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Photo- and thermal switching of blue phase film reflecting both right- and left-circularly polarized light

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Abstract:

A new kind of blue phase (BP) liquid crystals (LCs) films has been developed which could reflect both right- and left-circularly polarized light. Here a free-standing, three dimensional (3D) BP polymer template with around $2/3$ the cell thickness was firstly fabricated by nonuniform photopolymerization of a LC mixture at BP I temperature induced by ultraviolet light absorbing dye, in which the non-reactive components were removed to achieve porous polymer template with right-handed (RH) structure, retaining 3D structure of BP I. By refilling a photoresponsive and left-handed (LH) helical structure BP-LCs containing a chiral azobenzene switch into the polymer template, we finally obtain a BP-LC film with a segregation of RH and LH domains across the LC film, thereby reflecting both right- and left-circularly polarized light. Photo- and thermally switching characteristics of this BP film were detailedly investigated. These special optical properties make this kind of BP-LCs film an interest for great potential application in many fields such as flexible LCs display, optical components, LCs lasing, etc.

Introduction

Blue phases (BPs) of liquid crystals (LCs) are of considerable interests due to their unique self-assembled cubic structure and fascinating electro-optical characteristics.¹⁻⁵ As is well known that, thermodynamically stable blue phases, BP III, BP II and BP I, could be observed on cooling from isotropic phase to cholesteric (Ch) phase. BP III is amorphous with a local cubic lattice structure in the director field, whereas BP II and BP I have a fluid three-dimensional periodic structure in the director field with simple cubic and body-centred cubic symmetry, respectively.⁶⁻⁹ For BP I and BP II the lattice periods are of the order of the wavelength of visible light and give rise to Bragg reflections of circularly polarized light. These lead to potentially interesting photonic applications, such as reflective liquid crystal display, optical sensor and three-dimensional blue-phase lasers.^{4,5} Meanwhile, BP-LCs is currently considered the most promising materials for the next-generation LC display technology due to a fast electro-optic effect represented by an optical switching between isotropic and anisotropic states.^{1,5} However, narrow stable temperature range of BPs practical application has limited their utility to applications due to the existence of molecular misalignment spaces (disclination), and more efforts have been, therefore, paid to overcome this challenge, such as polymer stabilization, nanoparticles and special molecular design.¹⁰⁻¹⁹

Additionally, tuning of Bragg reflection of BPs by applying external stimuli, such as electrical field, temperature and optical field, is gaining increasing attention.²⁰⁻²⁶ As mentioned above, BP I and BP II exhibit self-assembled cubic structures, the reflection wavelength is given by:²²

$$\lambda = \frac{2na}{\sqrt{h^2 + k^2 + l^2}}$$

Where n and a are average refractive index and lattice constant of BPs, respectively, and h , k , and l are the Miller indices of a crystal plane. The cubic lattices of BPs can be changed by external fields because BPs has delicate and fragile structures, which leads to a shift of the reflection band. Based on the lattice change, optically and electrically tunable reflection colors of BP LCs have been demonstrated.²⁰⁻²⁶ Among them, dynamic optically tunable BPs behavior can be efficiently achieved by *trans-cis* photoisomerization of azobenzene dopant.²³⁻²⁶ Chanishvili *et al.* were the first to add an achiral azobenzene to a BP LCs, in which the shift of reflection wavelength and phase transition behavior were observed during light irradiation.²³ Q. Li *et al.* demonstrated a wide optical switching of the reflection colors accompanied with phase transition from BP II to BP I in BP-LCs doped with a chiral azobenzene switch.²⁴ Recently, we reported a stable BP I without any phase transitions can be reversible tuned by UV/Vis light irradiation over the visible region across red, green, and blue wavelength by employing a kind of hydrogen-bonded chiral azobenzene switches.²⁶ However, similar with Ch-LCs,²⁷ the Bragg reflection of incident unpolarized light of the BP-LCs is circularly polarized, matching the handedness of double twisted helical structure. Therefore, BP-LCs that reflects both right- and left- circularly polarized light simultaneously in a single LC film will gain more interesting based on the following two points. First, due to the nature of self-organization three-dimensional (3D) and isotropic structure, there is not any complicated fabrication process compared with the other photonic crystals fabricated by costly manufacturing processes, and no need of the orientation layer compared to Ch-LCs. Second, high reflectivity, high contrast and bicolor/multicolor reflections enable this kind

of BP material to be used in many potential fields, especially such as 3D tunable and multimode lasing, anti-counterfeiting optical material.

In this study, we develop a new kind of BPs film by using three-dimensional (3D) templated BP polymer nanostructure, in which the BP film could reflect both right- and left-circularly polarized light. Here we firstly fabricate a BP polymer template with right handed (RH) structure tethered to the upper surface of the cell and across around 2/3 the cell thickness by employing a nonuniform photopolymerization induced by ultraviolet light absorbing dye (ULAD). Furthermore, with the assistance of wash-out/refill method as demonstrated in our previous study,²⁸ a BP film reflecting both right- and left-circularly polarized light could be achieved due to a spatial segregation of the LH and RH domains occurs across the cell (in the z-direction). Furthermore, photo- and thermally switching of Bragg reflection of this BP film was demonstrated in detail. The optical tunable performance of this kind of BP film makes them interesting for a wide variety of applications ranging from optically LC display to tunable LC lasing.

Experimental

Materials

In this study, non-reactive nematic LCs, SLC1717 (589 nm, $\Delta n=0.201$) and 8CB (Slichem Liquid Crystal Material Co., Ltd.); S811 (chiral dopant, Beijing Lyra Tech); Diacrylate photo-polymerizable LCs monomer, C6M, LC 242, LC1057; Chiral LCs monomer (R-2-methylbutyl-4-hydroxybiphenyl-4'-[6-(acryloyloxy)hexyloxy] benzoic carboxylate, CMF-LCM); Ultraviolet absorbing dye, (ULAD, 2-{2,6-bis-[2-(4-dimethylamino-phenyl)-vinyl]-pyran-4-ylidene-malononitrile), 2,5-bis-[4'-(hexyloxy)-phenyl-4-carbonyl]-1,4,3,6-dianhydride-D-sorbitol

(ISO(C6OBA)₂) and photoinitiator, 2, 2-dimethoxy-2-phenyl-acetophenone (Irgacure 651, TCI Co. Ltd.) were used. C6M was synthesized according to the method suggested by D. J. Broer et al.²⁹ LC242 and LC1057, were purchased from BASF Co. Ltd. CMF-LCM has a similar preparation process with MF-LCM in our study,¹⁵ here we used chiral R-2-methylbutanol to replace achiral 2-methylbutanol. ULAD was lab-synthesized according to the earlier paper.³⁰ Figure 1 shows the chemical structures of 8CB, C6M, CMF-LCM, ISO(C6OBA)₂, S811, ULAD and Irgacure 651.

<Figure 1>

LC sample preparation

LC cells were made from two pieces of glass bonded together and no surface alignment layer were used, in which polyethylene terephthalate (PET) film of 12 μm thickness was used as the cell spacers. The LC mixture is filled into the cell at an isotropic state, and cooled down to BP temperature at a rate of 0.5 °C/min. Then the LC cell was irradiated with UV light for the purpose of photopolymerization and optical switching. Here, the temperature of the LC cell was carefully changed so as to keep the same texture and reflection band during the UV irradiation for the photopolymerization.

Preparation of a BP film reflecting both right- and left-circularly polarized light

BP film reflecting both right- and left-circularly polarized light was prepared by carrying out the following procedure. At first, a LC premixture was prepared, which is composed of 68.0 wt% 8CB, 4.5 wt% CMF-LCM, 18.5 wt% C6M, 7.0 wt% ISO(C6OBA)₂ and 2 wt% Irgacure 651. In order to induce nonuniform photopolymerization, 1 wt% ULAD was added to the above LC mixture. BP temperature range is from 41.5 °C to 36°C on the cooling process, here BP-LCs premixture was

photopolymerized by 3.0 mW/cm^2 UV light (365nm) for 5 min at $38 \text{ }^\circ\text{C}$ of BP I temperature range to form polymer stabilized BP, as shown of fabrication process in Fig. 2a. Here the BP temperature range of the RH BP sample after polymerization was about $40 \text{ }^\circ\text{C}$ ranged from $52 \text{ }^\circ\text{C}$ to 12°C due to the stabilization effect from the polymer network. Following that, the cells were immersed in cyclohexane for about 48 h and later in tetrahydrofuran for 20 min to remove the non-reactive components. After that the cells were kept in vacuum chamber at $60 \text{ }^\circ\text{C}$ for about 3 h. Thus the BP polymer template with a RH structure was achieved as shown in Figure 2a. Finally, the cell containing the BP polymer template was refilled with LC mixture containing a chiral azobenzene switch with a LH structure as shown in Figure 2a. Photo- and thermal switching of the cell A was investigated.

<Figure 2>

Measurements

The samples were observed using a polarizing light microscope (POM) (Leica, DM2500P) with a heating stage of an accuracy of $\pm 0.1 \text{ }^\circ\text{C}$ (LTS 420). The optical images were recorded using Linksys 2.43 software. The reflection spectra were obtained by fiber spectrometer (Avantes, AvaSpec-2048). The surface morphology and cross-section of the polymer network was observed by scanning electron microscopy (SEM) (Hitachi S-4700).

