Room temperature liquid crystal of symmetric gallic trimer containing cyanuric core: synthesis and mesomorphism

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Three novel gallic monomer 7, dimer 10 and trimer 12 with conjugated cyanuric core were designed and synthesised by Schiff-base condensation mode in yields of 80–90%. Their structures were characterised by fourier transform infrared spectroscopy, \textsuperscript{1}H NMR (nuclear magnetic resonance), electrospray ionization mass spectrometry and elemental analyses. Their mesomorphic behaviours were investigated by differential scanning calorimetry, polarising optical microscopy and X-ray diffraction. The gallic monomer 7 has no mesomorphic property, but the dimer 10 and trimer 12 possess good mesomorphic properties. The trimer 12 with high symmetry exhibited the typical hexagonal columnar liquid crystal at room temperature. The temperature range of mesophase is as wide as 149°C (14–163°C). The results suggested that the more gallic units and symmetric structures are favourable for excellent mesomorphic properties.

**Keywords:** gallic; cyanuric; trimer; mesophase; synthesis

1. Introduction

Columnar discotic liquid crystals (LCs), which were usually obtained by introducing several flexible aliphatic arms onto rigid polyaromatic cores, have been paid much attention extensively in the past decades due to their various potential applications for organic photovoltaic devices, organic field effect transistors, thin film transistors, organic light emitting diodes, gas sensors and photocopying machines, etc.[1–4] In order to prepare the columnar LCs effectively, the symmetric conjugated aromatic core with several aliphatic side chains attracted wide research interesting as excellent columnar LCs materials.[5–10] For example, triphenylene derivatives possessing 3–6 alkylic chains exhibited high tendency to form the required columnar phases.[11–16] Lately, gallic ether or its analogues with three long aliphatic chains on its phenolic groups were seen as good structural units to construct novel columnar LCs by connecting with conjugated aromatic core.[17–21] For instance, the saddle-shaped tetraphenylenes with peripheral gallic esters displaying columnar mesophases were described by Laschat’s group.[22] Detert reported the synthesis and mesomorphic properties of some tristriazolotriazines derivatives with 1,2-bis-substituted or 1,2,3-tri-substituted alkxy side chains on phenyl groups.[23,24] Our groups presented two gallic-perylene-gallic trimers with interesting mesomorphic properties.[25] Some metal complexes forming columnar mesophase containing gallic units,[26,27] luminescence LCs [28,29] and ionic LCs with gallic units[30,31] were also investigated as novel LC materials. Nevertheless, compared with the triphenylene columnar LCs, the varieties and amounts of gallic columnar LCs were less so far. In this paper, we designed and synthesised several new gallic derivatives with conjugated cyanuric core and investigated their mesomorphic behaviours. The results suggested that the symmetric gallic trimer presented good room temperature columnar LC property with a wide range of mesomorphic temperature, which was seldom observed for gallic LCs.

2. Results and discussion

2.1. Synthesis and characterisation

Scheme 1 illustrates the synthetic routes of gallic monomer, dimer and trimer containing cyanuric cores. Due to the different reaction activities of three chloric groups in cyanuric chloride, they could be substituted selectively under different temperature. Thus, according to the literature,[32] the 1,3,5-triazine-based derivatives 6, 9 and 11 with one, two and three aldehyde groups were designed and synthesised by simple procedures in high yields, respectively. One can see that compounds 6, 9 and 11 possess 1,3,5-triazine-based polyaromatic structures with overlapping conjugated areas, which were favourable for π–π stacking. Also, the gallic hydrazide derivative 3 was prepared by reacting methyl gallate 1 with bromodecane and then hydrazinolysis with hydrazine hydrate in yield of 82%.[33] Furthermore, by treating

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compound 3 with series of cyanuric derivatives 6, 9 and 11, respectively, the gallic monomer 7, dimer 10 and trimer 12 with cyanuric core were prepared in Schiff-base condensation mode in high yields of 80–90%. The structures of all new compounds were well confirmed by elemental analysis, IR, electrospray ionization mass spectrometry (ESI-MS) and $^1$H NMR (nuclear magnetic resonance) spectra. It was worth noting that compounds 7, 10 and 12 were the first example of gallic oligomers with the cyanuric core, which are important for studying the influences of different amounts of gallic units on the mesophase.

