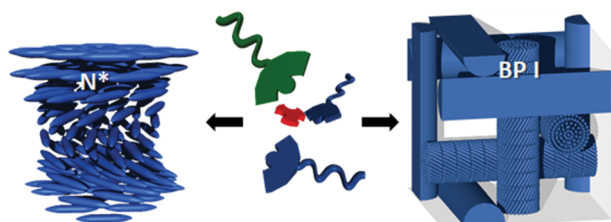c8tc06428h

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Hydrogen-bonded liquid crystals with broad-range blue phases

Marco Saccone, Michael Pfletscher, Ellen Dautzenberg, Ronald Y. Dong, Carl A. Michal and Michael Giese*

A series of hydrogen-bonded liquid crystals with broad blue phases formed by self-assembly of low molecular weight building blocks is described.

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COMMUNICATION

Hydrogen-bonded liquid crystals with broad-range blue phases†

Marco Saccone, [a] Michael Pfletscher,[a] Ellen Dautzenberg,[a] Ronald Y. Dong,[b] Carl A. Michal[bc] and Michael Giese [*a]

We report a modular supramolecular approach for the investigation of chirality induction in hydrogen-bonded liquid crystals. An exceptionally broad blue phase with a temperature range of 25 °C was found, which enabled its structural investigation by solid state ^{19}F-NMR studies and allowed us to report order parameters of the blue phase I for the first time.

Supramolecular chirality is omnipresent in nature and ensures structural integrity and the functionality of biological systems at the microscopic as well as macroscopic levels. An impressive example is the helical mesostructure of the exoskeletons of the jeweled beetle (*Chrysina gloriosa*), which generates brilliant structural colors induced by selective diffraction of light by its chiral nematic structure.[1] This structural coloration is a promising concept for materials chemists for developing new functional materials.[2] These architectures represent one-dimensional (1D) photonic band gap materials. They selectively reflect circularly polarized light of one handedness according to Bragg's law, when the helical pitch is on the order of the wavelength of visible light. Closely related to these helical structures are blue-phase liquid crystals (BP-LCs), which represent double-twisted superstructures with three-dimensional (3D) periodic cubic lattices (BPI and II).[3] BPs have attracted tremendous scientific attention within the past decades due to their exceptional optical properties. Although they are optically isotropic, they selectively reflect circularly polarized light, rendering them appealing for photonic applications.[4] In addition, they exhibit sub-millisecond response times, wide viewing angles and the Kerr-effect,[5] which makes them highly interesting for next-generation display technologies.[6] However, the major

limitation for applications of BPs is that they are only stable within a narrow temperature range (usually less than 2.0 °C) between the isotropic (I) and chiral nematic (N*) phases. Different approaches have been taken to enhance the temperature range of BPs such as the use of non-conventional nematic (N) host molecules (*e.g.* discotic[7] or bent-core[8]) or hydrogen bonding.[9] Most common are the addition of nanoparticles (NPs),[10] polymer stabilization,[11] or *in situ* polymerization of reactive mesogenic monomers to fix the 3D-structure of the BP.[12]

Although significant progress in the enhancement of the temperature range of BPs has been made, little is known about the structure–property relationships dictating the thermal stability of BP-LCs, with the notable exception of the study on polymer stabilized BP.[13] This is due to the fact that most systems, even those in the seminal report of Coles and Pivnenko,[14] consist of a complex composition of mesogens, chiral dopants and further additives (*e.g.* polymers or nanoparticles) which stabilize the BPs. The main challenge is to ensure the compatibility of all components to prevent phase separation and preserve fast phase transitions triggered by external stimuli such as electrical fields, temperature or light.

