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Playing with Liquid Crystals: (A Short Review)

SANTANU CHAKRAVORTY^{1*} and SOUMEN SAHA²

*1Department of Chemistry, Mathabhanga College, Mathabhanga, West Bengal 73 6146, India.
2Department of Sericulture, Raiganj University, Raiganj, West Bengal 733134, India.
*Correponding author E-mail: santanuorg34@gmail.com, thustu@gmail.com

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ABSTRACT

The liquid crystal structure is a distinct phase of matter seen between crystalline and isotropic regions. There are many types of glossy liquids, depending on the composition. They have a different type of electronic device and display. As a result, development and integration of this type of extraordinary materials is required. Here, we have outlined the preparation route and properties of the different compounds which show this type of properties.

Keywords: Liquid Crystal, Bent Shaped, Rod-Shaped, Disc-Shaped, HRXRD, DSC, POM.

INTRODUCIOTN

Liquid crystals are a different type of molecule that shows features between solids and liquids.1 This fourth mater state was first discovered by the Austrian botanist Reinitzer in 1888 but this became the subject of research after the great work of Prof. William Gray and G. H. Brown.² Subsequently a few scientists have developed a variety of methods for to generate them. These types of compounds are usually categories according to their behaviour and method of generation as thermotropic, lyo-tropic and metallo-tropic categories. Thermo-tropic materials are usually divided into three basic categories according to their shape, i.rod like liquid crystal³, ii. bent core liquid crystal⁴, iii. discotic liquid crystal.⁵ However, there are many other categories such as cholesterol-based liquid crystal, H-shaped,⁶ S-shaped,⁷ star shaped liquid crystal⁸ etc. Over a short period of our research on liquid crystal we have synthesized different type of thermotropic liquid crystals and characterized their unique properties with the help of POM, DSC and powdered XRD.

Rod shaped or linear type of liquid crystals

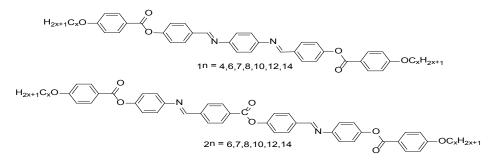
Discovery of room temperature liquid crystal⁹ motivates the scientist community to develop materials by inculpating aliphatic and aromatic moiety to the already existence materials or by introducing various substitution on it. They even develop materials only by modifying the spacer branched. In recent years rod like mesogenic materials are known where substituted or un-substituted aromatic rings having two or more in numbers interconnected via various linkage.¹⁰ In

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2007 we have designed and synthesized rod like materials where centre moiety connected to two symmetrical parts via imine types of linkages.¹¹ We

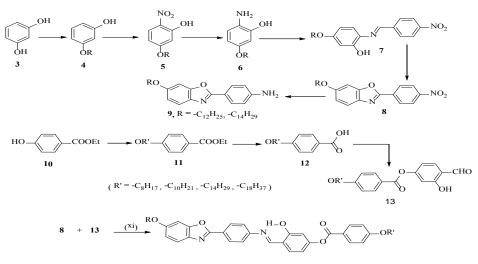
have varied the length of alkyl side chain and the number of aromatic ring of two series to observe any changes in properties (Scheme 1).



Scheme 1. Synthesis of linear dimer

Compound with short terminal alkyl chains of the series (1n = 4 and 2n = 6, 7) exhibited nematic, tilted SmC phase and higher order Smectic phase. On the other hand middle homologues (1n = 6-10 and 2n = 8-10) of the series exhibited same phase sequence as of lower homologues where as compound with large alkyl chain in terminal position displays SmA phase in addition to the all the other phases mentioned for rest of the series. Most interesting feature of this series is that by increasing end alkyl chain length we induced SmA phase in these two homologous series.

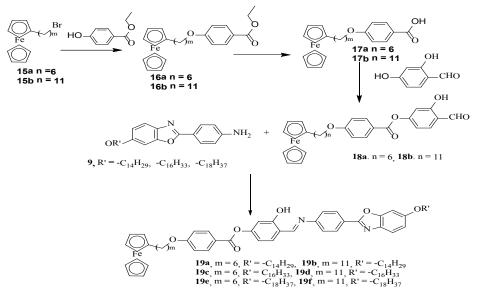
Synthesis of liquid crystalline materials with a hetero atom containing ring at the centre is the topic of interest for last few years.¹² Among the heterocyclic core 2-arylbenzooxazole derivatives found importance for their unusual molecular shape with elongated conjugation.13 Recently we have synthesized calamitic mesogen by incorporating alkyl substituted 2-arylbenzooxazole at the terminal position and investigated their mesogenic properties (Scheme 2).14 Benzoxazole amine derivative 9 was prepared by the condensation and subsequent reduction. Compound 6 in turn was synthesized starting from compound 3 by selective alkylation, nitration and hydrogenation. On the other hand compound 13 was synthesized by esterification reaction between acid derivative 12 and 2,4-dihydoxy benzaldehyde. The rod-shaped mesogens 14a-h was synthesized by the condensation reaction between aldehyde 13 and amine 9 (Scheme 2).



Polarising Optical Microscopic textures of the compounds of this series resembles the broken focal conic and the Schlieren texture of SmC phase. The phase exists over a wide temperature gap. Presence of SmC was further established by powdered XRD experiment. Possibly the presence of H-bonding the molecular structure increased the stability of SmC to the other compound with same heterocyles core.¹⁵

We also replaced the alkyl chain on

the benzene ring of the above series with alkyl ferrocenyl unit with variable chain spacer (Scheme 3) and investigated the change in liquid crystalline properties compared to the above series.¹⁶ Alkylation and hydrolysis of the resulting ester derivatives starting from the compound 15a/15b provide the compounds 17a or 17b. Compounds 17a-b was then converted to 18a,b by esterification reaction. 18a,b on condensation with benzooxazole amines gave the corresponding Schiff's base derivatives 19a-f.

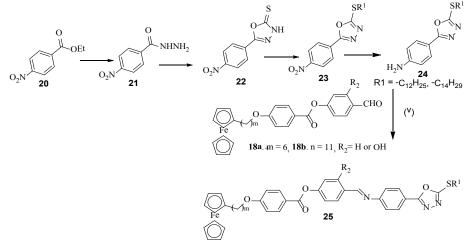




Compounds 19a-f exhibited typical texture of biaxial Smectic C phase (Picture 1). These indicate, changing the alkyl chain with ferrocene containing alkyl does not cause any change in the nature of the phase. Loubser and Imrie¹⁷ reported that in the case of ferrocene containing calametic liquid crystal presence of 3 to 4 aromatic ring is essential for stabilization of liquid crystalline phase which is nematic according to their observation. Seshadri & Haupt¹⁸ reported a ferrocene based liquid crystalline materials which exhibits interesting phase sequence of Chiral Smectic, TGB, SmA, N* phases. But at that time there was no any other reports of rod-like mesogens were found where feroccene and benzooxazole unit are interconnected with an imine type linkage.

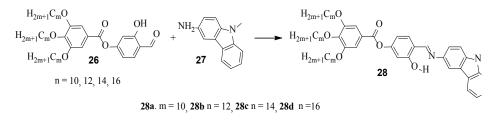
Recently we have performed another modification of the above series by replacing 2-arylbenzooxazole unit with S-alkylated oxadiazole unit.¹⁹ Thio alkylated oxadiazole unit was synthesized starting from ethyl-p-nitrobenzoate²⁰. The intermediate 22 was obtained by amidification followed by cycization in presence of carbondisulphide. S-alkylation followed by subsequent hydrogenation as depicted in Scheme 4 provided the corresponding S-alkylated oxadiazole derivatives 24.

The compounds 25a-g exhibited two mesophases but the compound 25h showed only one mesophase. From the PMO studies of the compounds 25a-h it was found that the lower temperature phase are N phases and the higher temperature one was SmC phase. On the other hand compound 25h only exhibited SmC phase. The key feature of this series is that the introduction of thio-oxadiazole unit instead of 2-arylbenzooxazole unit induces a new mesophase and lowered the melting and clearing temperatures as well as temperature of solidification. Our group also synthesized rod-like mesogen with terminal carbazole unit linked with tri-alkoxy substituted aromatic ring via a aromatic spacer having a Schiff's base linkage.²⁰ The compound 28 was prepared by simple condensation between amine 27 and hydroxyl–aldehyde 26.



 $\begin{array}{l} \textbf{25a}, R_2 = H, n = 6, R' = -C_{12}H_{25}, \textbf{25b}, R_2 = OH, n = 6, R' = -C_{12}H_{25}, \textbf{25c}, R_2 = H, n = 6, R' = -C_{14}H_{29}, \textbf{25d}, R_2 = OH, n = 6, R' = -C_{14}H_{29}, \textbf{25d}, R_2 = OH, n = 11, R' = -C_{12}H_{25}, \textbf{25f}, R_2 = OH, n = 11, R' = -C_{12}H_{25}, \textbf{25g}, R_2 = H, n = 11, R' = -C_{14}H_{29}, \textbf{25h}, R_2 = OH, n =$

Scheme 4





Compound 28a,c,d showed only one mesophase while compound 28b was non mesomorphic in nature. The non-mesomorphic behaviour of the compound 28b remained unexplainable. In POM studies of the compounds 28a-d (28C is non mesomorphic) displayed a strap-like appearance of columnar phase. Variable temperature powder X-ray studies were used to confirm the nature of the columnar mesophase. The powdered XRD pattern of compound 28a indicates that the columnar phase are stacks in rectangular fashion.²¹ An anti parallel association of two molecules of linear mesogens may be the cause of generation of rectangular columnar phase.²²

We also achieved another modification of the above series by replacing carbazole amine with 2-phenyl benzooxazole unit (Scheme 6) and investigated any changes in liquid crystalline property from the previous series.²³ The Schiff's bases analogous 29a-e were synthesized by condensation between aldehyde 26 and amine 9.

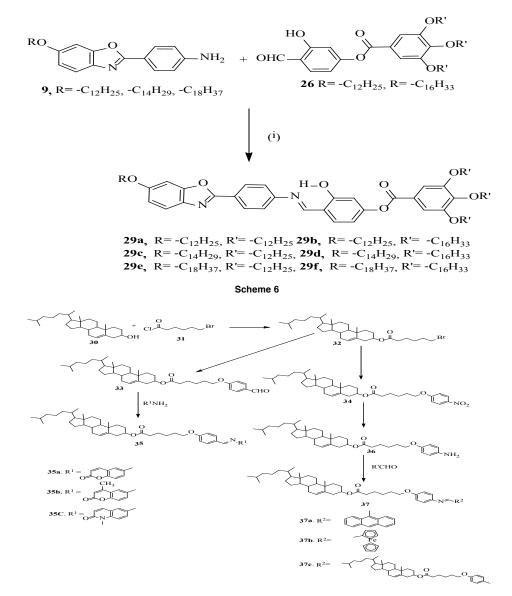
The compounds 29a-e exhibited higher order smectic phase (picture 2). Intramolecular H-bonding increases the stability of the smectic phase compared to the stability of phase reported by the others having the same heterocylic core unit.15, 24 The powder HRXRD of compound 29d indicates that the mesophase is monolayer in nature. Thus change in heterocyclic unit, changed the mesophase behaviour in this series compared to the previous one.

Cholesterol based liquid crystal

Chiral phases can be introduced in liquid crystalline materials either by the introduction of chiral moiety or by doping with a chiral component in a multi-component sysytem.²⁵⁻²⁶ Cholesterol with eight asymmetric centre, inflexible molecular structure, natural affluence attract the scientist to choose it as an alternative to synthesize chiral molecules. Unsymmetrical mesogenic dimers are formed by connecting Chloesterol unit with aromatic, aliphatic or heterocyclic core separated by variable alkyl chain unit. Liquid crystalline dimers are mostly unsymmetrical in nature though in symmetrical dimers are also known.²⁷

Liquid crystalline trimers are formed by interconnecting three segments. The three segments interconnects in such a way that they can formed linear trimer,²⁸ cyclic trimer,²⁹ star-shaped trimer³⁰ and trimer having rod-like and disc-like shape.³¹ Whereas tetramers are molecules in which four unit interconnected with each others. We have designed, synthesized and investigated the properties of several cholesterol based dimer, trimer and tetramer which are depicted in a combined form below.

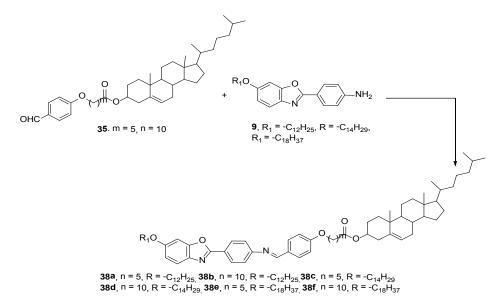
In 2009 we have published our work based on liquid crystalline cholesteryl oligomers with a Schiff base linkage.³² Compound 33 and 34 are obtained by esterification followed by alkylation. Compound 36 was obtained on hydrogenation of compound 34. Compounds 35a–c and 37a–c obtained by condensations reaction either with amines or aldehydes (Scheme 7).



Compound 35a exhibits the appearance of cholesteric phase (picture 3) SmA Phase (picture 4) TGB phase under POM studies. The compound 35c exhibited similar type of textures as of 35a where as 35b only displayed a cholesteric phase. Compound 37a exhibited an unknown mesophase but 37c exhibited an unknown mesophase along with a cholesteric phase. HRXRD experiment and molecular length calculation indicated the presence of partially bilayered SmA phase in 35a and 35c. Tamaoki *et al.*,³³⁻³⁵ reported dimers in which cholesterol unit was interconnected with a azobenzene core via

alkyal spacers, exhibits either intercalated smectic phase or cholesteryl phase. Therefore, variety of phases in compounds 35a and 35c in our study is quite significant in our studies.

Recently a modification by replacing the different heterocyclic unit of the above series with alkyl substituted 2-phenylbenzooxazole unit of different alkyl chain length was reported by our group.³⁶ The Synthesis is described in Scheme 8. The compounds 38a-f was synthesized by condensation reaction between amine 9 and aldehyde 35.

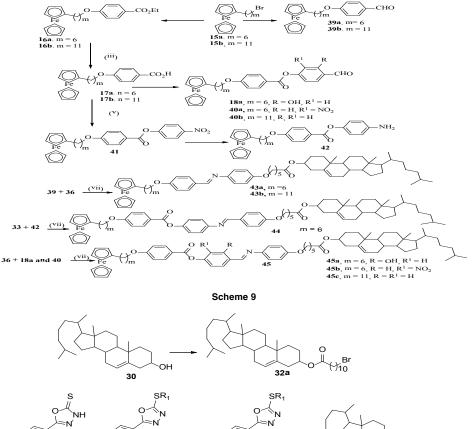


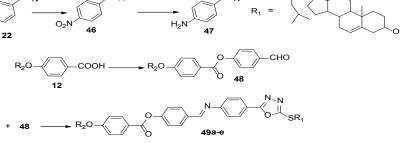
Scheme 8

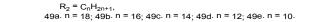
Compounds 38b and 38d showed the sudden appearance of BPI and BPII phases from homeotropic liquid on cooling under POM studies. The phase further transformed into chloesteric and smectic phases (SmA or SmC*) on lowering the temperature before going to solid phase. The other compounds showed typical Cr-SmC*-I transition. From the powdered XRD experiment and molecular length calculation it was confirmed that the SmA phase in this case is monolayer in nature.

We have also reported the synthesis of ferrocene-based metallomesogens with terminal ferrocenyl group (Scheme 9).³⁷ Compound 39a-b obtained from 15a or 15b by substitution of bromide with 4-formyl phenolate. Similar substitution with 4-hydroxy benzoate and hydrolysis of the resulting esters leads to compounds 17a-b. Esterification of 17a-b with either p-nitrophenols or substituted hydroxybenzaldehyde affords 41 and 18a, 40ab. 42 were obtained from 41 on hydrogenolysis. Compounds 43, 44 and 45 were obtained by condensation reaction as depicted in Scheme 9.

43a exhibited only cholesteric phase (N*). Replacing the hexa methylene spacer of 43a with undeca-methylene spacer (43b) induces two new phases TGB and SmA. On introduction another aromatic ring to the structure 43a i.e. compound 44 existences of cholesteric phase increases dramatically. Modification of the structure of 44 was done by introducing lateral hydroxyl (45a) and nitro (45b) groups. Both the compounds exhibits cholesteric phase but the only difference that arise by replacing a hydroxyl group with nitro group is that existence range of liquid crystalline texture and melting temperature of the nitro substituted compound decreases drastically compared to the hrdroxy substituted compound. Compound 45c on the other hand only possesses a stable of N^{*} phase. In general ferrocene based dimer needs three aromatic ring for appearance of liquid crystalline phase38 but we have observed twisted grain boundary phase in ferrocene based compounds with two aromatic rings. Recently we have reported dimers in which cholesterol substituted thiooxadiazole unit connected with alkyl substituted aromatic core via an imine linkage.³⁹ This material was prepared by the condensation reaction of amine 47 with the aldehyde 48 as depicted in Scheme 10.







Scheme 10

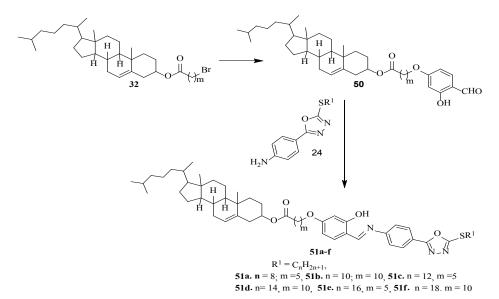
Compound 49a showed transient Blue Phase before transformation it into cholesteric phase N* phase. On further cooling TGBC* phase appeared (picture 6). The other homologues of the series do not exhibit any Blue phases but the rest

O₂N

of the phases are same as of 49a. The presence of TGBC* phase was confirmed by the POM study of the sample in as well as from HRXRD experiment.

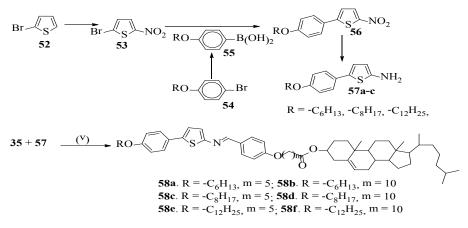
We have reported another series of

compounds with one aromatic ring less than the previous series by interconnecting cholesterol unit with thioalkyl-substituted oxadiazole unit.⁴¹ Synthetic procedure is described in Scheme 11. The compounds 51a-f was prepared by simple condensation between aldehyde 50 and amines 24 (Scheme 11). The compound 51a-f was synthesized by condensation reaction of amine 24 and aldehyde 50. The aldehyde 50 in turn was synthesized by substitution reaction between 2,4-dihydroxy benzaldehyde and bromo derivative 32 (Scheme 11).

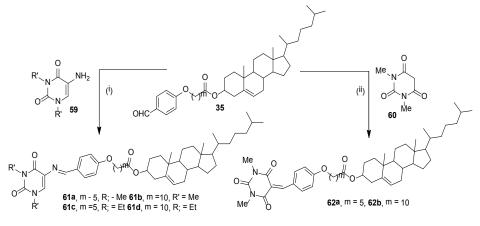


Scheme 11

The compounds 51b, 51d and 51f exhibited only one N* phase. On the other hand 51a exhibited only Cr-N*-I phase sequence, whereas the other two compounds exhibited I-N*-TGB-SmA-SmC-Cr (51c) and I-N*-SmA-Cr (51e) phase sequence. X-ray diffractogram of 51C at two different temperatures and calculation of lenth of the molecule from DFT studies indicated that SmA is mono layer in nature where as the SmC phase is tilted in nature. We⁴² have also reported compounds in which aryl substituted sulphur containing heterocycles linked with 4-substituted aromatic ring ended up with cholesterol via an imine linkage (Scheme 12). Compounds 57a–c was synthesized from 52 via nitration, Suzuki cross coupling, hydrogenation. The condensation reaction between the aldehyde 35 and the amines 57 yielded a series of dimers 58a-e (Scheme 12).



Liquid crystalline nature of all the dimers were established from POM and DSC experiments. POM study of the compounds 58a, 58b, 58d, 58f showed only cholesteric phase where as the compound 58e exhibited Cr-SmA-N*-I phase sequence. Several author reported cholesterol based dimer with thiophene moiety⁴³⁻⁴⁵ which exhibits either N* and/or Smectic phases but in our studies we have obtained number of new phases that was not reported by others for thiophene based compound of the above types. In continuation of our earlier effort to connect cholesteryl unit with heterocyclic unit, we have synthesized mesogenic compounds 61 and 62.⁴⁶ Synthetic method is described in Scheme 13. The compounds 61a and 61c were synthesized by condensation reaction between aldehyde 35 and either ethyl or methyl derivative of 5-aminouracile 59 on the other hand the compound 62b was synthesized by condensation reaction between compound 60 and aldehyde 35 (Scheme 13).



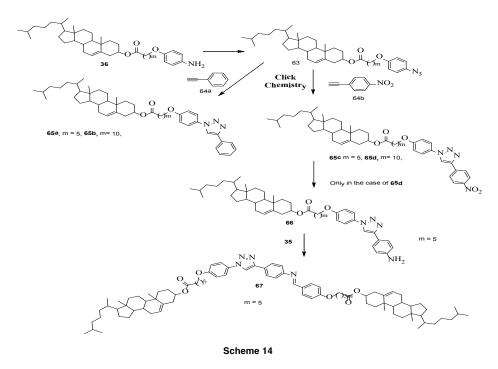


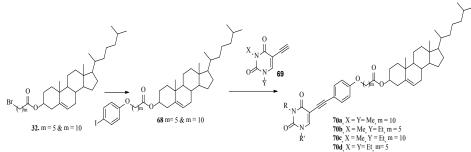
The compound 61a and 62a showed the phase sequence of crystal-SmA-TGB–N*-I, where as the compounds 61b-d and 62b are non-mesomorphic in nature. HRXRD indicated that SmA phase in 61a was monolayer in nature where as for 62a it was partial bi layer. The liquid crystalline phase may be generated in this case due to attraction of opposite poles that may be due to polar and non-polar ends within molecule though there is instance of forming liquid crystalline phase in compound of these types due to H-bonding as in waston-Crick base pairing⁴⁷.

Click-chemistry⁴⁸ is an important tool for the synthesis of cholesteric-based mesogenic material. Recently we have reported a series of compound having a triazole moiety and a cholesterol unit. Synthetic procedure is outlined in Scheme 14. Compounds 65 (a-d) and 67 were obtained from the compound 63. The dimesogens 65a–d was obtained in good yield yields by treating the azides 63 and alkynes 64 as depicted in Scheme 14. Compound 66 and 35 on condensation give the trimesogen 67 (Scheme 14).

Dimesogens of this series exhibited chiral smectic phase and cholesteric phase and in between the two phases TGB phase whereas the trimesogen exhibited Cr-SmC*-N*-I phase sequence. The presence of SmC* phase was established by HRXRD and DFT study.

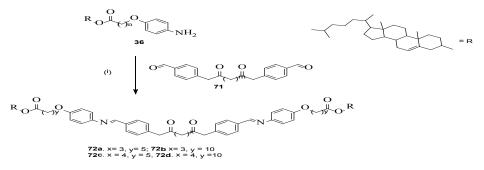
Over the years tolane based dimesogen compounds found importance due to their wide variety of phase sequence and necessity challenging synthetic strategy.⁴⁹ Recently we have also reported the synthesis of materials in which substituted pyrimidine core and cholesterol takes the two terminal end of a linear un-symmetric dimer interconnected via tolane unit.⁵⁰ The compounds 70a-d were synthesized by cross coupling between compound 68 and 69 using Sonogashira invented methodologies (Scheme 15).







Compound 70a-d only exhibits cholesteric phase After successful synthesis of the dimers and trimers cholesterol based tetramer was prepared by the condensation of aldehyde 71 and amine 36 as depicted in Scheme 16.⁵¹ Tetramers 72a-d was synthesized by condensation reaction between the aldehyde 71 and amine³⁶.



Scheme 16

The tetramer 72a, 72c-d exhibited only one mesophase where as the compound 72b exhibited

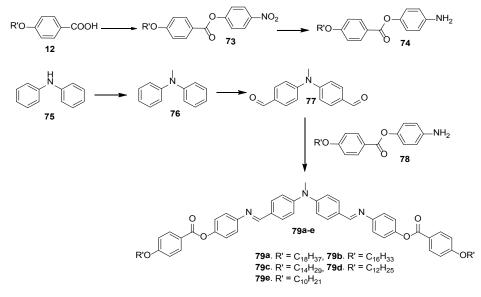
two mesophase transition in DSC experiment. From the POM study it was clear that the mesophase of 72a, 72c-d was cholesteric in nature whereas the compound 72b showed Cr-SmA-N*-I phase sequence. That the SmA phase was partial bilayer in nature was confirmed by HRXRD and molecular length calculation

Bent Shaped liquid crystal

Banana-shaped liquid crystals are an important field in the research of mesogenic material and supramolecular chemistry. The phases obtained from bent-shaped generally differ from those of the rod-shaped molecules.⁵²⁻⁵³ Banana mesogens having asymmetric centers at the end of the long-pendent or a unsymmetrical banana branch

exhibits ferroelectric behaviors. The bent-mesogens generals display the characteristics texture of B1, B2, B3, B4, B5 and B6 phases⁵⁴⁻⁵⁵ Synthesis of symmetrical and limited numbers of un-symmetrical bent-shaped molecules are known in literature.⁵⁶

In 2008 we have first reported bentcore molecule in which methylphenylaniline was used as a center core material.⁵⁷ The synthesis is depicted in Scheme 17. N-methyldiphenylamine 76 was formylated to access the core moiety 77 for the synthesis of banana-shaped molecules. Two symmetrical branched was synthesized by esterification followed by reduction. The homologous series 79a-e was prepared by the condensation of the aldehyde 77 with amine 78 (Scheme 17).

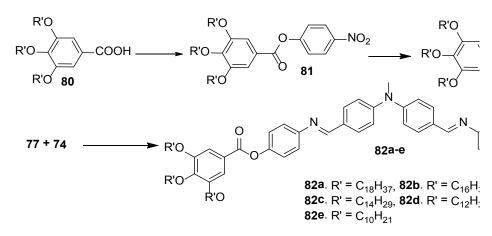




Compound 79a in POM exhibits a deformed smectic leaf texture which grew slowly with elliptical domains in different sites. On further cooling it is transformed into a more ordered twisted or coiled like texture. The compound 79b also exhibited similar type of behaviour. The compound 79c-e exhibited an unknown transition before displaying the texture of higher homologues. Powdered X-ray study and molecular length calculation at two different temperatures of 79a indicated the presence of SmA_d (higher temperature phase) and SmA₂ (lower temperature phase) phases. As the molecule deviated significantly from the bent-shape and approached a rod-like configuration,

thus the appearance of phases of rod-like liquid crystalline molecules was not unusual in this case. Disubstitution on TPA core with a CH₂NH linkage⁵⁸ generally resulted in non-mesophase behaviour but observation of SmA with di-substituted N-MeDPA core is quite significant in our study.

We had also reported another series of symmetrical banana-mesogen by changing the two symmetrical ends of above series.⁵⁹ The preparation of 82a-e is outlined in Scheme 18. Bent derivatives 82a-e was synthesized by the condensation reaction between amine 74 and aldehyde 77. The amine 74 in turn was synthesized by hydrogenation of the nitro derivative 81 (Scheme 18)



Scheme 18

82a-d displays the characteristic texture of columnar mesophase under POM whereas compound 82e is non-mesomorphic in nature. Probably the shorter chain length of 82e makes it non-mesmorphic. As the length of the terminal chain increases transition temperature decreases of the respective compounds. Although the molecules are bow-shaped but due to the presence of three alkoxy chain at the terminal position stacks the molecules in such a way that arrangement like a column within the molecular structure is possible resulted into the generation of columnar mesophase.

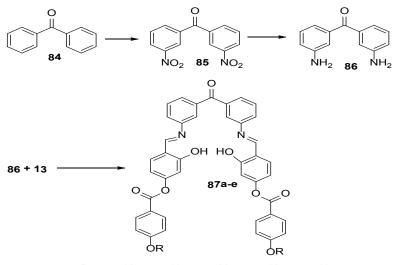
Later we had reported a series of symmetrical

mesogen with substituted benzophenone moiety.60

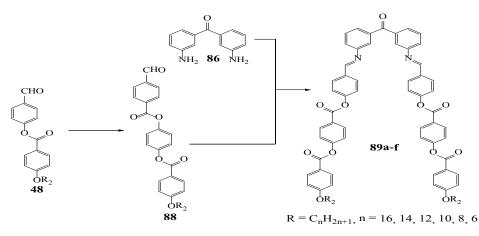
Syntheses is outlined in Scheme 19. The core moiety 86 was prepared by nitration followed by hydrogenolysis. Compound 87a–e was obtained by condensation between 86 and 13.

Higher homologues exhibited typical fan like texture characteristic of B2 phase where as the lower homologoues are non mesomorphic in nature. The existence of liquid crystalline phase was over a very short range undetectable by HRXRD experiment.

We had also synthesized another series of bent-shaped material by increasing one aromatic ring each in both the arm of the above series (Scheme 20).⁶²



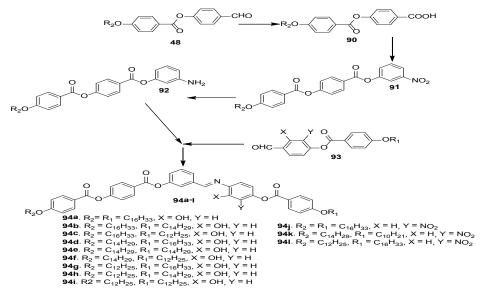
 $\mathsf{R} = -\mathsf{C}_{18}\mathsf{H}_{37}, \ -\mathsf{C}_{16}\mathsf{H}_{33}, \ -\mathsf{C}_{14}\mathsf{H}_{29}, \ -\mathsf{C}_{12}\mathsf{H}_{25}, \ -\mathsf{C}_{10}\mathsf{H}_{21}$



Scheme 20

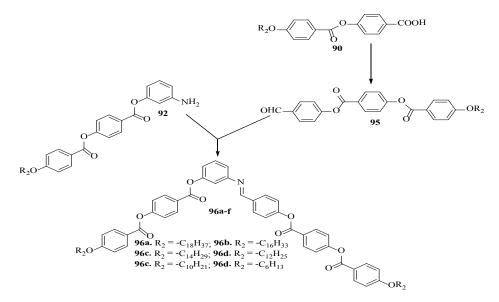
89a-d exhibits typical textures of B1 phase where as 89e-f exhibits only the textures of B6 phase. In order to determine the correct mesophase HRXRD was performed. HRXRD indicates the presence of B1 phase for compound 89C, on the other hand the mesophase of compound 89f identified as B6 phase which is in accordance with the observation under POM study. Though bending angle deviates significantly from ideal angle required for banana mesogens and also number of aromatic ring in the two branched is greater than ideal requisite value of exhibiting banana phase (exception are known^{63,64}) we observed only the phases that is expected from a banana shaped mesogens.

