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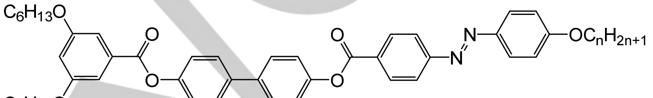
Isothermal Chirality Switching with Non-Polarized Light

Mohamed Alaasar,*[a,b] Silvio Poppe,[a] Qingshu Dong,[c] Feng Liu,*[c] and Carsten Tschierske*[a]

Abstract: Spontaneous mirror symmetry breaking is a fundamental process for development of chirality in natural and in artificial self-assembled systems. Here we report a new series of triple chain azobenzene based rod-like compounds, which for the first time show mirror symmetry breaking in an isotropic liquid occurring adjacent to a lamellar LC phase. The transition between the lamellar phase and the symmetry broken liquid is affected by *trans-cis* photoisomerization which allows a fast and reversible photoinduced switching between chiral and achiral states with non-polarized light.

Chirality of molecular systems has developed into a major topic in chemistry since its discovery by L. Pasteur.[1] It is considered as a prerequisite for emergence of life[2] and has numerous technological implications in pharmaceutical and agricultural chemistry[3] as well as for applications in nano-technology and advanced materials.[4] In condensed matter phases chirality can be based on permanent (configurational) or transient (conformational) molecular chirality and on chiral superstructures, arising from the (in most cases helical) organization of molecules (superstructural chirality).[5] Circular polarized light represents a chiral environment[6] and therefore has often been used, either to induce chirality, or to modify chirality by diastereomeric interaction with existing molecular and superstructural chirality.[7,8] In contrast, non-polarized light is achiral (racemic) and therefore cannot induce chirality in the absence of a properly oriented magnetic field or another source of chirality.[2b,6] Accordingly, chirality switching by non-polarized light requires *chiral molecules*, preferably organized in liquid crystalline (LC) phases combining the molecular order required for helix formation with the mobility required for switching. Photoisomerization changes the molecular shape and this modifies the self-assembly. Examples are light induced SmC*-SmA and SmA-N* transitions (see Scheme S2 in the SI) of azobenzene derived rod-like LCs. In the first case, photoisomerization removes the molecular tilt in the SmC* structure which is the basis of helix formation perpendicular to the layers.[9] In the latter case photoisomerization removes the layer structure (SmA), being incompatible with a helical twist between the molecules. Only in the photo-induced nematic phase without these layers the molecular chirality can couple with the nematic director field and a helical superstructure evolves (N* phase).[10] Recently, it was shown that for achiral bent mesogenic dimers with azobenzene units a helical superstructure is spontaneously formed in the twist-bend nematic phase (N_{TB}, see Scheme S2). This spontaneously chiral superstructure is removed by *trans-cis* photonisomerization with non-polarized light, leading to a transition to a nematic phase which, due to the absence of molecular chirality, is achiral (N).[11]



An (*n* = 4-8)

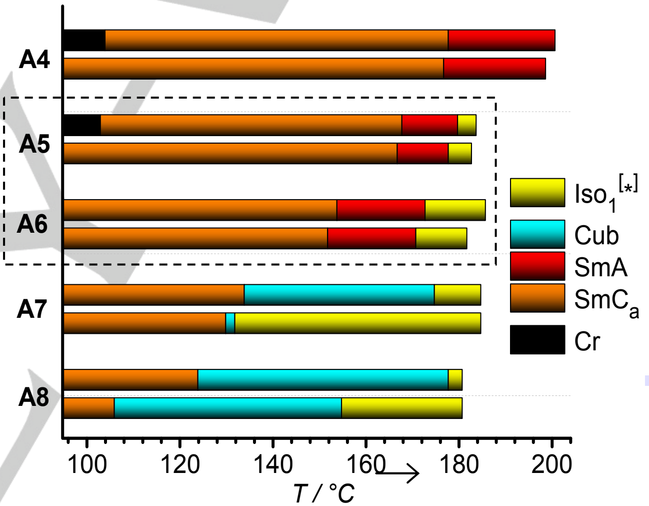


Figure 1. Phase transitions of compounds **A*n*** as determined from the 2nd heating (upper columns) and cooling DSC scans (lower columns) with rate 10 K min^{-1}; abbreviations: SmA = non-tilted lamellar phase; SmC$_a$ = anticlinic tilted lamellar phase; Cub = bicontinuous cubic phase with *Ia*$\bar{3}$*d* lattice; Cr = solid crystal; Iso$_1^{[*]}$ = chiral isotropic conglomerate liquid mesophase;[17] the superscript [*] indicates the presence of a chiral superstructure though the involved molecules are achiral; for numerical data and transition enthalpies see Table S1.

Herein we report the first example for the opposite process of *induction* of superstructural chirality by photoisomerization of *achiral* compounds with *non-polarized* light. In this case mirror-symmetry breaking takes place at the transition from a higher to a lower order phase which is contrary to common knowledge, where symmetry breaking occurs at the transition from lower to higher order (e.g. crystallization from solutions or melts). This discovery is based on our previous work on spontaneous mirror symmetry breaking in isotropic liquids (Iso$_1^{[*]}$ phases) formed by achiral multi-chained rod-like (polycatenar[12]) molecules.[13-16] The spontaneous mirror symmetry breaking in these liquids was proposed to result from the combination of a locally twisted cybotactic cluster structure with the chirality synchronization of the involved transiently chiral molecules.[15] The local twist is thought to be due to the local aggregation and parallel organization of the rods and the

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simultaneous clashing of the bulky terminal alkyl chains. These Iso$_1^{[*]}$ phases typically occur besides cubic or non-cubic mesophases with long range 3D lattice.[16,17] It was also shown that polycatenar compounds involving azobenzene units photo-isomerize in solution, though it was not possible to observe a measurable effect on the bulk state.[18] We attributed this to a kinetic hindrance of photoisomerization by the long range 3D lattice. In order to replace these 3D phases by fluid lamellar LC phases, we have designed a new class of polycatenar compounds involving a photoisomerizable azobenzene group[19] and having only three instead of the previously used four terminal alkyl chains (tricatenar compounds ***An***, see Figure 1). These compounds have been synthesized (Scheme S1), purified and analyzed as described in the Supporting Information.

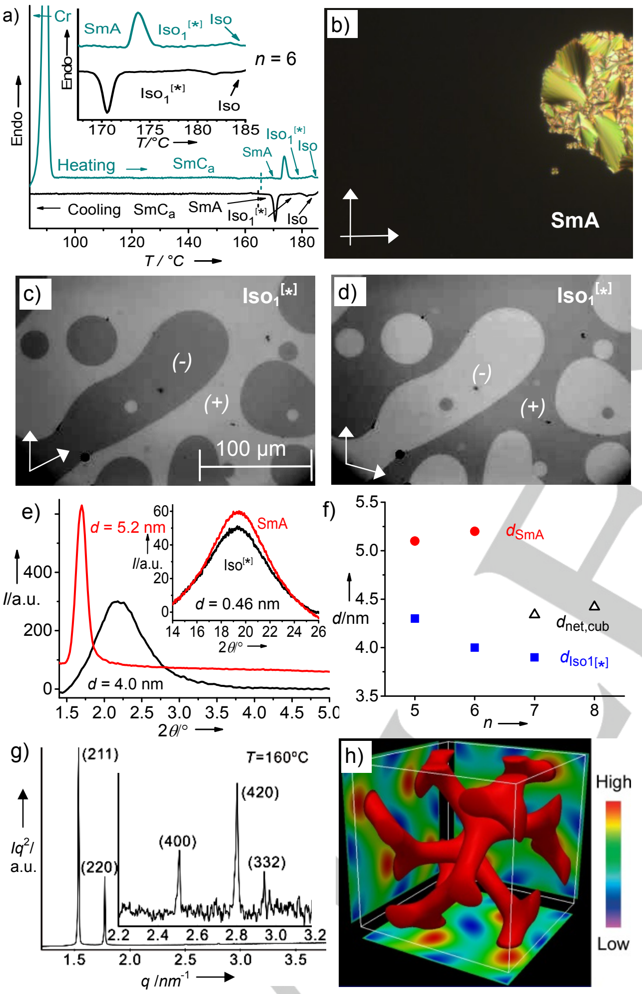

Figure 2. Characterization of the mesophases of compounds ***An***: a) heating and cooling DSC curves (10 K min^{-1}) recorded for **A6**. b) Texture of the SmA phase (T = 170 °C) of **A5** between crossed polarizers with coexisting homeotropic (dark area) and planar alignment (birefringent area). c, d) Chiral domains observed for the Iso$_1^{[*]}$ phase of compound **A6** in a thin film between two glass plates (~20 μm) at T = 181 °C, c) after rotating one polarizer by 12° from the crossed position in anticlockwise and d) in clockwise direction (see also Figure S12). e) SAXS pattern of the SmA and Iso$_1^{[*]}$ phase of **A6** at T = 180 and 160 °C, respectively; the inset shows the corresponding WAXS patterns. f) Development of the d-spacings $d_{\mathrm{Iso1[*]}}$ (2K below Iso-Iso$_1^{[*]}$ transition) and d_{SmA} depending on chain length (n); $d_{\mathrm{net,cub}} = (3½a_{\mathrm{cub}})/4$ is the distance between the two networks in the Cub/$Ia\bar{3}d$ phases. g) SAXS pattern of the Cub/$Ia\bar{3}d$ phase of **A7** (T = 160 °C) and h) reconstructed electron density map of the Cub/ $Ia\bar{3}d$ phase obtained from g), see Section 3.4.1 in the SI for more details.

The transition temperatures of compounds ***An*** are shown graphically in Figure 1 (for numerical data, see Table S1). All ***An*** compounds with n = 5-8 show spontaneous chiral conglomerate formation in a certain temperature range of their isotropic liquid phases (Iso$_1^{[*]}$). The formation of these Iso$_1^{[*]}$ phases from the achiral isotropic liquid state (Iso) is indicated under the polarizing microscope by the occurrence of chiral domains, identified upon slight rotation of the analyzer by a small angle out of the 90° position, leading to dark and bright domains which invert their brightness after rotating the analyzer by the same angle into the opposite direction (Figures 2c,d and S12). The domains have circular shapes and flow under gravity as typical for isotropic liquids.[20] The Iso$_1^{[*]}$ phases are accompanied by different types of LC phases, depending on the chain length n. As shown in Figure 1, a cubic phase with $Ia\bar{3}d$ lattice is found for compounds with chain length n = 7 and 8 (**A7**: a_{cub} = 10.0 nm, see Figure 2g, h and Table S2; **A8**: 10.2 nm, see Table S3; for more details of structure elucidation, see Section 3.4.1 of the SI). The cubic phases are achiral, in line with the racemic $Ia\bar{3}d$ structure being composed of two enantiomorphic networks of branched helical columns (Figures 2h and S16).[16] With decreasing chain length the optical isotropic cubic phase is replaced by less viscous and birefringent LC phases. Polarizing optical microscopy of the LC phases of compounds **A4-A6** indicates uniaxial SmA phases characterized by typical fan textures in planar alignment (layers perpendicular to the substrate) and dark isotropic textures in homeotropic alignment (layers parallel to the substrate, see Figure 2b). Small angle X-ray scattering (SAXS) confirms the lamellar organization and provides a layer thickness of d_{SmA}= 5.2 nm for the SmA phase of **A6**, as an example (Figure 2e, see also Tables S4-S6). This value is slightly larger than the molecular length (L_{mol} = 4.4 nm), indicating an antiparallel organization of the molecules with intercalation of the single alkyl chains between the biphenyl units of adjacent molecules (Figure 4a,c). In all cases on cooling the SmA phase an anticlinic tilted lamellar phase (SmC$_a$ phase) is observed (Figure 1). This is for some compounds followed by additional tilted and non-tilted smectic, hexatic and crystalline low temperature mesophases which will be discussed in a forthcoming report.

Though the Iso-Iso$_1^{[*]}$ transition is easily detected by POM, the associated enthalpies are very small and the corresponding DSC peaks are broad, more typical for a C_p-anomaly (see inset in Figure 2a).[14,17,21] Also the XRD patterns in the Iso$_1^{[*]}$ and Iso ranges are very similar. As shown in Figure 2e for the Iso$_1^{[*]}$ phase of **A6**, there are only two diffuse scatterings, one in the small and the other in the wide angle region of the XRD pattern (at d = 0.46 nm), in line with the presence of only short range order (black line, Figure 2e with inset). The intensity of the small angle scattering is significantly larger than the wide angle scattering, but it is also significantly broader compared to the smectic phases (red line, Fig 2e), as typical for liquids with a locally ordered structure (cybotaxis).[14,17,22] As previously shown, the change of the position and line width of the XRD small angle scattering at the Iso-Iso$_1^{[*]}$ transitions is continuous without any distinct jump.[14] This indicates that the Iso and Iso$_1^{[*]}$ phases are formed by small clusters which grow continuously on cooling and reach the critical size for chirality synchronization at the Iso-Iso$_1^{[*]}$ transition temperature.

Compound **A6** was selected for photoisomerization experiments in solution and in the distinct LC phases. Investigation in solution indicated almost complete *trans-cis* isomerization under UV light and back relaxation to the *trans*-isomer by thermal relaxation as shown in Figure 3a. Irradiation of the bulk sample with non-polarized 405 nm laser light (5 mW) lowered the SmA-Iso$_1^{[*]}$ transition temperature of **A6** by 10 K. An equilibrium state is reached after a couple of seconds (<2 s) and relaxes back to the initial transition temperatures (*cis*-to-*trans*) immediately after switching off the light source (<2 s). This allows an isothermal induction of chirality and a relatively fast and reversible on-off switching of chirality by non-polarized light, as shown in Figure 3b-d (see also the supporting video 1). The effect of irradiation on the Iso-Iso$_1^{[*]}$ transition temperature is much smaller, leading to a reduction by only 2 K, thus overall expanding the enantiotropic Iso$_1^{[*]}$ range of **A6** from 11 to ~20 K.[23]

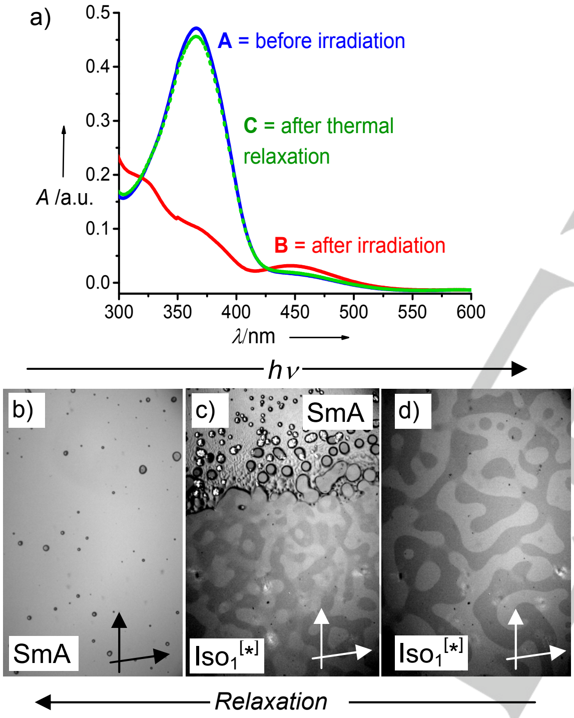

Figure 3. a) UV-vis spectra of **A6** dissolved in chloroform at 25 °C (c = 20 mol·L^{-1}). A: freshly prepared solution before irradiation; B: *cis*-isomer as obtained after 1 h irradiation with λ = 365 nm; C: after keeping the sample in dark overnight. b-d) Isothermal photo induced SmA-to-Iso$_1^{[*]}$ transition observed for compound **A6** in a thin film between two glass plates under slightly uncrossed polarizers (arrows) at T = 168 °C: b) SmA phase before illumination with non-polarized 405 nm laser light (5 mW); c) during SmA-to-Iso$_1^{[*]}$ transition upon starting of illumination and d) Iso$_1^{[*]}$ phase as observed during illumination. The reverse sequence is obtained immediately after switching off the light source (see also the supporting videos).

The sign of chirality in the chiral domains of the Iso$_1^{[*]}$ phase is retained during repeated switching cycles Iso$_1^{[*]}$-SmA-Iso$_1^{[*]}$ or Iso$_1^{[*]}$-Iso-Iso$_1^{[*]}$ (see supporting video 2), though the SmA and Iso phases, as well as the involved molecules, are achiral. It is therefore likely that the chiral information is stored at the surfaces of the glass substrates via surface stabilization of residual helical clusters or chiral surface defects, which then trigger the chirality sense in the developing bulk Iso$_1^{[*]}$ phase. In contrast, the Cub-Iso$_1^{[*]}$ transitions of compounds **A7** and **A8** are not affected by irradiation, as previously also observed for the tetracatenars.[18] Probably the long range 3D connected networks of columns in the Cub phase act as templates which kinetically hinder the isomerization.[24]

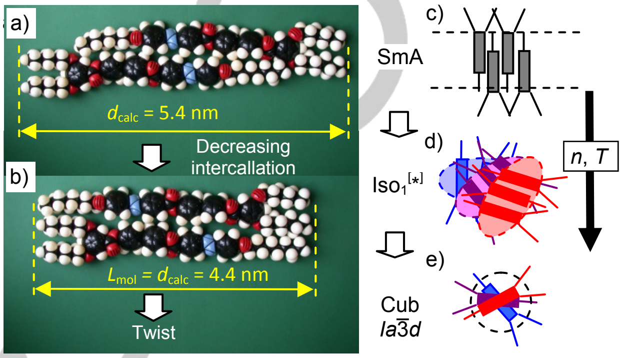

Figure 4. Molecular organization in the LC phases and in the cybotactic clusters of the Iso/Iso$_1^{[*]}$ phases: a, b) Molecular models of **A6** showing the molecular dimensions and the changing intercalation at the transition from a) the intercalated packing in the lamellar to b) the segregated packing in the Iso$_1^{[*]}$ and Cub/$Ia\bar{3}d$ phases (twist not shown). c-e) Schematic models showing the cross sections of c) the layers (SmA); d) the local organization in the twisted ribbons of the Iso$_1^{[*]}$ phases (from front to back the molecules are shown red, purple, blue) and e) the helical columns forming the networks of the Cub/$Ia\bar{3}d$ phase.

In order to understand the development of the Iso$_1^{[*]}$ phase in relation to the molecular structure and the self-assembly in the adjacent LC phases, the phase structures depending on chain length and temperature have been investigated in more detail. The maximum of the small angle X-ray scattering in the Iso$_1^{[*]}$ phases is $d_{\mathrm{iso1[*]}}$ = 3.9-4.3 nm, being significantly smaller than the layer spacing d_{SmA} in the adjacent SmA phase (d_{SmA} = 5.1-5.2 nm, Figure 2f) and only a bit smaller than the molecular length (L_{mol} = 4.4 nm for **A6**, Figure 4a,b). This suggests a non-intercalated antiparallel packing of the molecules with completely segregated alkyl chains and aromatic cores in the Iso$_1^{[*]}$ phase (Figure 4b). This non-intercalated packing is distinct from the intercalated organization in the adjacent SmA phase (Figure 4a) and provides an unbalanced ratio of two aromatic cores vs. three alkyl chains, leading to layer frustration which is released by development of curvature and twist. With growing alkyl chain length the twist of the aggregates increases, thus favoring the chirality synchronization of helical conformers. However, on cooling, the connectivity of the clusters increases, too, until a highly connected network is formed at the Iso$_1^{[*]}$-Cub/$Ia\bar{3}d$ phase transition (n = 7, 8) leading to the achiral cubic phase (Figure 2h). Accordingly, the distance between the nets forming the Cub/$Ia\bar{3}d$ phase (Figure 2h), $d_{\mathrm{net,cub}}$ = 4.4-4.5 nm is close to $d_{\mathrm{iso1[*]}}$ (Figure 2f), confirming that the segregated organization (Figure 4b) is retained in the Cub/ $Ia\bar{3}d$ phase. Figure 2f also shows that d_{Iso} decreases with growing n, from n =

5 to 7, whereas d_{SmA} grows, indicating that the local organization in the $\mathrm{Iso}_1^{[*]}$ phases becomes more intercalated, and hence, less sterically frustrated with decreasing chain length (Figure 4b→a), in this way favoring the lamellar organization. Deeper chain-core intercalation at decreased temperature (larger contribution of linear *trans*-conformers makes the chains more compatible with the rod-like cores) reduces the curvature further, and the local clusters can fuse to infinite layers with intercalated structure (SmA, *n* = 5, 6; Figure 4a,c). However, in the chiral isotropic liquids of compounds **A5** and **A6** a twisted organization is still retained in the aggregates and the local twist is obviously still sufficient for chirality synchronization to take place, thus leading to the chiral $\mathrm{Iso}_1^{[*]}$ phase. Only for the shortest homologue **A4** sufficient twist cannot develop and the SmA phase is directly formed from the achiral Iso phase. This provides an understanding of the formation of symmetry-broken $\mathrm{Iso}_1^{[*]}$ phases for compounds **A5** and **A6** occurring besides an SmA phase, just before the cross-over from lamellar to bicontinuous cubic organization in the adjacent LC phases.

In summary, isothermal switching from an achiral LC phase to a mirror symmetry broken isotropic liquid with non-polarized light was reported for achiral compounds, providing significant potential for technological and nano-technological applications.

Experimental Section

Compounds **A*n*** were synthesized according to the procedures given in the accompanying Supplementary Information, where also the used experimental methods are described.

Acknowledgements

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Keywords: Chirality, Photoisomerization, Azobenzene, Mirror-symmetry breaking, Liquid crystal

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[23] The SmA-Iso transition of **A4** is reduced by -4 K during irradiation, but no $\mathrm{Iso}_1^{[*]}$ phase is induced; the $\mathrm{SmC_a}$-SmA transition of **A5** is shifted by -3 K.

[24] Irradiation of **A7** and **A8** in the tilted $\mathrm{SmC_a}$ range can induce the cubic phase, i.e. isomerization to the bent *cis* isomer increases the interfacial curvature and the $\mathrm{SmC_a}$-Cub transition temperature is shifted by 7 K to lower temperature, expanding the cubic phase range by photoisomerization, similar as reported for azobenzene doped $\mathrm{SmC_s}$ phases.[26]

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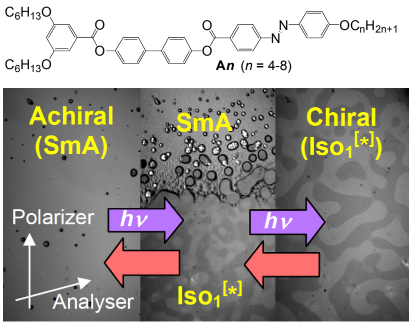
COMMUNICATION

Chirality switching in liquids

Achiral three chained azobezene compounds **A*n*** with proper chain length *n* show a fast isothermal switching between an achiral lamellar phase and a symmetry broken chiral liquid conglomerate by photoisomerization with non-polarized light.



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