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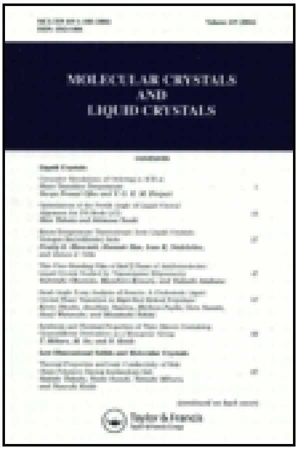

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Biaxial Nematic and Smectic A Phases in a "Peelable Banana-Shaped" Molecule

S. Krishna Prasad [a], Geetha G. Nair [a], D. S. Shankar Rao [a], Chethan V. Lobo [a], I. Shashikala [a] & C. V. Yelamaggad [a]

[a] Centre for Liquid Crystal Research, Jalahalli, Bangalore, India

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Biaxial Nematic and Smectic A Phases in a "Peelable Banana-Shaped" Molecule[†]

S. Krishna Prasad
Geetha G. Nair
D. S. Shankar Rao
Chethan V. Lobo
I. Shashikala
C. V. Yelamaggad
Centre for Liquid Crystal Research, Jalahalli, Bangalore, India

The article discusses the observation of a fluid phase having three-dimensional orientational order and its transformation to a similar phase but with an additional one-dimensional positional order in a low molar system. X-ray and optical observations establish the phases to be biaxial nematic and biaxial smectic A respectively. The organic system used is a unique combination of a bent core molecule linked to a rod-like unit through a flexible aliphatic spacer. We argue that this special combination is responsible for the observation of especially the biaxial nematic phase and propose possible molecular arrangements in the two phases.

Keywords: bent core molecule; biaxial nematic; biaxial smectic A; conoscopy, X-ray

Of the variety of phases exhibited by soft condensed matter, the liquid crystalline phases are probably the most fascinating [1,2]. The simplest of the liquid crystalline phases is the nematic phase, which is an orientationally ordered fluid, with the molecules, on an average, lined up along a common axis having an apolar symmetry, referred to as the director **n**. Known for over a century, this phase, possessing a uniaxial symmetry, forms the constituent of practically all liquid crystal display devices. In contrast, one of the most sought after phases in thermotropic liquid crystals is the biaxial nematic (N_b) phase. Although on the theoretical front some reports [3] have

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[†] Dedicated to Prof. S. Chandrasekhar

Address correspondence to S. Krishna Prasad, Centre for Liquid Crystal Research, Jalahalli, Bangalore 560 013, India. E-mail: skprasad1@yahoo.com

appeared on the properties of the N_b phase, experimentally its existence [4–8], first observed by Chandrasekhar *et al*. [4], is yet to be universally accepted [9]. On the other hand, when a one-dimensional translational order along the director is added to the orientational order, and within the planes perpendicular to it the arrangement of the molecule is still fluid-like, layered smectic phases are realized. The director could be either along the layer normal (smectic A or SmA phase) or tilted with respect to it (smectic C or SmC phase). Till recently, the biaxial character was invariably associated with tilted smectic phases, whereas the SmA was only uniaxial in character.

About thirty years ago the possibility of a biaxial smectic phase with a structure in which the molecules are still along the layer normal, but having an additional director in the plane of the layers, was pointed out [10] and labeled smectic C_M [Here M refers to McMillan, who proposed the first microscopic theory of the SmA-SmC transition.] Further development of this idea was put forth by Brand *et al*. [11] who proposed a biaxial SmA (SmA_b) phase with different kinds of symmetry depending on the type of constituent molecules: D_{2h}, if the molecules are board-like and C_{2V} if they are bent-core or banana-shaped. The observation of the $SmA_b$was first reported about a decade ago in a polymeric system [12] and subsequently has been seen in a binary mixture of rod-like and bent-core molecules [13], pure bent-core molecules [14], and a metallomesogen [15]. Except for the polymeric system no other material has shown a sequence of a transition from the N_b to the SmA_b phase. The obvious experimental difficulties of working with polymeric systems was also mentioned by the authors of Ref. 12. Further, the N-SmA is perhaps the most exhaustively studied phase transitions in liquid crystals, but is still the least understood with quite a few open questions [16]. Investigations on the N_b-SmA_b could perhaps answer some of these questions. Thus the need arises to observe a transition from the N_b to the SmA_b phase in a low molecular weight single component system.

