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"Synthesis, design and characterization of supramolecular self-assembly of calix[4]resorcinare substituted LCs"

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ABSTRACT

A new class of supramolecular calix[4]resorcinarene substituted alkoxy side chain (-OC₅H₁₁, -OC₈H₁₇, -OC₁₀H₂₁) has been synthesized and well characterized. These supramolecular compounds were investigated by polarizing optical microscope (POM), differential scanning calorimetry (DSC), thermogravimmetric analysis (TGA) and high-temperature X-ray diffraction studies (XRD). The present synthesized supramolecular derivatives are promising to stabilize the hexagonal columnar phase over a broad thermal range. All the synthesized derivatives showed hexagonal columnar phase at lower temperature and showed enantiotropical nature. Compound 2a with small alkyl spacer on eight side showed higher thermal stability as compare to higher alkyl spacer substituted compounds 2b and compound 2c. These research results suggest that calix[4]resorcinarene was a good platform to construct bowlshaped derivatives to exhibits the columnar liquid crystal phase and the observed liquid crystalline properties were greatly effected by the substituted alkoxy side chain on eight side of calix[4]resorcinarene skeletone. All the synthesized compounds were characterized by FT-IR, ¹H NMR and ¹³C NMR spectroscopy.

GRAPHICAL ABSTRACT



KEY WORDS

Calix[4]resorcinarene; enantiotropic; columnar phase; supramolecular LCs

1. Introduction

Study of the liquid crystalline state is a multidisciplinary subject and useful to mankind and various industrial applications through scientist, technologists [1-4] due to its

unique property to flow as liquid and optical properties as crystals. Study on LC state is planned with a view to understand and establish the effect of molecular structure on LC properties [5–6] as a consequence of molecular rigidity and flexibility [7–8]. Liquid crystals can be categorized into thermotropic, lyotropic and metallotropic phases. Thermotropic and lyotropic liquid crystals consist mostly of organic molecules although few minerals are also known [9]. Liquid crystals in the nematic group are most widely used in the production of liquid crystal displays (LCD) mainly due to their unique physical properties as well as wide range of temperature. In addition, the nematic phase in which liquid crystal molecules are oriented on average along to a particular direction. By applying an electric or magnetic field, the orientation of the molecules can be derived in a probable approach [10]. Literature survey revealed that the schiff base has been widely employed as a linking group for the synthesis of different forms of liquid crystals [11].

Calixarenes belong to the third generation of supramolecular platforms after crown ether and cyclodextrin [12–13][.] Calixarenes, possessing tunable 3D-shaped hydrophobic cavities belong to the important building blocks in supramolecular chemistry [14]. The calixarenes broaden functionalize at the upper and lower rim affords them with suitable binding sites for guest encapsulation and molecular assembly[15]. The four hydroxyl groups present in calix[4]arene interact by hydrogen bonding and also stabilize the cone conformation⁹. In recent years, the calixarene core was also successfully used as a rigid core to prepare various kinds of liquid crystalline compounds [16–18].

Liquid crystalline materials based on calix[4]arene rigid core was first reported in 1990 [19]. Calixarene based liquid crystalline compounds which are the most significant part of macrocycles based liquid crystals were investigated in literature [19]. Xu and Swager investigated the host-guest mesomorphism of calixarene compounds. They demonstrated how host-guest principles could be used to stabilize a columnar bowlic phase of a conformationally flexible calix[4]arene was found to display transient columnar bowlic mesophase [20]. For instance, the calizarene liquid crystals with substitution of long aliphatic side chains were prepared and displayed interesting liquid crystalline behaviour [21-22]. Yonetake et al. successfully synthesized two liquid crystal based on tert-butyl calix[8] arene to displayed smectic as well as nematic mesophase [23]. Yang et al. reported mesomorphic property in calix[4]arene cholesterol derivatives. The following group studied the mesophase appeared with the presence of columnar type molecular arrangement of calixarene with cholesterol derivatives [24]. In another investigation, the similar group has synthesised and studied the liquid crystalline behavior of calixarene-linked triphenylene derivatives [25]. Later, his group reported the mesomorphic properties of gallic-calizarene derivatives with stable cone conformation [26]. Recently, Yang et al. reported calix[4]resorcinarene cholesterol based columnar liquid crystals having schiff-base linking group [27]. Marcos and his coworkers synthesized and studied the mesomorphic property of calixarene schiff base derivatives, which have binding recognition and selectivity towards Zn⁺² ion [28]. Yang et al. reported bowlshaped liquid crystal based on cyclotriveratrylene derivatives with multiple triphenylene units. They demonstrated more triphenylene units linked with CTV derivatives to exhibit higher ordered hexagonal columnar phase [29]. Sharma et al. reported the two liquid crystalline series of calix[4]arene based on schiff-base and ester linking unit to