Results and Discussions

Firstly, we fabricate a free-standing, 3D BP polymer template with a RH structure, which retains the chiral 3D structure of BP I and attaches the top layer of the LC cell. The schematic of the fabrication process is shown in Fig. 2a, here the BP polymer template

was formed by using a polymer network from polymer-based stabilized BP system. The premixtures were composed of 7.0 wt% chiral dopant and 23 wt% reactive LC monomers (CMF-LCM and C6M) mixture, dissolved in the bulk LCs of 8CB. We note that, in order to induce the nonuniform UV photopolymerization, a small amount of ULAD (1.0 wt%) was added into the above LC mixture in this study, which has an absorption maximum at 334nm close to that of the photoinitiator (Irgacure 651, Ciba, 345nm) but an extinction coefficient two orders larger than that of Irgacure 651. As a result, a BP polymer template with RH structure tethered to the upper surface of the cell was obtained during the UV irradiation due to LC monomers diffusion induced by a gradient in UV intensity over the thickness of the film, as shown in Fig. 2a. It is worth noting that the depth of BP polymer template here was determined by many factors, including the ULAD concentration, UV light intensity, monomers concentration and so on. And then we use the solvents to remove non-reactive components in LC sample to create 3D BPs polymer nanostructures template, here a typical porous structure derived from the cubic symmetry of BP I with the size of several hundred nanometers could be observed in Fig. 2b, especially the inset of the magnified SEM image. Herein, the cross-linked polymer networks occupied in the disclination lines form this RH polymer template with a 3D lattice of disclination lines, in which this BP-like structure was re-transferred from the polymer to the refilled LC.

To verify the spatial distribution of BP polymer template in the LC cell, a photo-polymerizable nematic (N) LC mixture was prepared by mixing C6M, LC242 and LC1057 at a weight ratio of C6M : LC242 : LC1057 = 1 : 1 : 1 and 2% by weight of monomers of Irgacure 651 is added. After that, we refill this LC monomers mixture into the BP polymer template and subsequently carry out UV irradiation at N phase

temperature. Fig. 2c shows SEM image of the cross-section of this LC film, we observe that the LC cell possesses two distinct regions, which means that a spatial segregation structure occurs through the thickness of the cell. Herein, the top layer is the RH BP polymer scaffold, the thickness of which was measured to be around 8 μm with a 2/3 of the cell thickness of 12 μm ; while the bottom layer is the N-LC film with about 4 μm thickness. Therefore, we can confirm that the templated BP polymer structure attached to the top layer of the cell could be used to subsequently realize BP film reflecting both right- and left-circularly polarized light, where a spatial segregation of the RH and LH domains will occur across the cell.

Fig. 2d and 2e show the reflection spectra, POM images and digital photos of the above LC sample at different stage, respectively. As shown in Fig. 2d, the cell exhibits a Bragg reflection band of around 485 nm before polymerization (curve 1); while an obvious blue-shift of Bragg reflection occurred due to the volume shrinkage induced by photopolymerization as demonstrated in previous studies, where the reflection band shifts to around 420 nm (curve 2). After wash-out and drying process, the reflection band disappeared (curve 3), the polymer template exhibits a weak scattering state, which may be due to that the BP template has a porous structure which induces a light scattering at the interface between the polymer network and air. Additionally, from the POM images and digital photos as shown in Fig. 2e, the different stages of the same LC sample could also be observed, which is in accordance with the above reflection spectra.

To get a BPs film which reflects both right- and left-circularly polarized light, we fabricate another LC mixture with LH structure. Herein, we employ a chiral azobenzene switch (Azo-S) to achieve the switching of the Bragg reflection of a BPs film. The

molecule structure of chiral Azo-S and the optimized molecular conformation of *trans* and *cis* isomers induced by UV/Vis light were shown in Fig. 3a, the detailed characterization and optical performance of Azo-S was described in our previous study.³¹ Chiral Azo-S combined with bent-shape dopant (TBOA-MBIN) and S811 were doped into commercially available nematic LCs (SLC 1717) to induce BPs with LH structure. Here TBOA-MBIN was synthesized according to the procedure in our previous work.¹⁴ The weight ratio of SLC 1717: S811: Azo-S is 71.5:26:2.5, and 20 wt% of TBOA-MBIN were added into the above LC mixture. The BP temperature range of the LC mixture is from 54 °C to 49 °C. We note that, the POM textures (Fig. S1) and the temperature dependence of the reflection wavelength (Fig. S2) in the ESI, clearly shows that there is no BP II in our sample due to the highly chirality together with bent dopant similar to our previous studies.²⁶ The optical switching of BP-LC sample was performed at 52 °C of BP I and therefore the thermal effect can be eliminated. As is shown in Fig. 3b, upon UV irradiation (365 nm, 10 mW/cm²), the reflection band has a red-shift from around 430 nm to 610 nm. After 8s of irradiation, the reflection band of BP I no longer shifts and reaches a photostationary state with a reflection central band at 610 nm. Fig. 3c shows POM images for the BP I of this LC sample at 52 °C by UV light irradiation. As the irradiation time was increased, the various BP platelet colors of blue, green, and red were observed, which is corresponding to the digital images of LC cell with a continuous shift of reflection color under UV irradiation, as shown in Fig. 3d.