Supramolecular chemistry is an efficient tool for addressing the challenges in the design and synthesis of stable BP-LCs, since non-covalent interactions provide many advantages with respect to conventional methods such as facile fabrication, processing and recycling of functional materials. Supramolecular functional assemblies are derived from simple mixing of pre-tailored building blocks at room temperature, whereby the reversibility of the non-covalent bond allows for dynamic response to external stimuli, or damage (self-healing/-repair).[15]

Hydrogen bonds (HBs) have been the focus of intense research for the design of new functional materials.[16] A seminal work was contributed by Kato and Fréchet who investigated self-assembled liquid crystals based on the complementary interaction between benzoic acid groups and pyridyl derivatives.[17] Later Bruce *et al.* systematically investigated the LC behaviour of the HB assemblies.[18]

Since 2016, our group employs a modular approach for systematic structure–property relationship studies of hydrogen-bonded liquid crystals (HB-LCs). The obtained assemblies showed

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reversible phase transitions (N → I) upon irradiation with UV-light.[19] Moreover, the impact of fluorination in HB-LCs on the mesomorphic properties was investigated, showing that fluorination of the core unit yields broader mesophase temperature ranges due to the formation of stabilizing CF···HC bonds.[20]

Herein, we report a systematic study on the induction of chirality into HB-LCs, revealing an exceptional blue phase stabilization up to a temperature range of 25 °C. These results reported shed light into the structure–property relationships of blue phases and allow for elaborating the design criteria for improved mesophase stability – a crucial step towards the application of this exciting chiral mesophase.

In contrast to the general approach, where the chirality is induced by the addition of a chiral dopant to liquid crystalline hosts, we follow a supramolecular approach. None of the individual building blocks shows liquid crystalline properties. Upon self-assembly of the core unit with the chiral and achiral side-chains, however, a variety of chiral mesophases are observed. A set of HB-LCs derived from phloroglucinol (**PHG**) and 2-fluorophloroglucinol (**F-PHG**) as core moieties combined with alkoxyazopyridines (**Ap**) and alkoxystilbazoles (**St**) as side chains were obtained via simple self-assembly in acetone solution followed by subsequent removal of the solvent. The chirality was introduced by systematic substitution of the hydrogen bond accepting groups by a chiral derivative thereof, bearing a (*S*)-citronellyl alkyl chain (**Ap*** and **St***, see Scheme 1). It should be noticed that the 3 : 1 stoichiometry of the statistical assemblies is crucial for the stability of the mesophase.

An initial screening was performed to identify the most promising systems, and the most interesting results were found for the samples where 50% of the side chains were substituted by the chiral hydrogen-bond acceptor. The following discussion will focus on these samples.

The formation of HB adducts was confirmed by IR-spectroscopy. Typically, the broad vibration band of the OH-group of the **PHG** core ($\sim$3190 cm^{-1}) shifts to lower wavenumbers ($\sim$3050 cm^{-1}) when complexed with the pyridyl group of the side chain, with an additional signal at 2630 cm^{-1}.[19] For the assemblies based on **F-PHG**, the IR signals at $\sim$3200 cm^{-1} and $\sim$2690 cm^{-1} shifted to $\sim$3020 cm^{-1} and $\sim$2630 cm^{-1} upon formation of the HB assemblies (details see ESI†). The mesomorphic behavior of the HB-LCs was investigated by polarized optical microscopy (POM) and differential scanning calorimetry (DSC). The assemblies based on azopyridine side chains revealed exclusively monotropic phase transitions,
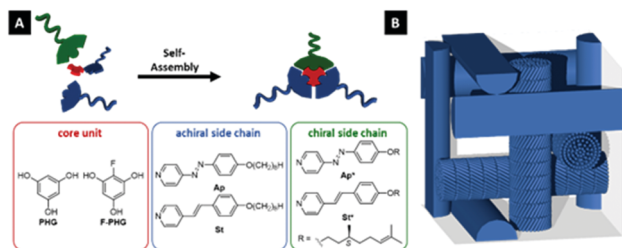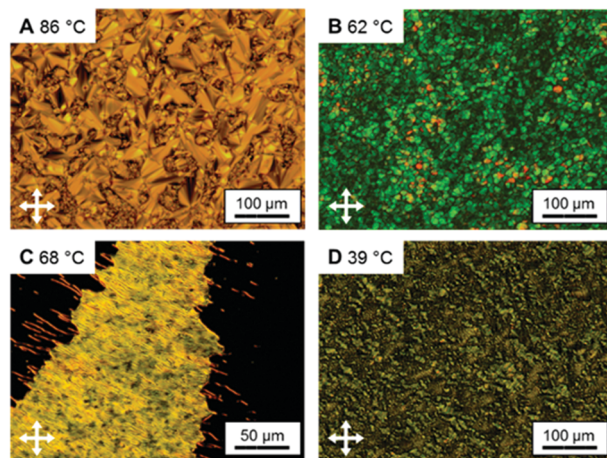
Fig. 1 POM pictures of the assemblies reported within the study (A–D). **PHG**···(**St$_{1.5}$St$_{1.5}$***): N*-phase (A). **F-PHG**···(**Ap$_{1.5}$St$_{1.5}$***): BPI (B). **PHG**···(**St$_{1.5}$Ap$_{1.5}$***): TGBA (C) and SmA*-phase (D).