2.2. Mesomorphic studies
The mesomorphic properties of gallic monomer 7, dimer 10 and trimer 12 with cyanuric cores were preliminarily investigated by differential scanning calorimetry (DSC). The results are shown in Figure 1 and Table 1. It can be seen that only one peak was observed for compound 7 upon second heating or cooling at 112.2°C and 107.3°C, respectively, indicating only crystalline-isotropic phase transition for compound 7. However, both compounds 10 and 12 exhibited two-phase transfer behaviours on second heating and cooling. For compound 10, two exothermal peaks on heating at 96.2°C and 180.2°C, and two endothermic peaks on cooling at 96.1°C and 160.1°C were clearly detected. Compound 12 also showed two exothermal peaks at 14.1°C and 163.6°C for heating and two endothermic peaks at 10.1°C and 156.4°C for cooling. These DSC data suggested that compounds 10 and 12 possessed the reversible phase transition of solid-state mesophase.
and mesophase isotropic phase on cooling and heating. The hysteresis phenomena observed for these could be attributed to supercooling, which was common for such viscous materials. Thus, judging from these DSC data, it might be deduced that compound 7 has no mesomorphic behaviour but compounds 10 and 12 possess mesophase on heating and cooling, which was confirmed further by polarising optical microscopy (POM) images. The temperature ranges of mesophase for compounds 10 and 12 are as wide as 84°C (from 96.2°C to 180.2°C) and 149°C (from 14.1°C to 163.6°C), respectively. By comparing with the structures of compounds 7, 10 and 12, it could be concluded that the more gallic units with alkyl chains are favourable for producing good mesomorphic properties. Due to rigidity of the polyaromatic core, only one gallic unit with alkyl chains in compound 7 is not enough for bearing mesomorphic property. Two gallic units with alkyl chains in compound 10 bring mesomorphic property, but the mesomorphic temperature is higher and the range of mesomorphic temperature is smaller than that of compound 12 with three gallic units with alkyl chains. Compound 12 is a typical room temperature LC with large temperature range of mesophase, which was seldom observed for gallic LCs.

Referring to the DSC results, the polarised optical microscopy was used to study the phase textures of compounds 7, 10 and 12 on heating and cooling. Compound 7 showed the crystal phase-isotropic phase transition on heating and cooling, and no mesomorphic texture was observed under POM. But for compounds 10 and 12, two phase transitions of solid-state mesophase and mesophase isotropic phase on heating and cooling were clearly presented. Compound 10 was pale solid at room temperature, a cloudy liquid between approximately 100°C and 180°C, and clear liquid above 200°C on heating. Moreover, the clear mesophase texture appeared on cooling slowly. Compound 12 was a soft pale solid after purification at room temperature. Upon heating, a cloudy liquid appeared till approximately 160°C. Upon slowly cooling, the mesophase texture emerged and existed at room temperature for several weeks. Figure 2 exhibits the textures of compounds 10 and 12

| Compounds | Phase transition | $T(\Delta H)$ heating | $T(\Delta H)$ cooling |
|-----------|------------------|-----------------------|-----------------------|
| 7         | Cr-Iso (Iso-Cr)  | 112.2 (21.4)          | 107.3 (19.8)          |
| 10        | Cr-LC (LC-Cr)   | 96.2 (9.9)            | 96.1 (10.4)           |
|           | LC-Iso (Iso-LC) | 180.2 (12.4)          | 170.1 (11.7)          |
| 12        | Cr-LC (LC-Cr)   | 14.1 (8.9)            | 10.1 (8.2)            |
|           | LC-Iso (Iso-LC) | 163.6 (6.7)           | 156.4 (7.1)           |

Notes: Cr, crystalline; LC, liquid crystalline; Iso, isotropic.

Figure 2. (colour online) Textures of compounds 10 and 12 obtained with polarised optical microscopy (×400) on cooling at 130°C.
at corresponding temperatures under POM. One can see that they were the typical fan textures, which were similar to the known textures for columnar phases. [34–38] These results of POM experiments were in agreement with the results of DSC experiments. Both DSC and POM experiments indicated that compound 7 showed no mesomorphic property, but compounds 10 and 12 exhibited columnar LC behaviours on cooling.