<Figure 3>

In order to evaluate the influence of phase state of the above LC mixture on the reflection characteristics of the complex LC film, we refill the above LC mixture into the

BP polymer template with a RH structure at 65 °C (isotropic state). As shown in Fig. 4a of the reflection spectra (curve 1), one reflection band was unexpectedly observed at around 412 nm. Meanwhile, the real image of the LC sample observed by circular polarizer shows that the LC sample at 65 °C only reflects right circularly polarized light, as shown in Fig. 4b. Therefore, we confirm that the Bragg reflection at 65 °C derives from BP polymer template with a RH structure. Here the circular polarizer used has two windows for RH and LH circularly polarized light, which is prepared by stacking multi-layer PVA films on TAC substrate and followed by dyeing and uniaxial tension process.³² At temperature down to 50 °C, the LH LC mixture refilled is in the blue phase. By comparing this state with the sample at 65 °C, the reflection band shifts to 428 nm and the reflectance intensity evidently increases. Furthermore, according to the images observed by the circular polarizer as shown in Fig. 4b, both right circularly polarized light and left circularly polarized light were reflected by this LC film. This suggests the lattice parameters of double twisted cylinder structure (DTC) between LH BP-LC mixture and RH BP template are very similar, which is coincident with each component as demonstrated above. The POM textures in Fig. 4c further demonstrates that both the top LH component and bottom RH component are blue phase at 50 °C. Here it should be noted that the refilled LH component dominates the textures of POM in complicated LC film, this may be mainly because of intact structure from its small molecule nature. Further cooling to 25 °C, the reflection band has a slightly red-shift with the reflection central band at 440 nm and the reflectance intensity of the LC film further increases, as shown in Fig. 4a (curve 3), which is due to the BP→Ch phase transition of the LH LC mixture. At this temperature, right circularly polarized light was reflected by the RH BP

polymer template and left circularly polarized light was reflected by the LH Ch-LC. Meanwhile, the overlap of the reflection band of these two components results in the increase of the reflectance intensity. We also confirm that the bottom LH layer is Ch phase according to the POM texture in Fig. 4c. Our results demonstrate that, by the use of the polymer template only on one layer of the LC cell, two separate heterogeneous RH and LH regions across the LC cell was fabricated. As a result, the top LC mixture and the bottom BP polymer template can keep their respective state. Therefore, the change of the phase state of the backfilled LH component could lead to different reflectance state as demonstrated above, for example, both BP and Ch phase could exist in the same LC cell. We note that, despite the different phase state of the bottom layer, there is no influence in performance of the top BP template. This result may have several explanations. First, as demonstrated in Fig. 2a, BP I was photopolymerized and followed by removing non-reactive components to obtain a porous polymer template, which retains the chiral 3D structure of the BP I, thereby it's easy to transfer BP-like structure from the polymer template to the refilled LC mixture, which was called "memory effect" and also suggested in Ch-LC system by our previous study.²⁸ Second, at 65 °C of isotropic phase of the LH LC mixture, the LC film exhibits a right circularly polarized reflection band induced by the BP polymer scaffold, which reveals that the dense polymer network has a strong anchoring effect for any rod-like molecules but may be not related with orientational order of the backfilled LC molecule, thereby realizing "structure recovery". Another phenomenon we need to clarify is that, the reflectance intensity of this LC sample based on BP polymer template is lower than that of the conventional LC sample in this study, we think that the observed reflection colors may arise from both reflection

and scattering, which is due to that interface scattering and multiple domains of BPs without no surface alignment layer.²²

<Figure 4>

To estimate the optical switching effect of the LC film we carry out different UV irradiation time at 52 °C. Fig. 5 shows the time-dependence of the reflection spectra of the LC film during UV irradiation, we just observe one and broad reflection band in the reflection spectra of the LC film as shown in Fig. 5a, in which the reflection central band of the LC film is about 410 nm. As demonstrated in the thermal switching section, at initial state (0s UV irradiation), right circularly polarized light and left circularly polarized light were reflected within almost the same reflection band by the LC film. That is, the lattice parameters of DTC of blue phase between LH layer and RH layer in the LC film are similar. As UV irradiation time is increased, we see that the LC film has two reflection bands while the reflectance intensity of the reflection band at around 410 nm decreases as shown in Fig. 5b-e. It is obvious from these spectra to observe directly the reflection band of LH component has a distinctly red-shift; While the reflection band of RH BP polymer template changes little (a slightly red-shift). Furthermore, Fig. 5f shows the reflection band of the LC film as a function of irradiation time during 365 UV light irradiation at 52 °C. The reflection band of LH component measured by fiber spectrometer has a red-shift from about 428 nm to 610 nm with an increase of the UV irradiation time, which is accordance with the aforementioned result. Meanwhile the reflection band of RH layer shifts a little, ranging from around 410 nm to 430 nm, which may be due to the change of the refractive index induced by molecular orientation change. Additionally, as the UV irradiation time increases, the various BP platelet colors from