while as soon as stilbazole-based side chains were employed, enantiotropic phase transitions were found.

For the stilbazole-based assemblies with **PHG** the characteristic fingerprint texture of an N*-phase was observed for all compositions (Fig. 1A) with mesophase ranges up to $\Delta T = 87$ °C (**PHG**···(**St$_{1.5}$St$_{1.5}$***), $T_{I–N*}$: 115 °C; $T_{N*–Cr}$: 28 °C). In contrast, the **PHG** assembly based on **Ap/Ap*** exhibited additionally the characteristic mosaic texture of BPI (Fig. 1B) between 75 and 67 °C. This unusually broad BP range ($\Delta T = 8$ °C) was the starting point for our systematic study on the stabilization of BPs in the HB-LCs. In a previous study we found that substitution of **PHG** by **F-PHG** leads to a stabilization of the mesophases of the HB-LCs, which we attributed to non-classical hydrogen bonds between **PHG** and **Ap** chains (C–F···H–C).[20] Therefore, the corresponding **F-PHG** aggregates were prepared and investigated with respect to their thermal behaviour. Again the stilbazole system showed a broad N*-phase ranging from 119 to 38 °C, while the azopyridine assemblies exhibited the BPI between 76–70 °C. As BPs only occur in systems with high helical twisting powers (HTP), these results indicate that the chiral transfer in the **Ap** systems seems to be more efficient than in the **St**-based assemblies.

Since generally the **St**-based assemblies[18a,21] showed superior mesophase ranges compared to the **Ap** systems, a series of mixed assemblies was prepared in order to improve the BP range by synergistic effects of the two side chains. Therefore, the chiral **Ap*** was combined with the achiral **St** as well as the achiral **Ap** with the chiral **St*** for both core moieties (**PHG** and **F-PHG**), yielding four new HB-LC compositions: **PHG**···(**St$_{1.5}$Ap$_{1.5}$***), **PHG**···(**Ap$_{1.5}$St$_{1.5}$***) as well as **F-PHG**···(**St$_{1.5}$Ap$_{1.5}$***), **F-PHG**···(**Ap$_{1.5}$St$_{1.5}$***). To our surprise, the mesomorphic behaviour of these materials differed largely from the previously described systems, which again proves the benefit of a modular approach.

The **PHG**···(**St$_{1.5}$Ap$_{1.5}$***) showed a complex thermal behaviour upon cooling with three different chiral mesophases – N* ($T_{I→N*}$: 98 °C), twist grain boundary (TGBA, $T_{N*→TGBA}$: 79 °C, Fig. 1C) and a chiral smectic A phase ($T_{TGBA→SmA*}$: 61 °C,

Scheme 1 (A) Modular approach towards the investigation of the chiral induction in HB-LCs. (B) Schematic representation of the BPI-structure.

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$T_{\mathrm{SmA^*\to Cr}}$: < 30 °C, Fig. 1D). Particularly, the chiral **Ap*** did not introduce a BP phase in this assembly. The inversed composition **PHG**···(**Ap**$_{1.5}$**St**$_{1.5}$*), however, exhibited a BP ($T_{\mathrm{I\to BP}}$: 79 °C) with a temperature range of ΔT = 10 °C and a N*-phase ($T_{\mathrm{BPI\to N^*}}$: 69 °C) with a range of ΔT = ~45 °C.