Recently, a novel type of molecular structure, referred to as banana- or bow-shape or bent core has been shown to give rise to new type of liquid crystalline phases, some of which show interesting electro-optic switching behavior [17,18]. Such molecules tilt about the *arrow* direction of the bows breaking the achiral symmetry of the layers leading to polar layers. During a short span of about seven years since the electro-optic switching was observed, several hundred compounds with bent core molecules have been synthesized and at least six different liquid crystalline phases have been identified, the most recent being the SmA_b phase. In fact, all the low molar mass non-metallomesogenic systems that have exhibited the SmA_b phase

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contain bent-core molecules. This should not be very surprising since the arrow direction of the bent-core molecules, if ordered, provides a natural choice for the in-plane director **m**, which in conjunction with the primary director **n** can give rise to a biaxial structure. However, with very few exceptions, bent core molecules give rise to smectic phases. Thus designing the molecular structure for realizing a thermotropic N_b phase is not a trivial task necessitating careful molecular engineering. However, five different classes of thermotropic mesogens have been realized in which the shape of the molecule resembles that of a bone, a spoon, a pipette, an ellipse or a board. In these molecular designs, the shape biaxiality [19] of the rod or disc-like entity is optimized essentially by breaking its shape symmetry. Considering the fact that the bent core banana-shaped systems have strong shape-biaxiality, we introduced a new molecular engineering concept in which the shape-biaxiality of a rod-like mesogen is enhanced by covalently linking it to a bent core molecule. Further, it was borne in mind that flexibility in the molecular structure, introduced using polymethylene units as spacers, generally stabilizes the nematic phase [20]. Additional support to this idea came from the results of the frustrated spin gas model [21], which has been used successfully to explain the reentrance of the nematic phase in strongly polar compounds. With these features in mind we designed and realized a non-symmetrical dimer consisting of a cyanobiphenyl unit, which is a rod-like molecule with a strongly polar terminal group and known to exhibit the nematic phase, covalently attached to a salicylaldimine-based unsymmetrical banana-shaped molecule generally supporting the formation of the smectic phases, through a hexamethylene central spacer. The synthetic route employed to prepare this unique bent-rod dimer, which we refer to as compound **I** (see Fig. 1), along with the preliminary characterization identifying the phases observed to be N_b and SmA_b is described elsewhere [22]. Due to the presence of the terminal polar group and the asymmetric nature of the structure the molecule can be regarded to be like a "peelable banana" in which the top (say, with the stalk) can be differentiated from the bottom. This feature, we feel, is

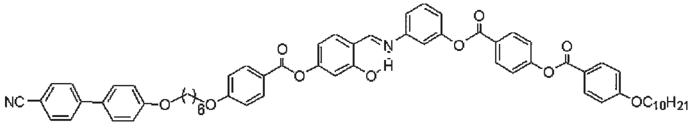

FIGURE 1 Structural formula and phase transition temperatures of the investigated compound having a covalent linkage between the bent core segment and the rod-like mesogen.

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important in stabilizing the type of mesophases observed for this material. The asymmetric nature has the additional role of keeping the transition temperature low.

Three mesophases could be clearly detected by calorimetry, polarizing microscopy and X-ray investigations. The lowest temperature mesophase was found to have a uniaxial character with a layered structure but with some unusual textures. Detailed characterization is underway to establish the nature of this phase and therefore here we just refer to it as **M** phase. From preliminary microscopic observations the other two phases could easily be identified as nematic and smectic respectively. First, we will discuss the characterization of the smectic phase that lies between the nematic and the M phase. X-ray measurements were carried out by taking the sample in a Lindemann capillary tube and aligning it in the presence of a magnetic field ($\sim$2T) by cooling it from the isotropic through the nematic phase. The X-ray diffraction pattern collected using a 2D imaging plate showed a pair of small-angle reflections, whose width was resolution-limited and a pair of wide-angle diffuse arcs orthogonal to the small-angle ones. The extracted spacing of the low-angle reflection is comparable to the length of the molecule in its most extended configuration (Here the length corresponds to the “bow-string” length, i.e., the distance measured between the two end-points of the bent-core molecule.) showing that the phase is SmA.

