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Synthesis and mesomorphism of ether–ester mixed tail C_3-symmetrical truxene discotic liquid crystals

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Truxene discotic liquid crystals are interesting for their polymesomorphism and as organic semiconductor candidates. We developed an efficient synthetic method for C_3-symmetrical truxene derivative 2,7,12-trihydroxy-3,8,13-trimethoxytruxene **5**, and further a series of ether–ester mixed tail truxene discogens **HTn**, $TR(OMe)_3[OOCC_6H_2(OC_nH_{2n+1})_3]_3$ were prepared. The truxene discogens **HTn** (n = 4–8, 10, 12) were characterised by using ^1H and ^{13}C nuclear magnetic resonance, and high-resolution mass spectrometry. Their thermal stability was studied with thermal gravimetric analysis and their liquid crystal properties were investigated by using polarising optical microscopy, differential scanning calorimetry and X-ray diffraction. They exhibit a hexagonal columnar (Col_h) mesophase in fairly wide temperature ranges.

Keywords: truxene; discotic liquid crystal; columnar mesophase; mixed tail discogen

1. Introduction

Since the first discovery of discotic liquid crystal mesophase in 1977, while investigating the optical and X-ray features of hexasubstituted benzene [1], the discotic liquid crystals of triphenylene [2, 3], phthalocyanine [4], hexabenzocoronene [5], porphyrine [6] and perylene [7, 8] have been successively reported. Discotic liquid crystals can self-assemble to be highly ordered hexagonal columnar (Col_{ho}) mesophases and exhibit fast mobility of charges and energies (9–14). As such, they have been studied as photoconductors (9–16), active materials in light emitting diodes [17], charge transport materials of photovoltaic cells [18], ion transport materials [19], gas sensor materials [20], optical materials of display devices [21, 22], and have shown huge potential industrial applications.

Truxene derivatives have been studied because of their unusual molecular structures and potential industrial application as active chemical components in a wide range of electronic and optoelectronic devices [23]. Recently, series of truxene dimers, oligomers and polymers have been synthesised (24–26). The hexasubstituted truxene derivatives **1** in Figure 1 from the literature are rich in mesophases [27]. Hexa-*n*-alkoxytruxenes **1a** exhibit only a Col_{ho} mesophase in fairly broad temperature ranges [28], whereas the hexa-*n*-alkanoyloxytruxenes **1b** [29] show an inverted mesophase sequence of $K\rightarrow N_D\rightarrow Col_h\rightarrow I$, while the familiar mesophase sequence is $K\rightarrow Col_h\rightarrow N_D\rightarrow I$. Furthermore, the hexa-*n*-alkoxybenzoyloxytruxenes **1c** display the reentrant N_D and Col_r mesophases, for example, to n = 12, $K\rightarrow Col_r\rightarrow N_D\rightarrow Col_r\rightarrow N_D\rightarrow I$ (see [30]). However, to our knowledge, only a few truxene discotic liquid crystals have been synthesised due to the synthetic difficulty.

Molecular symmetry is an important factor for liquid crystal design and phase transition temperature adjustment. We are interested in ether–ester mixed tail and functional truxene derivatives for their potential liquid crystal properties and fast charge carrier mobility as organic semiconductors. Here we report an efficient method for the synthesis of 2,7,12-trihydroxy-3,8,13-trimethoxytruxene **5** and based on it, a series of ether–ester mixed chain truxene discogens **HTn** are synthesised and their mesomorphism is investigated. The synthetic route is shown in Scheme 1.

2. Experimental details

^1H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on a Varian UNITY INOVA-400 (400 MHz) spectrometer, using Me_4Si as the internal standard. The high-resolution mass spectrometry (HRMS) was measured with a Varian 7.0T Fourier Transfer Ion Cyclotron Resonance Mass Spectrometer (FT-ICR-MS). The thermal stability of samples was tested with thermal gravimetric analysis (TGA) on a TA Q500 instrument, and the phase transition temperatures were measured with differential scanning calorimetry (DSC) on a TA Q100 instrument. The optical textures of the mesophase were observed and recorded with polarising optical microscopy (POM) on an instrument (XP-201) with a hot stage and a temperature controller

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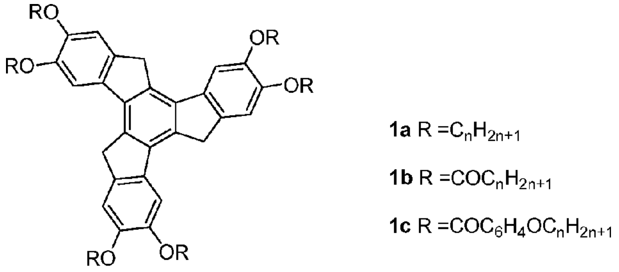

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Figure 1. Truxene discotic liquid crystals with six identical peripheral chains.

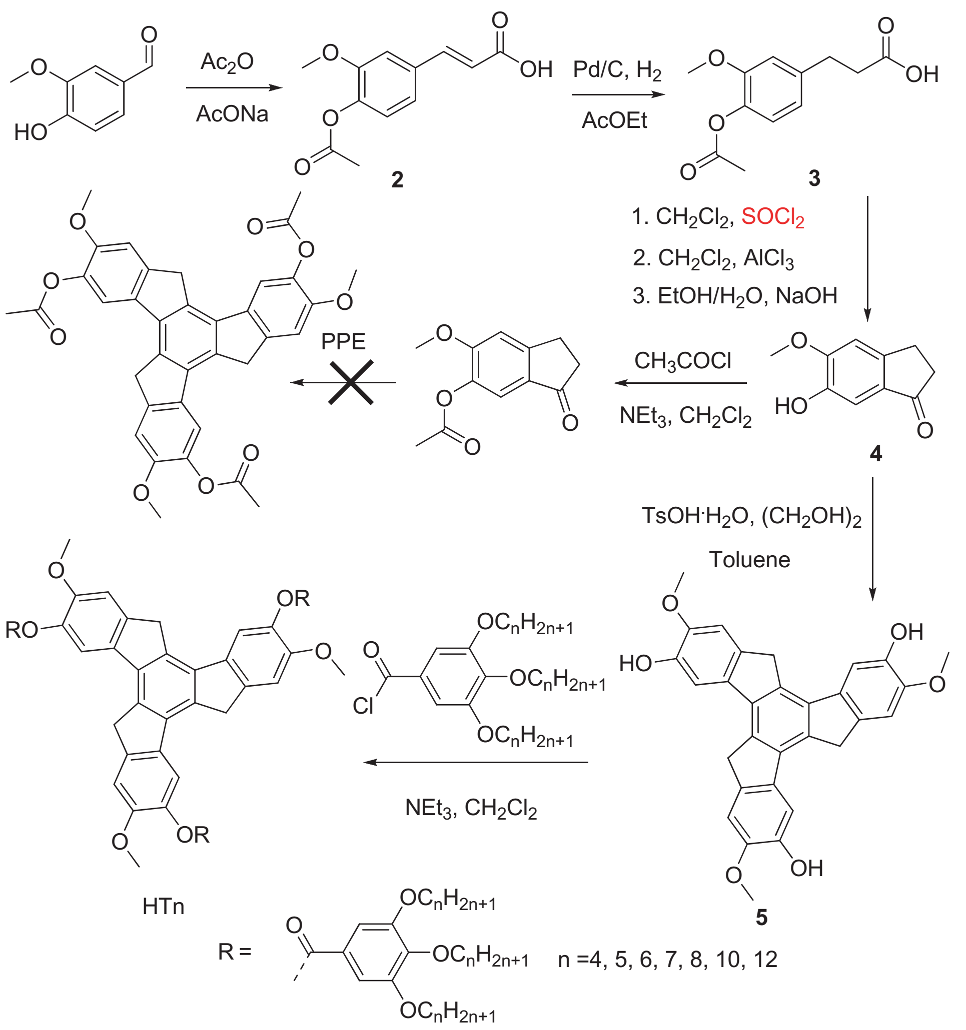
(XPR-201). The chemicals and reagents were commercially available and used without further purification.

2.1 *3-(4-Acetoxy-3-methoxyphenyl)acrylic acid (compound 2)*

3-(4-acetoxy-3-methoxyphenyl) acrylic acid was synthesised according to the modification of a reported method [31]. The mixture of vanillin (30 g, 197.4 mmol), sodium acetate (30 g, 365.9 mmol) and acetic anhydride (90 ml) were refluxed at 160°C in an oil-bath for 12 h. After cooling to 100°C, water (30 ml) was added carefully into the mixture and stirred at 100°C for 15 min. Then the mixture was poured on cracked ice. The yellow precipitate was filtered off and washed with water and acetone successively to give a white solid of compound **2** (30 g, yield 64.4%, m.p. 194–195°C).

2.2 *3-(4-Acetoxy-3-methoxyphenyl)propanoic acid (compound 3)*

The mixture of compound **2** (10 g, 42.3 mmol), 10% Pd/C (500 mg) and ethyl acetate (50 ml) was hydrogenated at 15 atm of H_2 for 12 h at 90°C. The catalyst was filtered off and the filtrate concentrated to give a white solid of **3** (9.3 g, yield 97%, m.p. 94–96°C, Lit. 94–95°C) [32].



Scheme 1. Synthesis of 2,7,12-trihydroxy-3,8,13-trimethoxytruxene **5** and ether–ester mixed tail truxene discogens **HTn** (n = 4–8, 10, 12).

2.3 6-Hydroxy-5-methoxy-2,3-dihydro-1H-inden-1-one (compound 4)

To the mixture of **3** (5.0 g, 21.0 mmol) and dimethylformamide (DMF) (0.2 ml), thionyl chloride (7.5 ml, 105 mmol) was added dropwise at 0°C. The mixture was stirred for 24 h at room temperature and then concentrated. The crude acid chloride in dry CH_2Cl_2 (10 ml) was dropped into the mixture of $AlCl_3$ (4.2 g, 31.5 mmol) in dry CH_2Cl_2 (50 ml) at 0°C. The mixture was stirred for 3 h at below 15°C and subsequently poured on crushed ice with concentrated hydrochloric acid. The resulting mixture was extracted with CH_2Cl_2, and washed with water and 10% aqueous $NaHCO_3$, dried with $MgSO_4$, and concentrated in a vacuum to give an oily liquid. To the liquid, NaOH (1.68g, 42 mmol), EtOH (30 ml) and water (10 ml) was added and the mixture was refluxed for 5 h. Then it was cooled, acidified and filtered to give a yellow solid of compound **4** (3.2 g, yield 85.5%, m.p. 194–196°C, Lit. 192–192.5°C) [33].

2.4 2,7,12-Trihydroxy-3,8,13-trimethoxytruxene $TR(OMe)_3(OH)_3$ (compound 5)

Compound **5** was synthesised according to the modification of a reported method [34]. The mixture of compound **4** (1.6 g, 9.0 mmol), *p*-toluenesulphonic acid monohydrate (1.7 g, 9.0 mmol), toluene (40 ml) and ethane-1,2-diol (20 ml) was refluxed in a three-neck flask equipped with a Dean–Stark apparatus for 24 h. The mixture was cooled and filtered to give a solid, which was washed successively with ethanol, acetone and CH_2Cl_2. The crude product was further stirred in DMF (4 ml) at 100°C for 2 h and the solid was washed with acetone to give a pink solid of **5** (1.1 g, yield 76.5%), which did not melt up to 400°C and only dissolved in dimethylsulphoxide (DMSO).

^1H NMR (d_6-DMSO, 400 MHz): δ (ppm) 9.01(s, 3H, **OH**), 7.33 (s, 3H, Ar**H**), 7.27 (s, 3H, Ar**H**), 4.03 (s, 6H, $3 \times \mathbf{CH_2}$), 3.88 (s, 9H, $3 \times \mathbf{OCH_3}$).

2.5 General procedure for the synthesis of 3,8,13-trimethoxy-2,7,12-tri- (3,4,5-trialkoxybenzoyloxy) truxene $TR(OMe)_3[OOCC_6H_2(OC_nH_{2n+1})_3]_3$ (HTn)

The mixture of compound **5** (40 mg, 0.083 mmol), Et_3N (0.5 ml), and $(RO)_3C_6H_2COCl$ (0.5 mmol) in CH_2Cl_2 (3 ml) was stirred for 12 h at room temperature and subsequently poured on crushed ice with concentrated hydrochloric acid (1 ml). The resulting mixture was extracted three times with CH_2Cl_2, the organic extractions were further washed with water and 10% aqueous $NaHCO_3$, dried with $MgSO_4$, and concentrated in a vacuum. The resulting solid was separated by using flash chromatography (silica gel, CH_2Cl_2/petroleum) and recrystallised from ethyl acetate to give a brown solid. The structural characterisation results of **HTn** are as follows.

HT4: 90 mg, yield 74.9%.^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.55(s, 6H, Ar**H**), 7.32(s, 3H), 7.19(br.s, 3H, Ar**H**), 4.13(s, 18H, $9 \times \mathbf{CH_2}$), 3.68(s, 9H, $3 \times \mathbf{CH_3}$), 1.84–1.74(m, 18H, $9 \times CH_2\mathbf{CH_2}$), 1.62–1.54(m, 18H, $9 \times CH_2CH_2\mathbf{CH_2}$), 1.01–0.98(m, 27H, $9 \times \mathbf{CH_3}$). ^{13}C NMR ($CDCl_3$, 400 MHz): δ (ppm) 165.17, 153.10, 150.13, 143.08, 139.08, 135.39, 134.32, 133.44, 123.88, 115.51, 109.79, 108.73, 69.00, 55.93, 35.70, 32.36, 31.40, 19.29, 19.17, 13.87, 13.83. HRMS (ESI) calculated for $C_{87}H_{108}O_{14}Na^+$: 1463.7428, found $(M + Na)^+$: 1463.7433.

HT5: 95 mg, yield 72.7%.^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.54(s, 6H, Ar**H**), 7.33(s, 3H), 7.18(br.s, 3H, Ar**H**), 4.09(s, 18H, J = 6.4 Hz, $9 \times \mathbf{CH_2}$), 3.68(s, 9H, $3 \times \mathbf{CH_3}$), 1.89–1.77(m, 18H, $9 \times CH_2\mathbf{CH_2}$), 1.60–1.50(m, 18H, $9 \times CH_2CH_2\mathbf{CH_2}$), 1.49–1.37(m, 18H, $18 \times \mathbf{CH_2}$), 0.98–0.90(m, 27H, $9 \times \mathbf{CH_3}$). ^{13}C NMR ($CDCl_3$, 400 MHz): δ (ppm) 165.16, 153.09, 150.16, 143.07, 139.11, 135.43, 134.33, 133.47, 123.88, 115.53, 109.81, 108.71, 69.32, 55.95, 35.72, 30.03, 29.89, 29.34, 28.27, 28.22, 22.55, 22.43, 14.07, 14.01. HRMS (ESI) calculated for $C_{96}H_{126}O_{14}Na^+$: 1589.8836, found $(M + Na)^+$: 1589.8850.

HT6: 104 mg, yield 73.7%. ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.52(s, 6H, Ar**H**), 7.33(s, 3H), 4.08(t, J = 6.4, 18H, $9 \times \mathbf{OCH_2}$), 3.69(s, 9H, $3 \times \mathbf{OCH_3}$), 1.89–1.75(m, 18H, $9 \times OCH_2\mathbf{CH_2}$), 1.54–1.47(m, 18H, $9 \times OCH_2CH_2\mathbf{CH_2}$), 1.38–1.34(m, 36H, $18 \times \mathbf{CH_2}$), 0.94–0.89(m, 27H, $9 \times \mathbf{CH_3}$). ^{13}C NMR ($CDCl_3$, 400 MHz): δ (ppm) 165.19, 153.08, 150.13, 143.08, 142.97, 139.03, 135.35, 134.30, 133.46, 123.82, 115.51, 109.76, 108.58, 69.28, 55.91, 35.65, 31.76, 31.57, 30.32, 29.31, 25.79, 25.75, 22.71, 22.63, 14.10, 14.03. HRMS (ESI) calculated for $C_{105}H_{144}O_{14}Na^+$: 1716.0245, found $(M + Na)^+$: 1716.0243.

HT7: 100 mg, yield 65.9%. ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.52(s, 6H, Ar**H**), 7.33(s, 3H, Ar**H**), 4.08(t, J = 6.4, 18H, $9 \times \mathbf{OCH_2}$), 3.73(s, 9H, $3 \times \mathbf{OCH_3}$), 1.89–1.75(m, 18H, $9 \times OCH_2\mathbf{CH_2}$), 1.54–1.46(m, 18H, $9 \times OCH_2CH_2\mathbf{CH_2}$), 1.41–1.29(m, 54H, $27 \times \mathbf{CH_2}$), (m, 27H, $9 \times \mathbf{CH_3}$). ^{13}C NMR ($CDCl_3$, 400 MHz): δ (ppm) 165.16, 153.09, 150.19, 143.08, 139.13, 135.46, 134.35, 133.49, 123.86, 115.57, 109.83, 108.70, 69.34, 55.97, 35.74, 30.38, 29.70, 29.38, 29.23, 29.06, 26.08, 26.04, 22.68, 22.60, 14.07, 14.06. HRMS (ESI) calculated for $C_{114}H_{162}O_{14}Na^+$: 1842.1653, found $(M + Na)^+$: 1842.1658.

HT8: 124 mg, yield 76.5%. ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.53(s, 6H, Ar**H**), 7.33(s, 3H, Ar**H**), 7.20(br.s, 3H, ArH), 4.08(t, J = 6.8 Hz, 18H, $9 \times \mathbf{CH_2}$), 3.67(s, 9H, $3 \times \mathbf{CH_3}$), 1.89–1.75(m, 18H, 9 ×

$CH_2\mathbf{CH_2}$), 1.58–1.46(m, 18H, 9 × $CH_2CH_2\mathbf{CH_2}$), 1.34–1.29(m, 72H, 27 × $\mathbf{CH_2}$), 0.91–0.86(m, 27H, 9 × $\mathbf{CH_3}$). ^{13}C NMR ($CDCl_3$, 400 MHz): δ (ppm) 165.18, 153.08, 150.15, 143.09, 142.99, 139.04, 135.37, 134.31, 133.47, 123.81, 115.52, 109.77, 108.58, 69.29, 55.69, 35.69, 31.92, 31.83, 30.39, 29.55, 29.37, 29.30, 26.13, 26.10, 22.71, 22.68, 14.11, 14.09. HRMS (ESI) calculated for $C_{123}H_{180}O_{14}Na^+$: 1968.3062, found $(M + Na)^+$: 1968.3064.

HT10: 98 mg, yield 53.6%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.52 (s, 6H, Ar**H**), 7.33 (s, 3H, Ar**H**), 4.08 (t, $J = 6.4$, 18H, 9 × $O\mathbf{CH_2}$), 3.74 (s, 9H, 3 × $O\mathbf{CH_3}$), 1.87–1.79 (m, 18H, 9 × $OCH_2\mathbf{CH_2}$), 1.55–1.48 (m, 18H, 9 × $OCH_2CH_2\mathbf{CH_2}$), 1.36–1.26 (m, 108H, 54 × $\mathbf{CH_2}$), 0.91–0.85 (m, 27H, 9 × $\mathbf{CH_3}$). ^{13}C NMR ($CDCl_3$, 200 MHz): δ (ppm) 165.15, 153.07, 150.16, 143.04, 139.08, 135.48, 134.32, 133.47, 123.82, 115.58, 109.77, 108.6, 69.31, 55.94, 36.00, 31.91, 30.40, 29.75, 29.60, 29.41, 29.37, 26.14, 22.69, 14.11. HRMS (ESI) calculated for $C_{92}H_{122}O_{14}Na^+$: 2221.5913, found $(M + Na)^+$: 2221.5914.

HT12: 122 mg, yield 66.6%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.52(s, 6H, Ar**H**), 7.33(s, 3H, Ar**H**), 4.13–4.04(m, 18H, 9 × $\mathbf{CH_2}$), 3.73(s, 9H, 3 × $\mathbf{CH_3}$), 1.87–1.76(m, 18H, 9 × $CH_2\mathbf{CH_2}$), 1.58–1.50(m, 18H, 9 × $CH_2CH_2\mathbf{CH_2}$), 1.47–1.25(m, 144H, 27 × $\mathbf{CH_2}$), 0.91–0.86(m, 27H, 9 × $\mathbf{CH_3}$). ^{13}C NMR ($CDCl_3$, 200 MHz) δ: 165.12, 153.09, 150.60, 143.05, 139.17, 135.59, 134.34, 133.47, 123.89, 115.60, 109.87, 108.70, 69.35, 69.25, 56.07, 35.85, 31.95, 30.41, 29.75, 29.65, 29.56, 29.45, 29.30, 26.16, 22.70, 14.09. HRMS (ESI) calculated for $C_{159}H_{252}O_{14}Na^+$: 2472.8693, found $(M + Na)^+$: 2472.8696.

3. Results and discussion

3.1 Synthesis and characterisation

Truxene discotic liquid crystals with six peripheral ester chains [35, 36] have been synthesised by the self condensation of 5,6-dimethoxy-2,3-dihydro-1*H*-inden-1-one in polyphosphate ethyl ester (PPE), resulting in hexamethoxytruxene, which has been demethylated yielding the key intermediate 2,3,7,8,12,13-hexahydroxytruxene, and further transferred to ester-functional truxene discogens by an esterification reaction. Hexaalkoxytruxenes have been prepared through the self-condensation dehydration reaction of 5,6-di-n-alkoxy-2,3-dihydro-1*H*-inden-1-one in PPE [28]. In order to synthesis ether–ester mixed tail hexasubstituted truxene derivatives, we have tried the self-condensation reaction of 6-methoxy-3-oxo-2,3-dihydro-1*H*-inden-5-yl acetate in PPE, which has resulted in a low yield of truxene. Subsequently, we have tried the trimerisation reaction of compound **4**, catalysed by *p*-toluenesulphonic acid monohydrate in a mixed solvent of toluene and ethane-1,2-diol. Water produced in the reaction was removed from the system through a Dean–Stark receiver. This method supplies the key intermediate of 2,7,12-trihydroxy-3,8,13-trimethoxytruxene **5** in a good yield (77%). The ether–ester mixed truxene discogens **HTn** are synthesised by further esterification of **5** with substituted benzoyl chloride in the presence of an organic base Et_3N.

3.2 Thermal stability

Truxene moiety is a large-sized rigid core and its discotic liquid crystals exhibit high clearing points. The ester bond often shows a weaker thermal stability than the ether bond. Therefore, it is necessary to check the thermal stability of the ether–ester mixed tail truxene discogen **HTn**. Figure 2 shows the TGA result of **HTn** in a N_2 atmosphere at the heating rate of 10°C min^{-1}. **HTn** loses little of its weight before 330°C, but only the clearing points of **HT12** and **HT10** are lower than 330°C, therefore the DSC measurement and POM observation of **HT12** and **HT10** are in the thermal stable temperature ranges of the discogens. The clearing points of the other five discogens are higher than the thermal decomposition temperature of 330°C; therefore, the DSC measurement only tested their clearing points with partial thermal decomposition.

3.3 Mesomorphism

The optical textures of **HT4**, **HT5**, **HT8** and **HT10** have been observed and recorded with a heating plate POM. The typical photomicrograph is shown in Figure 3.

The clearing point of **HT4** is higher than 380°C and it is partially decomposed at that temperature. The POM result shown in Figure 3(a) was taken at 280°C in the first heating run. The liquid-like flowing of the sample between glass plates was observed, but the optical texture is not typical. Figure 3(b) and (c) were recorded by heating **HT5** and **HT8** quickly above the clearing point and then fast cooling. Figure 3(b) is not a typical columnar texture, due to the partial thermal decomposition of the sample. However, Figure 3(c) has shown that it is a hexagonal column mesophase (Col$_h$). **HT10** is thermally stable at its clearing point (Figure 3(d)), which was taken from the cooling run and exhibits a typical optical texture of the Col$_h$. The POM results demonstrate that **HTn** exhibits the Col$_h$ mesophase.

The phase transition temperatures of **HTn** have been measured with DSC and the result is shown in Figure 4. As the decomposition temperatures of **HT4**–**HT8** are lower than that of their clearing

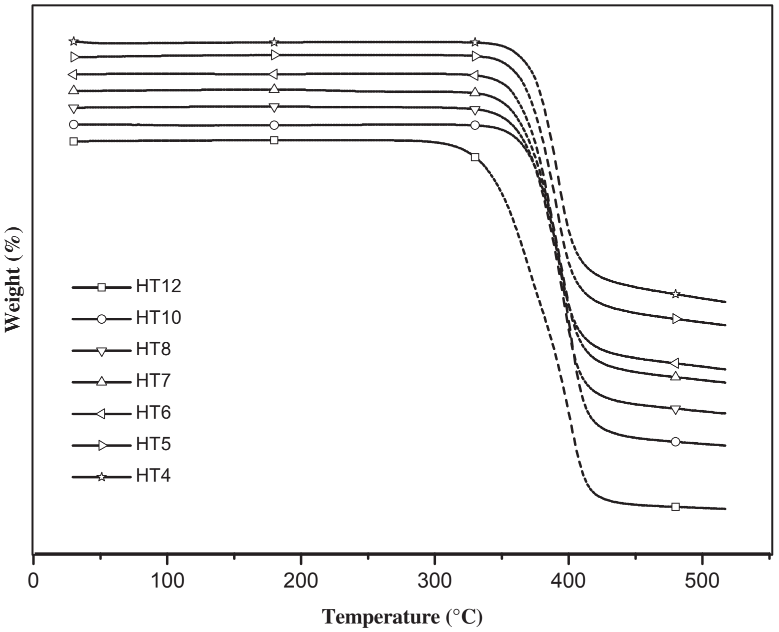


Figure 2. TGA diagram of ether–ester mixed tail truxene discogen **HTn**.

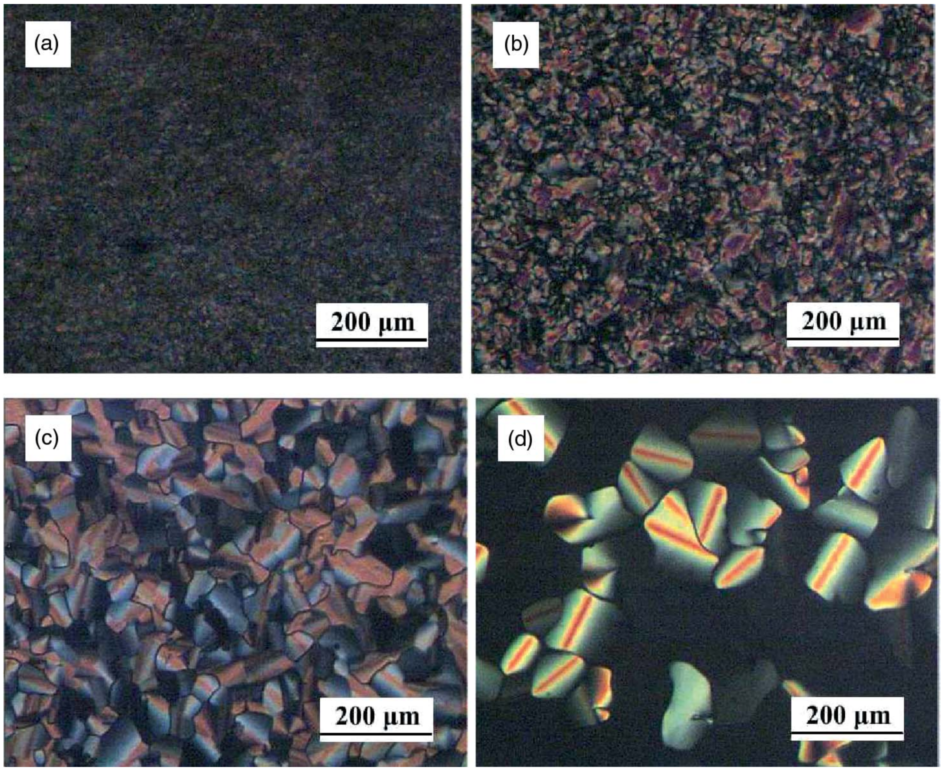
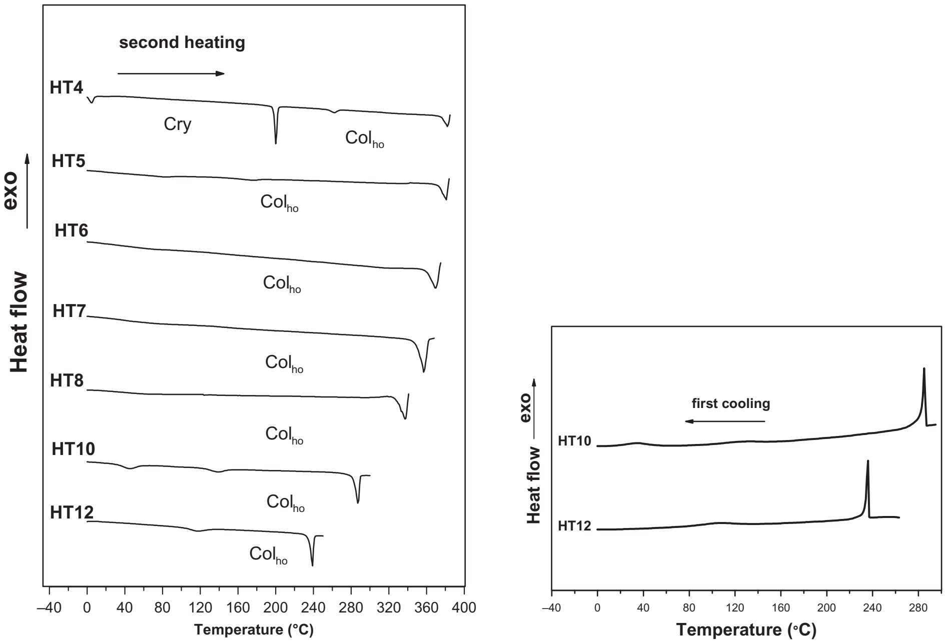


Figure 3. Optical textures of **HTn**. (a) **HT4** heated to 280°C from room temperature. (b) **HT5** cooled to 256°C from 420°C. (c) **HT8** cooled to 256°C from isotropic liquid. (d) **HT10** cooled to 250°C from isotropic liquid.

points, they are partially decomposed when heated to their clearing points in the heating run and no phase transition peaks are observed in the consequent cooling runs. The longer chain truxene derivatives **HT10** and **HT12** display lower clearing points and are thermally stable at that temperature, so both heating and cooling runs of DSC traces were measured and are shown in Figure 4.



Heating run of **HTn**   Cooling run of **HT10** and **HT12**

Figure 4. DSC traces of **HTn** for the heating and cooling runs (10°C min^{-1}).

The synthesised truxene derivatives **HTn** exhibit quite high clearing points, which are similar to the reported truxene derivatives **1**. However, the melting points of **HTn** are lower than that of **1**, as there are nine long aliphatic chains around the truxene core for **HTn**. The melting points (see Table 1) have been tested only in the first heating run, except for **HT4**, showing that the truxenes can only crystallise in an organic solvent and no crystallisation is observed in the bulk state. The POM and DSC results above demonstrate that **HTn** only have a Col$_h$ phase. In contrast with **1b** and **1c** which show several mesophases, **HTn** exhibited only one mesophase.

The melting points and clearing points of **HTn** from DSC are summarised in Table 1. The phase diagram and linear equation of their melting and clearing points are shown in Figure 5.

The melting points and clearing points of **HTn** display a quite regular change. The melting point decreases 29.1°C with the alkyl chain length increasing one unit and the clearing point decreases 22.4°C with the chain length *n* increasing one unit. As **HT4** and **HT5** began to decompose before the clearing points arrived, their observed clearing points are not very consistent with the linear equation of clearing points.

Table 1. Melting points and clearing points of **HTn**.

Compound	Mesophase and phase transition temperature (°C)
HT4	Cry 200.2 Col$_h$ 382.4 Iso[a]
HT5	Cry 165.2 Col$_h$ 380.3 Iso[a]
HT6	Cry 137.2 Col$_h$ 369.7 Iso[a]
HT7	Cry 112.8 Col$_h$ 357.2 Iso[a]
HT8	Cry 88.8 Col$_h$ 337.4 Iso[a]
HT10	Cry 45.7 Col$_h$ 287.5 Iso
HT12	Cry -17.6 Col$_h$ 239.0 Iso

Notes: Cry: crystal state; Col$_h$: hexagonal columnar mesophase; Iso: isotropic liquid.
[a] Partial thermal decomposition observed at the clearing points.

The mesophase structure has been further investigated with X-ray diffraction (XRD) and the three-dimensional (3D) stacking model for **HT10** is suggested in Figure 6. The XRD results reveal the diffraction peaks of 32.6 Å, 18.7 Å, 16.1 Å, 12.1 Å and 10.5 Å in the small angle region, showing the reciprocal spacing ratio of $1 : 1/\sqrt{3} : 1/2 : 1/\sqrt{7} : 1/3$, which can be assigned respectively to the (100), (110), (200), (210) and (300) reflections. This reciprocal spacing ratio is consistent with a hexagonal columnar (Col$_h$) mesophase, which belongs to the space group of *P6/mmm* (point group of D_{6h}) [37, 38]. The broad peak at around 4.6 Å in the wide-angle region represents the distance among the molten aliphatic chains surrounding the truxene core. Another diffraction peak is observed at 3.6 Å, indicating the average stacking distance between the ordered intra-columnar truxene cores. The lattice parameter of the Col$_h$ phase for **HT10** is calculated to be a = 37.6 Å from the XRD date (Figure 4). So, the above-mentioned results have confirmed that the mesophase is a Col$_h$ mesophase.

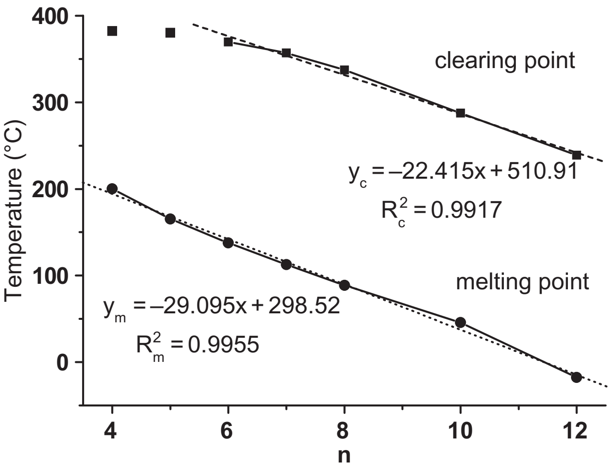


Figure 5. The phase diagram and linear equation of the melting point and clearing point of **HTn**.

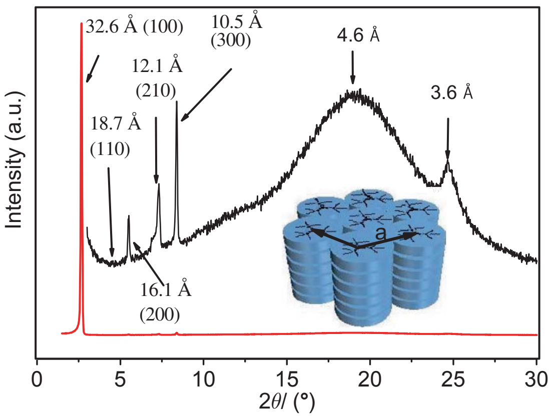


Figure 6. XRD pattern of **HT10** at 180°C: sample slowly cooled from the isotropic liquid state.

4. Conclusion

We have developed a simple and efficient method for the synthesis of ether–ester mixed tail truxene discotic liquid crystals. In contrast with the literature, where it is reported that truxene discogens **1b** and **1c** exhibit polymesomorphism, truxene **HTn** show only one mesophase of Col_h, with a very high mesophase stability and a wide mesophase range. 2,7,12-Trihydroxy-3,8,13-trialkoxytruxene is a key intermediate, and mixed tail or functional truxene discotic monomers, oligomers and polymers can be synthesised from it. Further studies on truxene discotic liquid crystals, based on the synthetic method developed here, are in progress.

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