Changing the core unit from **PHG** to **F-PHG** yielded **F-PHG**···(**St**$_{1.5}$**Ap**$_{1.5}$*) and **F-PHG**···(**Ap**$_{1.5}$**St**$_{1.5}$*) assemblies. The **F-PHG**···(**St**$_{1.5}$**Ap**$_{1.5}$*) revealed exclusively a N* ($T_{\mathrm{I\to N^*}}$: 108 °C before crystallizing at 44 °C). The most interesting finding within this series was made for the **F-PHG**···(**Ap**$_{1.5}$**St**$_{1.5}$*) showing a broad BP I (Fig. 1B) ranging from 75 to 50 °C followed by a chiral nematic phase ($T_{\mathrm{N^*\to Cr}}$: ~40 °C). To the best of our knowledge, this is the broadest BP range reported for a low-molecular-weight liquid crystal system. These results indicate that the chiral induction of the stilbazole is enhanced in an achiral azopyridine leading to the formation of the double-twisted BP structure, and we attribute the increased HTP to stronger $\pi-\pi$ interactions between the stilbazole and azopyridine in comparison to two stilbazoles (Fig. 2).

Hence the wide BP range is attributed to the high flexibility of HB-LCs, allowing the formation of a balanced structure to yield an efficient packing of the mesogens into a helical arrangement. In addition, the fluorine atom on the core unit may contribute to the stabilization of the BP through weak non-classical hydrogen-bonding (C−F···H−C) between the core moiety and the side chain. Furthermore, the strong electronegativity of the fluorine reduces the electron density of the aromatic core, which supports the efficient packing of the assemblies in the BP.

In order to get insight into the BP and N*-phases of **F-PHG**···(**Ap**$_{1.5}$**St**$_{1.5}$*), solid state ^{19}F-NMR study was performed on the sample as a function of temperature in an external magnetic field of 9.4 T. The first ^{19}F-asuppond ^{1}H-NMR studies on BPs were reported by Collings et al.[22] Later Meiboom and Sammon suggested that BPs have either a simple cubic or a body centered cubic (bcc) structure,[23] which was finally proven by Samulski and Luz[24] via deuterium NMR measurements.

More recently, a proton NMR relaxation study has also been reported to shed light on the dynamic processes in a BP phase.[25] In particular, rotation-mediated translation along the pitch axes became dominant below 50 kHz, whereas at higher Larmor frequencies the usual collective/individual motions were important.

The ^{19}F-NMR spectrum of **F-PHG**···(**Ap**$_{1.5}$**St**$_{1.5}$*) shows a single broad peak in the BPI. The peak shifts continuously upfield as temperature decreases, and the line-width broadens gradually in the BPI phase down to the N*-phase (Fig. S17, ESI†). By measuring the chemical shift anisotropy δ_{aniso} (= $\delta_{\mathrm{obs}} - \delta_{\mathrm{I}}$) with respect to the isotropic chemical shift δ_{I} = −9.12 ppm in the BPI and N*-phases, the order parameter of the molecular symmetry axis z_{m}, S_{zz}, can be determined in both mesophases. It is interesting to compare this chiral HB-LC with the discotic **F-PHG**···(**Ap**)$_3$ reported earlier[20] in which the z_{m} axis is normal to the **PHG** core plane and its ^{19}F signal shifts down-field as temperature decreases into the N-phase. This is an indication that the present chiral HB-LC has a rod-like shape with the molecular z_{m} axis lying in the core ring plane. For the **F-PHG** core, $S_{xx} - S_{yy} = 0$ is used. In the N*-phase, the directors form a planar distribution over a pitch length and the shift tensor is motionally averaged by fast rotations about the z_{m} axis. Here the z_{m} axis is normal to the pitch axis giving $\delta_{\mathrm{aniso}} = -(1/2)\delta P_2(\cos 90°)S_{zz}$, where the shift anisotropy $\delta = \sigma_{33} - \mathrm{Tr}\,\sigma$ = −78.67 ppm is from the reported fluorine chemical shielding tensor (CST) elements σ_{ii} of **F-PHG**···(**Ap-8**)$_3$ in the solid state.[20] Note that the factor −1/2 in δ_{aniso} arises from the helical axis being normal to the B-field (i.e. the angle θ = 90° between the B-field and the pitch axis) in the N*-phase. In the BP, the multi-domain cubic structures mean that the factor of −1/2 has to be replaced by −3/2 since the angle θ is now isotropically distributed.[26] Thus a reduction in the molecular order in the BPI by a factor of 3 occurs, leading to a large jump in S_{zz} at the BP-N*-phase transition (Fig. 3). The conventional fitting equation for the order parameter $S \equiv S_{zz}$ is the Haller power law:[27]

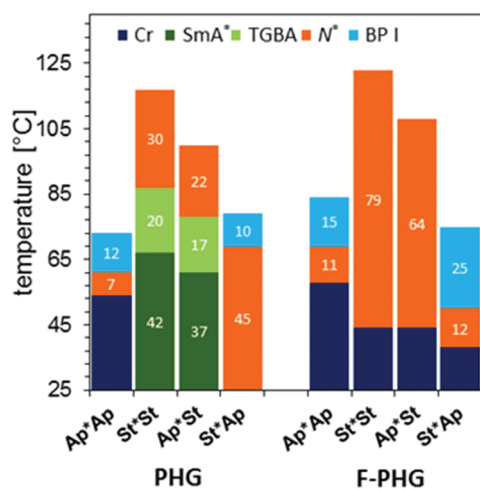$$S_{zz} = A\left(1 - \frac{T}{T_{\mathrm{c}}}\right)^{\beta}$$

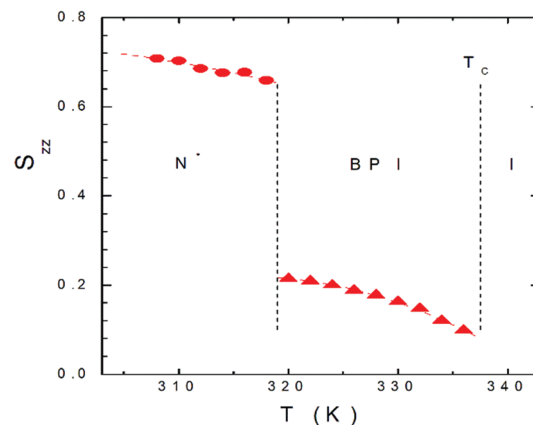Fig. 2 Overview of the transition temperatures of the HB-LCs as observed by POM upon cooling.

Fig. 3 Plot of the order parameter versus the temperature in **F-PHG**(**St***$_{1.5}$**Ap**$_{1.5}$) (N*: ●, BPI: ▲).

where the fit in each mesophase is found with $T_c = 337.5$ K. Fig. 3 shows the experimental order parameter as a function of T and the fits give pseudo-exponents β of 0.333 and 0.168 in the BPI and N*-phases, respectively. The corresponding A values are 0.075 and 0.027. The β value in the N*-phase is similar to those seen in calamitic (*viz.* between 0.1 and 0.2)[27] and bent-core nematogens (*ca.* 0.16).[28] The S values in the N*-phase are similar to those reported earlier in the N-phase of **F-PHG**⋯(**Ap-8**)$_3$.[20] It is also noted that the order parameter S in the BPII was reported earlier to be 0.06–0.08. The corresponding values could not be obtained in the BPI by their method owing to its smaller mono domains in comparison to those in the higher temperature BPII phase.[29]

Conclusions

We reported a systematic investigation on the induction of chiral mesophases in HB-LCs and found exceptionally broad BPI enabled by the modular supramolecular approach that allowed us to gain a deeper insight into the structure–property relationships of supramolecular chiral LCs. The broad temperature range of the BPI enabled the investigation of the orientational order by solid-state ^{19}F-NMR spectroscopy. A β (0.33) value has been determined for the first time in a BP phase, which is considerably larger than its value (0.17) in the N*-phase, which in line with the differences of their phase symmetries. Currently, we are working on the improvement of the stability of blue phases towards the application of these assemblies as tuneable 3D photonic reflectors and sensors.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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