Manipulating the twist sense of helical nanofilaments of bent-core liquid crystals using rod-shaped, chiral mesogenic dopants

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ABSTRACT
In some liquid crystal (LC) mixtures of bent-core host molecules that form helical nanofilaments (HNFs) and chiral, rod-shaped molecular guests, the spontaneous chirality of the HNFs is not influenced by the guest handedness. In other mixtures, the filaments become homochiral, responding to the handedness of the guest. We show that the important distinction between these two behaviours is the solubility of the guest material in the HNF phase. In our experiments, chiral LC mesogens doped into the HNF phase result in an enantiomeric imbalance and sometimes change the phase sequence on cooling from the isotropic melt.

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Introduction
A major challenge in the chemical and pharmaceutical industry has been the efficient resolution of chemical enantiomers.[1,2] Enantiomerically pure compounds are essential for the synthesis and development of pharmaceutical drugs, food additives and fragrances because many biological processes in humans, and indeed all living organisms, are sensitive to molecular chirality. Scientists have developed many techniques to synthesise, separate and identify chemical enantiomers, including asymmetric chemical synthesis,[3] high-performance liquid chromatography,[4,5] high-sensitivity chirality detection,[6,7] and bio-molecular sensing.[8] Many of these techniques are based on enantiospecific adsorption on chiral surfaces, [9] which generally requires the fabrication of surfaces that are chiral on molecular length scales.

Bent-core liquid crystals (LCs) are a prime candidate for application in such technologies because of the numerous examples of chiral phases which are formed by spontaneous reflection symmetry breaking. Such phases exhibit macroscopic chiral conglomerate domains and a wide variety of novel structural phenomena involving the interplay of chiral, polar and liquid crystalline order.[10–14] These include the B7 phase,[15] the spongy, dark conglomerate phase,[16] the twist-bend nematic phase,[17–22] and the helical nanofilament (HNF) phase (also known as the B4 banana phase). [23] In particular, the HNF phase formed by achiral, bent-core mesogens has proven to be quite complex and intriguing. Driven by intralayer structural mismatch, the tilted smectic layers have a spontaneous tendency for saddle-splay deformation, which couples with the layer chirality to form left- and right-handed HNFs. As in other helical superstructures that form upon spontaneous reflection symmetry breaking, such as those exhibited by achiral amino acid derivatives,[24] rod-like fd viruses,[25] helical
supermolecules,[26] dendrons,[27] the twist-bend nematic phase [17] and B7 spiral undulation defects, [28] left- and right-handed chiral structures in the HNF phase nucleate with equal probability.

The ability to control the handedness of a bent-core LC phase would allow the convenient self-assembly of chiral superstructures for applications in which the use of domains of a single handedness is desired. In the fluid B2 phase, a ‘sergeant-and-soldiers’ effect was identified, wherein only a small amount of a compatible chiral dopant was required to give a large excess of chiral domains of a given handedness. [29] It has proven much more difficult to induce homochirality in the more ordered bent-core phases by doping or indeed by any other method. Attempts at manipulating the chirality of the HNF phase by mechanically twisting the director field [30] and using biochiral amplification [31] have met with limited success. It has been shown previously that at least in some cases the handedness of the phases formed by bent-core LCs that are synthetically chiral is uniquely determined by the chirality of the molecules themselves.[32,33]

A common technique used to obtain a chiral phase of calamitic (rod-like) thermotropic LCs is to dope an achiral host mesogen with a chiral guest material which is sufficiently miscible with the host molecules in the given phase. It has proven to be difficult, however, to use this traditional approach in LC phases of achiral bent-core molecules, with the HNF phase being particularly problematic in this regard. Previous studies have found that many commonly used LC dopant molecules are virtually insoluble in the HNF phase, [34–36] and that even chiral dopants that are effective in inducing chirality in mixtures with a variety of rod-shaped mesogens do not appreciably alter the balance of left- and right-handed nanofilament domains.[37] When NOBOW [38] (an achiral, HNF-forming bent-core molecule) is mixed with many different classes of guest molecules, including many common, rod-like, chiral and achiral LC mesogens, the components mix well in the isotropic phase. On subsequent cooling of such mixtures, however, HNFs completely phase-separate from the isotropic melt, with both left- and right-handed chiral HNF domains forming as in neat NOBOW and the guest material filling space between the nanofilaments.[36,37] On the other hand, when achiral bent-core molecules are doped with chiral bent-core guest mesogens having nearly identical molecular structure, the enhanced miscibility of the chiral dopant can influence the enantiomeric balance of left- and right-handed domains in the sample.[39,40] It has also been reported [41] that doping the rod-shaped mesogen HOBAMBC into a bent-core host affected the enantiomeric balance in a phase later identified as HNF. Here, we characterise guest/host mixtures of HOBAMBC and several of its analogues with NOBOW. In these mixtures, we obtain homochiral HNF domains with 100% enantiomeric excess, and have observed phases and phase sequences not present in any of the individual components.

**Experimental**

Textural observations were made using a Nikon Eclipse E400 POL polarising light microscope equipped with an Olympus Camedia C-5050 Zoom digital camera. Optical rotation (OR) was measured by decrossing the polariser and analyser. The LC mixtures were filled into Instec 3.2-μm commercial cells by capillary action at high temperature in the isotropic phase and the cells were then cooled into the HNF phase, typically at a rate of about −2°C/min. Temperature was regulated using an Instec STC200D temperature controller.

Freeze-fracture transmission electron microscopy (FFTEM) experiments were carried out by sandwiching the LC between 2 mm × 3 mm glass planchettes and cooling the resulting cell from the isotropic to the desired LC phase while observing the sample in the microscope. The cell was then rapidly quenched to T < −180°C by immersion in liquid propane, and fractured under high vacuum at −140°C. The exposed LC surface was subsequently coated with 2 nm of platinum deposited at 45°, followed by ~25 nm of carbon deposited at 90° to increase the mechanical rigidity of the replica. After dissolving the LC, the Pt–C replica was placed on a copper TEM grid and imaged in a Philips CM 10 100 keV TEM, allowing the topography of the fracture plane to be observed. TEM images were obtained with a 1K × 1K Gatan Bioscan digital camera. The surfaces facing the platinum shadowing direction accumulate more platinum and therefore produce darker shadows in the TEM images.

Small- and wide-angle X-ray measurements of HOBAMBC/NOBOW mixtures at different temperatures were performed using a Bruker AXS NanoStar X-ray diffractometer (CuKα radiation, λ = 1.5418 Å) equipped with a HiStar area detector and crossed Goebel mirrors. X-ray diffraction experiments of NOBOW and racemic MOBAMBC/NOBOW mixtures were performed at Brookhaven National Laboratory on beamline X10A.

Differential scanning calorimetry measurements were carried out using a Mettler Toledo DSC823e/700.

NOBOW (4-[(E)-4-(nonyloxy)phenyl]iminio]methyl)-1,3-phenylene benzoate) was synthesised following the Matsunaga method, with a slight
modification of the condensation step using N,N’-carbonyldiimidazole instead of dicyclohexyl carbodiimide in the final esterification. [38] HOBAMBC, HOBACPC and racemic MOBAMBC were synthesised according to Refs. [42], [43] and [44] respectively.

**Results and discussion**

The chemical structures and bulk phase sequences of the investigated materials are shown in Figure 1. The morphology and phase behaviour of the bent-core LC NOBOW has been reported extensively, both neat and in mixtures.[23,34–36,45–47] The HNF phase of NOBOW appears dim and blue under crossed polarisers, with distinct left- and right-handed conglomerate domains revealed by decrossing the polarisers. The phase diagram of binary mixtures of HOBAMBC with NOBOW is similar to that of NOBOW with other LC guests,[37] as shown in Figure 2. At low guest concentration, for example, in a \( c = 25\% \) HOBAMBC/NOBOW mixture (where \( c \) is the weight percent of HOBAMBC), the B2 phase appears first from the isotropic, with some fraction of the HOBAMBC and NOBOW remaining isotropic. On further cooling, HNFs appear (Supplementary Fig. 1). At higher concentrations of the guest material, for example, in a \( c = 50\% \) HOBAMBC/NOBOW mixture, freezing-point depression causes the HNF phase to appear directly from the isotropic melt (Supplementary Fig. 2). At even higher concentrations of HOBAMBC, for example, in a \( c = 75\% \) HOBAMBC/NOBOW mixture, the smectic A* phase of HOBAMBC appears first from the isotropic melt, with the HNF phase appearing at lower temperature (Supplementary Fig. 3).

The chirality of the HNF domains of NOBOW is not influenced by chiral dopants such as 7O.5* [37] that are not miscible with NOBOW in the HNF phase (Figure 3(a)–(c)). In these cases, the HNF phase appears as a conglomerate with equal volumes of left- and right-handed domains, just as with neat NOBOW.

![Figure 1](https://example.com/figure1.png)

**Figure 1.** Chemical structures and phase sequences of NOBOW, HOBAMBC, HOBACPC and racemic MOBAMBC. Iso: isotropic; N: nematic; Sm: smectic; Cr: crystal. The ‘*’ symbol indicates a chiral phase.
In mixtures of NOBOW with chiral HOBAMBC, however, the HNF domains are observed to be homo-chiral. In a \(c = 50\%\) HOBAMBC/NOBOW mixture, for example, spherulitic HNF domains nucleate randomly from different sites in the isotropic melt and gradually grow outward. Every such domain throughout the cell is seen to be left-handed (Figure 3(d)–(f)). The magnitude of the optical activity of the HNF domains formed in the mixtures is lower than that of neat NOBOW, as shown in Supplementary Fig. 4. Because the HNFs form by nucleation and growth, the guest HOBAMBC molecules must bias the handedness of the HNF spherulites at the time of nucleation. Once nanofilament growth begins, this handedness is maintained.

Although the number of HNF domains within a cell and their size depend on the cooling rate, as is usually the case in a nucleation and growth process, in the mixtures we have studied we do not observe any dependence of chiral induction on cooling rate over the range experimentally accessible to us (from about \(-0.01^\circ\mathrm{C}/\text{min}\) to \(-15^\circ\mathrm{C}/\text{min}\)).

FFTEM experiments on HOBAMBC/NOBOW mixtures confirm the presence of HNFs with only one sign of twist. The HNFs appear to be ‘unwound’ to varying degrees (Figure 3(g)) and their widths vary from \(-30\) nm up to \(-105\) nm (Figure 3(h)), in contrast to neat or completely phase-separated NOBOW, shown in Figure 3(i), where the HNF widths are confined to the range 25–30 nm.[23] Variations in the appearance of the HOBAMBC-doped HNFs (Figure 3(g) and (h)) can be explained by considering the effects on the morphology of an HNF when infiltrated with HOBAMBC molecules. The twisted filament structure of the HNF is a consequence of the tilt of the molecular arms of the NOBOW molecules in nearly orthogonal directions within a smectic layer, with Gaussian curvature of the layers relieving the elastic strain.[23] We might expect that the inclusion of HOBAMBC molecules within the HNFs will reduce some of the strain produced by this intra-layer structural mismatch, thereby unwinding the HNFs to varying degrees and stabilising HNFs with larger diameters.

We characterised the mesophases found both in the neat materials and in the mixtures using x-ray diffraction (Figure 4). We have shown previously that in mixtures with 8CB (an achiral LC mesogen), NOBOW phase-separates from the isotropic melt and forms HNFs on cooling and that the HNF layer spacing is essentially the same as that found in the neat material.[23] However, when the guest is miscible in the HNF phase of NOBOW, as is the case with HOBAMBC, HOBCPC and racemic MOBAMBC, the smectic layer spacing and the intermolecular...
distance depend on the guest concentration. In the B2 phase, the layer spacing in HOBAMBC/NOBOW mixtures is larger than in neat NOBOW, as shown in Figure 4(a). In the HNF phase, however, the layer spacing is smaller (Figure 4(b)) than in neat NOBOW, while the intermolecular distance within each layer is larger (Figure 4(c)), an effect that becomes more marked as the HOBAMBC concentration in the mixtures increases. The variation of the layer spacing and intra-layer molecular distance indicates that a significant fraction of the chiral, rod-like molecules is homogeneously mixed into the smectic layers of the NOBOW.

We also characterised mixtures of NOBOW and HOBACPC, a molecule whose core and phase sequence strongly resemble those of HOBAMBC (the main difference is in the chiral centre, see Figure 1). In the optical microscope, the behaviour appears very similar to that of the HOBAMBC/NOBOW mixtures. In a $c = 50\%$ mixture of HOBACPC and NOBOW, for example, we also obtain a homochiral HNF sample, but one which rotates light in the opposite sense to the HOBAMBC/NOBOW mixtures (Supplementary Fig. 5). Otherwise, this mixture behaves very much like the 50% HOBAMBC/NOBOW samples: the HNF phase nucleates directly from the isotropic, and the HOBACPC material not incorporated into the HNFs shows phase transitions at roughly the same temperatures as the neat material.

An unexpected and rather interesting doping phenomenon was observed in mixtures of NOBOW with another rod-shaped mesogen, racemic MOBAMBC (see Figure 1). The length of the alkyl tail of MOBAMBC is significantly shorter than those of both HOBAMBC and HOBACPC but all three mesogens share a chemically identical core. In a $c = 50\%$ MOBAMBC/NOBOW mixture, we observe upon slow cooling from the isotropic ($-0.25°C/min$), instead of the expected B2 phase, a beautiful ribbon and filament texture characteristic of the banana B7 phase (Figure 5(a)). The B7 textures appear at $T = 129°C$, far above the I–N phase transition temperature of neat MOBAMBC.
At high temperatures, the B7 domains coexist with isotropic material, which we expect consists predominantly of isotropic MOBAMBC, with the B7 domains being rich in NOBOW. X-ray scattering of the B7 phase (Figure 5(b)) shows a smectic peak which is broad relative to the resolution limit (≈0.0002 Å\(^{-1}\)) and asymmetric, suggesting that the layers are modulated.\[15\] The B7 layer peak is at smaller q than that of the B2 phase of NOBOW (Figure 5(c)) at q = 0.144 Å\(^{-1}\) in the c = 50% MOBAMBC/NOBOW mixture, compared to q = 0.160 Å\(^{-1}\) in the B2 phase of neat NOBOW). This layer swelling is strong evidence that MOBAMBC guest molecules are incorporated into the smectic layers of NOBOW. This experiment reveals that there is a delicate balance between the B2 and the B7 phases that may be controlled by doping bent-core materials with the appropriate rod-shaped mesogens.

On further cooling, the mixture transitions into the HNF phase (at T = 107°C).

Finally, our experiments on mixtures of bent-core LCs with rod-like mesogens have revealed another interesting and unexpected effect. On cooling from...
the isotropic phase, the neat chiral bent-core material MHOBOW [48] first forms the B7 phase and on further cooling transitions into the HNF phase. In contrast to the behaviour of previously reported chiral bent-core mesogens,[32,33] however, the HNF phase of neat chiral MHOBOW exhibits a conglomerate texture, with domains with opposite OR that are revealed by decrossing the polarisers. On mixing chiral MHOBOW with 8CB, we observe the usual nucleation of spherulitic domains from the isotropic phase as seen in many other HNF mixtures. In contrast to the neat material, however, these domains are all of single handedness (Supplementary Fig. 6). In this case, dilution of the HNF phase by an immiscible, achiral calamitic dopant leads to the formation of homochiral nanofilaments throughout the sample.

**Conclusion**

The unique nanostructure, stability, reproducibility and controllability of HNFs of bent-core LCs promise great potential for applications in chiral technologies and nonlinear optics. Neat, achiral bent-core LCs form conglomerates of left- and right-handed HNF domains, but by doping in chiral, rod-like guest mesogens that are miscible with the bent-core molecules in the HNF phase, we have obtained uniformly homochiral preparations of HNFs. Polarised optical microscopy, differential scanning calorimetry, FFTEM and X-ray analysis indicate that these chiral dopants infiltrate the smectic layers of the bent-core mesogens, enabling the dopant to determine the chirality of the entire sample. We conclude that chemical compatibility of the rod-like guest molecules with the arms of the bent-core host favours the
miscibility of the guest mesogens in the HNF phase and plays a significant role in influencing the phase morphology of the system.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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