In order to further investigate the columnar mesomorphic properties of compounds 10 and 12, their mesophases were studied by X-ray powder diffraction. The results are illustrated in Figure 3. In the small angle region, one peak at 2.60° and a broad peak at 4.31° were observed for compound 10 and two clear peaks at 2.13° and 2.47° appeared for compound 12. In the wide angle region, both compounds 10 and 12 showed similar broad halos at 17–25° and a reflection at approximately 23.1°. These data certainly support that compounds 10 and 12 possess columnar mesophase. The d-spacing derived from 2.60° (34.1 Å) and 2.13° (41.4 Å) were assigned to the average distances of inter-column spacing. The broad halos at 2θ = 17–25° were assigned to the average distances of the molten alkyl chains on gallic units (3.6–5.2 Å). The reflections at 23.1° suggested the spacing of approximately 3.8 Å, which were the typical characteristic of intra-columnar order of discotic LCs with π–π interactions. Moreover, for compound 12, the d-spacing calculated from 2.13° and 2.47° were 41.4 Å and 35.7 Å, respectively, which were in accordance with the ratio of 2/√3, indicating the hexagonal columnar LC. However, it was difficult to deduce the concrete columnar structures due to the complex and broad peaks at 4.31° for compound 10, which might indicate the mesogens typically stack to form columns (not necessarily simple one-molecule-wide columns), but no positional order among the columns. These phenomena could be explained by their structures. Due to compound 10 having two gallic units with low symmetry, it is not favourable for the positional order among the columns. But compound 12 possessed three gallic units with high symmetry, resulting in the ordered hexagonal columnar LC.

Based on the above DSC, POM and X-ray diffraction (XRD) experimental results, it could be concluded that the amounts of gallic units had great influence on the mesomorphic properties. The gallic monomer 7 with cyanuric core showed no mesomorphic property. The dimer 10 and trimer 12 exhibited good mesomorphic properties. The trimer 12 with high symmetry possessed not only room temperature LC behaviour with large temperature range of mesophase but also the hexagonal columnar LC.

3. Conclusions

In conclusion, we reported the design and synthesis of three gallic monomer 7, dimer 10 and trimer 12 with cyanuric core in Schiff-base condensation mode in high yields of 80–90%. Their structures were characterised by fourier transform infrared spectroscopy, 1H NMR, MS and elemental analysis. Their mesomorphic behaviours were investigated by DSC, POM and XRD. The results suggested that the mesomorphic properties were greatly influenced by the amount of gallic units. The gallic monomer 7 with cyanuric core showed no mesomorphic property. But the dimer 10 and trimer 12 exhibited good mesomorphic properties. The trimer 12 was a typical hexagonal columnar LC at room temperature, and the temperature range of mesophase was as wide as 149°C (14–163°C).

4. Experimental

The organic solvents and inorganic reagents were purified according to standard anhydrous methods before use. All chemical reagents were obtained from Aladdin Co., Ltd. (Shanghai, China) and used without further purification. Thin-layer chromatography (TLC) analysis was performed using precoated glass plates. Column chromatography was performed by using silica gel (200–300 mesh). IR spectra were recorded on a Perkin-Elmer PE-983 infrared spectrometer (PerkinElmer, Waltham, MA, USA) as KBr pellets with absorption in cm⁻¹. NMR spectra were recorded in CDCl₃ on a Bruker-ARX 400 instrument (Bruker, Rheinstetten, Germany) at 30°C. Chemical shifts are reported in ppm, using tetramethylsilane as internal standard. ESI-MS spectra were obtained from DECAX-30000 LCQ Deca XP mass
4.1. Synthesis of gallic monomer with cyanuric core 7

Under N₂ atmosphere, the mixture of triazine derivative 6 (0.0575 g, 0.15 mmol) and gallic hydrazide 3 (0.104 g, 0.15 mmol) was refluxed in 20 mL of methanol/CHCl₃ (3:1, V/V) by using 0.10 mL of acetic acid as catalyst. After reaction overnight, TLC detection indicated the disappearance of starting materials. Then the solvent was evaporated under reduced pressure. The residue was treated by 10 mL of methanol and precipitate was formed. The precipitate was filtered and washed by small amounts of methanol three times. After vacuum dryness, compound 7 was obtained as white solid in 84% yield. Compound 7: FT-IR(KBr), v/cm⁻¹: 3414, 2924, 2855, 1672, 1578, 1487, 1446, 1341, 1232, 1205, 1114, 809; ¹H NMR (400 MHz, CDCl₃) δ (ppm): 0.89 (t, J = 8.4 Hz, 2H, CH₂), 1.25–1.80 (m, 60H, C₆H₂⁺), 3.60–4.01 (m, 6H, ArOCH₃), 6.70–7.41 (m, 18H, ArH and ArNH), 8.35 (s, 1H, CH=CN), 9.58 (s, 1H, CONH). MS (m/z (%): 1053.7 (M⁺, 100). Anal. calcd for C₆₅H₅₉N₇O₅: C 74.08, H 9.08, N 9.28.

4.2. Synthesis of gallic dimer with cyanuric core 10

Under N₂ atmosphere, the mixture of triazine derivative 9 (0.0412 g, 0.1 mmol) and gallic hydrazide 3 (0.138 g, 0.2 mmol) was refluxed in 20 mL of methanol/CHCl₃ (3:1, V/V) by using 0.15 mL of acetic acid as catalyst for 6 h. TLC detection indicated the disappearance of starting materials. Then the solvent was evaporated under reduced pressure, and the residue was treated by 10 mL of methanol to afford precipitate. The precipitate was filtered and washed by small amounts of MeOH three times. After vacuum dryness, compound 10 was obtained as pale solid in 88% yield. Compound 10: FT-IR (KBr), v/cm⁻¹: 3417, 2924, 2855, 1618, 1573, 1491, 1423, 1373, 1209, 1110, 809; ¹H NMR (400 MHz, CDCl₃) δ (ppm): 0.97 (t, J = 8.0 Hz, 18H, CH₃), 1.26–1.84 (m, 120H, C₁₀H₂₀), 3.63–4.02 (m, 12H, ArOCH₃), 6.69–7.38 (m, 18H, ArH and ArNH), 8.41 (s, 2H, CONH), 8.66 (bs, 2H, CH=SN). MS (m/z (%): 1754.3 (M⁺, 100). Anal. calcd for C₁₆₉H₁₄₁N₉O₁₅: C 74.61, H 9.88, N 6.39; found C 74.64, H 9.84, N 6.38.

4.3. Synthesis of gallic trimer with cyanuric core 12

Under N₂ atmosphere, the mixture of triazine derivative 11 (0.0221 g, 0.05 mmol) and gallic hydrazide 3 (0.104 g, 0.15 mmol) was refluxed in 30 mL of methanol/CHCl₃ (3:1, V/V) by using 0.2 mL of acetic acid as catalyst. TLC detection indicated the disappearance of starting materials in 12 h. Then the solvent was evaporated under reduced pressure. The residue was treated by 10 mL of methanol to afford white precipitate. The precipitate was filtered and washed by some methanol for three times. After vacuum dryness, compound 12 was obtained as pale soft solid in yield of 80%. Compound 12: FT-IR(KBr), v/cm⁻¹: 3414, 2924, 2860, 1646, 1564, 1491, 1364, 1205, 1114, 841; ¹H NMR (400 MHz, CDCl₃) δ (ppm): 0.87 (s, 27H, CH₃), 1.25–1.83 (m, 180H, C₁₀H₂₀), 3.64–4.05 (m, 18H, ArOCH₃), 6.88–7.94 (m, 18H, ArH), 8.51 (bs, 3H, CH=SN), 8.90 (bs, 3H, CONH). MS (m/z (%): 2453.8 (M⁺, 100). Anal. calcd for C₁₅₅H₁₄₇N₁₉O₁₅: C 74.86, H 10.22, N 5.14; found C 74.84, H 10.26, N 5.10.

Disclosure statement
No potential conflict of interest was reported by the authors.

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Supplemental data
Supplemental data for this article can be accessed here.

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