nematic phase is favoured in higher methylene spacer containing members this behaviour is unexpected [8–9].

A plot of the cooling transition temperatures as a function of the number of carbon atoms in the connecting spacer and terminal  $n$ -alkoxy chain for the compounds **4a–f**, **4g–l** and **4m–r** is shown in Fig. 3(a), (b) and (c). It can be seen that the clearing temperature curve gradually increases with increasing in the number of carbon atoms in the terminal alkyl chain for the sets of **4a–f**, **4g–l** and **4m–r** homologous series of compounds.

From this we deduce that the type of phase depends on the length of spacer alkoxy chains and not on the length of the terminal chains. In shorter spacer chain members ( $n = 6$ ) the SmA is observed regardless the length of terminal alkyl chain. As the length of the spacer chain increases to  $n = 8$  and  $10$ , the nematic (N) phase is observed as in the compounds **4g–r**. The nematogenic behaviour between compounds **4g–r**, suggests that when the spacer carbon chains reach a certain length ( $n \geq 8$ ) the nematic (N) phase at the higher temperatures, is induced [25]. On the other hand, as mentioned above, the melting points increase when the number of carbons in terminal alkoxy chains increases. This may be due to the excessive Van der Waals attractive forces between the terminal alkyl chains or between the alkoxy chains where the melting temperatures obviously increase from  $n = 6$  to  $18$  [33].

### 3.2. Molecular modeling

In order to get a better understanding of the relationship between the structure and type of phase, molecular modeling studies have been carried out by HyperChem program. The variation of length at the terminal alkyl chain did not change the type of phase. Surprisingly, the type of phase is mainly depending on the length of spacer alkoxy chain. Molecular models of compounds **4a**, **4g** and **4r** are given in Fig. 4, in which the length of spacer alkoxy chain varied from  $n = 6, 8$  and

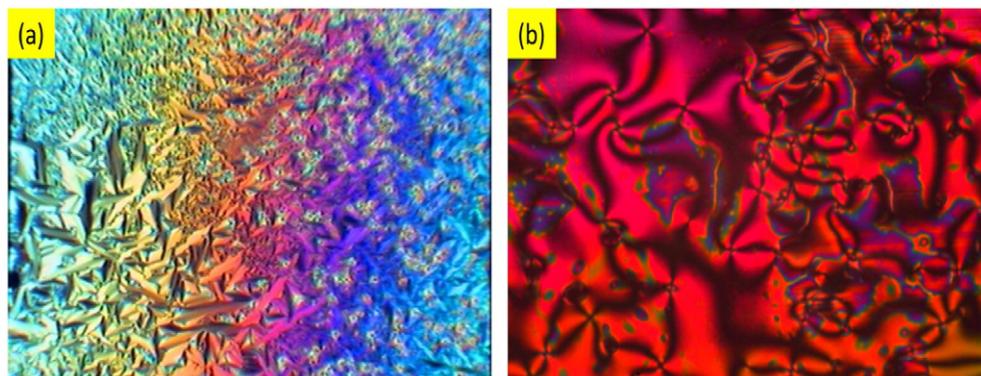


Fig. 1. Optical photomicrographs of (a) SmA phase shown by **4a** at  $117\text{ }^{\circ}\text{C}$  upon heating; (b) Nematic phase shown by **4p** at  $170\text{ }^{\circ}\text{C}$  upon heating.

