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Liquid Crystals

Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t713926090

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Online publication date: 14 December 2009

To cite this Article Kim, Jinsoo, Jung, Young Ju, Ka, Jae-Won, Kim, Eun Chol, Ahn, Taek and Yi, Mi Hye(2009) 'Mesomorphic phase transition behaviour of photopolymerisable liquid crystalline triphenylene ether compounds', Liquid Crystals, 36: 12, 1451 – 1457

To link to this Article: DOI: 10.1080/02678290903295735 URL: http://dx.doi.org/10.1080/02678290903295735

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Mesomorphic phase transition behaviour of photopolymerisable liquid crystalline triphenylene ether compounds

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(Received 29 July 2009; final version received 28 August 2009)

This report discusses the preparation and the unusual mesomorphism of three homologues of photopolymerisable triphenylene ether compounds. These homologues showed 'a cold crystallisation' on heating differential scanning calorimetry curves, and this phenomenon is attenuated with increase methylene units, spacers between the triphenylene core and the terminal photopolymerisable acrylate group. Classical textures of hexagonal columnar (Col_h) mesophase were observed by polarising optical microscopy for all three photopolymerisable mesophase compounds described here. Also described is the mesomorphism of their intermediates, hydroxylalkoxytriphenylenes. In some cases, the discotic columnar hexagonal mesophases were confirmed by wide angle X-ray scattering techniques.

Keywords: mesomorphism; photopolymerisable triphenylene ether compound; cold crystallisation; hexagonal columnar mesophase

1. Introduction

Triphenylene-based discotic liquid crystals possessing intrinsic uniaxial negative birefringence show either discotic nematic or discotic columnar phases, depending mainly upon the variation of lateral substituent of the triphenylene core (1-4).

Photopolymerisable triphenylene discotic nematic mesogens with a photo-initiator have been cured to form the stable anisotropic films by the irradiation of ultraviolet light (5–6) and used commercially in phase compensation films to improve the viewing angle of liquid crystal display devices (6–11). In addition, columnar arrangement of triphenylene discotic mesogens has been known to provide anisotropic materials with potential applications such as field effect transistors and photovoltaic devices due to their extraordinary one-dimensional electrical conductivity (12–17).

Considering the practical use of the photopolymerisable discotic mesogens, a wide mesophase range and stable supramolecular assembly of mesophase should be satisfied. Although there are reports of many homologous series of non-reactive triphenylene discotic liquid crystals (18-21), very little is known about the corresponding reactive discotic mesogens (22-26).

In this report, we describe the synthesis and mesophase behaviour of a homologous series of photopolymerisable acrylate triphenylene ether compounds **2a-2c** as well as their intermediates, hydroxyalkoxytriphenylenes **1a-1c** (Scheme 1). Also discussed is the unusual phase transition behaviour of the compounds

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2a–2c in comparison with corresponding alkyloxytriphenylenes **HAT**-*n*s (n = 6, 8, and 12) (Scheme 1).

2. Experimental

2.1 Characterisation

All reactions were analysed by thin layer chromatography on silica gel ${}^{60}F_{254}$ and column chromatography was performed on a silica gel 60 (Merck, 70–230 mesh). ¹H NMR spectra were taken on a Bruker BZH300/52 spectrometer at 300 MHz. And IR spectra were obtained using a Smith ChemID FTIR spectrometer. The molecular mass results were obtained from a JEOL JMS-DX303 Mass Spectrometer.

Differential scanning calorimetry (DSC) was performed on a Perkin-Elmer Diamond DSC Autosampler. A Nikon Optiphot-POL polarising optical microscope (POM) equipped with an INSTEC HCS402 hot stage and a STC200 temperature controller was used to observe mesophase textures. In order to more closely investigate the mesophase structure, wide angle X-ray scattering (WAXS) measurements were carried out by Bruker D8 Discover X-ray diffractometer equipped with a General Area Detector Diffraction System and a high-temperature stage.

2.2 Synthesis

The materials including 2,3,6,7,10,11-hexahydroxytriphenylene (HHTP), 6-bromo-1-hexanol, 8-bromo-1octanol, 12-bromo-1-dodecanol, and acryloyl chloride



Scheme 1. Chemical structures of a homologous series of triphenylene ether compounds (1a-1c, 2a-2c, and HAT-*n*s) with different terminal groups discussed in this study.