blue, green, and red were also observed under POM during the UV irradiation as similar to the results in Fig. 3c, which derives from the LH component and is also consistent with the digital images of the LC film observed by LH window of circular polarizer. From the above discussion, we can clearly see that the reflection band of the LC film was reversibly switched from “hyper-reflective” state to dual reflection bands state. What’s more, the tuning process is a reversible by UV/visible light irradiation.

<Figure 5>

The mechanism of the optical switching of LH component of the LH film is mainly due to the variation of the chiral strength of the added chiral Azo-S induced by photo-isomerization from *trans* state to *cis* states. Fig. 6 shows the illustrations of the LC film reflecting both right- and left-circularly polarized light and the structure change of DTC of the LH layer doped with chiral Azo-S before and after UV/Vis light irradiation. Here the photoresponsive behavior arises from azo group, in which chiral Azo-S has two azo linkages resulting in reversible *trans–cis* isomerization of azo configurations on light irradiation. As demonstrated in Fig. 3a, because of the decrease of molecular aspect ratio (the length *L* to the width *D*, *L/D*) of *cis*-isomer with a bent shape,³¹ the photoinduced *trans–cis* isomerization was found to decrease the chirality of chiral Azo-S and correspondingly increase the lattice constant of DTC structure of BP-LCs as shown in Fig. 6, which was verified by the above spectra data in Fig. 5. These results are also similar with our previous study, in which a H-bonded Azo-S was used to tune the reflection color in BP-LC system.²⁶ We note that, the demonstration of feasibility of achieving this separated double layer in a BP-LC cell, along with the fact that it allows for photo-/thermal switching of the BP-LC film reflecting both right- and left-circularly

polarized light, may allow for extending our approach to other types of LCs and other switching strategies, such as electric field and so on.

<Figure 6>

Conclusions

To conclude, we demonstrate a new method toward a novel kind of BP films reflecting both right- and left-circularly polarized light. Our results show that a 3D and LH BP polymer template, which is attached to just one of the substrates and goes across around 2/3 of the LC cell gap, was formed by utilizing a nonuniform photopolymerization of a LC mixture at BP I temperature induced by ultraviolet light absorbing dye. As a result, a spatial segregation structure of RH and LH domains occurring through the thickness of LC cell was fabricated by refilling the BP-LC mixture with LH structure containing a chiral Azo-S, in which each region of the hybrid BP film reflects its own handedness polarized light. Furthermore, the Bragg reflection of the hybrid BP film could be reversibly switched by photo- and thermal fields. These hybrid BP films with the distinct optical properties are expected to be promising applications for flexible LCs display, optical components, LCs lasing, etc.

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Figure captions

Fig. 1. The chemical structures of the materials used.

Fig. 2. (a): Schematic of the procedure for preparing the BP film; (b): SEM images of the surface morphology of the BP polymer template, the inset of the magnified SEM image; (c): SEM image of the cross-section of the polymer template by infiltrating into N-LC monomers and followed by UV irradiation; (d): The reflection spectra of the cell before and after UV irradiation (curve 1 and curve 2), and after removing the non-reactive components (curve 3) and (e): POM images and digital images of the above sample at different stage.

Fig. 3. (a) Molecule structure of chiral Azo-S and the optimized molecular conformation of *trans* and *cis* isomers induced by UV/Vis; (b): The time-dependence of the reflection spectra of LH LC mixture doped with chiral Azo-S during UV irradiation; (c): POM images of LH LC sample under UV irradiation; (d) The corresponding digital images of the LH LC sample.

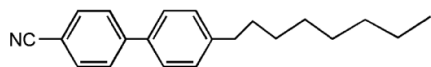
Fig. 4 (a): Reflection spectra of the BP film at different temperature; (b): The digital images of the BP film observed by circular polarizer; (c): POM images of the BP film at different temperature. The inset: R-CPL and L-CPL: right- and left-circularly polarized light.

Fig. 5 (a)-(e): Reflection spectra of the BP film during UV irradiation; (f): The reflection band of the LC film as a function of UV irradiation time. The inset: R-CPL and L-CPL: right- and left-circularly polarized light.

