



Hydrogen-bonded ionic liquid crystals based on multi-armed structure: synthesis and characterization

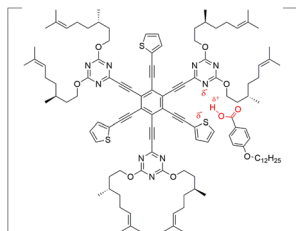
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Abstract

A new type of six-armed π -conjugated structure was built up from benzene as the central core and electron-rich triazine rings as the peripheral group. The hexasubstituted product was liquid and did not exhibit any liquid crystal properties while the equimolar mixtures in 1:1 ratio of the hexasubstituted compound and 4-(dodecyloxy)benzoic acid resulted in an organic salt exhibiting a columnar mesophase characterized with a dendritic growing texture. Examination of the relationship between structure and properties and the presence of the number of carbon atoms in the alkyl chain suggested that, the higher increase led to the phase transition at low temperature to form a columnar mesophase as an ionic liquid crystal composed of cations and anions. These are often stabilized by resonance with strongly delocalized charges. The liquid crystalline properties of the organic salt were investigated by differential scanning calorimetry (DSC) and polarizing optical microscopy (POM).

Graphical abstract



Keywords Liquid crystals · Shape-persistent materials · π -Conjugated systems · Sonogashira coupling

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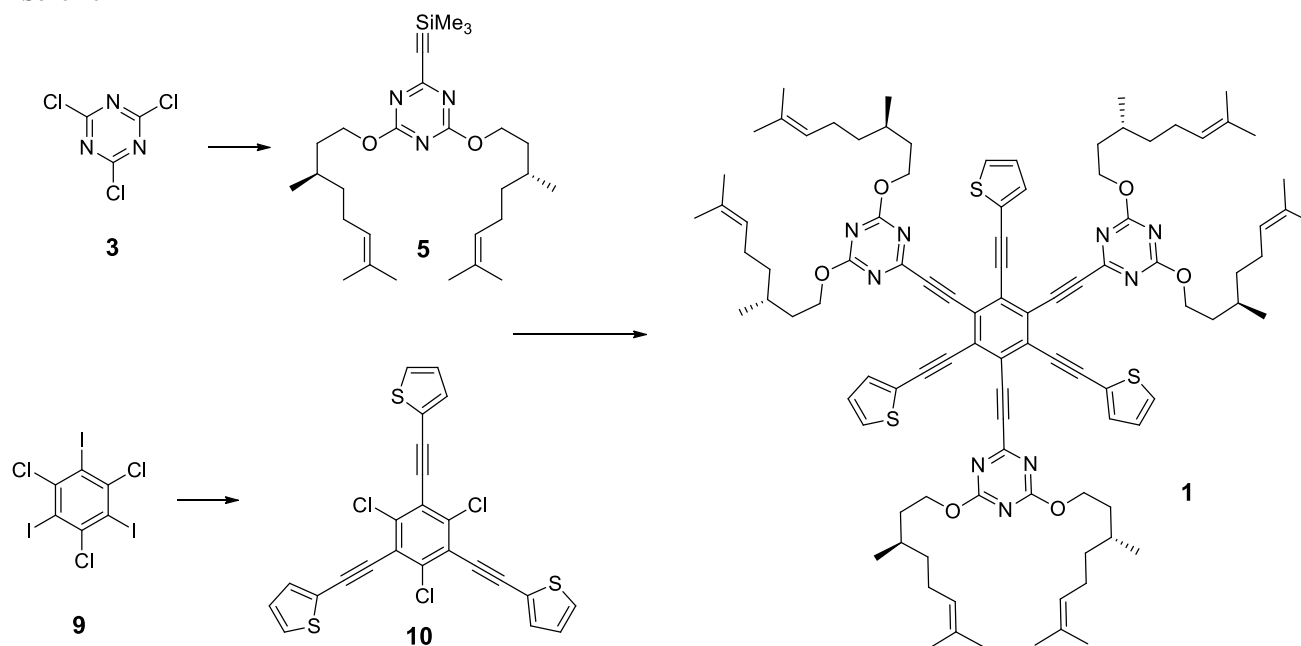
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Introduction

Heterocyclic compounds containing nitrogen have been found in a wide spectrum of scientific research and industrial applications. Triazine [1] with alternating nitrogen and carbon atoms on a six-membered ring is a basic building block of many chemical materials [2–4], and commonly present in natural and synthetic compounds with significant bioactivity profiles [5–7] as antitumor agents [8], CRF receptor antagonists [9], antimicrobial and anti-inflammatory agents [10], antitumor [11] and anti-AIDS agents [12].

Triazine-based materials are rich in nitrogen and show higher thermal stability as compared to carbon-based materials [13, 14]. Some derivatives of triazines such as melamine, cyanuric chloride, and cyanuric acid are produced on industrial scale. In addition, inexpensive production cost

Scheme 1



makes triazine-based donor- π -acceptor structures suitable candidates for developing organic dyes that do not contain metals. Such structures are excellent photo stimulants and have a high molar absorption coefficient. Such materials are easy to synthesize [15] and present good electrical, optical properties [16–20]. In search of developing novel molecular architectures with unique structural, optical and electronic properties significant attention has been devoted to 1,3,5-triazine-based materials, due to the favorable LUMO levels of triazine moiety [21].

Cyanuric chloride and cyanuric fluoride can be subjected to sequences of nucleophilic substitution reactions in a controlled manner to prepare derivatives of triazine in aliphatic, aromatic, alkynic nature. It is possible to connect three different C-nucleophiles, N-nucleophiles, S-nucleophiles and/or O-nucleophiles in a desired order to the triazine unit. Many examples of this have been reported in the literature [22–26].

The basic principle in the design and production of synthetic materials is to bring together the building blocks possessing certain principles such as hydrogen bonding, van der Waals, π - π stacking, etc. to introduce desired functionalities to the final structures. In this context, triazine-based structures have important places in the literature and play key roles in imparting various functionalities to the molecular architectures [27–29].

Star-shaped macromolecules possessing well-defined and monodisperse architecture have found potential application as materials of OLED and electro-optic devices [30, 31]. Such macromolecules with rigid arms directing radially symmetrical away from the center have void regions where

appropriate guest molecules can be hosted via electrostatic or hydrogen bonding motif resulting materials with unique properties [19, 20].

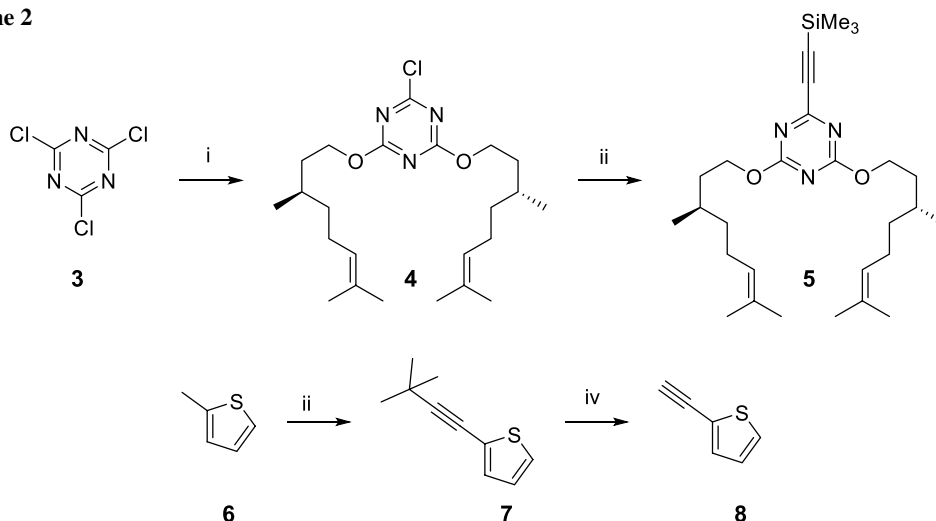
In an ongoing investigation to develop π -conjugated functional materials containing triazine units, which are strategically placed in the star-shaped macromolecular, we have developed tri-armed mesogens of 2,4,6-tris[[4,6-bis(dodecyloxy)-1,3,5-triazine-2-yl]ethynyl]-1,3,5-triazine [19] and 2,4,6-tris[[4,6-bis((*S*)-citronellyloxy)-1,3,5-triazine-2-yl]ethynyl]-1,3,5-triazine [20] in which alkoxy-substituted triazines were connected to the central core with rigid acetylenic bridges, which demonstrated Smectic C (SmC) mesophase at low temperatures.

In this work, we present a six-armed macromolecule **1** with C_3 symmetrical arrangements of the rigid side group on the central aromatic core. Chiral dialkoxy substituted three triazines were connected to the central benzene at 1,3,5-positions via rigid bridges. The remaining alternating position of the central benzene possessed three acetylenic thiophene units. We also prepared organic salt of **2** by mixing macromolecule **1** with 4-(dodecyloxy)benzoic acid (4-DBA) (Scheme 1) [20], which shows enantiotropic mesophases, ‘inequimolar’ ratio under ultrasonic conditions.

Result and discussion

The synthesis of the targeted macromolecule **1** was accomplished by a cross coupling reaction between the key intermediates **5** and **9** in the presence of $\text{Pd}(\text{PPh}_3)_4$ (Scheme 2). Intermediate **5** was prepared in two steps starting from

Scheme 2



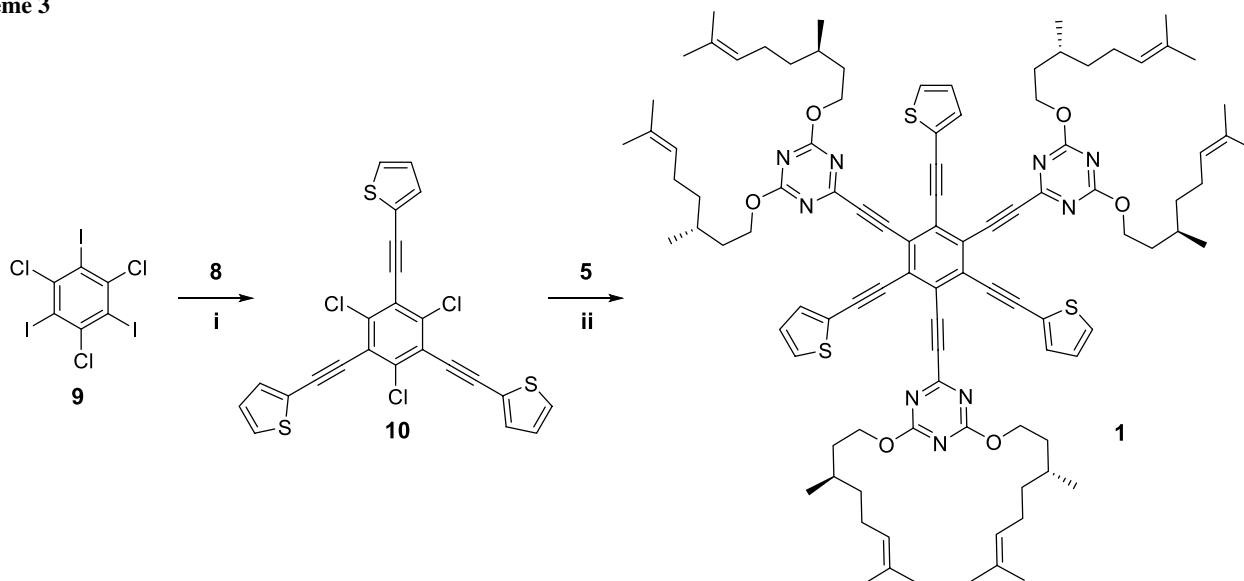
i) (S)-Citronellol (2 eq), K_2CO_3 , THF, 50 °C, 6 h. ii) Ethynyltrimethylsilane, K_2CO_3 , $Pd(PPh_3)_4$, CuI, THF, reflux, 6 h. iii) 2-Methylbut-3-yn-2-ol, $Pd(PPh_3)_4$, CuI, K_2CO_3 , THF, reflux. iv) K_2CO_3 , toluene, 80 °C, 12 h.

2,4,6-trichloro-1,3,5-triazine (**3**), which was reacted with two equivalents of (S)-(-)- π -citronellol followed by treating the resulting compound **4** with trimethylsilyl acetylene in the presence of palladium catalyst. The ethynylthiophene **8** was prepared as outlined in Scheme 2. Intermediate **9** was prepared by a cross coupling reaction between 1,3,5-trichloro-2,4,6-triiodobenzene, which was prepared with a reported procedure [32] from 1,3,5-trichlorobenzene, and 2-ethynylthiophene (**8**) in the presence of tetrakis(triphenylphosphine)palladium catalyst.

Six-armed shape persistent material **1** was found to be colourless oily substance at room temperature and converted to an organic salt by mixing with 4-(dodecyloxy)benzoic acid in a 1:1 ration in dry THF and sonicating the mixture at room temperature for 15 min. Macromolecule **1** and organic salt **2** were characterized by 1H NMR, ^{13}C NMR, FT-IR, and QTOF analyses (Schemes 3, 4).

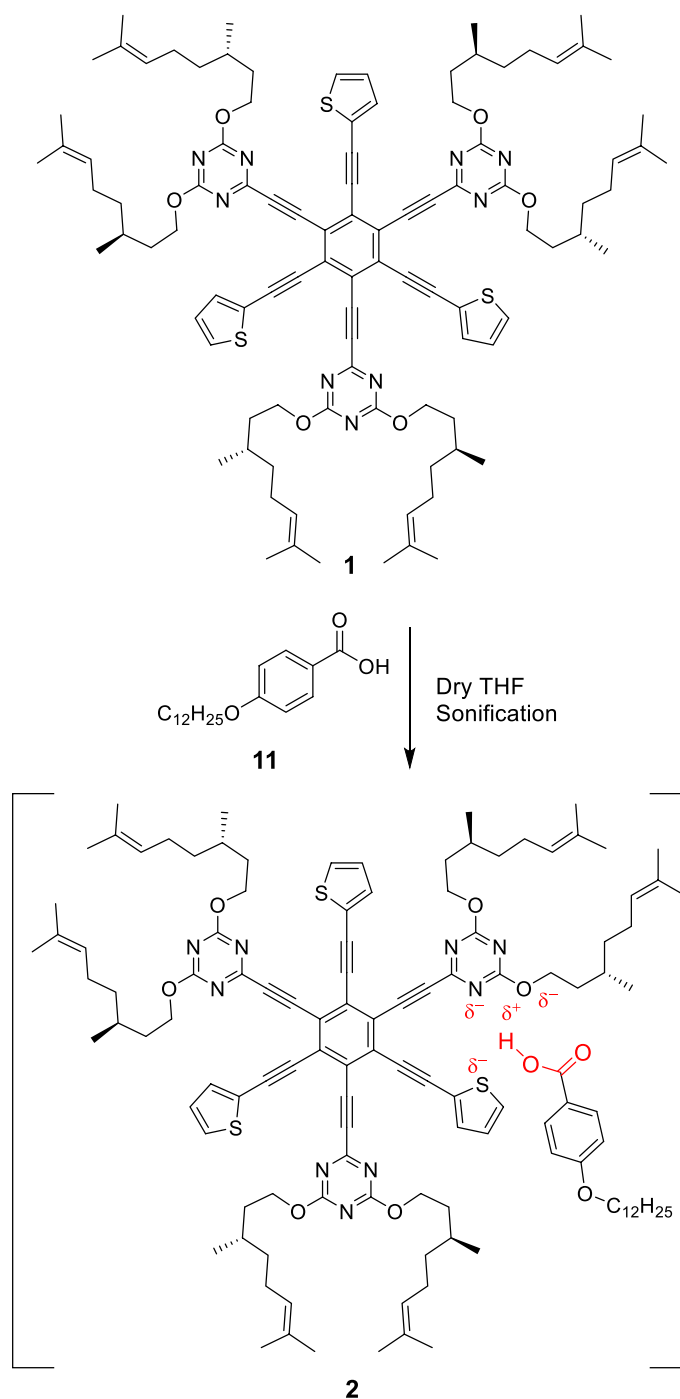
The formation of organic salt between six-armed π -conjugated system and the mesogenic carboxyl group took place by ionic interaction and mainly studied by FT-IR (Fig. 1). Asymmetric stretching of the sharp carbonyl peak

Scheme 3



i) $Pd(PPh_3)_4$ (0.02), CuI (0.04), Et_3N (3.2 eq), dioxane, 75 °C, 6 h.
ii) $Pd(PPh_3)_4$, CuI, K_2CO_3 , dioxane, 80 °C, 16 h.

Scheme 4



of the carboxylic acid 4-DBA at 1680 cm^{-1} shifted to 1570 cm^{-1} .

The formation of organic salt **2** was also followed by NMR spectroscopy. The signals belong to the aromatic protons of the alkoxy benzoate unit shifted from 8.05, 6.95 ppm to 7.85, 6.70 ppm, respectively, due to changes in electron density of the aromatic ring. Similarly, the signals of oxymethylene protons of 4-DBA shift to higher field 3.8 ppm as compared with the signals of pure 4-DBA at 4.05 ppm

(Fig. 2). Additionally, the signals of thiophene protons in pure macromolecule **1** at 7.32, 6.95 ppm were shifted after the formation of the salt with alkoxybenzoic acid organic salt **2** to 7.15, 6.82 ppm. The changes in chemical shift values are considered as a result of the complex formation between macromolecule **1** and 4-(dodecyloxy)benzoic acid. However, the signals of oxymethylene protons which belong to compound 4-DBA experienced a lesser shift from 4.25 to 4.13 ppm due to the fact that their electronic environment

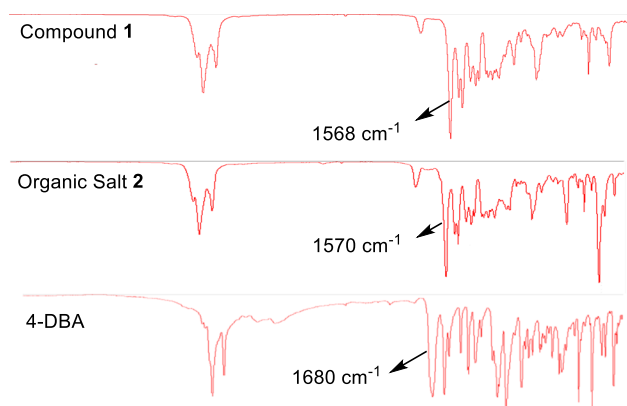


Fig. 1 FT-IR spectra of compound **10**, the organic salt **2** and benzoic acid 4-DBA

did not change much. Furthermore, the ^{13}C NMR spectra show that the carbonyl carbon shifted to a higher field 166.8 ppm as compared with the signals of pure 4-DBA at 171.6 ppm. On the other hand, slight shifting was observed of the triazine ring carbon from 171.5 to 170.8 ppm (Fig. 3).

The mesomorphic properties of the six-armed organic salt **2** were investigated by differential scanning calorimeter (DSC) and using polarized optical microscope (POM). Compound 4-DBA with a *n*-dodecyloxy terminal chain which were previously reported [19, 20], shows enantiotropic tilted smectic phase (SmC) as well as nematic (N) phase. The phase transitions of the corresponding molecules are given in Table 1.

As shown in Fig. 4a, the differential thermograms of 4-DBA show three endotherms for a phase transition sequence of crystal (Cr)—smectic C (SmC)—nematic (N)—isotropic phase (iso). On cooling from isotropic phase, the same behaviour of reverse transitions was

observed. Additionally, a calorimetric peak corresponding to Cr2–Cr1 transition at 65.86 °C was detected in cooling DSC thermogram.

On heating DSC, the organic salt **2** shows transition from crystal to isotropic state at 86.13 °C. On cooling from isotropic, a dendritic growing texture started to appear at 72 °C and the texture was preserved until 51 °C under POM. This observation was in agreement with the two exotherms at 65.24 °C and 54.84 °C, respectively, corresponding to a phase transition sequence of isotropic phase (iso)-liquid crystalline (LC) mesophase-crystal (Cr) on DSC cooling curve (see Fig. 4b). The liquid crystalline mesophase was assigned as Col mesophase which the columns are stacked in either rectangular or hexagonal 2D lattices. Typical textures of organic salt **2** observed on cooling are given in Fig. 5.

Herein, we would like to mention the mesomorphic properties of the organic salt **12** (see Fig. 6) prepared from the tri-armed macromolecule with chiral citronellyloxy side groups and 4-DBA by mixing 1:1 ratio [20]. The comparison of organic salts **2** and **12** with two chiral citronellyloxy side groups attached to three 1,3,5-triazine units at the periphery shows that the structural change on macromolecule of organic salt from the tri-armed to six-armed macromolecule based on central benzene core with three acetylenic thiophene units lead to the formation of columnar mesophase which was traditionally observed for disc-shaped molecules. As a result of the decreased non-covalent intermolecular interaction due to steric hindrance at the periphery, the thermal range of columnar mesophase of organic salt **2** is about 10 °C whereas SmC phase range of organic salt **12** is significantly much higher as well as the mesophase is more stable. One point we noted here for organic salt **2** that the introducing of three acetylenic thiophene units which leads to six-armed structure gives rise

Fig. 2 The comparison of ^1H NMR spectra (in CDCl_3) of macromolecule **1**, organic salt **2** and benzoic acid 4-DBA

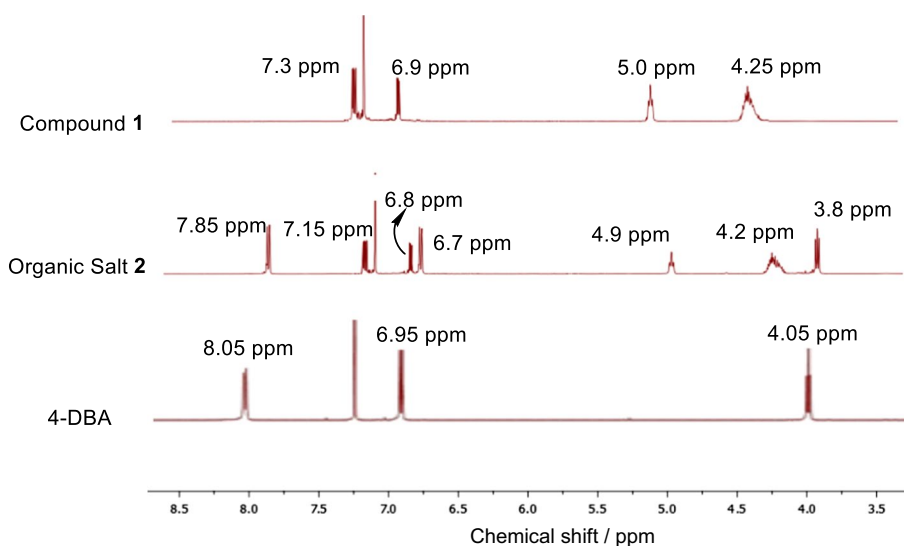


Fig. 3 ^{13}C NMR spectra in CDCl_3 of macromolecule **1**, organic salt **2** and benzoic acid 4-DBA

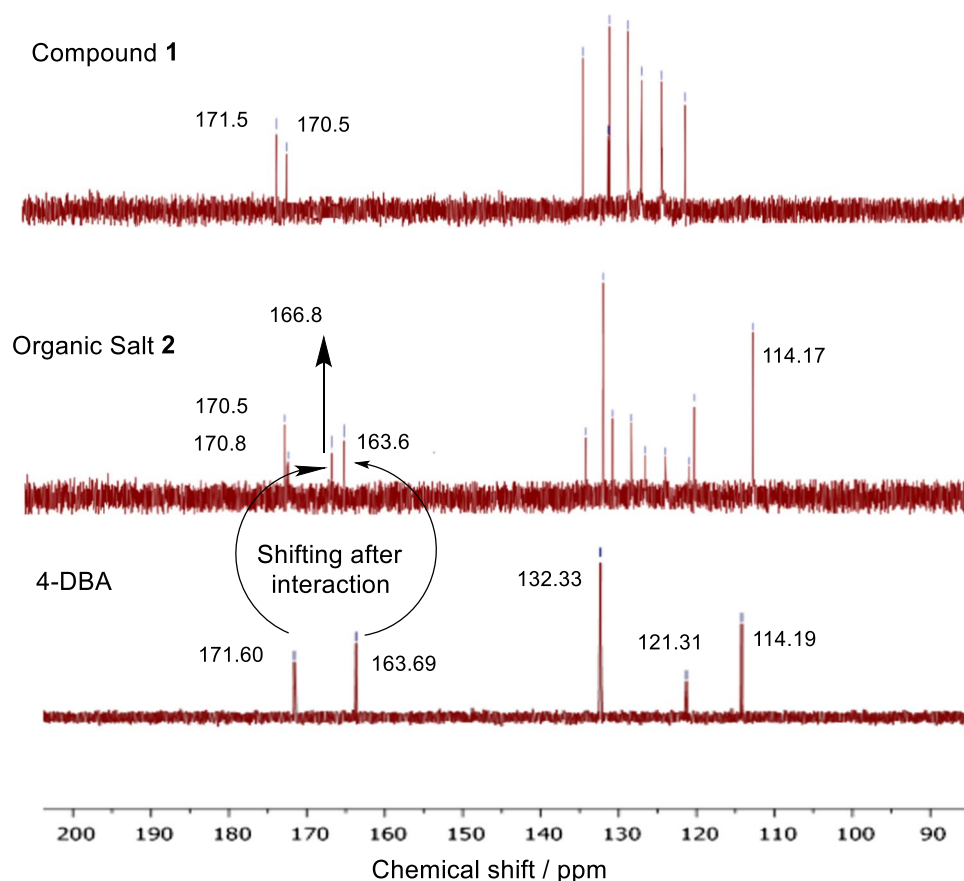


Table 1 Mesophases and phase transition temperatures as observed on heating ($\text{H} \rightarrow$) and cooling ($\leftarrow \text{C}$) and corresponding transition enthalpies of the compounds 4-DBA and organic salt **2** (OS2)

Comp	$T/^\circ\text{C}$ [$\Delta H / \text{kJ mol}^{-1}$]
4-DBA	$\text{H} \rightarrow$: Cr 99.98 [39.01] SmC 132.43 [2.39] N 138.42 [2.05] Iso
	Cr1 65.86 [-6.40] Cr2 86.95 [-10.77] SmC 128.12 [-2.08] N 134.12 [-2.48] Iso: $\leftarrow \text{C}$
OS 2	$\text{H} \rightarrow$: Cr 86.13 [32.66] Iso
	Cr 54.84 [-14.67] Col 65.24 [-] Iso: $\leftarrow \text{C}$

^aPerkin–Elmer DSC-6; enthalpy values in italics in brackets taken from the 1st heating and cooling scans at a rate of $10^\circ\text{C min}^{-1}$

Cr crystal, SmC tilted smectic phase, N nematic phase; Col columnar mesophase, Iso isotropic phase

to a drastic change on melting and clearing temperatures as well as inducing columnar phase at lower temperatures.

Conclusion

In summary, we have successfully synthesized and characterized a six-armed macromolecule, which is composed of a benzene unit as the central core, three thiophene units and

three 1,3,5-triazine rings carrying two (*S*)-citronellyloxy branched chains which are connected to the benzene core by acetylene bridges. The presence of three triazines substituted with chiral citronellyloxy and three thiophene rings on the central molecular was unable to induce mesomorphism of triazines derivatives. Therefore, the final product was mixed with 4-DBA through hydrogen-bonded interaction that may increase the ability to form liquid crystal material. The organic salt of the six-armed macromolecule with 4-(dodecyloxy)benzoic acid prepared by mixing 1:1 ratio in THF exhibited a monotropic columnar mesophase at lower temperatures in a narrow mesomorphic range.

Experimental

The following reagents and solvents were acquired without additional purification from Merck: cyanuric chloride, ethynylbenzene, 1,3,5-trichlorobenzene, 2-iodo-thiophene, 2-methylbut-3-yn-2-ol, copper iodide, potassium carbonate, tetrakis(triphenylphosphine)palladium, tetrahydrofuran, and dioxane. Thin-layer chromatography was carried out on aluminum plates (20×20 cm) covered with silica gel 60 F254 (Merck), whereas column chromatography was carried out on Merck's silica gel 60 (0.063–0.200 mm).

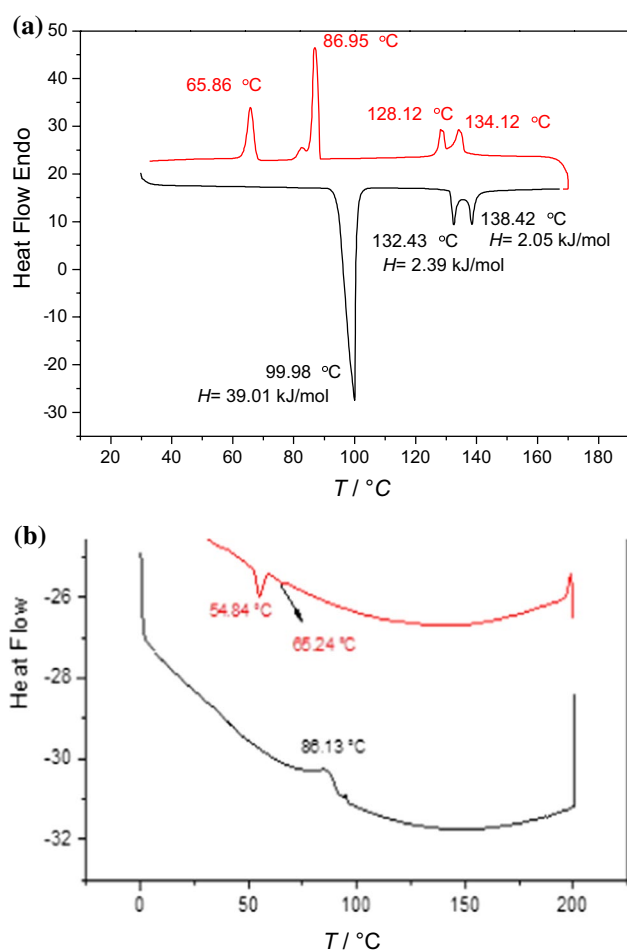


Fig. 4 DSC thermogram of compounds 4-DBA (a) and OS 2 (b) on 1st heating and cooling (10 °C min^{-1})

High-resolution mass spectrometry (HRMS), FT-IR (Shimadzu Prestige-21, KBr discs), ^1H NMR (500 MHz) and ^{13}C NMR (125 MHz) (CDCl_3 , standard internal TMS) spectrometers were used to determine the structure of compounds. Differential scanning calorimetry (DSC) and polarized optical microscope (POM) was used to evaluate the organic salts' liquid crystalline state.

The preparation of compound 4, 5, 7, 8, and 9 were reported in the references [20, 26, 31], respectively.

2,2',2''-[(2,4,6-Trichlorobenzene-1,3,5-triyl)-tris(ethyne-2,1-diyl)]trithiophene (10, $\text{C}_{24}\text{H}_9\text{Cl}_3\text{S}_3$) A mixture of 0.5 g 1,3,5-trichloro-2,4,6-triiodobenzene (9, 0.89 mmol), 0.48 g 2-ethynylthiophene (8, 2.69 mmol), 0.1 g $\text{Pd}(\text{PPh}_3)_4$ (0.089 mmol), 0.03 g CuI (0.178 mmol), and 0.39 g K_2CO_3 (2.84 mmol) were dissolved in 10 cm^3 of dioxane then stirred at 75 °C for 6 h. under argon atmosphere. The solution was poured into a mixture of 20 cm^3 ethyl acetate and 20 cm^3 water. The organic layer was washed and dried over sodium sulfate. The solvent was removed under vacuum and the residue was purified by column chromatography with hexane/ethyl acetate (5% EtOAc) as an eluent to give pale yellow powder. Yield 0.37 g (84%); m.p.: $124\text{--}127\text{ °C}$; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.33$ (m, 3H, Ar-H), 7.15 (m, 3H, Ar-H), 6.75 (dt, $J = 6.8, 3.4$ Hz, 3H, Ar-H) ppm; ^{13}C NMR (126 MHz, CDCl_3): $\delta = 145.7, 140.8, 136.9, 131.5, 128.9, 126.3, 100.4, 98.1$ ppm; HRMS: m/z calcd for $\text{C}_{24}\text{H}_9\text{Cl}_3\text{S}_3$ (M^+) 499.88, found 499.8921 (M^+) and 500.8859 ($[\text{M} + \text{H}]^+$).

6,6',6''-[[2,4,6-Tris(thiophen-2-ylethynyl)benzene-1,3,5-triyl]-tris(ethyne-2,1-diyl)]tris[2,4-bis[(3,7-dimethyloct-6-en-1-yl)-oxy]-1,3,5-triazine] (1, $\text{C}_{99}\text{H}_{123}\text{N}_9\text{O}_6\text{S}_3$) Compound 10 (0.14 g, 0.28 mmol), 0.41 g 2,4-bis[(3,7-dimethyloct-6-en-1-yl)-

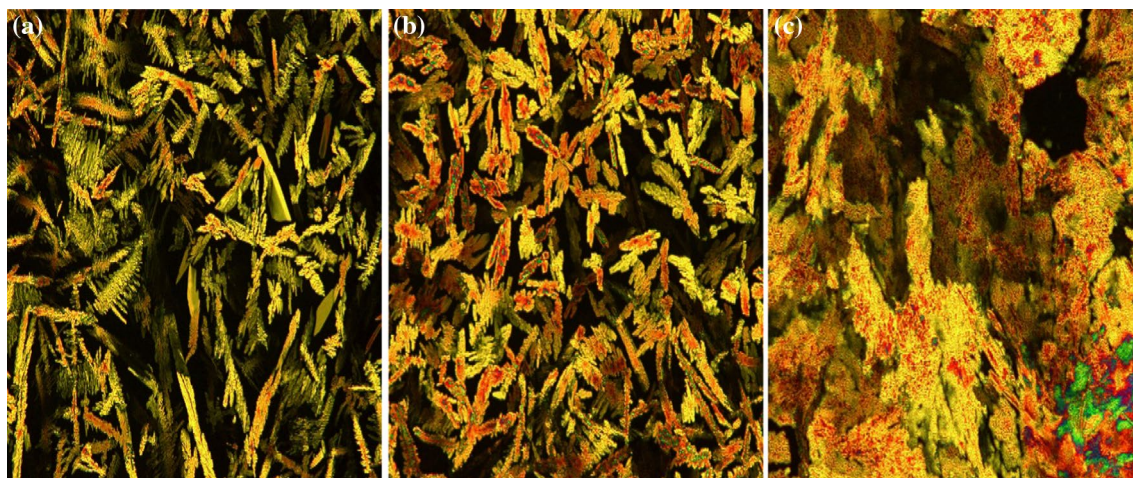


Fig. 5 Optical textures of organic salt 2 as observed between crossed polarizers in ordinary glass plates on cooling: **a** dendritic texture of Col phase at 62 °C and **b** 57 °C ; **c** crystalline phase at 42 °C