In the optical studies with substrates treated for planar configuration of the molecules, a focal conic type of texture was observed. However, even glass plates treated with octadecyltriethoxysilane (ODSE), failed to produce a perfectly homeotropically aligned sample. Instead a low-birefringence schlieren texture consisting of dark brushes emanating from some points was observed (see Fig. 2a). Interestingly,

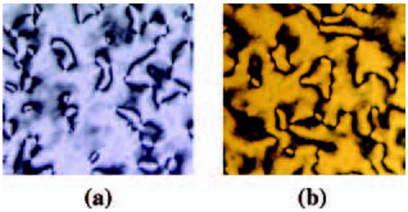

FIGURE 2 Schlieren texture observed in the (a) SmA_b (temperature, 133°C) and (b) N_b (temperature, 160°C) phases when the sample was taken between glass plates treated with ODSE and polyimide-coated but unrubbed, respectively. Notice that the defects are exclusively 1/2 strength disclinations.

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the texture consisted of exclusively two-brush (strength $S = \pm 1/2$) disclinations. It is well known that the schlieren texture is an imprint of the director field in the plane of the substrates and therefore the observation of exclusively two-brush disclinations has important implications. First of all it rules out the phase being a smectic C phase, in which the director is tilted away from the smectic layer normal direction resulting in the presence of an additional director **m** in the plane of the layers and unlike the primary director **n**, **m** is polar in nature, i.e., **m** and $-$**m** are not equivalent, owing to which $\pm 1/2$ defects cannot occur. On the other hand, in a layered structure $\pm 1/2$ defects are predicted [11] as well as experimentally observed for a biaxial smectic A ($\mathrm{SmA_b}$) phase [12–15]. But it should be pointed out that the ordering of the bent-core molecules in a layer such that their "arrow" directions are uniformly oriented would result in a polar layer with an electric polarization appearing parallel to the in-plane director **m**. In such a case, since **m** is polar the structure will not support the formation of $\pm 1/2$ defects for arguments mentioned above. But if the direction of **m** alternates between adjacent layers then it can exhibit a two-brush schlieren pattern and the structure would be anti-ferroelectric in character. To verify whether it is so, we carried out electrical switching measurements using planar oriented samples. Both square wave and triangular wave low frequency (1–10 Hz) fields did not reveal any switching of the sample ruling out the antiferroelectric structure. In order to confirm the optical biaxiality of this phase, conoscopic experiments were carried out. For this purpose samples in which both the **n** and **m** directors are well aligned have to be used, a requirement that was obtained in the following way. The sample was sandwiched between two glass plates treated with ODSE and separated by two metal spacers of ~8 μm thickness; the gap between the metal spacers being ~1 mm. While the ODSE treatment facilitates a homeotropic alignment of the molecules with the **n** director uniformly oriented normal to the substrate, application of a 1 kHz sine wave electric field (70 V magnitude) aligned the in-plane director **m**. The conoscopic pattern obtained (Fig. 3a) shows clear separation of the isogyres, a characteristic feature of biaxial phases. Two other aspects that need to be mentioned are the observations of strong intensity fluctuations and the formation of a striped texture (Fig. 4), both the features being reported in the literature [13,14]. All these features unambiguously prove that this phase is indeed a biaxial smectic A phase. Quantitative measurements of the in-plane birefringence (Δm) using a tilting plate compensator yielded a value of 0.013 at ~10°C away from the transition to the nematic phase. We shall later return to the possible molecular arrangement in this phase.

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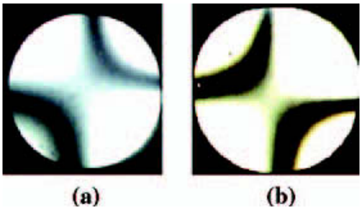

FIGURE 3 Conoscopic patterns in the (a) SmA_b and (b) N_b phases obtained with the **n** director oriented with ODSE coating on the substrate surfaces and **m** director by an in-plane field. The temperatures of the SmA_b and N_b were 132°C and 139°C respectively. The fact that the isogyres are widely separated is a clear indication of the biaxiality of the phases.

When the sample was taken between either untreated or polyimide-coated unrubbed plates, the nematic phase showed the typical schlieren texture, except that the pattern consisted entirely of 2-brush disclinations (see Fig. 2b). As was argued in an earlier paper [6], this is certainly an indication of biaxiality, since the escape mechanism for the $S = 1$ (4-brush) defect does not eliminate the singularity in a N_b phase (as it does in the uniaxial case) $S = 1$ probably becomes energetically unfavourable. This argument is supported by the topological considerations of Mermin [23] and the recent computer simulations [24]. To ensure that the biaxiality is not introduced by the glass surfaces we examined the textures formed in both the N_b and SmA_b phases in a free-standing film obtained by spreading the fluid over a hole drilled in an aluminium plate. Again only $S = \pm 1/2$ defects were

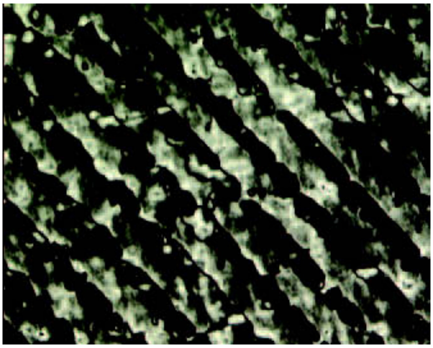

FIGURE 4 Photomicrograph of the alternate dark and bright stripe pattern seen in the SmA_b phase. The width of the stripe is $\sim 50\,\mu m$. Temperature of the sample was 128.5°C.

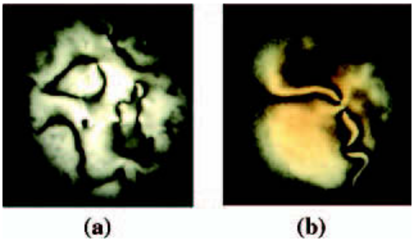
(a) (b)

FIGURE 5 Free standing films of (a) SmA_b and (b) N_b phases showing defects consisting of 1/2 strength disclinations.

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observed (see Fig. 5). The conoscopic observations carried out in the manner described above, showed clear separation of the isogyres (Fig. 3b) confirming the biaxial nature of the phase. The birefringence of the plane perpendicular to the principal director **n** was smaller than that in the SmA_b phase having a value of 0.005. The X-ray pattern obtained in this phase (Fig. 6b) was identical to that in the SmA_b phase (Fig. 6a) except that the full-width-at-half-maximum of the reflections was more indicating lack of positional order along any direction. It may also be pointed out that samples taken between kapton sheets that were coated with a polyimide solution and unidirectionally rubbed also yielded essentially similar patterns in both the SmA_b and N_b phases.

Now we shall discuss the possible molecular configuration in the SmA_b and the N_b phases. (i) One possible structure is that in the SmA_b phase the bent core molecules order in such a fashion as to make the "arrow" direction of the molecules oriented uniformly within a layer (leading to the formation of the **m** director) giving rise to a ferroelectric

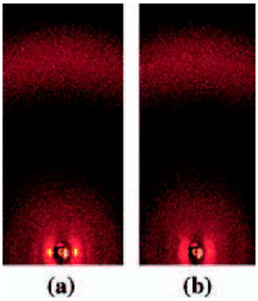
(a) (b)

FIGURE 6 X-ray pattern obtained in the (a) SmA_b and (b) N_b phases. The temperatures of SmA_b and N_b phases were 126°C and 140°C respectively.

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layer. But to explain the presence of the $S = \pm 1/2$ defects the direction of the **m** director has to alternate from one layer to another (Fig. 7a). However, the absence of the any antiferroelectric-like electric switching signal rules out this possibility. (ii) Although a packing of the molecules such that the **m** director of the neighbouring molecules lies in the molecular plane but pointing randomly either to the left or to the right (Fig. 7b) can explain the observed features, such a structure is not efficient from the excluded volume point of view. (iii) The quartet structure [14] (see Fig. 7c), wherein four bent core molecules form a group is a possibility. The quartet as an entity has a D_{2h} symmetry and accounts for all the features observed for the SmA_b phase. The quartet model can also be used to explain a non-polar N_b phase, in which the quartets are arranged in such a fashion as to lose the one-dimensional positional order of smectic phase leading to the appearance of the nematic phase. But it may be noticed that for the nematic ordering, model (ii) mentioned above does not place any serious restrictions on the packing of the molecules. In our opinion, such a structure (Fig. 7d) is more probable than the quartet structure. Further, from a molecular point of view, the appearance of the nematic phase in a system of bent core molecule of the type that we have synthesized can be explained on the basis of the quite successful frustrated spin gas model. This model developed by Berker and

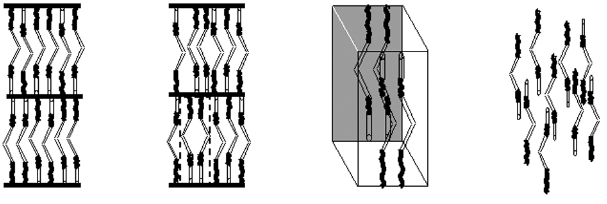
(a) (b) (c) (d)

FIGURE 7 Schematic representation of the different possible molecular packing in the (a, b and c) SmA_b phase and (d) N_b phases. In (a) the layers are polar in the plane, but apolar in the direction normal to it (polar **m** director, apolar **n** director) and thus have a C_{2v} symmetry; the successive layers are antiferroelectrically stacked. In (b) even the **m** director becomes apolar within the layer, thus having D_{2h} symmetry, indicated by the dotted rectangular box. An alternate possibility for a structure with D_{2h} symmetry is the quartet structure (diagram c) in which a pair of molecules in the plane of the paper have their **m** director antiparallel with respect to the pair below that plane. The arrangment in the N_b phase is based on ideas of the frustrated spin gas model.

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collaborators [21] is essentially designed for rod-like molecules that have a strong polar group at one end like the ones used in this study. In the SmA phase of such systems, the model considers a two-body dipolar potential with ferroelectric and antiferroelectric interactions, and then looks for the effect on a third dipole, with the dipoles oriented parallel to the layer normal. When the dipolar forces between two particles cancel, the third dipole experiences no force and is therefore free to permeate or diffuse from one smectic layer to the next one, thus "frustrating" the smectic order. Consequently, the nematic phase appears. On the other hand, if the cancellation is not complete, the presence of the triplet is supposed to stabilize the smectic order through short-range dipolar interactions.

Now let us look at how this model can be applied to the case of bent core molecules with a strongly polar group at one end, like the ones discussed in this paper. Just as in the case of rod-like molecules, here too the system would first of all like to reduce the electrostatic contribution to the free energy. Therefore the molecules are arranged so that the terminal polar groups are away from each other. Without the presence of the flexible aliphatic spacer connecting the two rigid parts of the molecules, namely, the bent-core and the rod-like parts, the permeation or the diffusion of the molecules appears to lead to situations wherein the excluded volume of the resulting structure is large. Therefore molecules in which the terminal polar group is directly attached to the bent-core may not find it favorable to exhibit a nematic phase, but rather have layered phases. By allowing the molecules to have arbitrary amounts of shift along the primary director direction, the presence of the central spacer group between the two entities helps, in simultaneously satisfying the criterion of positioning the terminal polar group as well as the bent region of the molecule such that both the electrostatic and also the excluded volume contributions are minimized. This helps in stabilizing a nematic phase and the additional ordering of the **m** director leads to the biaxiality of the phase (see Fig. 7d).

We have also prepared several other bent-rod dimers in which the molecular structures of the two mesogenic cores are identical to that for compound **I**, while the length and parity of the central spacer have been varied. In addition, schiffs base analogues have also been synthesized. Our preliminary studies [25] show that some of the bent-rod dimers exhibit interesting phase sequences, involving columnar, uniaxial smectic and biaxial nematic phases. Furthermore, the length and parity of the spacer seems to have a significant effect on the thermal behavior of these dimers, a feature reminiscent of other dimeric liquid crystals.

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In summary, we have described the occurrence of the biaxial nematic to biaxial smectic phase transition in a low-molar mass liquid crystalline system. This has been achieved by a careful design of the constituent molecules that incorporate a novel concept of covalently linking a bent-core molecule to a rod-like mesogen through a flexible spacer group. Realization of a biaxial nematic phase in a low molar system achieves significance since it opens up the possibility of fabricating new types of liquid crystal display devices. A device employing such a material is attractive as the switching of the in-plane director (**m** director) is expected to be much faster than the switching of the **n** director.

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