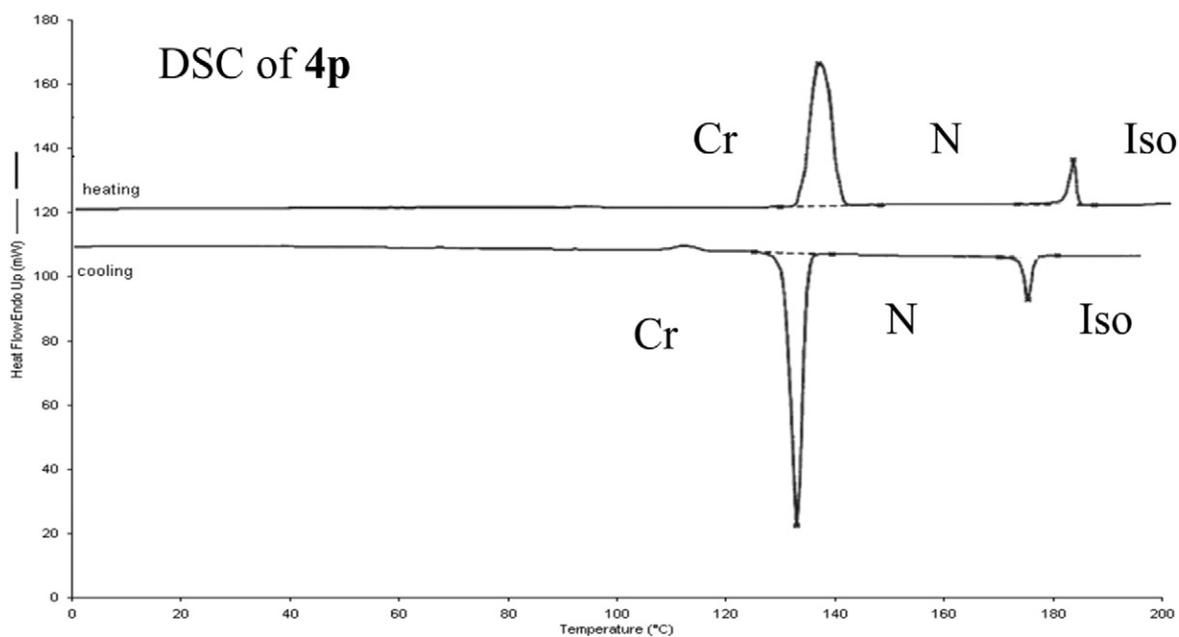


Fig. 2. DSC thermogram (Endothermic/Exothermic scans) of compound 4p.

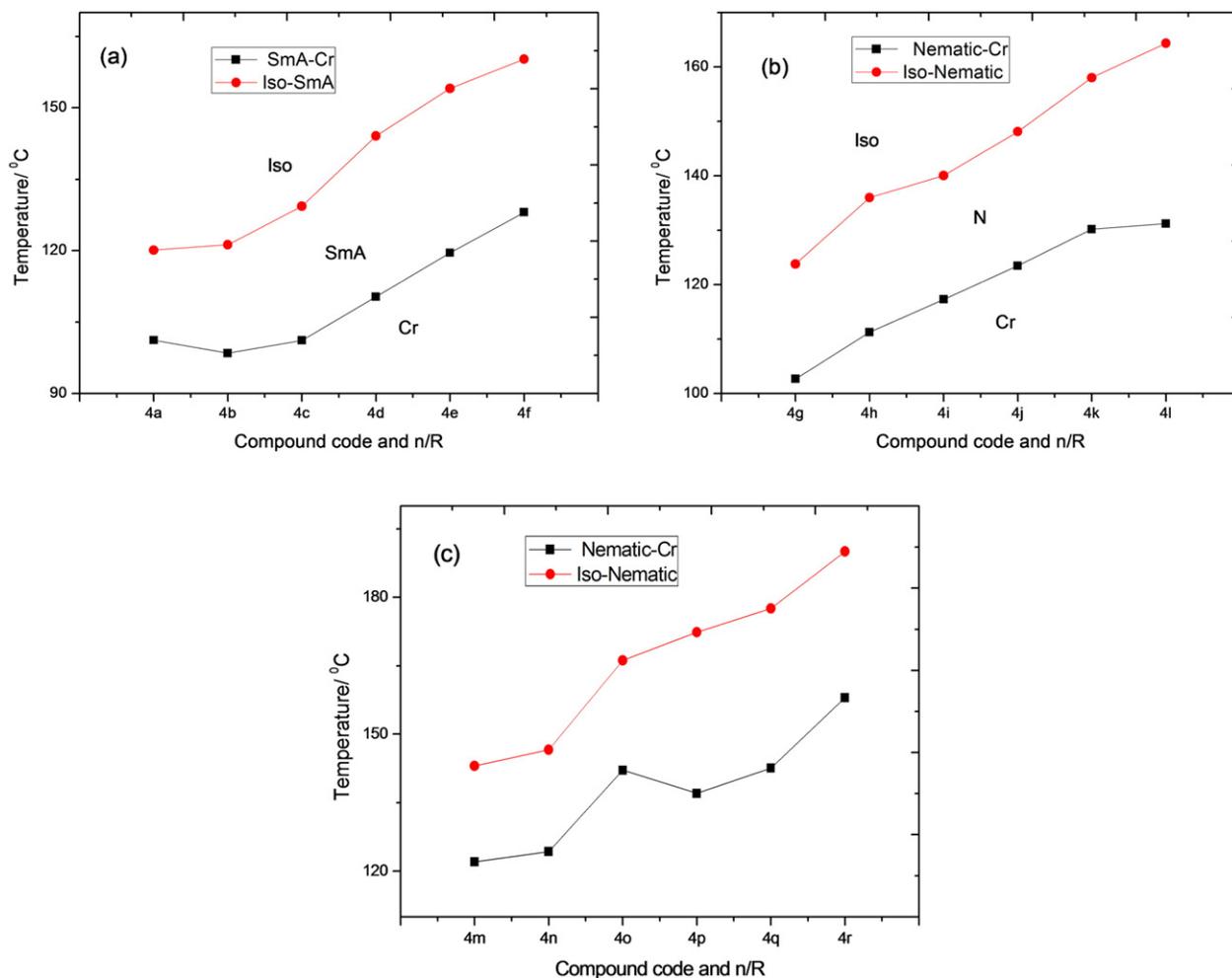


Fig. 3. A plot of cooling transition temperature as a function of the number of carbon atoms in the methylene spacer and terminal chains for compounds (a) 4a–f, (b) 4g–l and (c) 4m–r.

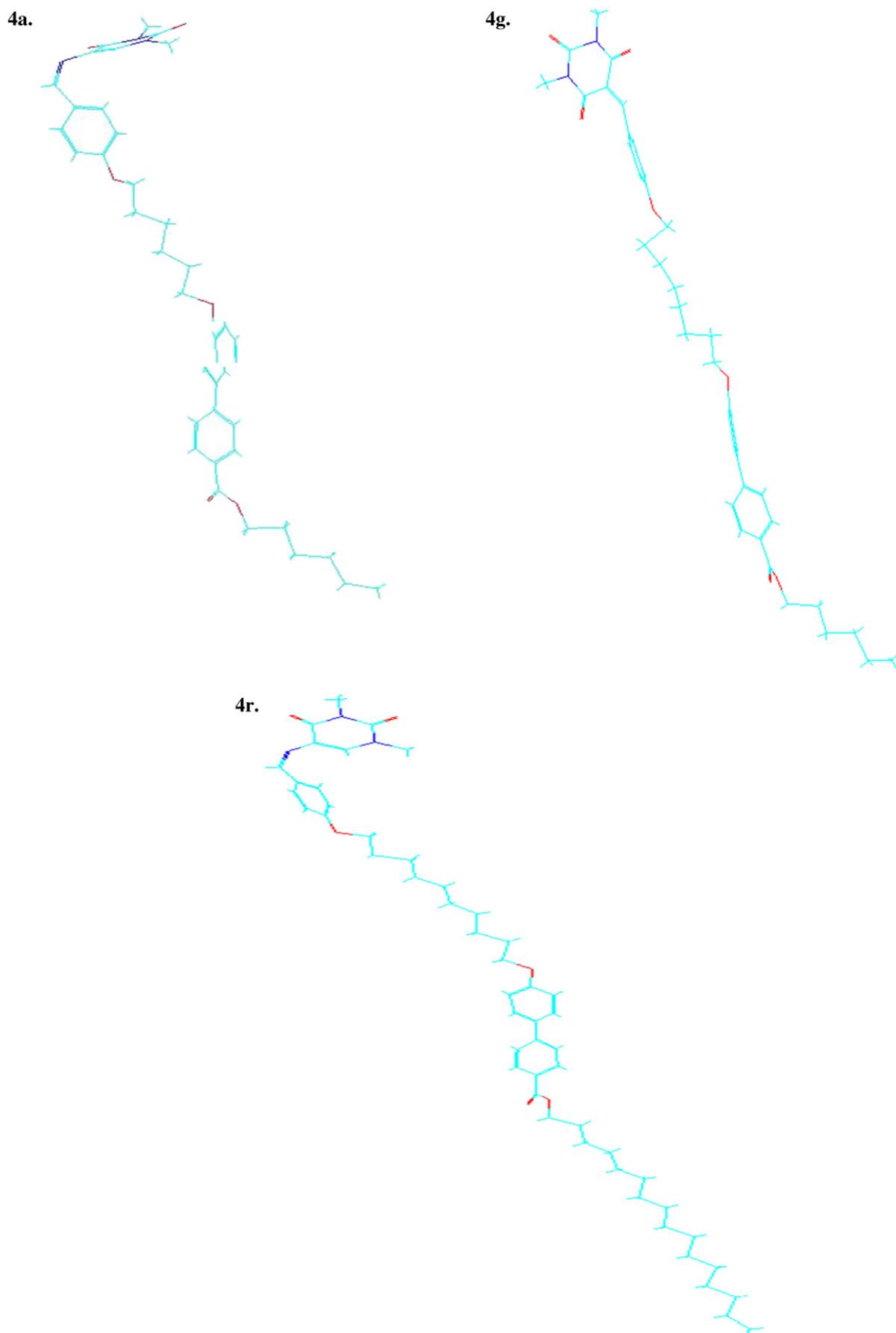


Fig. 4. Molecular models of compound 4a, 4g and 4r using HyperChem program.

10, respectively. The data are also in agreement with the experimental results of title compounds **4a–r**.

As shown in Fig. 4, the 1,3-dimethyluracil ring and the two phenyl rings (or biphenyl moiety) which is adjacent to the spacer appear at different positions depending on the number of carbons at the alkoxy spacer [34–36]. The models indicate that 1,3-dimethyluracil and biphenyl core groups take at different side where the number of carbons at spacer change from  $n = 6$  to 10. Non-planar geometry of 1,3-dimethyluracil and biphenyl rings was observed in compound **4a** and this geometry tends to exhibit smectic A. However, a more planar geometry of 1,3-dimethyluracil and biphenyl was found in compounds **4g** and **4r** and this molecular conformation favoured arrangement of a nematic phase.

#### 4. Conclusion

In this paper the synthesis, characterization, mesomorphic and theoretical study of a new dimeric liquid crystalline compounds with 1,3-dimethyluracil and biphenyl core systems have been presented. All synthesized compounds exhibit liquid crystal properties and the smectic A phase is observed when the spacer alkoxy chain  $n = 6$  and terminal chain are even parity numbers from 6 to 18. However, for longer spacer chain ( $n = 8$  and 10), it turned to nematogenic materials without exhibition of smectic A regardless of the length of alkyl terminal chain.

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