



Ionic interaction of tri-armed structure based on benzene ring: synthesis and characterization

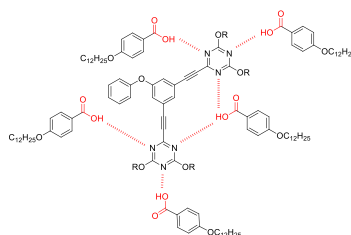
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Abstract

Liquid crystalline is a state of matter which combines the properties of liquid and a crystalline solid. Ionic liquid crystals are a new class of materials which exhibit phase transition at low temperature. A new structure was synthesized based on a benzene ring carrying three arms, two arms of triazine ring and the third arm is phenoxy benzene. The presence of alkyl chain groups on the periphery reduced the mesophase transitions which are desirable for low-temperature applications of liquid crystals. The substitution was achieved efficiently by Sonogashira coupling. The tri-armed compound was synthesized successfully through cross-coupling. However, it was oily and no transition phase was observed. Therefore, to increase the liquid crystal possibility, the compound was mixed with 4-(dodecyloxy)benzoic acid (4-DBA) with 1:1 ratio to produce an organic salt through hydrogen bonding. The organic salt obtained by ionic interaction between the tri-armed compound and 4-DBA exhibited a phase transition at low temperature. However, ionic liquid crystals are important class of mesomorphic materials and have considered as sealants due to their very low vapour pressure which demonstrated a proficient electrolyte systems for their anisotropic phase such as high ionic conductivity and diffusion along with excellent thermal stability. All the intermediate compounds and final compounds were confirmed by spectroscopic techniques (¹H NMR, ¹³C NMR, FT-IR, and HRMS).

Graphic abstract



Keywords Tri-armed structure · Triazine ring · Hydrogen bonding · Ionic interactions

Introduction

New compounds containing a 1,3,5-triazine unit showed much in the synthesis of liquid crystals molecular and investigated their properties according to the literature [1]. The liquid crystal state combines the properties of the liquid phase (capacity of flowing) with the solid phase (capacity of an ordered and crystalline structure) [2]. Star-shaped systems containing 1,3,5-triazine derivatives have been used as acceptors in materials chemistry. The important structures that contain the s-triazine core and other units as donors

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substituent's allows the optoelectronic properties which led these materials to use in luminescent liquid crystals [3, 4]. The donor substituent such as styryl benzene [5], thiophene [6], 1,2,3-triazole [7], tetrathiafulvalene [8], ferrocene [9], 2-pyridyl [10], and bisphenylaminobenzene [11].

Triazine has been also used as functional materials from hard to soft porous frameworks [12], supported catalysts [13], absorption of surfactants from the aqueous solution [14], cathodes for lithium batteries [15], and nonporous membranes for desalination [16]. The chemistry of triazines since past two centuries has been studied intensively due to their physical and chemical properties especially in pharmaceutical and industry. However, recently the research using 1,3,5-triazines unit as starting material carried out on the antitumor activity [17, 18].

1,3,5-Triazine has been used as a core [19]. Besides, cyanuric chloride has been easily interacted with various nucleophilic side groups to obtain liquid crystals molecularly depending on their structures which can exhibit calamitic morphological behaviours or discotic [20].

Soft materials of liquid crystals are unique groups that have both the solid state and the liquid state and exhibit a molecular organization [21]. The molecules display a long-range order by occupying fixed positions at regular distances in the crystalline phase, while the molecules are free to move around in both positional and orientational order in the liquid phase [22]. Therefore, there is an exceptional intermediate state demonstrated between isotropic liquids and isotropic solids in the liquid crystalline materials. The fourth state of matter was related to this intermediate which is called a mesophase [23].

Liquid crystal mesophases depends on the molecular size, shape of the structure, the therapeutic objective and the analytical significance for their identifications well as their applications in other fields [24]. Among these materials, disc-like liquid crystals are particularly gathering attention especially concerning to their unique optical properties.

Triazine-based chemistry has a wide range of applications as agrochemicals, inhibitors, industries, antitumor, and flame retardants, but also shows a high affinity for liquid crystal properties and the interest in triazine-containing processes [25].

1,3,5-Triazine unit due to their high electron affinity and symmetric structure is widely used to construct optoelectronic materials as a strong electron-accepting center. The s-triazine and its derivatives have their importance in heterocyclic compounds due to their very good activities [26]. The heterocyclic compounds with nitrogen or sulfur atoms have been an interest in the design of new drugs or material due to their physiochemical properties [27]. Liquid crystals are widely used both scientifically and industrially; many compounds based on triazine unit were employed as electron injection and transport materials. The class of star-shaped

glassy nematic liquid crystals based on pentaerythritol as a flexible core was investigated in detail [28].