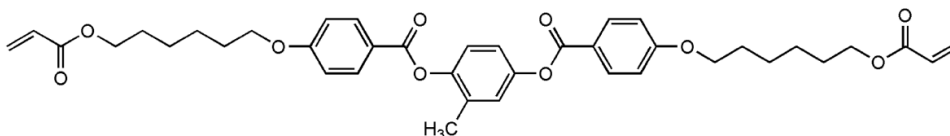
Fig. 6 Illustration of the BP film reflecting both right- and left-circularly polarized light

(R-CPL and L-CPL) and Schematic representation of switching mechanism of LH component doped with chiral Azo-S, demonstrating partial enlargement of DTC structure before and after UV irradiation.

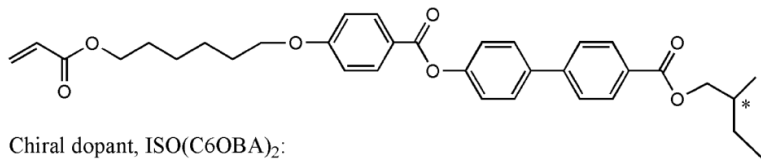
4-cyano 4'-octylbiphenyl, 8CB:



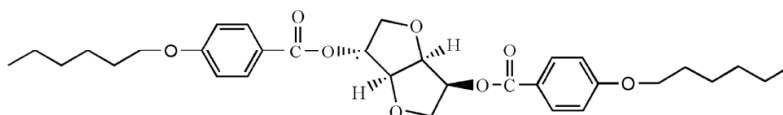
Di-functional liquid crystalline monomer, C6M:



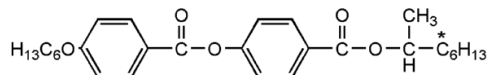
Chiral Mono-functional liquid crystalline monomer, CMF-LCM:



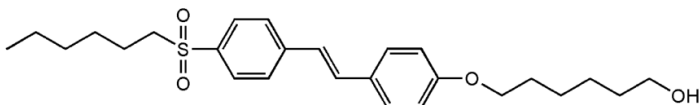
Chiral dopant, ISO(C6OBA)₂:



Chiral dopant, S811:



Ultraviolet light absorbing dye, ULAD:



Photoinitiator, Irgacure 651:

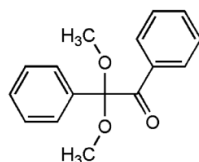


Figure 1

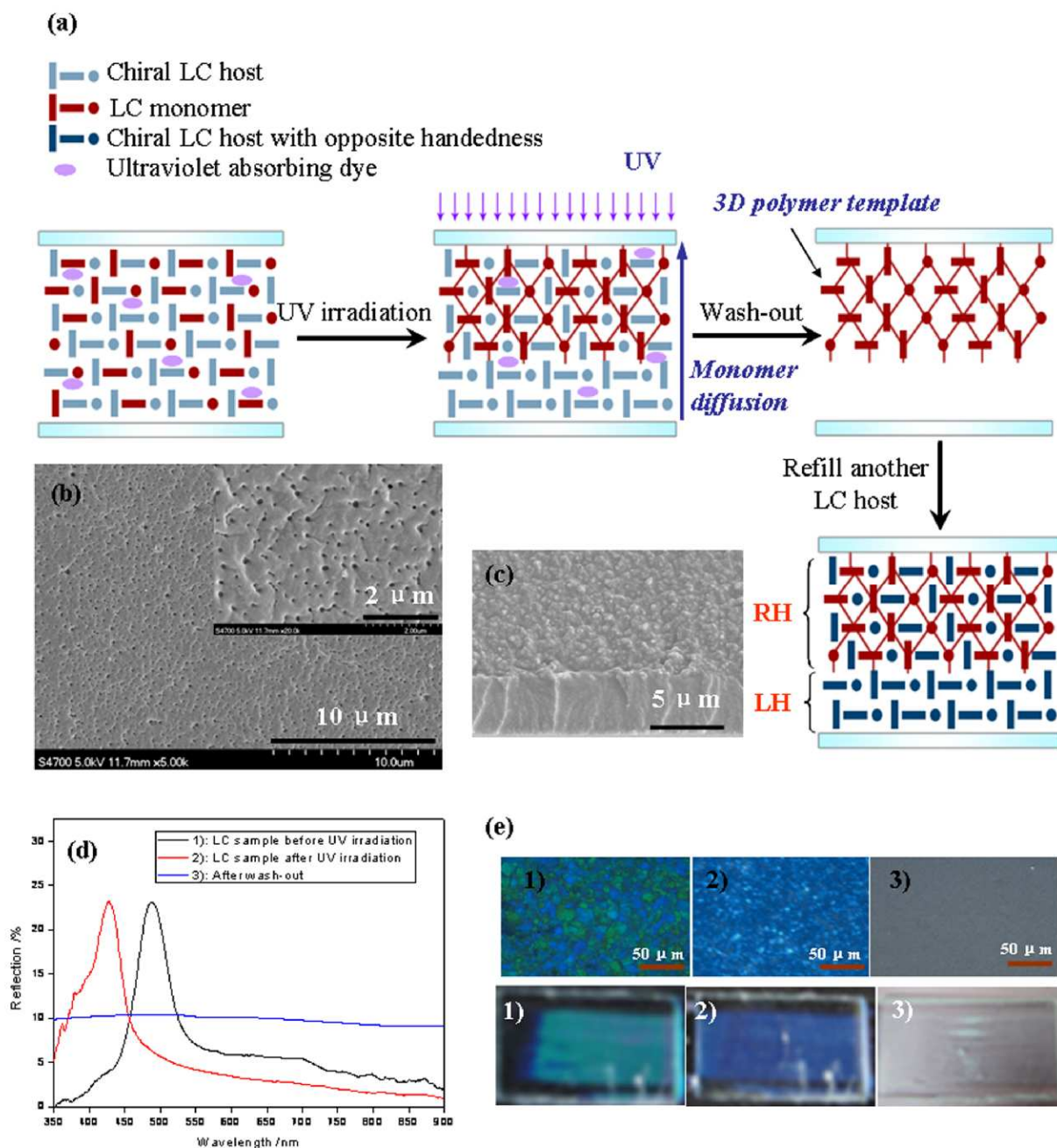


Figure 2

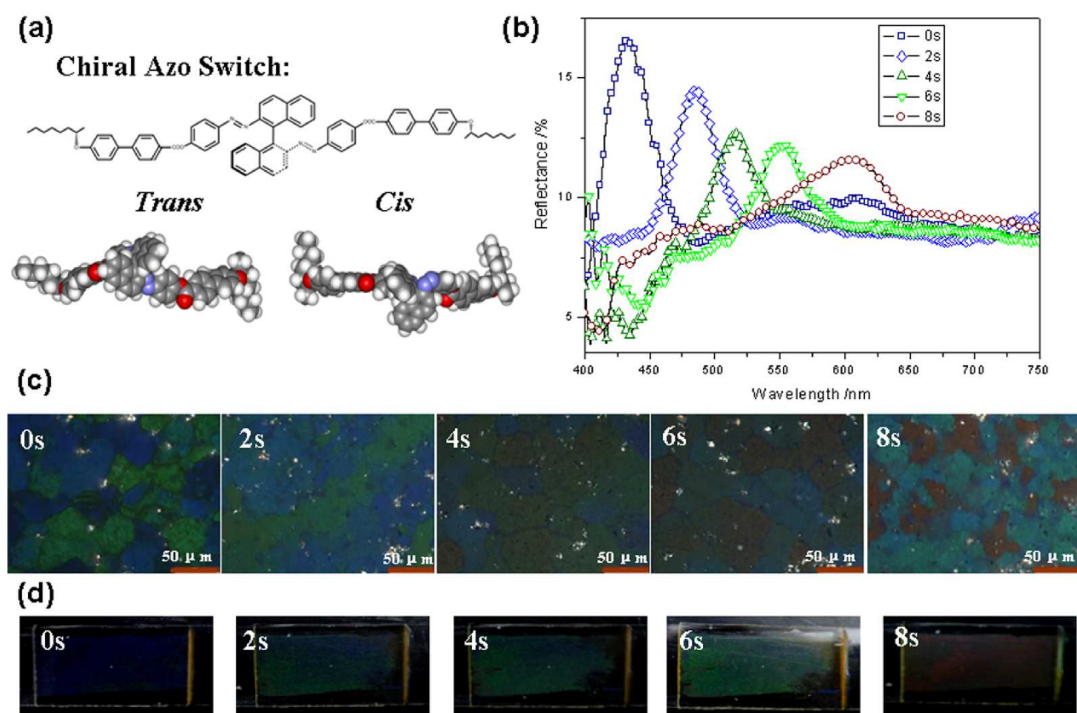


Figure 3

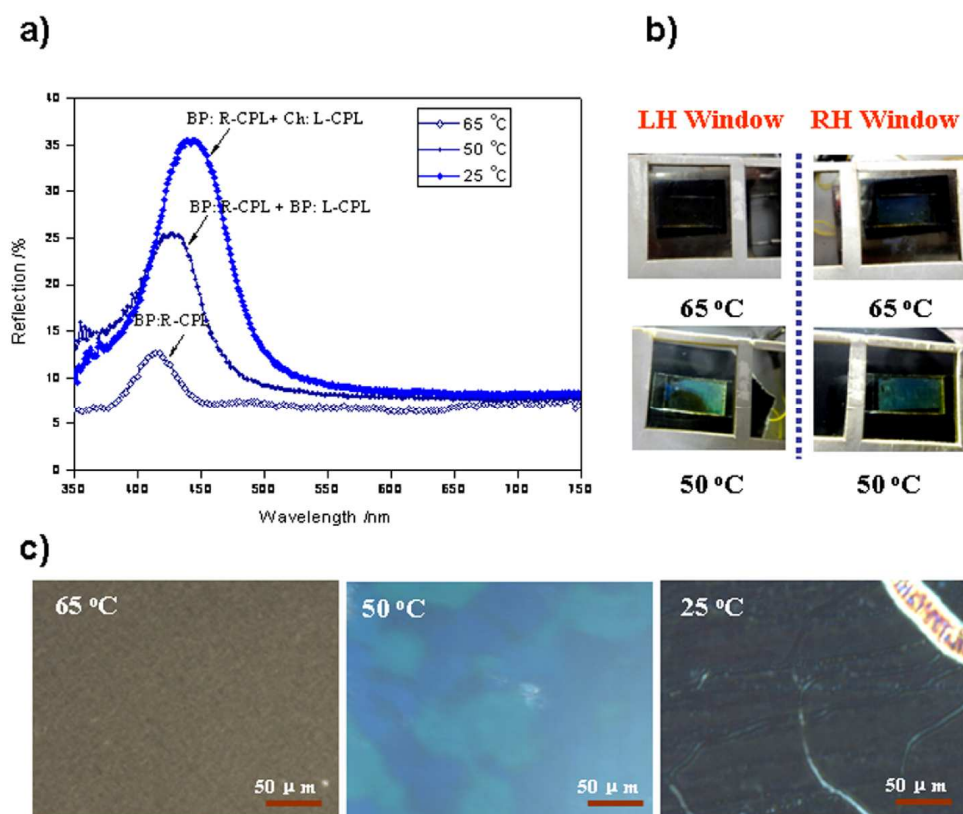


Figure 4

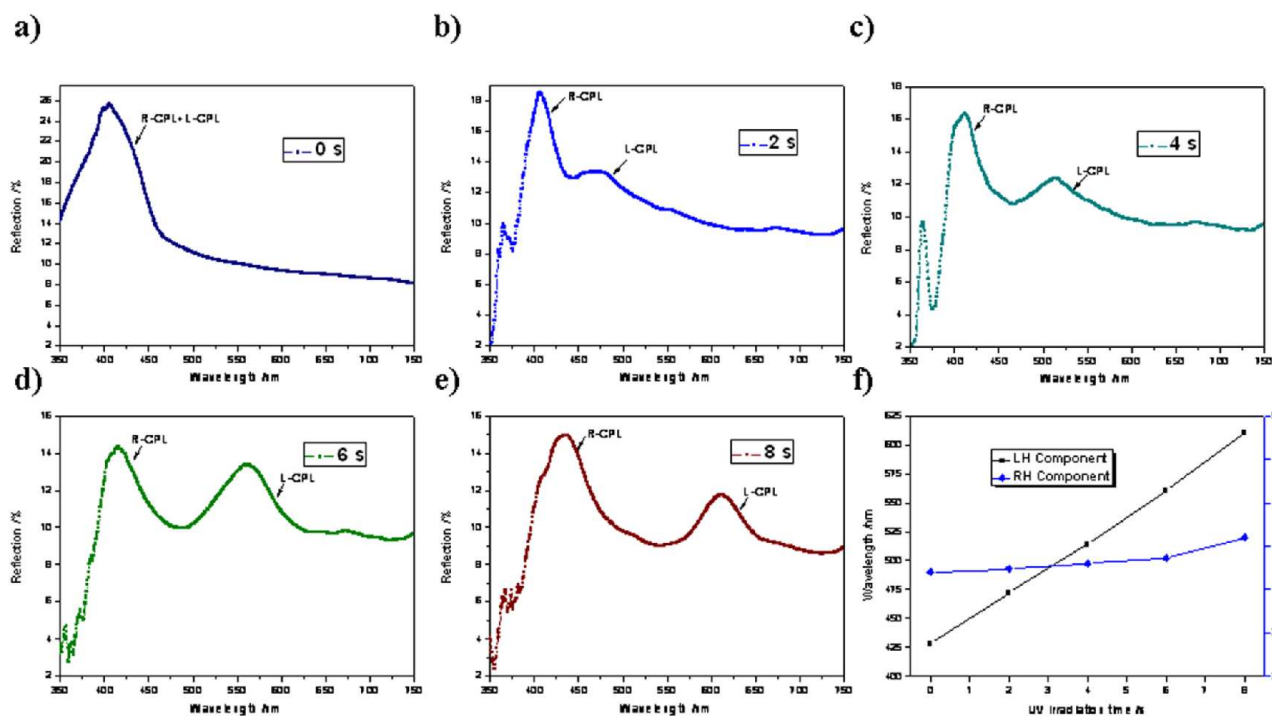


Figure 5

Graphic Abstract:

Table of contents entry

Text:

A new kind of blue phase (BP) liquid crystals (LCs) films which could reflect both right- and left-circularly polarized light and photo- and thermal switching performance has been demonstrated.

Color graphic:

