Chapter VI

Four-ring Bent-Core Mesogens with Different Terminal Alkyl Chains
6.1. Unsymmetrical Four-ring bent-core mesogen

The discovery of polar order in the bent core compounds promoted the importance of these bent or banana or V-shaped liquid crystals as a new sub-field of liquid crystals. Because of the sterically induced packing of these bent molecules several new smectic modifications can be realized which have no counterpart in the field of calamitic liquid crystals [1]. On the other hand, structural features of the new mesophases can lead to unusual physical properties which are not only interesting from the theoretical point of view but also in practical applications. As a consequence of the special molecular packing of bent molecules, smectic layers with $C_{2v}$, $C_2$, and possibly also $C_{1h}$ or $C_1$ symmetry are realized [2] giving rise to ferro-, ferri- or antiferro-electric properties. The ferro- or antiferro-electric switching and the second harmonic generation (SHG) activity of some phases offer, in principle, interesting possibilities for practical applications.

In the last decade there has been a revolution in the molecular design, synthesis and characterization of bent or bow or banana shaped achiral molecules exhibiting banana mesomorphism apart from chiral phases, dark conglomerate phases, two dimensional polar columnar phases etc. Most importantly till date various modifications had been attempted and these modifications are confined to five and six ring systems or else four ring systems separated by an alkylene spacer.

In the year 2010, Rao et al. [3] have designed and successfully synthesized the four-ring bent core system exhibiting banana mesomorphism. The modification of angular 3,4'-disubstituted biphenyl central unit with the introduction of ester linkage between the two phenyl rings realized two novel polar central cores for possible extension of bent core molecular architectures. The central part is a bent-core molecule with nonlinearly substituted two phenyl rings connected through a flexible ester linkage. Unlike the bent mesogenic dimers and/or hockey-stick molecules the reported compounds represent a novel class of bent shaped molecules comprised of four phenyl rings.

In this chapter the preparation and investigation of bent-core liquid crystals containing four aromatic rings with ester and imine linkage and having unsymmetrical end alkoxy chains will be discussed.
The modification of 3,4'-disubstituted biphenyl central unit with the introduction of an ester linkage between the phenyl rings can yield two different central cores 3-aminophenyl-4-aminobenzoate and 4-aminophenyl-3-aminobenzoate. On extending both the ends with two different alkoxy salicylaldehyde yielded two homologous series of compounds as presented in Table 6.1.

Table 6.1: List of Compounds designed and synthesized

![Chemical structures](image)

6.1.1. Results and Discussion
[4-(N-4'-alkyloxysalicylideneimino)benzoate]-[3-(N-4'-undecyloxysalicylideneamino) phenyl] [1-n-11]:

The important requirement for achieving polar order and chirality in any of the materials is that the individual constituent molecule should possess a bent shape in the central core and non-coplanarity of the phenyl rings in the molecule to result into noncentrosymmetric and non-coplanar architecture. In attempting to devise a strategy for the synthesis of bent-shaped molecules, we reasoned that Carsten’s angular 3,4' and 4,3'-disubstituted biphenyl central unit [4] can be separated by an ester linkage which can still be sufficiently bent shaped and unsymmetrical to match the perturbation produced in the form of a shape factor by the introduction of a bridging group between two phenyl rings to yield a unsymmetrical molecule. The intermediate two side wings of this unsymetrical bent-core liquid crystal was based on reaction of 4n-alkoxysalicylaldehyde moiety, one with 3-aminophenol and another with 4-aminobenzoic acid in ethanolic solution in presence of glacial acetic
acid as catalyst. But the reaction of undecyloxysalicylaldehyde with 3-aminophenol is fixed at one end with the variation of alkoxy chain at the other end. The synthesis of the designed compounds 1-n-11 \((n = 6, 10, 12, 13, 14, 15 \text{ and } 16)\) are presented in Scheme 6.1.

The starting material in the present study 4-n-alkyloxysalicylaldehyde was prepared by Williamson etherification of 2,4-dihydroxybenzaldehyde with appropriate n-alkyl bromide. The target bent-core material \([4-(N-4'-dodecyloxysalicylidene)benzo-ate]-[3-(N-4'-undecyloxysalicylidene-amino) phenyl] (1-12-11)\) was prepared by coupling 3-(N-4'-undecyloxysalicylidene-amino)phenol and 4-(N-4'-dodecyloxysalicylideneamino)benzoic acid in dry dichloromethane (DCM) and dicyclohexylcarbodiimide (DCC) in presence of \(N,N'\)-dimethylaminopyridine (DMAP) as a catalyst at 25°C. The precipitate of dicyclohexylurea obtained as side product was filtered off from the reaction mixture. The compounds were further recrystallized repeatedly by absolute ethanol to get the pure compound as yellow solid.

The formation and purity of all of the compounds was confirmed by CHN analysis, \(^1\)HNMR and IR spectroscopy. The other compounds of 1-n-11 homologue with \(n = 6, 10, 12, 13, 14, 15 \text{ and } 16\) were also synthesized by the same procedure and
characterized. The details of the experimental procedures along with the spectroscopic data for 1-n-ll homologue of compounds are presented in the experimental part of Chapter 3. The liquid-crystalline behaviour of the synthesised compounds had been investigated by polarising optical microscopy (POM) and differential scanning calorimetry (DSC).

6.1.2. Mesomorphic properties
The transition temperatures, enthalpies and entropies associated with the phase transitions of all the compounds of the homologous series 1-n-ll (n = 6, 10, 12, 13, 14, 15 and 16) obtained from DSC at a scan rate of 5°C min⁻¹ in the heating and cooling scans are presented in Table 6.2. The DSC thermogram of compound (1-12-11) is shown in Figure 6.1.

All the compounds were found to exhibit one mesophase only exhibiting almost same texture. The observed optical textures exhibited by a representative compound [4-(N-4'-dodecyloxysalicylidenemino)benzoate]-[3-(N-4'-undecyloxy salicylideneamino) phenyl] (1-12-11) are presented in Figure 6.2.

![Figure 6.1: DSC thermogram of 1-12-11](image-url)
Table 6.2: Transition Temperatures (°C), Enthalpies (italics kJ/mol) and entropies (italics in brackets J/mol/K) of homologues series 1-n-11.

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<tr>
<th>Comp</th>
<th>n</th>
<th>Cr</th>
<th>Heating Cooling</th>
<th>Heating Cooling</th>
<th>B7</th>
<th>Heating Cooling</th>
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<td></td>
<td>[22.8, 78.9]</td>
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<td>•</td>
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<td>107.5</td>
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<td></td>
<td></td>
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<td>[2.2, 8.3]</td>
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<td>•</td>
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<td>106.6</td>
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<tr>
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<td>1-16-11</td>
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<td>[27.5, 90.4]</td>
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<td>[6.6, 21.2]</td>
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Upon very slow cooling the isotropic liquid, the mesophase defect textures of 1-12-11 compound exhibited by (Figure 6.2a) resembles the B₇ phase texture of fascinating long spiral domains. The other remarkable feature of these compounds is circular domains texture with dark extinction crosses (Figure 6.2b) coinciding with the direction of polarizer and analyser. Optical textures of the compound 1-12-11 in untreated glass plate and coverslip are presented in Figure 6.2c and 6.2d. All the compounds of the homologous series 1-n-11 exhibit banana liquid crystalline behaviour over a moderate temperature region. The phase transition temperature as a function of number of carbons in the end alkyl chain length is depicted in Figure 6.3.
Figure: 6.2: Different type of domains observed for B phase of 1-12-11 (a) and (b) Fan-shaped texture in 5 micron cell at 109.8°C and 103°C respectively; (c) in normal glass plate at 109°C and texture (d) full growth at 102°C.

Figure: 6.3: Phase transition temperatures of 1-n-11 homologue as a function of number of carbons in the alkyl chain length.

All other compounds of this homologue shows typical textural features observed for this series of compound which are shown in Figure 6.4. All the samples on cooling
exhibited characteristic textures viz., (i) growth of focal conic texture with spiral filaments (ii) only spiral texture (iii) fan-shaped texture (iv) low birefringence 2D focal conic and (v) circular domains texture resembling the textures observed in B7 phase of known reference compounds [3a,3b]. In particular majority of the textural features observed are similar to those observed for the 2-nitro-1,3-phenylene bis-[4-(4-n-alkoxyphenylimino- methyl)benzoate] (n-OPIMB-NO₂) [5], which is a representative standard material exhibiting B7 mesophase.