Therefore, the aim of this research is to synthesize new macromolecular structure based on benzene ring as a central core, the central core connected to three armed, 1,3-dichloro-5-phenoxybenzene arm, which obtained from the reaction of 1,3,5-trichlorobenzene and phenol and two arms of 2-chloro-4,6-bis(3,7-dimethyloct-6-en-1-yloxy)-1,3,5-triazine which obtain from the reaction between 1,3,5-triazine and citronellol. These arms may be linked to the central core through rigid linkers or flexible or semi-flexible.

Results and discussion

In this study, a new tri-armed compound was successfully obtained through cross-coupling starting from 2,4,6-trichloro-1,3,5-triazine and 1,3,5-trichlorobenzene as a starting material. The target compound and the intermediate compounds were confirmed by spectroscopic techniques. The result showed no liquid crystals of the tri-armed compound. Therefore, it was reacted with 4-(dodecyloxy)benzoic acid (4-DBA) resulting in an organic salt via intermolecular hydrogen bonding to increase the possibility of liquid crystalline of the mixture. 4-DBA, its known compound which already has liquid crystal properties. The obtained organic salt is confirmed mainly by FT-IR, ^1H NMR, ^{13}C NMR, and HRMS.

The ionic interaction between the tri-armed π -conjugated system and the mesogenic carboxyl group was mainly studied by FT-IR. The carboxylic peak corresponding to 4-DBA at 1680 cm^{-1} shifted to 1670 cm^{-1} in organic salt (Fig. 1) [1].

Additionally, the mass spectrometry was also confirmed the molecular weight of the organic salt **11** by the presence of $(M + 2\text{H})^{+2}$ peak at $m/z = 650.43$ ($650 \times 2 - 2 = 1299.8$), which calculated for $\text{C}_{81}\text{H}_{114}\text{N}_6\text{O}_8$ 1299.8 (Fig. 2).

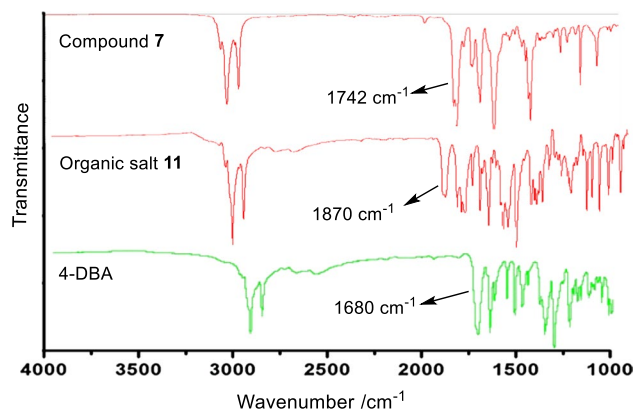
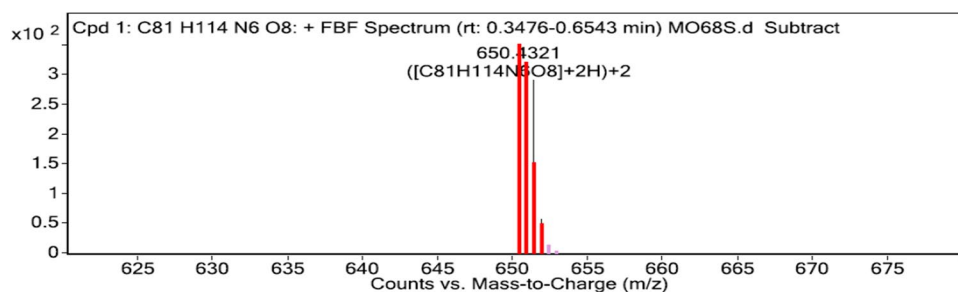


Fig. 1 FT-IR spectrum of 4-DBA, organic salt **11**, compound **7**

Fig. 2 Mass spectrum of the organic salt **11****MS Spectrum Peak List**

<i>m/z</i>	<i>z</i>	Abund	Formula	Ion
650,4321	2	351,47	C ₈ H ₁₁ N ₆ O ₈	(M+2H)+2
650,9344	2	89,13	C ₈ H ₁₁ N ₆ O ₈	(M+2H)+2
651,4484	2	290,53	C ₈ H ₁₁ N ₆ O ₈	(M+2H)+2
651,9315	2	55,84	C ₈ H ₁₁ N ₆ O ₈	(M+2H)+2

Table 1 Chemical shifts (δ /ppm) of relevant protons in CDCl₃ as the solvents (25 °C)

Compound	Aromatic Ha/ ppm	Aromatic Hb/ ppm	-OCH ₂ -/ppm
Compound 7	–	–	4.05
4-DBA	8.05	6.95	4.15–3.97
Salt 11	7.80	6.73	3.75

¹H NMR showed the signals corresponding to the aromatic protons of an alkoxy benzoate unit in organic salt observed at 7.8, 6.7 ppm as compared to pure benzoic acid at 8.05, 6.95 ppm. These shifts of the aromatic protons of organic salt compared to pure 4-DBA was due to the ionic interaction between triazine and a carboxyl group of the mesogenic unit which increased the electron density of the aromatic ring. Besides, the signals of oxymethylene protons of 4-DBA in ion complex shift to higher field 3.75 ppm as compared with the signals of pure 4-DBA at 4.05 ppm. Whereas, the protons corresponding to benzene rings observed at 7.4, 7.3, 7.20 ppm (Table 1).

Also, the organic salt proved by the ¹³C NMR spectra. The signal of the carbonyl carbon in an organic salt shifted from 171.6 to 166.7 ppm. These changes in chemical shift due to increase of electron density after the ionic interaction, whereas the aromatic carbon next to alkoxy group shift slightly from 163.6 to 163.9 ppm, that's lead to decrease in electron density of carbon in case of ionic interaction form. Also, the triazine ring carbon observed at 172.01 and 170.98 ppm (Table 2).

The organic salt was investigated by differential scanning calorimeter (DSC). Upon heating curves, the organic salt **11** shows two peaks at 51.9 °C and 11 °C. On cooling from isotope phase, one peak was observed at 96.3 °C (Table 3).

Table 2 Chemical shifts (δ /ppm) of relevant carbon of 4-DBA before and after interaction in CDCl₃ as the solvents (25 °C)

Compound	Aromatic Ca/ppm	Aromatic Cb/ppm
4-DBA	171.6	163.6
Salt 11	166.7	163.9

Table 3 Phase transition as observed on heating (H→), cooling (←C) of the organic salts **11** and 4-DBA

Compound	<i>T</i> /°C/ ΔH /kJ mol ⁻¹
4-DBA ^b	H→: Cr 99.98 [39.01] SmC 132.43 [2.39] N 138.42 [2.05] Iso
OS (11)	H→: Cr 11.3 [– 35], 51.9 [– 11], 96 [– 42] Iso

The tri-armed compound was oily due to the citronellyloxy chains which are positioned at the peripheries of triazine. The compound showed no liquid crystals and no transition phase of liquid properties in its pure state. Therefore, the compound was mixed with 4-DBA in 1:1 ratio via intermolecular hydrogen bonding to obtain the organic salt through ionic interaction to increase the liquid crystal possibility. However, this method showed significant points in producing liquid crystal molecules but at low temperature.

Ionic interactions through hydrogen bonding have a propensity to be stable for lamellar mesophases. However, ionic liquid crystals exhibit phase transition at low temperature which are more organized than the smectic A phase at a lower temperature [29].

The ability of the molecular to show liquid crystals properties depends on the structure and side chains. In other ways, increasing the length of the alkoxy chain leads to decrease in the temperature of the transition phase. That

is why the organic salt **11** which obtained via ionic interaction between tri-armed compounds and 4-DBA with a long alkoxy side chain showed liquid crystal at low temperature [30].

Ionic liquid crystal behaviour

Ionic liquid crystals can be considered as materials that contain anions and cations which combine the properties of liquid crystals and ionic liquids. However, the character of the ionic liquid crystals are different from the conventional liquid crystals. That ionic interactions play an important role in the stabilization of mesophases.

Most of the ionic compounds that studied showed a smectic A phase, but a columnar phase was observed for some of the salts (containing 64 terminal NH_2 groups).

The phase behaviour of liquid crystals are depending on the degree of protonation of primary amino groups as compared with that of the tertiary ones. Thus, protonation of the tertiary amino groups of the compound is more pronounced for the higher generation dendrimers than for the low generation dendrimers.

Ionic liquid crystals based on benzene rings have been created by the interaction of carboxylic acids in the compound of 4-DBA with triazines that are functionalized on their periphery with alkoxy chains. An ion pair is formed by the transfer of the proton of the carboxylic acid to the nitrogen, resulting in an organic salt. The salts obtained have higher transition temperatures due to the presence of hydrogen bonds within the structure.

In hydrogen-bonded liquid crystals, thermotropic mesophases are displayed by associations of a hydrogen-bond donor and a hydrogen acceptor. The building blocks of hydrogen-bonded liquid crystals are often not mesomorphic themselves, but the resulting supramolecular aggregates which exhibit a variety of ordered structures. These ionic liquids could be also used to immobilize transition metal catalysts in the liquid phase of two-phase catalytic reactions. Other applications include their use as a solvent for extraction processes and as an electrolyte for batteries, fuel cells, and dye sensitive solar cells [31] (Fig. 3).

The π - π stacking of the aromatic rings plays an important role in the mesomorphic properties and thermal stabilities of observed phases. The effect of changing the core moiety of 4-DBA molecules on the mesophase behaviour of 1:1 molar mixtures, comparison was constructed between the mesophase stabilities. Our study revealed that increasing the length and rigidity of the acid mesogenic core resulted in an increase of the stabilities and mesomorphic ranges of liquid crystal properties. Although the chain length of the alkoxy terminal of the carboxylic acid and the tri-armed compound increases the dimensional parameters with considerable

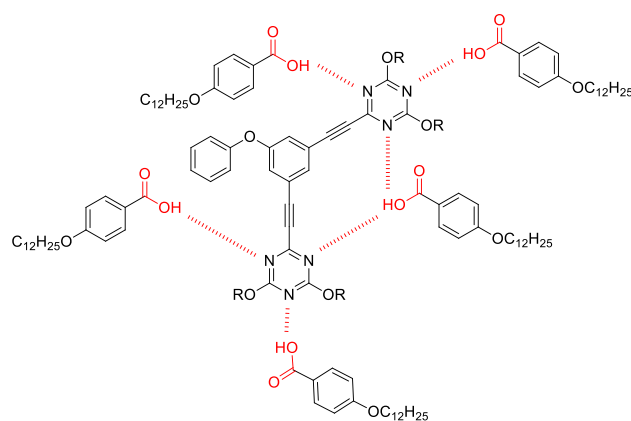


Fig. 3 Ionic interaction of 4-DBA with triazines in tri-armed compound [32]

value which led to decreases the stability of mesophase mixture, with increases of either the alkoxy chain of both 4-DBA and tri-armed structure. Therefore, the shorter chain length of the base and/or the acid resulted in the increment of the lateral interaction to enhance the more ordered phases range and stability (Fig. 4) [20].

Conclusion

Ionic liquid crystals are organic salts having synergistic properties of ionic liquids and liquid crystalline materials endowed with non-covalently bound. The main driving force for discovering ionic liquids is that these compounds have too low vapour pressure to be candidates to replace volatile organic solvents in organic reactions. Ionic liquids are generally considered designer solvents. New star-shaped compound was successfully obtained through cross-coupling which composed 2,4,6-trichloro-1,3,5-triazine carrying dodecyloxy chains positioned at the peripheries and 1,3,5-trichlorobenzene. The tri-armed compound was a non-liquid crystal. Therefore, the compound was mixed with 4-DBA via ionic interaction and through hydrogen bonding to obtain organic salt. The organic salt investigated by differential scanning calorimetry (DSC), two transition phases was formed after the ionic interaction between the compound **7** and 4-DBA resulting in an organic salt. All the synthesis compounds were confirmed by spectroscopic techniques (^{13}C NMR, ^1H NMR, FT-IR, and HRMS).

Experimental

1,3,5-Trichlorobenzene, cyanuric chloride, dodecan-1-ol, ethynyltrimethylsilane, copper iodide, tetrakis(triphenylphosphine) palladium, potassium carbonate,