Scheme 2. Synthetic route of a series of photopolymerisable discotic mesogens and their intermediates, $R^1 = C_n H_{2n}OH$; $R^2 = C_n H_{2n}OCOCH = CH_2$, **1a** and **2a**: n = 6; **1b** and **2b**: n = 8; **1c** and **2c**: n = 12: (a) Br(CH₂)_nOH, K₂CO₃, EtOH or DMF, 70°C, (b) TEA, MEHQ, acryloyl chloride, CHCl₃, reflux.

were commercially available and used without further purification.

The compounds 1a-1c and 2a-2c were prepared from the HHTP by previously reported methods shown in Scheme 2 (22–26). A description of the synthesis of compounds 1a-1c and 2a-2c is given.

2.2.1 2,3,6,7,10,11-hexakis(6-hydroxyhexyloxy) triphenylene (**1a**)

HHTP (5.0 g, 15.0 mmol) and K_2CO_3 (51.0 g, 370.0 mmol) were heated at 70°C in ethanol (400 ml). To this solution, was added 6-bromo-1-hexanol (32.5g, 180.0 mmol) dissolved in ethanol (100 ml) dropwise for 1 h and remained at 70°C for 48 h. The resulting mixture was cooled to room temperature and diluted with deionised (DI) water followed by extraction with ethyl acetate. The organic layer was separated and dried with MgSO₄ and the solvent was evaporated in vacuo. The crude product was purified by column chromatography (silica gel, methylene chloride: methanol = 8:1) to afford **1a** (solid, 4.2 g, 30%). ¹H NMR (300 MHz, methanol-d₄) δ 7.36 (ArH, s, 6H),

3.94 (ArOC<u>H₂</u>, t, 12H), 3.48 (C<u>H₂</u>OH, t, 12H), 1.76(ArOCH₂C<u>H₂</u>, quint, 12H), 1.48 (HOCH₂C<u>H₂</u>, quint, 12H), 1.40 ((C<u>H₂</u>)₄, m, 24H); FT-IR (KBr): 3340 (OH) cm⁻¹; MS (FAB) m/z = 924 (M⁺).

Compounds 1b (solid) and 1c (solid), homologues of 1a, were prepared using a similar procedure to 1a, with corresponding ω - bromo-1-alkanol in DMF solution instead of ethanol.

2.2.2 2,3,6,7,10,11-hexakis(6-hydroxyoctyloxy) triphenylene (**1b**)

Yield 20%. ¹H NMR (300MHz, CDCl₃) δ 7.17 (Ar<u>H</u>, s, 6H), 3.93 (ArOC<u>H₂</u>, t, 12H), 3.58 (C<u>H₂OH</u>, t, 12H), 1.84 (ArOCH₂C<u>H₂</u>, quint, 12H), 1.59 (HOCH₂C<u>H₂</u>, quint, 12H), 1.31 ((C<u>H₂</u>)₄, m, 48H); MS (FAB) m/z = 1093 (M⁺).

2.2.3 2,3,6,7,10,11-hexakis(12-hydroxydodecyloxy) triphenylene (**1c**)

Yield 15%. ¹H NMR (300MHz, CDCl₃) δ 7.83(Ar<u>H</u>, s, 6H), 4.22(ArOC<u>H</u>₂, t, 12H), 3.62(C<u>H</u>₂OH, t, 12H),

1.95(ArOCH₂C<u>H₂</u>, quint, 12H), 1.53(HOCH₂C<u>H₂</u>, quint, 12H), 1.38((C<u>H₂</u>)₈, 96H); FT-IR(KBr): 3363(OH) cm⁻¹; MS(FAB) m/z= 1430(M⁺).

2.2.4 2,3,6,7,10,11-hexakis(6-acryloyloxyhexyloxy) triphenylene (**2a**)

To a stirred solution of **1a** (4.0 g, 4.3 mmol), TEA (10.8 ml, 77.8 mmol) and 4-methoxyphenol(MEHQ) 100 ppm at 0°C was added a solution of acryloyl chloride (4.2 ml, 51.8 mmol) in CHCl₃ (50 ml) for 1 h and remained under reflux for 24 h. The resulting mixture was cooled to the room temperature and diluted with DI water followed by extraction with chloroform. The organic layer was separated and dried with MgSO₄ and the solvent was evaporated in vacuo. The crude product was purified by column chromatography (silica gel, hexane: ethyl acetate = 2:1) to afford **2a** (waxy, 1.7 g, 31%).

¹H NMR(300MHz, CDCl₃) δ 7.82 (Ar<u>H</u>, s, 6H<u>)</u>, 6.42 (acryl.<u>H</u> m, 6H), 6.12 (acryl.<u>H</u> m, 6H), 5.82 (acryl.<u>H</u> m, 6H), 4.11 (ArOC<u>H₂</u>, t, 12H), 4.23 (acryl.OC<u>H₂</u>, t, 12H), 1.98 (ArOCH₂C<u>H₂</u> quint, 12H), 1.72 (acryl.OCH₂C<u>H₂</u> quint, 12H), 1.48 ((C<u>H₂</u>)₂, m, 24H); FT-IR (KBr): 1722 (C=O), 1181, 1260 (C–O) cm⁻¹; MS (FAB) m/z = 1248 (M⁺).

Compounds **2b** (waxy) and **2c** (solid) were prepared using a similar procedure to **2a**.

2.2.5 2,3,6,7,10,11-hexakis(8-acryloyloxyoctyloxy) triphenylene (**2b**)

Yield 44%. ¹H NMR (300MHz, CDCl₃) δ 7.84 (Ar<u>H</u>, s, 6H), 6.42 (acryl.<u>H</u> m, 6H), 6.12 (acryl.<u>H</u> m, 6H), 5.81 (acryl.<u>H</u> m, 6H), 4.23 (ArOC<u>H₂</u>, t, 12H), 4.15 (acryl.OC<u>H₂</u>, t, 12H), 1.94 (ArOCH₂C<u>H₂</u>, quint, 12H), 1.64 (acryl.OCH₂C<u>H₂</u>, quint, 12H), 1.54 ((C<u>H₂)</u>₄, m, 48H); FT-IR (KBr): 1722(C=O), 1170, 1256(C–O) cm⁻¹; MS (FAB) m/z = 1417 (M⁺).

2.2.6 2,3,6,7,10,11-hexakis(12-acryloyloxydodecyloxy) triphenylene (**2c**)

Yield 34%. ¹H NMR(300MHz, CDCl₃) δ 7.83 (Ar<u>H</u>, s, 6H), 6.42 (acryl.<u>H</u> m, 6H), 6.12 (acryl.<u>H</u> m, 6H), 5.82 (acryl.<u>H</u> m, 6H), 4.22 (ArOCH₂ t, 12H), 4.14 (acryl.OCH₂ t, 12H), 1.93 (ArOCH₂CH₂ quint, 12H), 1.66 (acryl.OCH₂CH₂ quint, 12H), 1.52 ((CH₂)₈, m, 96H); FT-IR (KBr): 1722 (C=O), 1170, 1256 (C-O) cm⁻¹.

3. Thermal characterisation and mesophase behaviour

The mesophase behaviour of the liquid crystalline compounds was investigated by DSC and POM. In some cases, a WAXS measurement was performed for mesophase compounds aligned at room temperature by cooling from the isotropic liquid phase. Phase transition temperatures, corresponding enthalpy changes and mesophase textures of the synthesised compounds, **1a**-**1c** and **2a**-**2c**, as well as three corresponding non-reactive hexaalkoxytriphenylenes, **HAT**-*n*s, are summarised in Table 1.

There are many reports available regarding the phase transition temperature and occasionally enthalpy changes of non-reactive discotic liquid crystals **HAT-6**, **8** and **12** (*2*, *4*, *18–21*). They were known to have a discotic hexagonal columnar mesophase (Col_h) and their isotropic phase transition temperatures were in the reverse order of an alkyl chain length, **HAT-6**> **HAT-8**> **HAT-12** due to the increasing chain mobility. In this discussion, **HAT-***n* were adopted as references to explain the effect of the

Table 1. Phase transition temperatures (peak temperature) and corresponding enthalpy changes for triphenylene ether compounds.

Compound	End group	Thermal transition (°C) (enthalpy changes (J g^{-1}))	
		Heating scan	Cooling scan
1a	ОН	Cr 42 (34.0) Col _h 87 (3.7) I	I 84 (3.0) Col _h –6 (18.3)Cr
1b		Cr 54 (27.7) I	I 39 (23.9) Cr
1c		Cr ₁ 93 (0.8) Cr ₂ 104 (47.2) I	I 83 (1.0) Col _h 87 (23.2) Cr
2a	OCO-CH=CH ₂	X –22 (2.7) Cr –5 (–31.9) Cr 30 (52.0) I	I –26 (2.6) Col _h
2b		X 11 (15.0) Cr 20 (33.0) I	I –11 (31.0) Col _h
2c		X 33 (132.4) Cr 44 (42.3) I	I 19 (66.7) Col _h
HAT-6 ^a	Н	Cr 68 (42.5) Col _h 97 (4.4) I	n.d. ^c
HAT-8 ^a		Cr 67 (74.0) Col _h 86 (4.4) I	n.d.
HAT-12 ^b		Cr 55 (n.d.) Col and Col 63 (n.d.) I	n.d.

Cr = Crystal, $Col_h = hexagonal$ columnar liquid crystalline phase, Col = columnar phase, X = A yet-to-be-identified phase, and I = isotropic.

^aData available from reference (2).

^bData available from reference (20).

 c n.d. = no data available.

terminal group modification with the acrylate and hydroxyl groups, respectively, on the mesomorphism of triphenylene ether compounds.

3.1 Hexakis(hydroxyalkoxy)triphenylenes (1a-1c)

Among three synthesised hexakis-alkanols, compound 1a with six methylene units in the side chain spacer exhibits a classical thermotropic mesophase transition behaviour on heating DSC curve, melting point at 42°C; the clearing point is at 87°C (Figure 1). Despite the hydrogen bonding effect, the phase transitions of 1a are detected at lower temperatures compared with the reference, HAT-6 (Cr 68°C Col_h100°C I). This observation suggests that intra-molecular hydrogen bonding between neighbouring hydroxyhexyl chains breaks down the inter-digitations of the non-polar alkyl chains, inducing the higher energy state. The slight increment of the mesophase range of compound 1a can be ascribed to the inter-molecular hydrogen bonding effect that stabilises both of the long range orders in inter-columnar and inter-disc triphenylene cores. Figure 2 shows a pseudofocal conic mesophase texture classified as a discotic columnar mesophase. The structure of the mesophase of 1a was investigated by WAXS and two peaks are seen in the low angle region and the d-spacings are in $d_{100}:d_{110} = 1:\frac{1}{\sqrt{3}}$. This is a characteristic reflection for a hexagonal arrangement of molecule (Figure 3). In the wide angle region. **1a** shows broad and sharp reflections. one broad reflection centred at ca. 4.3 Å corresponding to the hydroxy-hexyl hydrocarbon chains and another sharp reflection centred at ca. 3.6 Å corresponding to the stacking of the triphenylene cores.

Compound 1b, with two additional methylene units on each alkyl side chain compared with that of 1a, exhibits only one phase transition, crystal to isotropic liquid on DSC heating run (see Figure 1). With the help of the POM, the phase transition behaviour of



Figure 1. Differential scanning calorimetry curves of compounds 1a, 1b, and 1c (second heating and second cooling at scanning rate of 3° C/min). C and H stand for cooling and heating cycles, respectively.



Figure 2. Mesophase texture of compound 1a at 6°C on cooling from the isotropic liquid (magnification 500×).



Figure 3. Wide angle X-ray scattering analysis at room temperature: 1D profile (a) and 2D pattern (b) of compound **1a**, respectively.



Figure 4. Mesophase texture of compound 1c at 90°C on cooling from the isotropic liquid (magnification 200×).

1b is interpreted as follows: melting at 56° C to form the isotropic liquid followed by a transition into a crystal state at 39° C on a subsequent cooling.

Compound **1c** contains six more methylene units on each alkyl side chain compared with **1a** and exhibits a crystal to another crystal phase transition at 93° C, followed by isotropic melting at 104° C, according to the DSC heating curves. Subsequent cooling from the isotropic liquid phase exhibits a mesophase within a very narrow temperature range, between 87° C and 83° C (Figure 1), and POM shows a dendritically growing texture allowing the assignment of a discotic columnar mesophase (Figure 4).

As a result, hexakis–alkanol intermediates show a shift of the transition temperatures compared with the corresponding non-reactive hexaalkoxytriphenylenes, **HAT-n**s (n = 6, 8, and 12, respectively), but not in a systematic way. The mesophase range is narrowed as methylene units increase, reflecting the increasing flexibility as in the case of **HAT-n**s. In addition, the mesophase textures are not affected by the introduction of terminal hydroxy functional groups.

3.2 Hexakis(acryloyloxyalkoxy)triphenylene (2a-2c)

To our surprise, we observed the exothermic enthalpy changes on heating DSC curves shown in Figure 5 at a scanning rate of 5°C/min. This abnormal exothermic phenomenon could be caused by polymerisation of the reactive acrylate groups (8, 27) or more highly ordered phase transition to crystal, so called 'cold crystallization' (28). In this case, cold crystallisation is more likely because the same DSC curves were observed repeatedly for all three acrylate compounds



Figure 5. Differential scanning calorimetry curves of compounds 2a (a) and 2b and 2c (b) (second heating and second cooling at scanning rate of 5°C/min). A negative phase transition enthalpy change on a heating run is indicated as a circle. C and H stand for cooling and heating cycles, respectively.

2a–2c on repetitive second and third heating runs. Although it is not as obvious as in **2a**, the same phenomenon was also observed in the case of compounds **2b** and **2c**. DSC results are summarised in Table 1 with the help of corresponding POM results (Figures 5–8), although it requires in-depth investigation in order to elucidate more precisely their thermal behaviour. It should be noted that there are phases remained to be determined, denoted as X.

Compound **2a** shows a first phase transition at -22° C, second and third at -5° C and 30° C, respectively, on the second heating DSC scan. The distinctly large amount of heat emission (31.9 J g⁻¹) at -5° C illustrates a more rigid ordering and could be directly associated to the large enthalpy (52.0 J g⁻¹) of the isotropic transition of the compound **2a** at 30° C on heating. It is noteworthy that the liquid crystal phase is seen at -28.4° C on the cooling DSC run from an isotropic liquid phase, maintained below -40° C. It



Figure 6. Mesophase texture of compound 2a at -40° C on cooling from the isotropic liquid (magnification $500 \times$).



Figure 7. Mesophase texture of compound 2b at -20° C on cooling from the isotropic liquid(magnification $200 \times$).

was repeatedly observed on subsequent third heating and cooling DSC scans.

In the case of compounds **2b** and **2c** possessing longer methylene units than **2a**, the phase transition temperatures are increased, presumably due to the strengthening of van der Waals interactions, and the cold crystallisation phenomenon is diminished.

Discotic hexagonal columnar mesophases (Col_h) were identified for all three acrylate compounds based on the mesophase textures observed by POM on cooling



Figure 8. Mesophase texture of compound 2c at $17^{\circ}C$ on cooling from the isotropic liquid (magnification $200 \times$).

from the isotropic phase (Figures 6–8). In the case of compound **2c**, discotic hexagonal columnar mesophase is confirmed by WAXS, revealing two distinct scattering peaks in the small angle range, the *d*-spacings of which accurately match the characteristic ratio of $1:\frac{1}{\sqrt{3}}$ the (100) and (110) reflections in 2D hexagonal columnar mesophase (Figure 9). In the wide angle region, **2c** shows broad and sharp reflections as in the case of **1a**, one broad reflection centred at ca. 4.0 Å corresponding to the acrylate-dodecyl hydrocarbon chains and another sharp reflection centred at ca. 3.5 Å corresponding to the stacking of the triphenylene cores.

It has been expected that the photopolymerisable acrylate groups result in the depression of phase transition temperatures of compounds 2a-2c compared with the non-reactive analogues HAT-*n*s (n = 6, 8 and 12, respectively) (29) and DSC curves of 2a-2c on heating, they agree well with our expectation (see Table 1 for the comparison). In addition, reactive acrylate groups delay the transition to the higher order phase on cooling (super cooling) as shown in Figure 5.

4. Conclusions

Three homologues of photopolymerisable liquid crystalline triphenylene ethers and their intermediates were synthesised. Corresponding non-reactive alkoxytriphenylenes (**HAT**-ns, n = 6, 8 and 10, respectively) were used as references to discuss structure–mesophase behaviour.

DSC measurements and the texture observation by POM showed that photopolymerisable acrylate homologues are thermotropic liquid crystals with a hexagonal columnar (Col_h) mesophase. It is remarkable that



Figure 9. Wide angle X-ray scattering analysis at room temperature: 1D profile (a) and 2D pattern (b) of compound 2c.

hexakis-acrylates of triphenylene ethers exhibit an exothermic phenomenon, 'cold crystallisation', on DSC heating runs. They also show lower mesophase transition temperatures compared with corresponding HAT-ns, as expected. Corresponding intermediates, hexakis-alkanols, exhibit a discotic hexagonal columnar mesomorphism in some cases. As methylene units are increased in flexible side chains, the more ordered states are stabilised in both hexakis-acrylate and hexakis-hydroxy triphenylene ethers.

In conclusion, (i) thermal behaviours of the three homologous photopolymerisable triphenylene ether compounds and their intermediates are affected by both acrylate and alcohol terminal functional groups as well as by the length of methylene units in the side chain spacers; and (ii) the discotic hexagonal columnar mesophase textures are maintained, in comparison with corresponding HAT-ns.

Acknowledgements

This research was supported by a grant from the Fundamental R&D Program for Core Technology of Materials funded by the Ministry of Knowledge Economy, Republic of Korea.

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