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show smectic and nematic phase and also studied their biological behaviour [30]. Recently, Sharma et al. reported calix[4]arene core based LCs which showed stable mesomorphism over long temperature range and good photophysical behaviour in solution as well as thin film [31–32].

The comprehensive literature studied reveals that the supramolecular columnar liquid crystalline compounds are very rarely reported. In this present study, we wish to report first newly functionalized bowl-shaped supramolecular resorcinarene derivatives based on resorcinarene as a central core substituted by the variable peripheral alkoxy side chain on eight side and studied their liquid crystalline properties. In present study, we have introduced total three novel compounds having pentyloxy, octyloxy and decyloxy side group.

2. Experimental and Instrumentation

2.1. Materials

Resorcinol, cinnamaldehyde, various alkyl bromides (R-Br) were purchased from Sigma Aldrich. All the other chemical reagents were purchase from Sigma Aldrich. Solvents were dried and purified in the usual manner. Column chromatography was performed using silica gel (60-120 mesh).

2.2. Measurements

The FT-IR spectra were recorded as KBr pellet on Shimadzu in the range of 3800-600 cm⁻¹. The ¹H and ¹³C NMR spectra were obtained on a Bruker Advance 400 MHz spectrometer using tetramethylsilane as the internal standard. The mesophase type was identified by visual comparison with known phase standards using a Nikon Eclipse LV-100 POL polarizing optical microscope (POM) fitted with a hot stage temperature-controlled LTSE 420 heating stage (Linkam Scientific Instruments, Tadworth, Surrey, UK) with temperature controlling accuracy of 0.1K. The transition temperatures and associated enthalpy changes were determined by differential scanning calorimeter under nitrogen atmosphere. Thermo gravimetric analysis (TGA) was performed using a Perkin Elmer-STA 6000 apparatus under high purity nitrogen. The X-ray diffraction (XRD) measurements were performed on a Rigaku-Ultima IV powder diffractometer equipped with a Cu k α source ($\lambda = 1.5418$ A° and 1.6 kW, X-ray tube with applied voltage and current values as 40 kV and 30 mA power) and also Philips X'PERT MPD.

3. Result and discussion

3.1. Synthesis and characterization

The synthetic approach for the preparation of the derivatives of oxadiazole is presented in below Scheme 1. Calix[4]resorcinarene (1a) was prepared by the reaction of resorcinol with cinnamaldehyde in presence of sodium hydroxide [33]. The final target compounds (2a-2c) were prepared by the reaction of compound (1a) with various alkyl bromides in the presence of anhydrous K_2CO_3 and dry acetone [34]. The resultant



Scheme 1. Reaction and condition: (i) Cinnamaldehyde, NaOH, Reflux; (ii) R-Br, Anhydrous.

crude residue was purified by using column chromatography on silica gel eluting with methanol: chloroform as eluent (1:4). The ¹H NMR, ¹³C NMR and IR results also well supported to the corresponding structures of derivatives and final target compounds.

Comp.2a: Yield 74%, FT-IR (KBr) in cm⁻¹: 2910 (-C-H- Str in aromatic), 1260 (-C-O str), 891 polymethylene (-CH₂-)n of $-OC_5H_{11}$, 930 (alkene group). ¹H NMR: 0.88-0.90 (t, 16H, $-OC_5H_{11}$), 1.31 (m, 32H, $-OC_5H_{11}$), 1.77 (p, 16H, $-OC_5H_{11}$), 2.63 (16H, $-ArCH_2Ar$ -), 4.08 (16H, $-OC_5H_{11}$), 4.06 (t, 16H, $-OC_5H_{11}$), 3.63 (s, 4H, -CH-), 6.55 (s, 4H, Ar-H), 7.10 (s, 4H, Ar-H), 7.19 (d, 8H, Ar-H), 7.24 (d, 8H, Ar-H), 6.87 (s, 1H, Ar-H); ¹³C NMR: 160.4, 152.7, 147.3, 146.3, 128.8, 128.1, 96.7, 69.8, 69.0, 37.2, 34.7, 29.3, 28.1, 22.7, 14.1.

Comp.2b: Yield 71%, FT-IR (KBr) in cm⁻¹: 2930 (-C-H- Str in aromatic), 1250 (-C-O str), 748 polymethylene (-CH₂-)n of $-OC_8H_{17}$, 930 (alkene group). ¹H NMR: ¹H NMR: 0.88-0.90 (t, 16H, $-OC_8H_{17}$), 1.28-1.31 (m, 64H, $-OC_8H_{17}$), 1.47 (sext, 16H, $-OC_8H_{17}$), 1.77 (p, 16H, $-OC_8H_{17}$), 2.63 (16H, $-ArCH_2Ar$ -), 4.08 (16H, $-OC_8H_{17}$), 4.06 (t, 16H, $-OC_8H_{17}$), 3.63 (s, 4H, -CH-), 6.55 (s, 4H, Ar-H), 7.10 (s, 4H, Ar-H), 7.19 (d, 8H, Ar-H), 7.24 (d, 8H, Ar-H), 6.87 (s, 1H, Ar-H); ¹³C NMR: 160.4, 152.7, 147.3, 146.3, 128.8, 128.1, 96.7, 69.8, 69.0, 37.2, 34.7, 29.3, 28.1, 22.7, 22.1, 21.6, 14.1.

Comp.2c: Yield 76%, FT-IR (KBr) in cm⁻¹: 2910 (-C-H- Str in aromatic), 1260 (-C-O str), 624 polymethylene (-CH₂-)n of $-OC_{10}H_{21}$, 930 (alkene group). ¹H NMR: 0.88-0.90 (t, 16H, $-OC_{10}H_{21}$), 1.28-1.31 (m, 84H, $-OC_{10}H_{21}$), 1.47 (sext, 16H, $-OC_{10}H_{21}$), 1.77 (p, 16H, $-OC_{10}H_{21}$), 2.61 (16H, $-ArCH_2Ar$ -), 4.06 (16H, $-OC_{10}H_{21}$), 4.06 (t, 16H, $-OC_{10}H_{21}$), 3.63 (s, 4H, -CH-), 6.55 (s, 4H, Ar-H), 7.10 (s, 4H, Ar-H), 7.19 (d, 8H, Ar-H), 7.24 (d, 8H, Ar-H), 6.87 (s, 1H, Ar-H); ¹³C NMR: 160.4, 152.7, 147.3, 146.3, 128.8, 128.1, 96.7, 69.8, 69.0, 37.2, 34.7, 29.3, 28.1, 22.7, 14.1.

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	Phase sequence	
Compound	Heating	Cooling
2a	Cr 183.60 (1.7) Col _h 218.92 (7.4) I	l 216.72 (4.2) Col _h 181.32 (9.3)
2b	Cr 179.58 (1.2) Col _h 210.98 (9.5) I	l 208.67 (3.1) Col _h 178.41 (7.6)
2c	Cr 164.41 (1.1) Col _h 191.01 (6.6) l	l 190.82 (2.6) Col _h 163.37 (9.4)

Table 1. Phase transition temperatures (°C) and corresponding enthalpies (kJ/mol) of synthesized compounds.

^aPeak temperatures in the DSC thermograms obtained during the first heating and first cooling cycles at 10° C/min; Col_h = columnar hexagonal phase.





3.2. Reaction and Scheme

 K_2CO_3 , dry Acetone, Reflux, (R = C_nH_{2n+1} , n = 5,8,10).

3.3. DSC investigation

The mesomorphic behavior of present synthesized resorcinarene core based derivatives **2a**, **2b** and **2c** were preliminarily studied by using differential scanning calorimetry (DSC) represented in Figure 3. The transition temperatures and the associated enthalpy changes obtained from the DSC scans of first heating and cooling cycles are mentions in Table 1 respectively. Figure 1 and Figure 2 indicates the thermal behavior of these synthesized compounds in first heating and first cooling cycle. In DSC scans, compound 2a-2c exhibited two endothermic peaks corresponding to crystal to mesophase (Cr-Colh) and mesophase to isotropic (Colh-I) on heating and cooling condition. From these results, it can be noted that all the synthesized derivatives showed enantiotropical liquid crystalline properties. Compound **2a** with a pentyloxy tail group exhibited two endothermic peaks at 183.60 °C and 218.92 °C on heating cycle while on cooling cycle, two endothermic peaks traced at 181.32 °C and 216.72 °C. Similarly, Compound **2b** with octyloxy tail group exhibited two endothermic peaks at 179.58 °C and 210.98 °C on heating cycle while on cooling condition it appeared at 178.41 °C and 208.67 °C. In



Figure 2. Bargraph showing the thermal behaviour of compounds 2a-2c (cooling condition).



Figure 3. The DSC traces of compounds 2a (a), 2b (b) and 2c (c) on first heating and cooling (scan rate 10°C/min).

addition, compound **2c** showed phase transition at 164.41 °C and 191.01 °C on heating condition and on cooling it exhibited at 163.37 °C and 190.82 °C with a lower enthalpy change value to form Col_{h} -I phase transition peak noticed in DSC scans on heating and cooling condition. It can be noted that the compounds **(2b-2c)** with higher alkyl chain



Figure 4. Phase diagram of comp.2a-2c (heating and cooling condition) by POM.

showed lower transition temperature as compare to compound (2a). The result obtained from DSC study is further confirmed by using POM and XRD analysis.

3.4. POM investigation

The mesophase phase transition temperatures of present resorcinarene based derivatives (2a-2c) were further investigated by polarizing optical microscope (POM) study. It can be observed that the texture pattern of compounds (2a-2c) showed same textural pattern of hexagonal columnar phase. The mesomorphic temperature ranges for the synthesized compounds (2a-2c) are 41.0 °C, 39.0 °C, 32.0 °C on heating cycles while it exhibited at 36.0 °C, 30.0 °C and 26.0 °C on cooling cycles respectively. One can remark the mesophase temperature range of compound 2a is higher as compare to compound 2b and 2c due to the presence of short alkyl chain spacers on eight side substitutions on resorcinarene core. The phase transition temperatures for all compounds were noticed on heating condition at 181.0 °C, 177.0 °C and 166.0 °C which is nearer to the phase transition temperature obtained from the DSC analysis. On cooling condition, same textural pattern of Colh mesophase was observed at 178.0 °C, 180.0 °C and 162.0 °C before the formation of solid crystal at lower temperature. It can be noted that, the phase transition temperature is decreased with increasing alkoxy side chain on terminal position which enhance the flexibility of molecule and weaken the core-core interaction inbuilt with resorcinarene. Thus, an enantiotropic mesomorphic behavior over a good thermal range of mesophase was seen for the present synthesized supramolecular calix[4]resorcinarene based LCs. The texture pattern of hexagonal columnar phase is additionally confirmed by XRD analysis.

3.5. XRD investigation

To confirm the mesophase observed through a polarizing optical microscope (POM) and differential scanning calorimetry (DSC), XRD studies have been performed to confirm the observed liquid crystalline phase in synthesized calix[4]resorcinarene based supramolecular



Figure 5. XRD profiles depicting the intensity against the 2Θ obtained for the Colh phase of compound **2a** at 182.0 °C (a); Colh phase of compound **2b** at 177.0 °C (b) on cooling from isotropic temperature.

derivatives. All the samples have been studied for their LC phase in their transition temperature by using XRD study. The sample was filled by melting as isotropic state and cooled down to their LC state and scanned for X-ray studies. The X-ray patterns of LC phases of all the supramolecular calix[4] resorcinarene derivatives exhibited three reflections in small angle region and two other diffuse scattering peak in the wide angle region as shown in Figure 5 (a-b). Compound 2a showed three reflections at 1.89°, 2.78° and 4.13° in the small angle region and 19.01° and 20.08° in the wide angle region. Similarly, Compound 2b at 1.76°, 2.81° and 5.23° in small angle region while 19.61° and 20.12° in wide angle region. The d-spacing value of compound 2a is 38.37Å, 28.51Å and 18.42Å in the small angle region. Similarly, 42.77Å, 25.24Å and 17.51Å for compound 2b, The ratio of these present reflections agreed with $1:1/\sqrt{3}$: $1/\sqrt{7}$, signifying the [100], [110] and [200] plane of hexagonal lattice [35]. The presence of small diffuse peak at wide angle region at 19.01°, 20.08° in compound 2a and 19.61° and 20.12° in compound 2b, this is due to the presence of core-core interactions of ordered hexagonal phase in addition of formation of self-assembly of bowlic compounds. The literature survey indicated that the similar XRD traces were reported for supramolecular calixarene based liquid crystalline compounds with different substituted derivatives on lower and upper rim [35]. The estimated molecular length of present synthesized compounds is nearer to the observed molecular length. One can see that the presence of broad halos at $2\Theta = 19 \sim 20^{\circ}$ indicating the ordering alkoxy side chain group on the terminal side of octa-substituted side alkoxy chain on calix[4]resorcinarene derivatives. The possible schematic diagram of self-assembly of bowl-shaped LCs derivatives arranged into the form hexagonal columnar arrangement in compounds 2a-2c are mention in Figure 6. Based on all the data from DSC, POM, and XRD, it is confirmed that all the synthesized resorcinarene derivatives exhibited stable hexagonal columnar liquid crystalline mesophase with good thermal stability.

3.6. Thermogravimetric analysis

Thermal stability of compounds 2a, 2b, and 2c were studied by thermogravimetric analysis (TGA) under nitrogen atmosphere shown in Figure 7. All the synthesized



Figure 6. Proposed mechanism of hexagonal columnar arrangement in comp.2a-2c



Figure 7. TGA curves of the compounds 2a-2c carried out at a rate of 10 °C/min.

calix[4]resorcinarene based supramolecular derivatives were stable at least up to $\approx 250^{\circ}$ C and complete degradation occurs at around 400°C which showed the good stability of this synthesized compounds respectively. As there is no loss seen up to temperature at around 150°C that means no water or other any solvent entrapped in mesophase formation. All the synthesized compounds show good thermal stability as also confirmed by POM temperature range as well. From the POM study, compound 2a-2c showed mesophase temperature range 41 degree, 39 degree and 32 degree which clearly indicates that the increasing side alkyl chain inbuilt with alkoxy group on the core of resorcinarene as a result the mesophase temperature range is decreased respectively.

4. Conclusion

Supramolecular n-alkoxy calix[4]resorcinarene derivatives are a promising class of bowlic shaped columnar liquid crystals. The self-assembly of synthesized supramolecular

derivatives to form an ordered hexagonal columnar phase. The calix[4]resorcinarene based LCs is prepared by the single step process by the reaction of calix[4]resorcinarene and n-alkyl bromides. The length of the side spacer alkyl chain had no effect on the absorption and emission properties in the solution state. The increase of flexible alkyl chain length on terminal side is effect on the mesophase range. The compounds with lower alkyl chain group shows higher temperature range of mesophase as compare to higher alkyl chain substituted compounds. All the synthesized derivatives showed higher thermal stability and stabilize columnar mesophase.

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