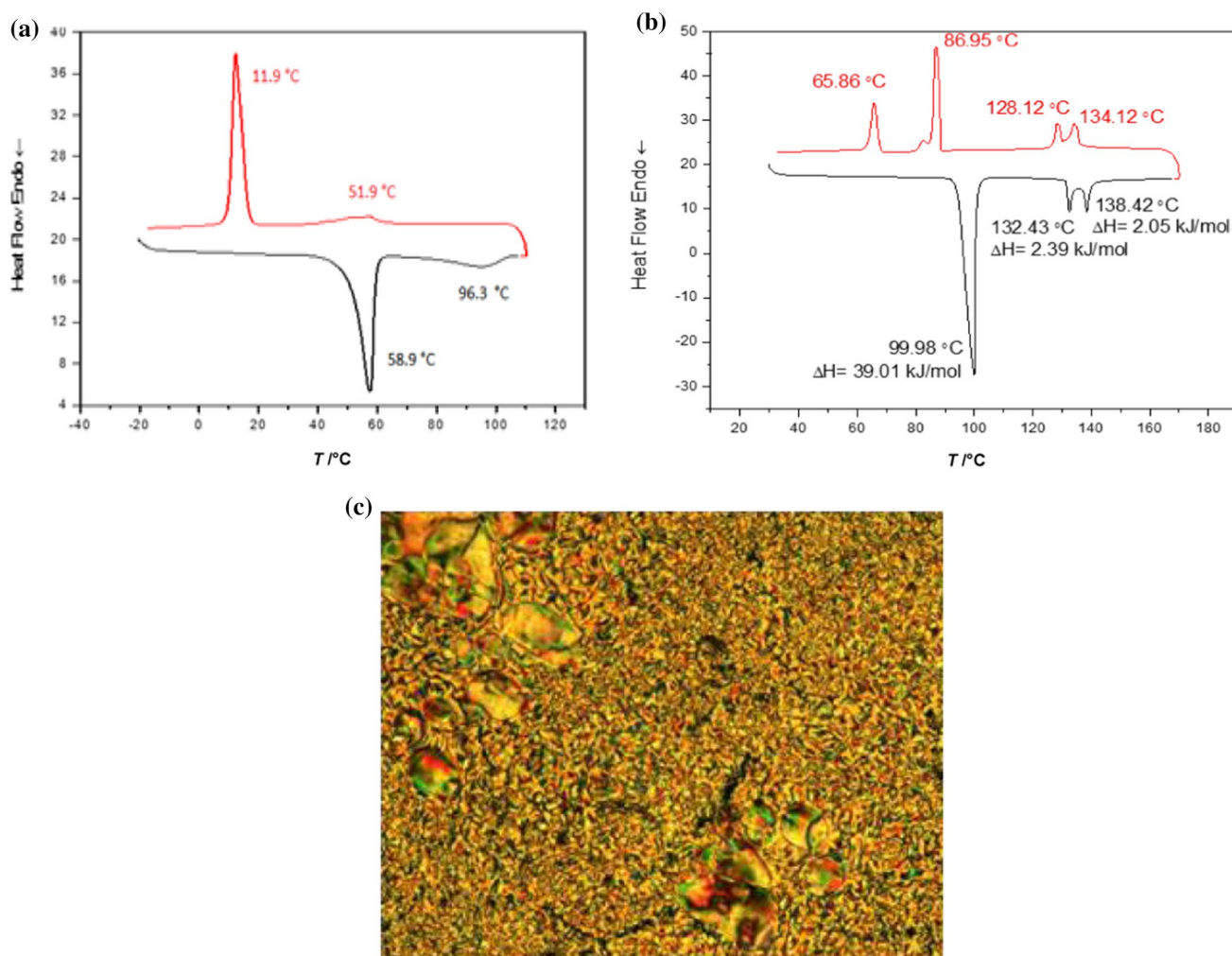


Fig. 4 DSC of organic salt **11** (a), DSC of compound 4-DBA (b), and a typical texture of smectic C mesophase (c)

triethylamine, dimethylformamide, tetrahydrofuran, and dioxane. Thin-layer chromatography was performed using aluminium plates (20×20 cm) coated with silica gel 60 F254 (Merck), while column chromatography was carried out using silica gel 60 (0.063–0.200 mm) from Merck. The reagents and solvents were used without further purification.

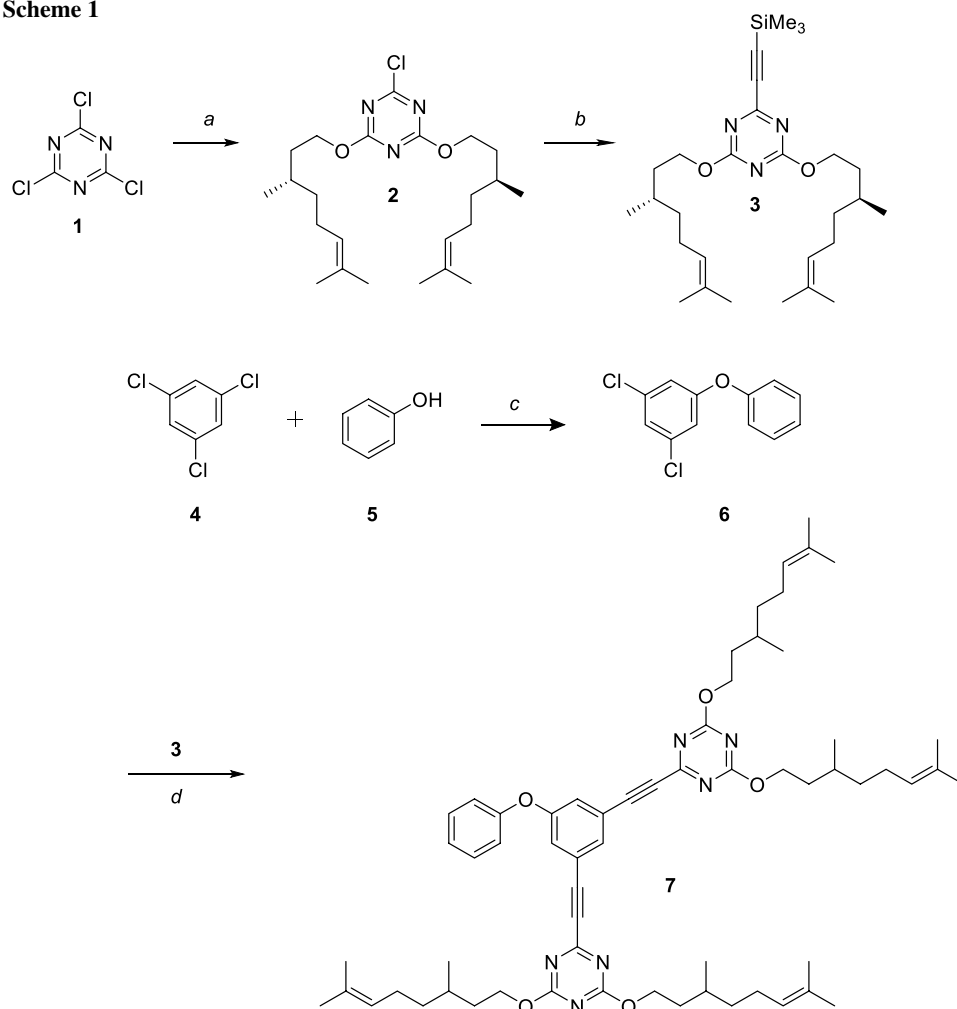
The ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance III (500 MHz) in CDCl_3 . The chemical shift values are expressed as δ value (ppm) down field with residual protons of the solvents (CDCl_3 , $\delta = 7.26$ ppm) as internal standard. FT-IR (Shimadzu Prestige-21, KBr discs), and HRMS (high-resolution mass spectrometry).