Next we move from symmetrical bent-core molecule to an un-symmetrical one.⁶⁵ We have prepare the material by condensation of amine 92 with substituted aldehyde 93 (Scheme 21). The amines in turns prepared in five steps starting from alkoxy substituted acids.



Scheme 21

94a-I exhibited a stripe-like texture the B2 phase. Further confirmation of B2 phase was established from HRXRD experiment. The B2 phase is stable over a range of 20°C for 94a-i and around 3-4°C for 94j-I. We did not any other phase rather than the B2 phase though it is very common to observe the phases of rod-like mesogens in compounds with substitution in arms⁶⁶. In continuation of our earlier study we have modified the above series by varying the length of two arms.⁶⁷ Compound 96a–f was synthesized by usual condensation reaction between 92 and 95 (Scheme 22).



Scheme 22

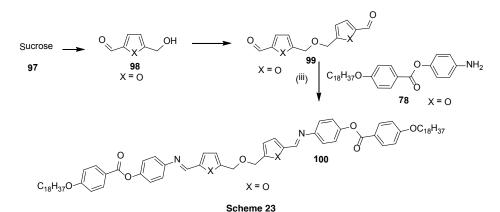
The compounds bearing the longest chain length (18, 16 and 14) showed the B2 phase. Middle members of the homologous series (compounds with chain length 12, 10) displayed a B1 phase (C12 is enantiotropic while C-10 is monotropic in nature, , while the lowest-member of the series (chain length 6) exhibited a B6 phase.

The X-ray diffraction diagram of the compound 96b indicates the presence of the B2 phase, where as the phase of 96d and 96f identified as B1 and B6 from their small angle reflaction pattern.

Such a type of banana mesogen generally possesses higher melting points or B1 to B2 over a

short temperature range⁶⁹⁻⁷⁰. Thus the appearance of B2, B1, B6 with variation of chain length over a relatively wide temperature range made our series quite significant.

So far we discussed the liquid crystalline property of pure bent core liquid crystalline material synthesized by us, but we also synthesized some bent core material unusual in shape. We had reported bent-shaped dimeric liquid crystalline material in which bis-furan-2-aldehyde was used as a core moiety.⁷¹ Bis-furan-2-aldehyde 99 obtained from sucrose by dehydration was condensed with 78 to obtained the compound 100 (Scheme 23)

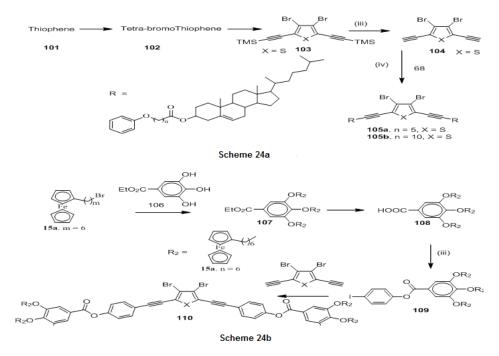


The sample displayed the texture of higher order smectic phases.

Several authors synthesized mesogen with banana-calamatic hybrid to obtain some unusual properties⁷²⁻⁷³. Recently we too reported a thiophene-containing materials that may be designated as a mixture of bent and rod-shaped materials.⁷⁴

Compounds 105a-b was synthesized by reaction between intermediate 104 and cholesteryl iodide 68. The intermediate 104 was synthesized by three step procedures starting from thiophene by bromination, sonogashira cross coupling followed by deprotection (Scheme 24a). On the other hand the acid derivative 108 was obtained by reaction of the compound 15a alkylation and subsequent hydrolysis of the ester. Compound 109 was obtained from 108 by usual ester formation reaction. Final product 110 was prepared by cross coupling reaction between 104 and 109 (Scheme 24b).

The compounds 105a-b showed enantiotropic phase sequence of Cr-SmA-N*-I. However, compound 110 did not show any mesomorphic phase behavior.



Disc-shaped liquid crystal research on Disc-shaped liquid crystal was flourished after the discovery of Disc-shaped liquid crystal by Chandrasekhar and the coworkers almost 45 years ago.⁷⁵⁻⁷⁶ Disc-shaped liquid crystal has profound application solar photovoltaic cells.⁷⁷⁻⁷⁸ Disc-shaped liquid crystal generally aggregate among themselves into hexagonal or rectangular fashion to display characteristic columnar phase.

In 2007 our group first published the synthesis and characterization of disc-shaped molecules derived from triphenyl amine.⁷⁹ Compound 111 was prepared from amine 74 and trialdehyde 87 via condensation reaction (Scheme 25).

The compound 111 when placed in a thin

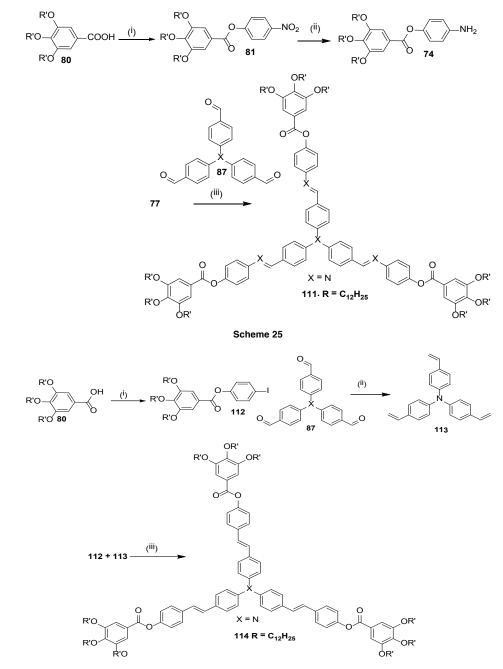
cell it showed typical texture of columnar phases. HRXRD indicates that mesophase is arranged in hexagonal columnar fashion. The liquid crystalline behaviour could be explained by micro-segregation of the promesogenic polar ester with branched chain attached at the end.

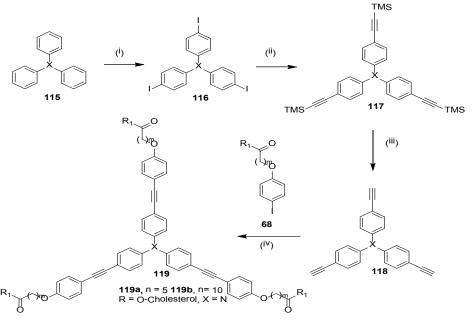
We have synthesized another compound by replacing C=N bond of Scheme 26 with C=C bond. The synthesis is depicted in Scheme 26.⁸⁰ The target compound was prepared by Heck cross coupling reaction between 112 and 113 (Scheme 26).

Compound 114 under POM shows the presence of only one mesophase and DSC study confirm the finding of POM. X-ray diffraction study suggested that the mesophase was a rectangular columnar one. Liquid crystalline materials with a triphenyl amine core and a alkenyl linkage are generally non-mesomorphic in nature⁵⁸ so the observation of columnar mesophase in our case is quite promising in our case.

Modification of the above series was done recently by replacing double bond bond

with triple bond along with replacement of aromatic trialkoxy side chains with cholesterol unit. The synthesis is outlined in Scheme 27.⁸¹ Compound 117 the intermediated of the synthesis was prepared from triphenylamine in three steps by iodination, sonogashira cross coupling and subsequent TMS-deprotection (Scheme 27).



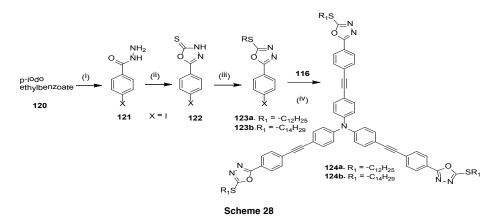


Scheme 27

Compound 119a-b was prepared by coupling reaction between two precursors 118 and 68.

Textural analysis was performed using POM, DSC and HRXRD experiments. None of the compound of this series exhibited the properties typical of disc-shaped molecules instead it displayed the phase sequence of cholesteric compounds. Both the compounds showed SmB-TGB-chloesteric phase sequence. X-ray also confirmed the presence of order smectic phase in compound 119a.⁸² Observation of above phase sequence is contrary to our expectation of showing the phases of either pure cholesterol base dimer⁸³ or the phases of purely disc-shaped one.⁸⁴

We also carried out another modification of the series by replacing cholesterol side chain with thioalkyl substituted heterocycle unit (Scheme 28).⁸⁵ Precursors 123a-b was prepared from p-iodobenzoate in three steps via amidation, cyclization and subsequent thioalkylation (Scheme 28).

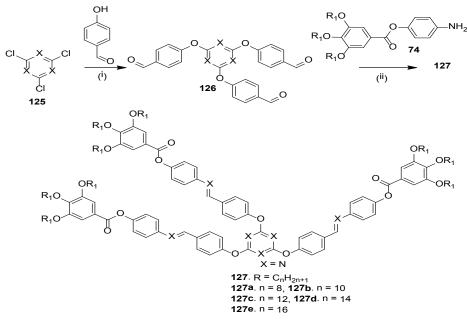


Final cross coupling between 123a-b and 116 afforded the target compounds 124a-b. Unfortunately both the compounds of this series are non mesomorphic however they exhibited excellent fluorescence behavior.

1,3,5-triazine based discotic liquid crystal was synthesized recently by condensation reaction

between precursor 126 and 74^{5,86} (Scheme 29).

All the compounds of the series displayed only one mesophase as evident from DSC experiment. From the POM study liquid crystalline phase was identified as columnar one. HRXRD pattern also confirms the observation of POM study. It is assumed that the molecules are stacked over each other one after another, forming a single column and the columns were positioned symmetrically in a hexagonal fashion. 1,3,5 trizine based reported compounds generally exhibits SmA phases⁸⁷⁻⁸⁸ but in our case we observed mesophase.



Scheme 28

CONCLUSION

The short review mainly described the synthesis, liquid crystalline property of various shapes of liquid crystals which were designed, synthesized and characterized by us over a brief period. In our opinion, it may be a helpful for those who are engaged in this field of research.

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