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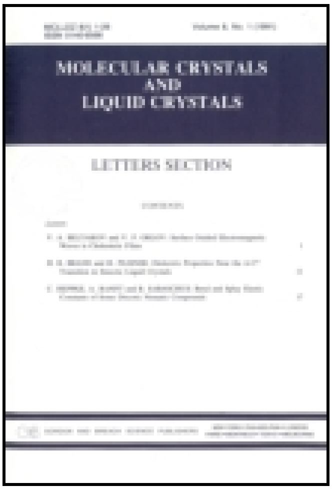

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Synthesis and Mesomorphic Properties of New Columnar Liquid Crystal Containing 1,3,5-triiminebenzene with Pendant 1,3,4-thiadiazole Group

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Synthesis and Mesomorphic Properties of New Columnar Liquid Crystal Containing 1,3,5-triiminebenzene with Pendant 1,3,4-thiadiazole Group

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A novel discotic liquid crystal series based on 1,3,5-benzenetrisazomethine derivatives with three pendant 2-amino-5-(4′-n-alkoxy)phenyl-1,3,4-thiadiazole has been synthesized, which is the first columnar molecules containing 1,3,4-thiadiazole moiety exhibiting a discotic liquid crystal. The molecular structure of compounds was confirmed by FT-IR, ^{1}H-NMR, and mass spectroscopy and elemental analysis. The electron excitation properties of these compounds were investigated by UV-vis absorption spectroscopy. Their liquid crystalline properties were studied by polarizing optical microscopy and differential scanning calorimetry. The formation of a columnar mesophase was found to be dependent on the number of methylene unit in alkoxy side chains.

Keywords Columnar phase; liquid crystals; Schiff base; thiadiazole

Introduction

Discotic liquid crystal molecules have flat or nearly flat cores surrounded by six or eight (sometimes four) long chain substituents. The first discotic liquid crystal were observed in the hexa-alkanoyloxybenzenes and the hexaalkoxy- and alkanoyloxy-triphenylenes and was found in 1977 by the Indian researcher Chandrasekhar [1]. Since then, a large number of discoid mesogenic compounds have been discovered in which triphenylene, porphyrin, phthalocyanine, coronene, and other aromatic molecules [2,3] are involved.

Many a new discotic liquid crystals have been developed to induce functions such as improved charge transportation [4,5], anisotropic ion transportation [6–8], gas and molecular separation [9], photonics [10], and biological [11] functions.

During the last decades many series of liquid crystalline compounds containing heterocyclic groups have been synthesized due to their potentially wide range of applications,

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such as in the optical, electrical, and biological medical fields [12–15]. Among these heterocyclic, 1,3,4-thiadiazole derivatives have been studied as excellent candidates for material applications due to their excellent thermal and chemical stabilities, their electron-deficient nature and good electron accepting ability [16,17]. Numerous examples of these based compounds have been applied as electron-transporting materials [18]. On the other hand, most examples of mesomorphic 1,3,4-oxadiazole derivatives were generally calamitic liquid crystals exhibiting nematic/smectic phases [19–31] and electron-transporting capability [32–35].

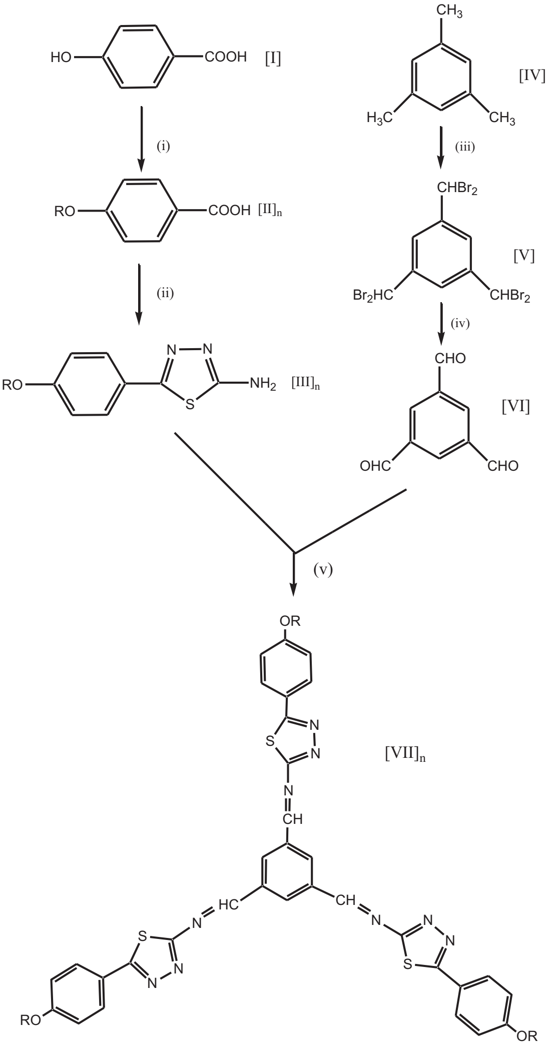
Limited number of discotic 1,3,4-oxadiazole derivatives have been reported [36–47]. For example, a novel three-armed discotic liquid crystal based on 1,3,5-triethynylbenzene as a core and 2,5-diphenyloxadiazole as rigid arms has been synthesized, which is the first star-shaped molecule exhibiting a discotic nematic phase [43]. A new class of discotics derived from tris(*N*-salicylideneaniline) with outer 1,3,4-oxadiazole wings have been synthesized and their thermal and photophysical properties are investigated by Yelamaggad et al. [44]. A novel discotic liquid crystalline material composed of 1,3,5-tri-substituted benzene core and 2-(4-hexloxyphenyl)-oxadiazole-5-yl arms was synthesized and characterized by Kim et al. [45]. Lai et al. reported a series of polycatenar 2,5-bis (3,4,5-trialkoxyphenyl)-1,3,4-oxadiazoles [37] and their corresponding palladium(II) complexes (metallomesogens) [38], which showed columnar phases, and some of them were room temperature liquid crystals. Recently, columnar liquid crystalline materials based on 1,3,5-benzenetrisamide derivatives with three pendant 2-phenyl-5-(mono-, di-, and/or tri-*n*-alkoxyphenyl)-1,3,4-oxadiazole arms were reported by Barbera et al. [46]. A series of hexacatenar columnar liquid crystals containing the 1,3,4-oxadiazole group as a rigid core were reported by Li et al. [39,42]. A lollipop-shaped molecule containing 1,3,4-oxadiazole moieties display columnar phase was designed and synthesized by Choi et al. [47].

As mentioned above most of the reported heterocyclic compounds display columnar mesophase containing 1,3,4-oxadiazole. Recently, Parra et al. [48] reported the synthesis of columnar liquid crystals based on amino-1,3,4-thiadiazole derivatives. Thus we designed and synthesized a novel discotic liquid crystalline composed of 1,3,5-triiminebenzene and three identical arms containing 1,3,4-thiadiazole moiety. To the best of our knowledge, there have been no reports on the mesomorphic behavior of discotic columnar liquid crystals containing 1,3,4-thiadiazole derivatives.

Experimental

General

All the chemicals were supplied from Aldrich-Sigma Chemicals Co. and used as received. FTIR spectra were recorded using potassium bromide discs on a 8400s Shimadzu spectrophotometer. The ^{1}H NMR spectra were recorded on Bruker AMX-300 spectrometer at 300 MHz, using deutrated chloroform or DMSO as solvent with TMS as an internal standard. Elemental analysis (C,H,N) were carried out using a Perkin-Elmer model 2400 instrument. Mass spectra were recorded on IEOL JMS-7 high resolution instrument. UV-vis absorption spectra were reported on a Cintra 5 UV-Visible spectrometer. Transition temperatures and enthalpies were scanned in TA instruments Q1000 DSC, differential scanning calorimeter with a heating rate of 10.0°C/min in air and it was calibrated with indium (156.6°C, 28.45 J/g). The temperatures were read as the maximum of the endothermic peaks. The textures of the mesophases were studied with Olympus BX40 microscope equipped with a Leitz Laborlux 12 Pols hot stage and PR600 controller.



Scheme 1. Reagents and conditions; (i) MeOH, KOH, appropriate RX, reflux, overnight; (ii) $POCl_3$, thiosemicarbazide, reflux, 7 hr; (iii) Br_2, UV light, 170°C; (iv) anhydrous morpholine, reflux, 4 hr; and (v) absolute EtOH, reflux, overnight.

Synthesis and Characterization

The steps of the synthesis of homologous series 1,3,5-tri-[5–4′-nalkoxyphenyl-1,3,4-thiadiazole-2-yl-]-trimesilydine $[VII]_n$ are shown in the sequence of reactions depicted in Scheme 1. Where *n* designate the number of carbon atom in terminal alkoxy group substituent.

Synthesis of 4-n-alkoxybenzoic acid. 4-*n*-alkoxybenzoic acid $[II]_n$ was obtained following the procedure described in [49].

Synthesis of 5-(4-n-alkoxyphenyl)-2-amino-1,3,4-thiadiazole $[III]_n$. A mixture of appropriate 4-*n*-alkoxybenzoic acid (10 mmol) and (0.91 g, 10 mmol) of thiosemicarbazide with 5 mL of phosphorus oxychloride was refluxed gently for 5 hr. After cooling

50 mL of water was added, the mixture was then refluxed for 7 hr and filtered, neutralized with potassium hydroxide. The precipitate was washed with water and recrystallized from ethanol-water to give titled compound $[\text{III}]_n$.

Compound	$[\text{III}]_1$	$[\text{III}]_2$	$[\text{III}]_3$	$[\text{III}]_4$	$[\text{III}]_5$	$[\text{III}]_6$	$[\text{III}]_7$	$[\text{III}]_8$
Yield%	65	65	60	65	60	70	50	45
m.p. °C	195	200	188	190	187	180	170	165

The spectroscopic and elemental characterization of $[\text{III}]_5$ example is given: FT-IR (KBr, cm^{-1}): 3241 ($\nu_{\text{asy, N–H}}$, NH_2), 3101 ($\nu_{\text{sy, N–H}}$, NH_2), 2936–2864 ($\nu_{\text{C–H}}$, aliphatic), 1609 ($\nu_{\text{C=N}}$, thiadiazole ring), 1018 ($\nu_{\text{C–O–C}}$, OCH_2). ^{1}HNMR (DMSO, δ in ppm): 7.10–7.70 (d-d, 4H, arom. H), 7.35 (s, 2H, NH_2), 4.02 (t, 2H, OCH_2), 1.80 (m, 2H, $OCH_2\,\underline{CH_2}$), 1.40 (m, 4H, CH_2CH_2) 0.95 (t, 3H, CH_3). Anal, Calcd. for $C_{13}H_{17}ON_3S$; C: 59.32%, H: 6.46%, N: 15.97%, and S: 12.17%, and found C: 59.24%, H: 6.54%, N: 16.02%, and S: 12.32%. MS: m/z+1 = 264.

Synthesis of $\boldsymbol{\alpha,\alpha,\alpha',\alpha',\alpha'',\alpha''}$***-hexabromomesitylene [V].*** To a 100 mL round bottomed flask containing 23 mL of mesitylene, 15 mL of bromine was added within 2 hr at 155°C, another 15 mL of bromine was added also within 6 hr at 170–180°C. The addition of bromine was administrated under UV light. The mixture was then refluxed for another 1 hr at 170°C. After cooling to room temperature the mixture was filtered to give 80% yield of the titled compound [V], m.p. = 183 °C. Anal, Calcd. for $C_9H_6Br_6$; C: 18.18% and H: 1.01% and found C: 17.71% and H: 1.54%. MS: m/z+1 = 595.

Synthesis of benzene-1,3,5-tricarboxyaldehyde [VI]. A (2.97 g, 5 mmol) of [V] was dissolved in 22 mL of anhydrous morpholine. The solution was then refluxed for 4 hr by using water bath, after that the solution was poured into a 25 mL of concentrated hydrochloric acid with ice, the solution was then filtered, and the precipitate was then dissolved again in 200 mL boiled water. The solution was neutralized by potassium hydroxide (60% yield from the titled compound [VI] was obtained by cooling to room temperature), m.p. 154 °C. Anal, Calcd. for $C_9H_6O_3$; C: 66.67% and H: 3.70% and found C: 67.08% and H: 4.11. MS: m/z+1 = 163.

Synthesis of 1,3,5-tri-[5(4′-n-alkoxyphenyl)1,3,4-thiadiazole-2-yl]-trimesilydene $\boldsymbol{[\text{VII}]_n}$***.*** A mixture of appropriate 2-amino-5-(4-*n*-alkoxyphenyl)-1,3,4-thiadiazole [III]n (30 mmol) and 1,3,5-triformylbenzene [VI] (1.62 g, 10 mmol) dissolved in 35 mL of absolute ethanol. The mixture was then refluxed for 24 hr with stirring. After cooling to room temperature the solvent was evaporated, and the precipitate was recrystallized from ethanol.

Compound	$[\text{VII}]_1$	$[\text{VII}]_2$	$[\text{VII}]_3$	$[\text{VII}]_4$	$[\text{VII}]_5$	$[\text{VII}]_6$	$[\text{VII}]_7$	$[\text{VII}]_8$
Yield%	60	63	50	55	65	60	45	45
m.p. °C	195	200	188	190	187	180	170	165

The spectroscopic characterization of $[\text{VII}]_5$ example is given: FT-IR (KBr, cm^{-1}): 3100 ($\nu_{\text{C–H}}$ aromatic), 2930–2961 ($\nu_{\text{C–H}}$ aliphatic), 1665 ($\nu_{\text{C=N}}$, imine), 1609 ($\nu_{\text{C=N}}$, thiadiazol). ^{1}HNMR (DMSO, δ in ppm) 8.97 (s, 3H, N = CH), 8.80 (s, 3H, arom. H of central benzene ring), 7.15–8.15 (d-d, 12H, arom. H), 4.15 (t, 6H, 3 × OCH_2), 1.78 (m, 6H, 3 × $OCH_2\,\underline{CH_2}$), 1.45 (m, 12H, 6 × CH_2), 0.91 (t, 9H, 3 × CH_3). MS: m/z+1 = 786.

Table 1. Elemental analysis data for the series $[VII]_n$

Compound	Molecular formula	Molar mass g/mol	Analysis % found (calculated)			
			C	H	N	S
$[VII]_1$	$C_{36}H_{27}O_3N_9S_3$	729	59.04 (59.26)	3,72 (3.70)	17.58 (17.28)	13.19 (13.17)
$[VII]_2$	$C_{37}H_{29}O_3N_9S_3$	743	59.68 (59.76)	3.10 (3.90)	16.78 (16.96)	12.97 (12.92)
$[VII]_3$	$C_{38}H_{31}O_3N_9S_3$	757	60.32 (60.24)	4.21 (4.10)	16.53 (16.64)	12.81 (12.68)
$[VII]_4$	$C_{39}H_{33}O_3N_9S_3$	771	59.90 (60.70)	4.36 (4.28)	16.35 (16.34)	12.60 (12.45)
$[VII]_5$	$C_{40}H_{35}O_3N_9S_3$	785	61.10 (61.15)	4.50 (4.46)	16.10 (16.05)	12.28 (12.23)
$[VII]_6$	$C_{41}H_{37}O_3N_9S_3$	799	61.69 (61.58)	4.68 (4.63)	15.82 (15.77)	12.04 (12.02)
$[VII]_7$	$C_{42}H_{39}O_3N_9S_3$	813	61.97 (61.99)	4.68 (4.80)	15.58 (15.50)	11.93 (11.81)
$[VII]_8$	$C_{43}H_{41}O_3N_9S_3$	827	62.36 (62.39)	5.04 (4.96)	15.39 (15.24)	11.76 (11.61)

Results and Discussion

Synthesis

The 5-(4-*n*-alkoxyphenyl)-2-amino-1,3,4-thiadiazole $[III]_n$, which is precursors of this series, were synthesized starting with the reaction of thiosemicarbazide with 4-n-alkoxybenzoic acid $[II]_n$ ($n = 1$–8) followed by dehydration in $POCl_3$ to form thiadiazole ring. The synthesis of benzene-1,3,5-tricarboxyaldehyde [VI] was achieved by the reaction of $\alpha,\alpha,\alpha',\alpha',\alpha'',\alpha''$-hexabromomesitylene [V] with anhydrous morpholine. The1,3,5-tri-[5(4′-*n*-alkoxyphenyl)1,3,4-thiadiazole-2-yl]-trimesilydene $[VII]_n$ were obtained by condensation of compounds $[III]_n$ with the benzene-1,3,5-tricarboxyaldehyde [VI] in absolute ethanol according to the well-known procedure for the synthesis of Schiff bases.

The structures of all products were identified by using FTIR and ^{1}NMR, and mass spectrometry as already described in the experimental section. The all resultant data of the spectra were in accordance with expected values. The purities of compounds were confirmed by using an elemental analysis. The elemental analysis of series $[VII]_n$ compounds synthesized above is listed in Table 1. The observed values are in well agreement with theoretical values indicating structure of respective compounds.

Mesomorphic Properties

The phase transition of compounds of the $[VII]_n$ series were studied using polarizing optical microscopy (POM) and differential scanning calorimetry (DSC). The phase transition temperatures observed by POM agree well with the corresponding DSC thermograms.

Table 2. Phase transition temperatures (°C) and enthalpies (kJ/mol, in parentheses) as determined by DSC, for compounds of series [VII]$_n$

Comp.	Phase behavior
[VII]$_1$	Cr $\xrightleftharpoons{220\ (61.21)}$ I
[VII]$_2$	Cr $\xrightleftharpoons{237\ (67.85)}$ I
[VII]$_3$	Cr $\xrightleftharpoons{215\ (63.00)}$ I
[VII]$_4$	Cr $\xrightleftharpoons{200\ (16.20)}$ Col$_x$ $\xrightleftharpoons{219\ (3.75)}$ I
[VII]$_5$	Cr $\xrightleftharpoons{205\ (11.60)}$ Col$_x$ $\xrightleftharpoons{210\ (2.72)}$ I
[VII]$_6$	Cr $\xrightleftharpoons{195\ (20.35)}$ Col$_x$ $\xrightleftharpoons{212\ (4.68)}$ I
[VII]$_7$	Cr $\xrightleftharpoons{198\ (13.71)}$ Col$_x$ $\xrightleftharpoons{200\ (4.01)}$ I
[VII]$_8$	Cr $\xrightleftharpoons{190\ (26.88)}$ Col$_x$ $\xrightleftharpoons{205\ (5.71)}$ I

Note: Cr = crystal phase; Col$_x$ = undefined columnar phase; I = isotropic phase; all data are obtained from the second heating cycle.

The phase transitions and thermodynamic data for compounds of the series [VII]$_n$ are summarized in Table 2.

The first three homologous [VII]$_1$–[VII]$_3$ do not reveal any liquid crystalline behavior, but simply changes from the solid crystalline state to the isotropic liquid at 220°C, 237°C, and 215°C for compounds [VII]$_1$, [VII]$_2$, and [VII]$_3$, respectively. Compounds [VII]$_4$–[VII]$_8$ display enantiotropic mesomorphism. The texture observed by POM on cooling from the isotropic liquid are consistent with the presence of columnar mesomorphism, with fan-shaped typical to columnar phases for compounds [VII]$_4$, [VII]$_6$, and [VII]$_8$ and fan shaped focal conic typical to a columnar hexagonal for compound [VII]$_5$ as shown in Fig. 1.

The formation of a columnar mesophase was found to be dependent on the number of methylene units in alkoxy terminal chains attached to the rigid core. The mesophase creation probably results from two competitive tendencies: stiffening of the molecular core by dipole-dipole molecular interaction of highly polar methoxy, ethoxy, and propoxy terminal substituent, which depresses the mesomorphic phases, and elongation of that substituent promoting these mesophases. The long alkoxy chains probably biased the rotation of both molecular parts. However, this molecular core is less rigid than that in compounds [VII]$_1$, [VII]$_2$, and [VII]$_3$, resulting in the formation of columnar phase, which is stable in narrow temperature range. The mesophase's stability was found to be poor for the last five compounds. This suggests that the two strong stiffening of the molecular core depresses the columnar phase formation. It is worth wide to mention that an increase in the molecular length with little or no change in the width allow an increase in the anisotropy of the polarizability for molecules with three thiadiazole rings, which favor molecular interaction and liquid crystalline properties for these compounds, [VII]$_4$–[VII]$_8$.These results illustrate the importance of the influence of the number of the methylene unit in terminal alkoxy chain in the occurrence of columnar order.

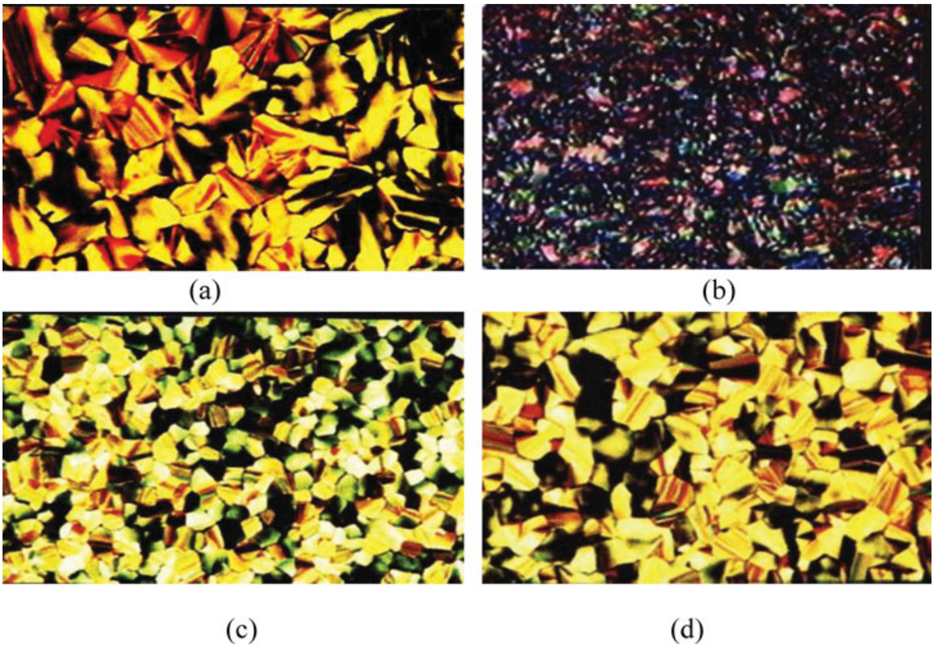


Figure 1. Cross-polarizing Optical textures of the columnar mesophase obtained on cooling (Magnification 200×) for compounds of series $[VII]_n$, (a) $[VII]_4$ at 211°C, (b) $[VII]_5$ at 206°C, (c) $[VII]_6$ at 198°C, and (a) $[VII]_8$ at 200°C.

The effects of the terminal chain length on the transition temperatures and phases behavior observed in this series are in accordance with those observed for columnar discotic mesogenes [49–52].

The UV absorption properties of these liquid crystalline molecules have been studied. The absorption spectra in DMSO solution for these compounds exhibited a distinctive absorption peak at 310–312 nm which attributed to the π-π^* transition. These results showed that these liquid crystalline compounds could emit a blue light in DMSO solution on photoexcitation.

Conclusions

A new series of discotic liquid crystalline based on 1,3,5-benzenetrisazomethine derivatives with three pendant 2-amino-5-(4′-*n*-alkoxy)phenyl-1,3,4-thiadiazole were designed and synthesized by varying alkoxy terminal chain length ($n = 1$–8). The formation of a columnar mesophase was found to be dependent on the number of methylene unit in alkoxy terminal chains. The compounds with $n \geq 4$, exhibited an anantiotropic columnar phase; however, compounds with $n = 1$, 2, and 3 formed a crystalline phase.

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