2-Chloro-4,6-bis(3,7-dimethyloct-6-en-1-yloxy)-1,3,5-triazine (2) [1] 2,4,6-Trichloro-1,3,5-triazine (**1**, 0.29 g, 1.6 mmol), 0.5 g 3,7-dimethyloct-6-en-1-ol (3.2 mmol), and 0.45 g K_2CO_3 (3.2 mmol) were dissolved in 10 cm^3 of THF under argon atmosphere and stirred at 50 °C overnight (Scheme 1). The solution was poured into a mixture of 10

cm^3 ethyl acetate and 10 cm^3 water. The organic layer after separation dried with sodium sulfate. Under vacuum, the solvent was evaporated and the crude product purified by column chromatography with hexane/ethyl acetate (2.5% EtOAc) as an eluent to yield 0.25 g (41%). ^1H NMR: $\delta = 5.14$ (dd, $J = 7.1, 1.3$ Hz, 2H), 4.5 (m, 4H, OCH_2), 2.09 (m, 4H, CH_2), 1.9 (m, 6H, CH_3), 1.8 (m, 6H, CH_2), 1.6–1.3 (m, 10H, CH_2), 1.07–0.95 (m, 6H, CH_3) ppm; ^{13}C NMR: $\delta = 172, 171, 131.5, 124, 69, 36.8, 35, 29, 25.7, 25.3, 19.2, 17.6$ ppm; HRMS: m/z calcd. for $\text{C}_{23}\text{H}_{38}\text{ClN}_3\text{O}_2$ (M^+) 423.27, found 424.27 ($[\text{M} + \text{H}]^+$), 441.311 ($[\text{M} + \text{NH}_4]^+$).

2,4-Bis(3,7-dimethyloct-6-en-1-yloxy)-6-(trimethylsilylethynyl)-1,3,5-triazine (3) [20] 2-Chloro-4,6-bis(3,7-dimethyloct-6-en-1-yloxy)-1,3,5-triazine (**2**, 1.2 g, 2.5 mmol), 0.29 g ethynyltrimethylsilane (2.96 mmol), 0.4 g K_2CO_3 (21.9 mmol), 0.28 g $\text{Pd}(\text{PPh}_3)_4$ (0.24 mmol), and 0.09 g CuI (0.49 mmol) were dissolved in 10 cm^3 of THF under argon atmosphere then refluxed for 6 h (Scheme 1). The solution

Scheme 1



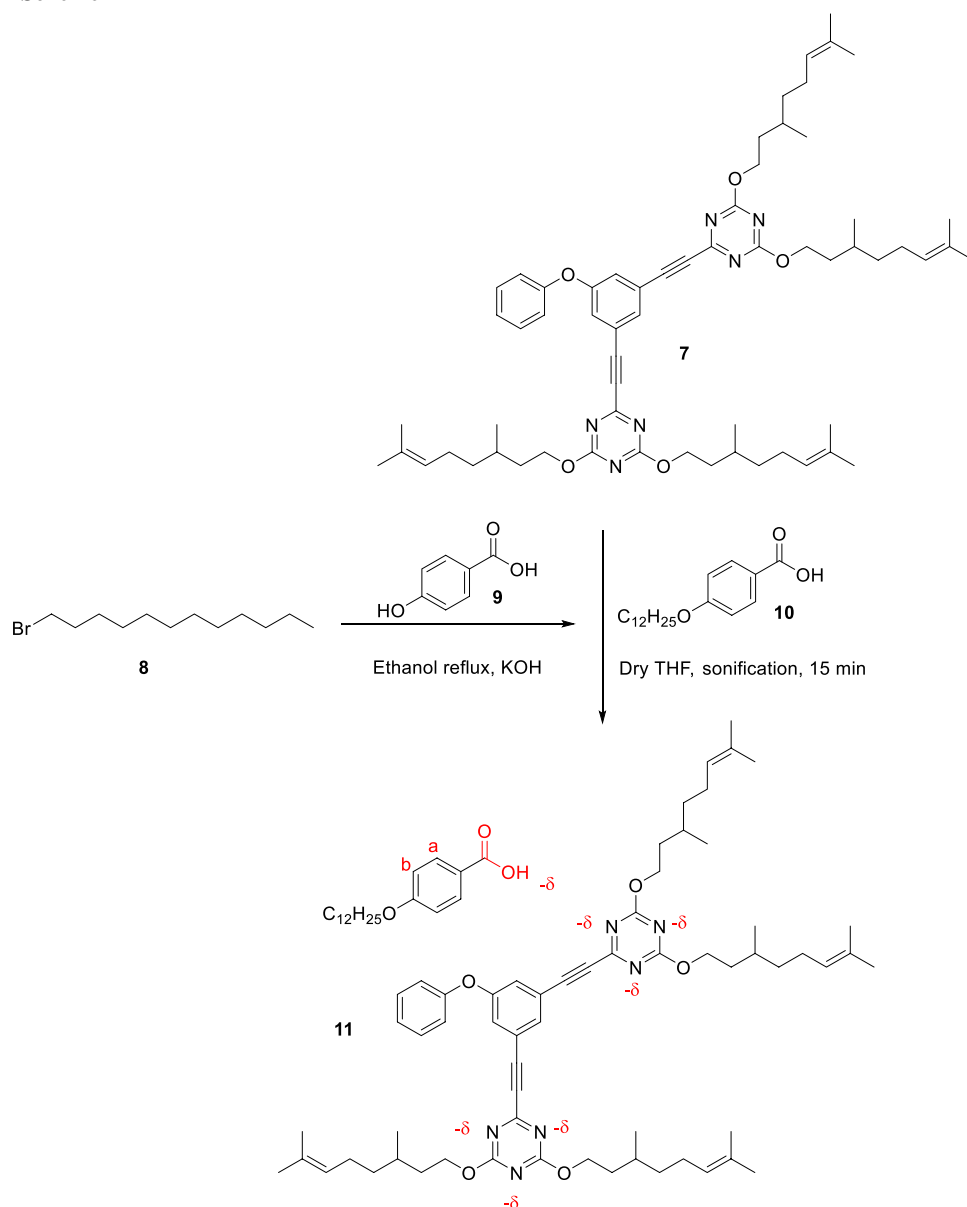
was poured into a mixture of 20 cm³ ethyl acetate and 20 cm³ water. The organic layer after separation dried with sodium sulfate. The solvent was removed under vacuum to give brown oily material with yield 1.1 g (81%). HRMS: *m/z* calcd. for C₂₈H₄₇N₃O₂Si (M⁺) 485.34, found 486.33 ([M + H]⁺), 508.32 ([M + Na]⁺).

1,3-Dichloro-5-phenoxybenzene (6) [33] A mixture of 2.0 g 1,3,5-trichlorobenzene (4, 11.1 mmol), 1.04 g phenol (5, 11.1 mmol), and 3.37 g K₂CO₃ (24.4 mmol) were dissolved in 10 cm³ of dioxane under argon atmosphere. The mixture was stirred for 6 h at 70 °C, then the temperature increased to 70 °C for 4 h (Scheme 2). The solution was poured into a mixture of 10 cm³ ethyl acetate and 10 cm³ water. Then the organic layer was dried over sodium sulfate and under vacuum the solvent was removed. The residue was purified by column chromatography with ethyl acetate/hexane (5% EtOAc) as an eluent to give white solid material (2.16 g, 81.8%). ¹H NMR: δ = 7.27 (s, 3H, Ar-H), 7.22 (d, 2H, Ar-H), 6.8 (d, 3H, Ar-H) ppm; HRMS: *m/z* calcd. for

C₁₂H₈Cl₂O (M⁺) 238.00, found 477.982 ([2 M]⁺), 715.988 ([3 M]⁺).

6,6'-[(5-Phenoxy-1,3-phenylene)bis(ethyne-2,1-diyl)]-bis[2,4-bis(3,7-dimethyloct-6-en-1-yloxy)-1,3,5-triazine] (7, C₆₂H₈₄N₆O₅) 1,3-Dichloro-5-phenoxybenzene (6, 0.2 g, 0.84 mmol), 0.46 g 2,4-bis(3,7-dimethyloct-6-en-1-yloxy)-6-(trimethylsilylethynyl)-1,3,5-triazine (3, 1.1 mmol), 0.09 g Pd(PPh₃)₄ (0.084 mmol), 0.031 g CuI (0.168 mmol), and 0.23 g K₂CO₃ (1.68 mmol) were dissolved in 10 cm³ of dioxane under argon atmosphere. The mixture was stirred for 16 h at 80 °C. 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. Under vacuum, the solvent was removed and the residue by column chromatography was purified with hexane/ethyl acetate (5% EtOAc) as an eluent to yield 0.30 g (60%). ¹H NMR: δ = 7.4 (s, 2H, Ar-H), 7.3 (s, 1H, Ar-H), 7.25 (d, 2H, Ar-H), 7.0 (m, 3H, Ar-H), 4.9 (s, 4H), 4.15 (t, 8H, OCH₂), 1.8 (m, 14H, CH₂), 1.6 (t, 8H, CH₂), 1.4 (m, 12H, CH₃), 1.25–0.9 (m, 42H,

Scheme 2



CH_2), 0.75 (m, 12H, CH_3) ppm; ^{13}C NMR: δ = 171.8, 170, 165, 164, 135, 133, 131, 124.9, 124.8, 123, 103, 64.6, 41.6, 41.4, 37.2, 35.9, 35.4, 29, 25, 19.5, 17.6, 13.5, 13.1 ppm; FT-IR (KBr): $\bar{\nu}$ = 2967, 2925, 2850, 1742, 1570, 1522, 1504, 1429, 1340, 1340, 1299 cm^{-1} ; HRMS: m/z calcd. for $\text{C}_{62}\text{H}_{84}\text{N}_6\text{O}_5$ (M^+) 993.37, found 992.65, 993.66 (M^+).

4-(Dodecyloxy)benzoic acid (10) [34] A solution of 4-hydroxybenzoic acid (16.43 mmol), 11 cm^3 1-bromododecane (46 mmol, 2.8 eq), and 2.58 g KOH (46 mmol, 2.8 eq) in 50 cm^3 ethanol was heated under reflux for 3 days. The hydrolysis was carried out by adding 10% aqueous KOH (25 cm^3) and the mixture refluxed overnight. After cooling down, the reaction mixture was acidified with HCl (6 M),

the precipitate filtered, washed with water, and recrystallized from ethanol to obtain the pure product 4-dodecyloxybenzoic acid as white solid material (yield 4.55 g, 91%). ^1H NMR: δ = 8.10 (d, 2H, Ar-H), 6.98 (d, 2H, Ar-H), 4.07 (t, 2H, OCH_2), 1.84 (m, 2H, CH_2), 1.48 (m, 2H, CH_2), 1.37–1.28 (m, 16H, CH_2), 0.91 (t, 3H, CH_3) ppm; FT-IR (KBr): $\bar{\nu}$ = 2914, 2848, 2559, 1670, 1604 cm^{-1} .

6,6'-[(5-Phenoxy-1,3-phenylene)bis(ethyne-2,1-diyl)]-bis[2,4-bis(3,7-dimethyloct-6-en-1-yloxy)-1,3,5-triazine] 4-(dodecyloxy)benzoate (11, $\text{C}_{81}\text{H}_{114}\text{N}_6\text{O}_8$) 4-DBA mesogenic unit (10) and 6,6'-[(5-phenoxy-1,3-phenylene)bis(ethyne-2,1-diyl)]bis[2,4-bis(3,7-dimethyloct-6-en-1-yloxy)-1,3,5-triazine] (7), with one to one ratio. The

sonication was done in dry THF for 10 min until a transparent solution was obtained. Then, the solvent was removed in vacuum. $^1\text{H NMR}$: $\delta=7.8$ (d, 2H, Ar-H), 7.4 (s, 2H, Ar-H), 7.25–6.9 (m, 6H, Ar-H), 6.7 (d, 2H, Ar-H), 4.8 (s, 4H), 4.15 (t, 8H, OCH₂), 3.8 (t, 2H, OCH₂), 1.75 (m, 12H, CH₂), 1.6 (m, 8H, CH₂), 1.4–1.3 (m, 24H, CH₃), 1.1–0.9 (m, 28H, CH₂), 0.7 (m, 15H, CH₃) ppm; $^{13}\text{C NMR}$: $\delta=172$, 170, 166, 163, 151, 134, 133.6, 133.3, 132.5, 131.4, 129.9, 126.7, 125, 123, 122, 121, 114, 69.3, 68.5, 41.6, 37.4, 37.1, 36.1, 35.4, 32.4, 29.98, 29.9, 29.8, 29.6, 29.5, 26.2, 25.99, 25.7, 25.6, 22.9, 19.7, 17.9, 14.4, 12 ppm; FT-IR (KBr): $\bar{\nu}=2914$, 2849, 2560, 1670, 1604, 1577, 1469, 1430, 1331 cm^{-1} ; HRMS: m/z calcd. for C₈₁H₁₁₄N₆O₈ (M⁺) 1299.8, found 650.43 ([M + 2H]²⁺).

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