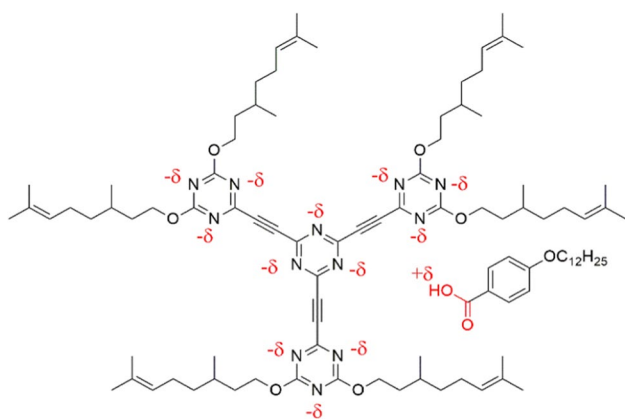


Fig. 6 The macromolecule structure of organic salt 2,4,6-tris[(4,6-bis(*S*)-citronellyloxy)ethynyl]-1,3,5-triazine (**12**)

oxy]-6-[(trimethylsilyl)ethynyl]-1,3,5-triazine (**5**, 0.84 mmol), 0.032 g Pd(PPh₃)₄ (0.028 mmol), 0.01 g CuI (0.056 mmol), and 0.136 g K₂CO₃ (0.98 mmol) were dissolved in 10 cm³ of dioxane then stirred at 80 °C for 16 h under argon atmosphere. The solution was poured into a mixture of 20 cm³ ethyl acetate and 20 cm³ water. The organic layer was washed and dried over sodium sulfate. The solvent was removed under vacuum and the residue was purified by column chromatography with hexane/ethyl acetate (5% EtOAc) as an eluent to give colourless oil. Yield 0.39 g (86%); ¹H NMR (500 MHz, CDCl₃): δ = 7.32 (m, 6H, Ar-H), 6.95 (m, 3H, Ar-H), 5.07 (s, 6H), 4.37 (t, 12H, OCH₂), 1.97 (m, 12H, CH₂), 1.75 (m, 18H, CH₃), 1.63 (m, 8H, CH₂), 1.25–0.95 (m, 30H, CH₂), 0.75 (m, 9H, CH₃) ppm; ¹³C NMR (126 MHz, CDCl₃): δ = 171.5, 170.2, 134.4, 131.1, 128.9, 127.4, 127.1, 124.8, 124.6, 121.9, 101.2, 93.1, 77.8, 65.6, 41.6, 37.7, 37.5, 36.4, 36.3, 31.9, 30.2, 29.9, 29.4, 25.7, 25.5, 22.7, 19.5, 17.6, 14.1, 13.5, 13.1 ppm; FT-IR: $\bar{\nu}$ = 2922, 2853, 1734, 1568, 1622, 1522, 1503, 1458, 1430, 1412, 1382 cm⁻¹; HRMS: *m/z* calcd for C₉₉H₁₂₃N₉O₆S₃ (M⁺) 1631.29, found 816.4493 ([M + 2H]²⁺).

Organic salt 2 (C₁₁₈H₁₅₃N₉O₉S₃) 4-DBA mesogenic unit **10** with a carboxyl group was added into a solution of compound **1** in 10 cm³ of dry THF with one-to-one ratio. The resulting solution was sonicated for 15 min until observing a transparent solution. Then, the solvent was removed in vacuum. ¹H NMR (500 MHz, CDCl₃): δ = 7.85 (m, 1H, Ar-H), 7.15 (m, 6H, Ar-H), 6.82 (m, 3H, Ar-H), 6.75 (m, 1H, Ar-H), 4.92 (s, 6H), 4.17 (t, 12H, OCH₂), 3.82 (t, 2H, OCH₂), 1.85 (m, 18H, CH₃), 1.65–1.32 (m, 24H, CH₂), 1.24–0.85 (m, 60H, CH₂), 0.75 (m, 18H, CH₃) ppm; ¹³C NMR (126 MHz, CDCl₃): δ = 171.7, 170.6, 165.6, 163.6, 134.4, 132.2, 131.1, 128.9, 127.2, 124.7, 121.9, 121.2, 114.1, 68.2, 65.6, 41.5, 37.2, 37.1, 35.6, 35.8, 35.5, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 25.9, 25.7, 25.4, 22.6, 19.4,

17.6, 14.1 ppm; FT-IR: $\bar{\nu}$ = 2922, 2850, 1738, 1570, 1521, 1431 cm⁻¹; HRMS: *m/z* calcd for C₁₁₈H₁₅₃N₉O₉S₃ (M⁺) 1937.73, found 387.4189 (M⁵⁺) and 426.3900 ([M + 5 K]⁵⁺).

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s00706-022-02969-x>.

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