Figure: 6.4: Typical textures observed in B7 phase of 1-n-11 homologue (a) growth of focal conic texture with spiral filaments of 1-6-11; (b) spiral texture of compound 1-10-11; (c) full growth of texture b; (d) and (e) resembling fan-shaped texture of compounds 1-13-11 and 1-14-11; (f) low birefringence 2D focal conic of 1-16-11.
6.1.3. X-ray studies:
To confirm the mesophase structures X-ray diffraction studies are performed on a representative compound 1-12-11 belonging to the homologous series 1-n-ll. A typical diffraction pattern obtained for the compound 1-12-11 at 98°C and 105°C are shown in Figure 6.5. Multiple sharp peaks in the wide angle region at 98°C indicate the sample became crystalline. At 105°C the diffraction pattern indicated a strong reflection at $2\theta = 1.95^\circ$ corresponding to 43.07Å. This corresponds to the molecular length which is slightly lower than the estimated molecular length of 47.3 Å (Figure 6.6) in all trans conformation which indicates a tilted layer with a tilt angle 24°. In the small angle region ($2\theta = 1 - 8^\circ$) low intense peaks are observed for the 2D polarization splay modulated layer undulated structure, a structural feature associated with the B? and the B_{RevTilted} phases with a 2D lattice. In addition to these reflections a broad diffuse wide angle reflection at $2\theta = 19.3^\circ$ corresponding to 4.57 Å reflects the spacing between the aliphatic chains indicative of a liquid-like in-plane order of the fluid phase.

![Figure 6.5: Intensity profile of the X-ray diffraction pattern of the compound 1-12-11 at 105 °C](image)

The formation spiral domains and 2D focal conic textures, the multiple peak reflections at low angles, the tilt in molecular layering and non-switchable phase structure are indicative of a B? phase structure and suggest that the phase represents
2D polarization splay modulated layer undulated molecular structure of SmCPU family [6-14]. Further, electro-optical experiments on the compounds are in progress to compliment the X-ray results and to assign the reflections and to identify the exact phase structure.

If we take a look at the most stable conformational molecular structure of the compound 1-12-11 as shown in Figure 6.6, exhibit distinct twisted molecular conformation. The plane of the first two phenyl rings separated by the ester linkage from the plane of other two phenyl rings in compound 1-12-11 is twisted to an extent that the two planes are almost perpendicular to each other. Hence it is believed that the twisted conformation aided by the lateral H-bonding in these molecules promotes the helical arrangement in 1-12-11.

![Molecular structure and energy minimised conformation of 1-12-11.](image)

Molecular length = 47.3 Å

**Figure: 6.6**: Molecular structure and energy minimised conformation of 1-12-11.

### 6.1.4. UV-Visible Study:

UV-Visible spectra of all the compounds of the homologue 1-n-11 (n = 6, 10, 12, 13, 14, 15 and 16) at 10^{-5} M concentrations were recorded in dichloromethane (CH2Cl2). The representative compound 1-6-11 of the 1-n-11 homologue has absorption peak centered at ~349 nm (3.5 eV, ε = 119000 Lmol^{-1}cm^{-1}) is due to the π-π* transition of the highly π-conjugated system having the substituted phenyl benzoate unit as the core. UV-Visible spectra of 1-n-11 homologue series are shown in (Figure 6.7).
6.1.5. [3-(N-4'-undecyloxysalicylidene-amino)benzoate]-[4-(N-4'-alkyloxysalicyli-
-cdenemino) phenyl]: (2-n-11)

The modification of angular 3,4'-disubstituted biphenyl unit with the introduction of
an ester linkage viz., COO moiety between the phenyl units leads to structural
variation of polar groups in bent core molecules. It had been demonstrated earlier
that the ester linkage as well as reduction of phenyl rings in general reduces the
melting and clearing temperatures in the five and four ring systems. So to examine
the effect of direction of the ester linkage on the phase transitions and phase
structure of four ring systems, homologues of compounds have been synthesized and
their properties were characteristics by different technique. The molecular structure
of this homologues series was presented above (Table 6.1). Moreover, with respect
to the unequal distribution of the two side chains or wings of the central aromatic
rings these molecules could also be regarded as hockey stick molecules.

The bent-core material with unsymmetrical side chains of two phenyl rings yields an
unsymmetrical molecule. The intermediate two side wings of this unsymmetrical
bent-core liquid crystal was based on reaction of 4-n-alkoxysalicylaldehyde moiety,
one with 3-aminophenol and another with 4-aminobenzoic acid in ethanolic solution
in presence of glacial acetic acid as catalyst. The synthesis of the designed
compounds 2-n-11 homologue (n = 10, 12, 14 and 16) are presented in Scheme 6.2.
The starting material in the present study 4-n-alkyloxysalicylaldehyde was prepared by Williamson etherification of 2,4-dihydroxybenzaldehyde with appropriate n-alkyl bromide. The target bent-core material \([3-(N-4'-\text{undecyloxysalicylidene-amino})\text{benzoate}]-[4-(N-4'-\text{alkyloxysalicylic denemin})\text{phenyl}]\) \((2-12-11)\) was prepared by coupling \([3-(N-4'-\text{undecyloxysalicylidene-amino})\text{benzoic acid}]-[4-(N-4'-\text{alkyloxysalicylic denemin})\text{phenol}]\) in dry dichloromethane (DCM) and dicyclohexylcarbodiimide (DCC) in presence of N, N'-dimethylaminopyridine (DMAP) as a catalyst at 25°C. The precipitate of dicyclohexylurea formed as side product was filtered off from the reaction mixture. The compounds were further recrystallized repeatedly by absolute ethanol to get the pure compound as yellow solid. The formation and purity of all of the compounds was confirmed by CHN analysis, \(^1\)HNMR and IR spectroscopy. This homologues with \(n = 10, 12, 14\) and 16 were also synthesized following the same procedure and characterized. The details of the experimental procedures along with the spectroscopic data for all the homologous series of compounds are presented in the experimental section of Chapter 3. The liquid-crystalline behaviour of the synthesised compounds had been investigated by polarising optical microscopy (POM) and differential scanning calorimetry (DSC).

**Scheme 6.2:** Reagents and conditions i. Ethanol, drops of glacial acetic acid, reflux 4 h; ii. DCC, DMAP, DCM, 48h
6.1.6. Mesomorphic properties:
The transition temperatures, enthalpies and entropies associated with the phase transitions of all the compounds of the homologues series 2-n-11 \((n = 10, 12, 14 \text{ and } 16)\) as a function of number of the carbon atoms in the terminal alkyl chains of one end keeping other end fixed with undecyloxysalisaldehyde \((n = 11)\) obtained from DSC at a scan rate of \(5^\circ C \text{ min}^{-1}\) in the second heating and first cooling scans are presented in Table 6.3.

![DSC thermogram of 2-12-11](image)

**Figure: 6.8: DSC thermogram of 2-12-11**

All compounds of the 2-n-11 homologues exhibit banana liquid crystalline behaviour over a moderate temperature region. The calorimetric study on a representative compound 2-12-11 of the homologue 2-n-11 exhibited two phase transitions in heating and cooling cycles. The differential scanning calorimetric thermogram of compound 2-12-11 is presented in Figure 6.8.

The phase transition temperature as a function of number of carbons in the end alkyl chain length is depicted in Figure 6.9. All the compounds were found to exhibit one mesophase only exhibiting similar textures. On increasing the length of end alkoxy chain the mesomorphic range increases from 17° \((n=10)\) to 27° \((n=16)\). A decreasing trend of the melting and clearing transition temperatures with the increasing chain length of the terminal alkoxy chains is observed.
### Table 6.3: Transition Temperatures (°C), Enthalpies (italics kJ/mol) and entropies (italics in brackets J/mol/K) of homologous series

<table>
<thead>
<tr>
<th>Comp</th>
<th>n</th>
<th>Heating Cooling</th>
<th>Cr</th>
<th>B1</th>
<th>Heating Cooling</th>
<th>I</th>
</tr>
</thead>
</table>
| 1-10-11 | C_{10}H_{21} | 90.7  
[3.9, 14.1] | 137.4-138.9  
[24.0, 91.5] | 147.5  
[29.3, 50.1] | ● |
|        |     | 82.7  
[2.6, 9.7] | 121.9  
[17.3, 57.4] | 138.3  
[15.7, 50.7] | ● |
| 1-12-11 | C_{12}H_{25} | 95.3  
[24.3, 83.4] | 135.6  
[52.6, 162.7] | 140.4  
[17.5, 53.7] | ● |
|        |     | 80.3  
[10.8, 38.9] | 115.2  
[49.0, 180.2] | 139.3  
[17.3, 53.1] | ● |
| 1-14-11 | C_{14}H_{29} | 98.7  
[16.7, 53.9] | 130.8  
[30.9, 90.9] | 138.8  
[13.3, 39.5] | ● |
|        |     | 77.8  
[32.3, 103.4] | 137.0  
[8.6, 25.8] | ● |
| 1-16-11 | C_{16}H_{33} | 93.1  
[13.6, 43.8] | 127.5  
[29.5, 87.0] | 137.7  
[11.4, 33.0] | ● |
|        |     | 80.8  
[12.2, 40.9] | 109.8  
[32.5, 100.3] | 136.1  
[8.0, 23.3] | ● |

Figure: 6.9: Phase transition temperatures as a function of number of carbons in the alkyl chain length in the cooling cycle.

The observed optical textures exhibited by a representative compound of 2-<em>n</em>-<em>ll</em> homologue are presented in Figure 6.10. Upon very slow cooling from the isotropic liquid, compounds of 2-<em>n</em>-<em>ll</em> (n= 10, 12, 14 and 16) exhibited developable fan like textures shown in Figure 6.10a-f. The developable domain-like textures resemble the rectangular columnar B\textsubscript{1} phases which has been widely related to the existence
of smectic undulated phases [15]. On further cooling, the material enters completely
to a structured columnar fan like texture under the polarization microscope.

Figure: 6.10: Typical textures observed of 2-n-11 homologue (a) at 133.3°C of 2-
10-11, developable domains; (b) 2-12-11 in normal glass plate; (c) and (d) focal
conic B1 of 2-12-11 in different cell thickness 3.5 & 9 μm respectively; (e) at
135.6°C of compound 2-14-11; (f) 2D modulated smectic phases of compound 2-16-
11.

However, such type of textures are exhibited by 2D modulated smectic phases of
bent core molecules based on locally ferroelectric layering and spontaneous splay of
the polarization. Based on polarization splay modulation (either syn- or anti-order),
slopes of undulated layers and layer continuity versus layer discontinuity at the
intermediate defects, twelve different phases are proposed. Only some of the phase structures had been realized till now. The textural features of 2-n-11 homologue in the present work resemble the textures observed in compounds exhibiting 2D polarization splay modulated layer undulated B2/B2RevTilted phases [16-18].

If we take a look at the most stable conformational molecular structure of the compound 2-12-11 as shown in Figure 6.11 it exhibit planar molecular conformation. The plane of the first two phenyl rings separated by the ester linkage from the plane of other two phenyl rings in compound 1-n-11 is twisted to such an extent that the two planes are almost perpendicular to each other but the compound 2-n-11 exhibit planar conformation i.e all the four phenyl rings lie on the same plane which is drastically different from the 1-n-11 series. Hence it is believe that the direction of ester linkage (-COO or -OOC) promoted twist or planar conformation.

Figure: 6.11: Molecular structure and energy minimised conformation of 2-12-11

6.1.7. UV-Visible study:
The UV-Visible absorption spectroscopic properties of the representative compound 2-n-11 in solution were studied in dichloromethane as solvents of 10^{-5} M concentrations to obtain the information regarding absorption. The compound 2-10-11 exhibited absorption peaks in the UV-Visible spectrum at ~344nm (3.6 eV, \varepsilon \sim 101000 \text{ Lmol}^{-1}\text{cm}^{-1}). The absorption band with large molar absorption coefficients reflects the \pi-\pi^* transition of the highly \pi-conjugated system among the four planar phenyl group. The UV-Visible spectra of 2-n-11 homologues as are shown in Figure 6.12.
6.1.8. Discussions:

The bent core molecules because of bent shape exhibit polar order and some of them possess tilt order. In such polar LCs the polarization vector is free to rotate continuously in the layer plane under the influence of external fields. To escape from macroscopic polar order of the molecular structure the splay of polarization stabilizes the polarization modulation. The spontaneous breaking of chiral symmetry leads to a coupling between the polarization splay and molecular tilt order [19, 20]. Hence the coupling disturbs the homogeneous layered structure to manifest two-dimensional (2D) modulated structures viz. the polarization modulated either with a continuous periodically undulated smectic layers (PMLU, Classical B₂ phases) or with a discontinuous structure of blocks (columns) of smectic layer fragments (B₁Rev/Tilt) organized into two-dimensional columnar like lattices. Recently it had been shown that banana molecules exhibiting rectangular columnar phases, manifested from broken layers, can possess spontaneously ordered dipoles [21-28]. The broken layers or the fragmented layers can form rectangular columns.

Figure: 6.12: The absorption profile for 2-n11 homologue in dichloromethane (10⁻² M).
Figure: 6.13: Polarization-modulated and layer-undulated structures in bent-core liquid crystals. **a)** synclinic layer (block) structure; and **b)** anticlinic layer (block) structure. Side views and top views of the layers are shown. Arrows represent polarization direction, and lines (|) the director position in space, their length being a measure. In the side view the ending | represents the part of the director that is tilted towards the observer. $\lambda_m$ is the modulation wavelength, $\lambda_w$ is the width of the domain wall (the wall between two regions of favourable polarization splay). $h$ is the longitudinal shift of the synclinically tilted blocks.

6.1.9. Conclusions:

Unsymmetrical bent-core compounds are successfully synthesized which exhibiting B$_1$ and B$_7$ phase structure. In summary, the fundamental phase structure of the banana shaped molecules based on non-symmetric bent-core four ring systems described in this chapter apparently seems to belong SmCP family as evidenced by three dimensional image of orientational pattern. The hydroxyl group in the wings of these molecules participate in inter and/or intra-molecular H-bonding which may promote the formation of undulated and modulated smectic phases.
References:


