

A Luminescent Dicyanodistyrylbenzene-based Liquid Crystal Polymer Network for Photochemically Patterned Photonic Composite Film

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Electronic Supplementary Information

Abstract A novel photonic composite film based on a luminescent dicyanodistyrylbenzene-based liquid crystal polymer network (LCN) was fabricated by using a silica colloidal crystal as a template. The upper part of inverse opal structure and the luminescence characteristics of dicyanodistyrylbenzene-based moiety endowed the resulting bilayer photonic film with structural color arising from coherent Bragg reflection and fluorescence properties, respectively. A fluorescence enhancement phenomenon was observed in the photonic film due to the overlap between the reflection band and emission band of the fluorescent LCN. More importantly, the photo-induced irreversible *Z/E* photoisomerization of dicyanodistyrylbenzene-based moiety in the photonic film led to both a reflection spectral shift and an observable fluorescence variation. On the basis of this effective phototuning process, microscopic patterning of photonic film was developed under both fluorescence mode and reflection mode. The work demonstrated here provides a new route to construct photo-responsive photonic film.

Keywords Luminescent liquid crystal; Polymer networks; Photonic composite; Photopatterning

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INTRODUCTION

The photonic crystals (PCs) which can be exploited to control and manipulate the flow of light continue to be a hot topic, since PC assemblies have emerged as key materials in the building blocks of various devices for applications in optics, electronics, and sensors^[1–4]. In order to promote PCs materials for practical applications, much effort has been paid to achieve the goal of patterning a PC. On one hand, some conventional methods including lithography, template directed approaches, microimprint lithography and inkjet printing have been employed to pattern PCs^[5–7]. However, some challenges still need to deal with, such as complex template requirements, pattern precision, time-consumption and so on. On the other hand, of particular interesting is to construct PCs patterns triggered by external stimuli such as electric, magnetic fields or chemicals^[8–11].

Crosslinked liquid crystal polymer, including liquid crystal elastomer (LCE) and liquid crystal polymer network (LCN), is one of the most interesting stimuli-sensitive polymers due to their ability to change their shape reversibly upon a certain external stimulus^[12–14]. A variety of works

have recently been carried out to manipulate the reflection band/color and shape of the PCs based on LCE or LCN, where the tuning mechanism should be attributed to the changes of the LC molecule alignment induced by temperature, UV/Vis light or electric field^[15–20]. However, there are only a few studies on the patterning of the LCN-based PC *via* photo-triggered isomerization of photoresponsive molecules. In particular, simultaneous modulating of reflection and fluorescence colors in the system has not been reported to date.

In this work, we propose a novel photochemical patterning of bilayer photonic composite film with the upper inverse opal structure, based on a luminescent dicyanostyrylbenzene-based LCN. As is well known, dicyanodistyrylbenzene-based molecule is a π -conjugated platform with various optical properties, such as tunable luminescence emission in the visible spectra and aggregate-induced enhanced emission (AIEE) properties. Additionally, the dicyanostyrylbenzene derivatives can undergo *Z/E* isomerization upon the radiation of ultraviolet (UV) light^[21–24]. Thus, dicyanodistyrylbenzene-based materials have recently attracted tremendous research interest for using the construction of multi-responsive systems^[25–31]. Here, due to the luminescence emission and *Z/E* photoisomerization of the dicyanodistyrylbenzene-based moiety, irreversible photo-tuning both reflection structural color and fluorescence

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color as well as fluorescent enhancement phenomenon are observed in the photonic film. Based on these observations, a reflection-fluorescence dual-mode LC photonic pattern could be achieved (Fig. 1a), where the microscopic pattern was recorded through ultraviolet (UV) light. These results are promising for the fabrication of LC nanocomposite films for photonic applications.

EXPERIMENTAL

Materials

Dicyanostyrylbenzene-based monomer was synthesized in our lab, and the details of synthesized process and characterizations were provided in electronic supplementary information (ESI). C6M, a diacrylate LC monomer, was synthesized according to the method described in the

previous work^[32]. Three monoacrylates with a carboxylic acid group were purchased from Bayi Space LCD. Photoinitiator Darocur 784 was purchased from J&K Scientific Ltd. Thermal polymerization inhibitor, *tert*-butylhydroquinone, was obtained from J&K Scientific Ltd. Figure 1(b) shows the chemical structures of dicyanostyrylbenzene-based monomer (1), C6M (2), three monoacrylate carboxylic acids (3), Darocur 784 (4) and *tert*-butylhydroquinone (5).

Fabrication of the Photonic Composite Film

Firstly, we synthesized monodispersed silica microspheres with a certain diameter based on the standard Stöber method. After that, a close-packed multilayer opal PC was fabricated by a vertical deposition method in anhydrous ethanol of the resulting silica microspheres. Then a special blank cell was

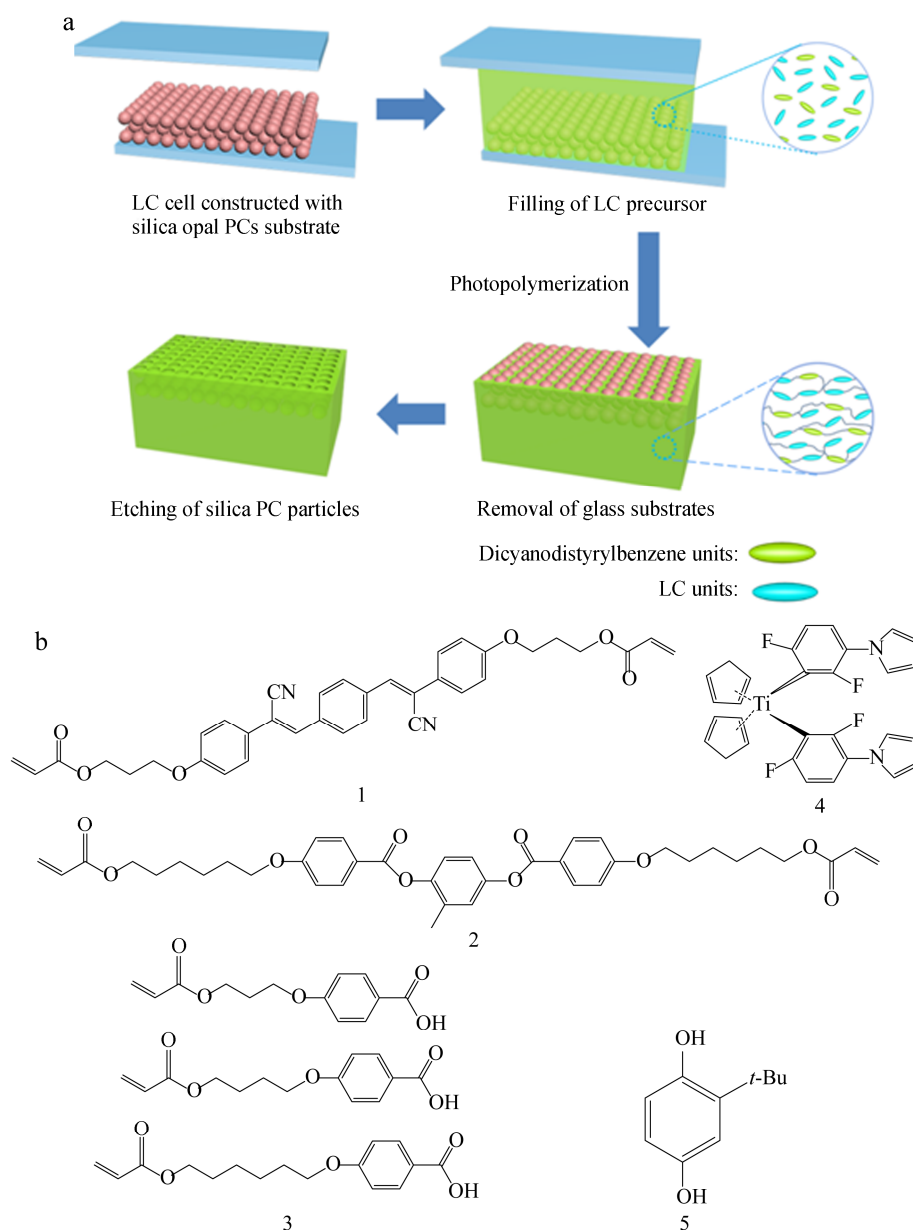


Fig. 1 (a) Schematic illustration of preparation process of the photonic LC films and the LCN formation; (b) The chemical structures of the materials used to prepare the photonic film

set up, where inner-coated poly(vinyl alcohol) (PVA) glass after rubbing process was used as the top substrate and the glass with silica opal PCs was employed as the bottom substrate. Here the spacers were pasted on the glass substrate with silica opal PCs, therefore the spacers do not affect the silica opal PCs on the bottom layer. In order to further obtain a photonic film with upper layer of inverse opal structure, a LCN precursor was prepared, which was composed of C6M, carboxylic monoacrylates, dicyanodistyrylbenzene-based monomer with a weight ratio of 1:8.5:0.4, and 1.0 wt% of Darocur 784 as visible light photoinitiator, and trace of *tert*-butylhydroquinone used as thermal polymerization inhibitor were added into the LC mixture. Following that, the cell was filled with LC precursor at the temperature of 120 °C and then cooled to 95 °C with nematic phase. The polymerization was conducted under visible light for 30 min, and a post-exposure heat treatment at 115 °C was employed to increase the conversion of the acrylate groups. The film was peeled off from glass substrates and was immersed in 3% hydrofluoric acid for a few hours to remove opal matrix. The schematic illustration of the preparation process of the LC photonic polymer films is shown in Fig. 1(a). After drying at room temperature, the final bilayer photonic composite film was obtained with certain reflectance color.

Measurements

The photoluminescence intensity was measured by fluorescence spectrum photometer (Hitachi, F-4500) and ultraviolet absorption spectrum was measured by UV/visible spectrophotometer (G91/G9PC). The mesomorphic properties of the LC monomer mixture and microscopic patterns of the photonic composite film were observed using a polarizing optical microscope (POM, Leica, DM2500P)

equipped with a hot stage calibrated with an accuracy of ± 0.1 °C (Linkam, THMS-600) under transmission and reflection mode, respectively. Morphologies of silica opal PCs and the photonic composite film were observed using a scanning electron microscope (SEM, Hitachi S-4700). The thermodynamic properties were measured using a differential scanning calorimeter (DSC, Pyris Diamond) with a heating and cooling rate of 10 °C/min under a dry nitrogen purge. The reflection spectra were obtained by a fiber spectrometer (AvaSpec-2048) with a Y-type optical fiber in reflection mode, and the incident light and collected light were both vertical to the tested samples.

RESULTS AND DISCUSSION

To fabricate reflection-fluorescence two-mode photonic film, here a new dicyanostyrylbenzene-based LC monomer was synthesized based on three-step synthetic procedure including etherification, Knoevenagel reaction and acylation reaction. A specific description of the preparation procedure and the structure characterizations of the monomer are provided in Figs. S1 and S2 (in ESI). The phase-transition temperatures of the dicyanodistyrylbenzene-based monomer determined by DSC are shown in Fig. S3 (in ESI), which suggests that the monomer exhibits a LC phase with a broad temperature range. The typical texture of nematic phase in POM images could be observed (Fig. S4, in ESI). We firstly investigate the *Z/E* photoisomerization of the monomer in solution (Fig. 2a). The ultraviolet absorption spectra of the monomer in methanol solutions are shown in Fig. 2(b); the changes of two main peaks were successively observed upon irradiation with 365 nm UV light (10.0 mW/cm²) for 0 min to 20 min. The

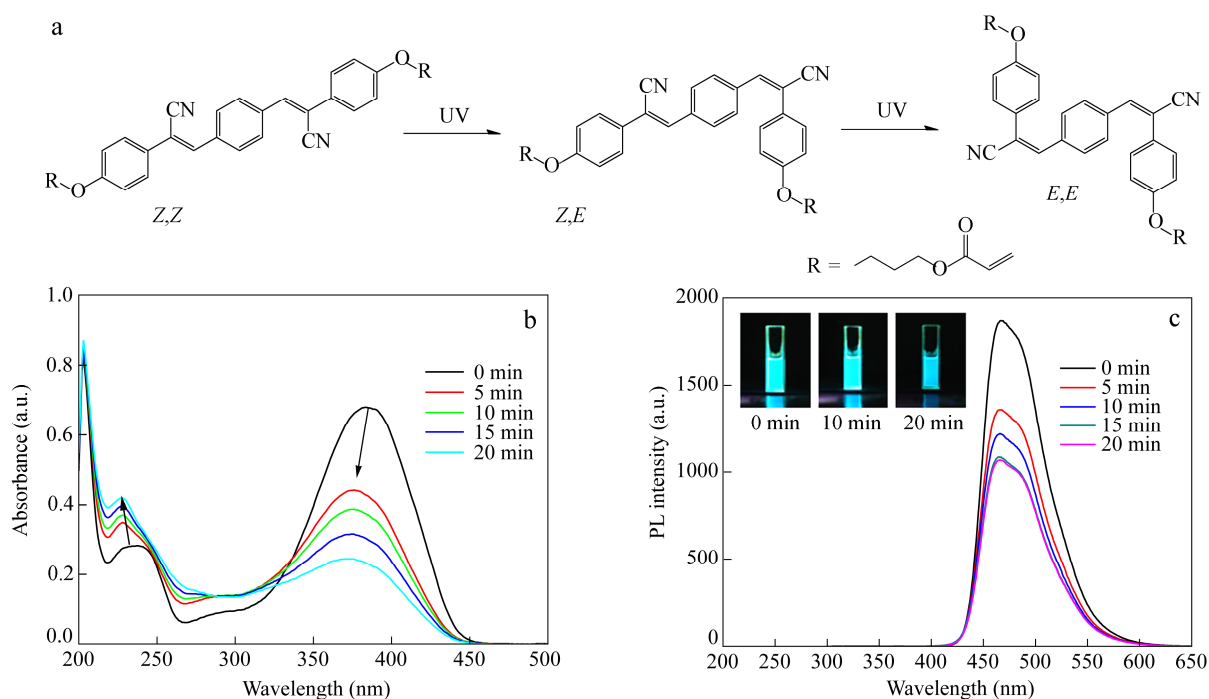


Fig. 2 (a) Schematic diagram of three isomers of dicyanostyrylbenzene-based monomer; (b) UV-Vis absorption spectra and (c) fluorescence spectra of the dicyanostyrylbenzene-based monomer (The inset pictures are fluorescent photographs of the dicyanostyrylbenzene-based monomers dissolved in chloroform.)

absorption maximum peak at 378 nm showed an obvious decrease of the intensity, accompanied by a blue-shift from 384 nm to 370 nm, and the photostationary state (P_{ss}) was achieved after approximately 20 min of 365 nm UV irradiation. The blue-shift in the absorption should be ascribed to the Z/E isomerization due to the decrease of effective conjugation length from the initial Z,Z -isomer to bent Z,E -isomer and E,E -isomer. Meanwhile, the absorbance peak at 228 nm increased and a corresponding blue-shift was also observed. The Z/E photoisomerization process was also monitored by $^1\text{H-NMR}$ characterization. As shown in Fig. 3, according to the orderly changes in protons of $^1\text{H-NMR}$ spectrum, the dicyanodistyrylbenzene-based monomer molecules were all in the Z,Z -form at the initial state. After irradiation with 365 nm UV light for 20 min, the intensity of some peaks decreased and new peaks appeared, demonstrating that partial Z,Z -form molecules were transferred to the Z,E - and E,E -isomers. Moreover, the spectrum shows little changes upon further UV light exposure, which reveals that the isomerization reaches the photostationary state (P_{ss}) when exposing under 365 nm UV light radiation for 20 min. In addition, an obvious decrease in fluorescence intensity of dicyanodistyrylbenzene-based monomer dissolved in chloroform solution was observed in the emission spectrum as shown in Fig. 2(c). The observed decrease of fluorescence intensity may also be attributed to the Z/E isomerization of the monomer. In the dilute solution state, rotational motions of phenyl rings are unconstrained, and thus the isomerization takes place easily as the π -conjugation length reduces, resulting in a decrease in fluorescence intensity^[33]. As the inset images shown in Fig. 2(c), the fluorescence color of the monomer turned to be blue after 20 min UV radiation. Meanwhile, the fluorescence intensity became weaker than that of the initial state. It should be noted here that, the absorption spectra of the monomer shown in Fig. S5 (in ESI) had almost no changes under

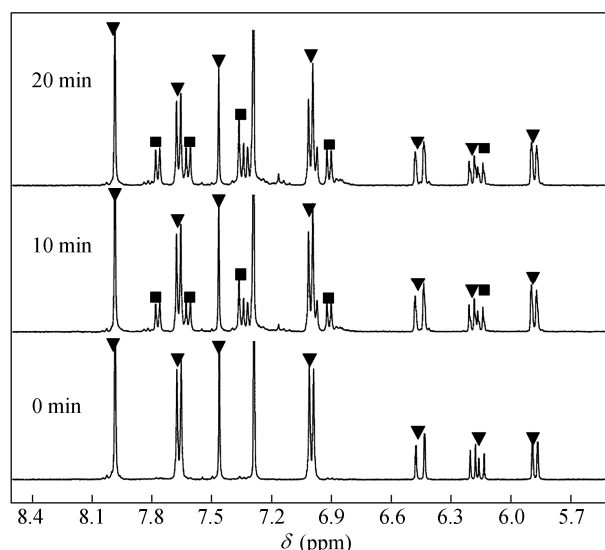


Fig. 3 $^1\text{H-NMR}$ spectra of dicyanodistyrylbenzene-based monomer exposed to UV irradiation (365 nm, $\sim 10 \text{ mW/cm}^2$) for 0, 10, 20 min ($\blacktriangledown$ represents Z,Z isomers, $\blacksquare$ represents new peaks (configurational isomers) after exposure under UV radiation.)

exposure of 254 nm UV radiation accompanied by the heating, which means that Z/E photoisomerization of the monomer is irreversible.

Based on luminescent dicyanodistyrylbenzene-based monomer, we developed a reflection-fluorescence two-mode LC photonic film by employing opal silica PC as the template. Here the photonic film with the upper layer of inverse opal structure was obtained through the infiltration of LC precursor into the silica PC template, UV induced photo-polymerization and followed by the removal of the template as shown in Fig. 1(a). The obtained photonic film showed switchable optical behavior when exposed to UV light radiation. The cross-sectional SEM images of the photonic composite film after the etching process are shown in Figs. 4(a) and 4(b). It is clearly seen that the photonic film is basically composed of two layer including the top inverse opal layer and the bottom pure LCN layer, wherein the thickness of the whole photonic film is about 30 μm and the inverse opal layer is around 4.0 μm . The surface morphologies of the inverse opal layer in the photonic film shown in Figs. 4(c) and 4(d) exhibited periodically ordered air hole structure, which replicated the structure of the silica opal template.

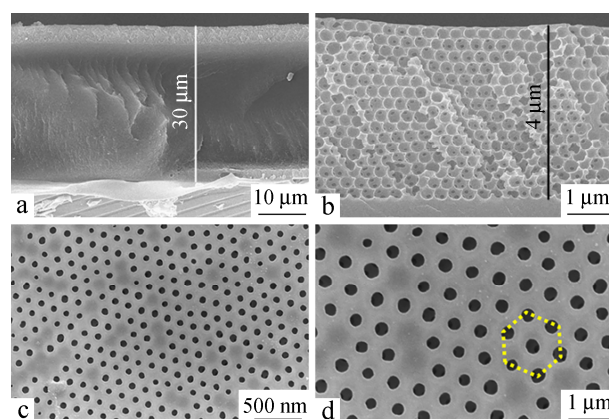


Fig. 4 (a) Cross-sectional SEM image of the LCN photonic film, (b) high magnification cross-sectional SEM image of the composite film; (c) SEM image of the inverse opal layer, (d) high magnification SEM image of the LCN inverse opal layer

To estimate the relationship between reflection and fluorescence of the LC photonic composite film, we prepared a special structure composite film. We removed half of the SiO_2 opal structure and immersed this non-opal section into 3.5 wt% PVA solution. After that, this section was rubbed to induce planar orientation after hot-stage treatment at 85 $^\circ\text{C}$ for 1 h. Then the blank cell was made by assembling this specific substrate and the top PVA-coated planar-orientation substrate. Finally, the LC composite film was prepared according to the steps mentioned above. The reflectance spectrum of the photonic composite part is shown in Fig. 5(a), and the reflectance spectrum shows a main peak at about 480 nm. The fluorescence spectrum of photonic composite part and pure LC part are plotted in Fig. 5(b). The inset pictures in Figs. 5(a) and 5(b) are the photonic composite film taken under lamp-light and 365 nm UV light, respectively. As we can see in the inset picture of

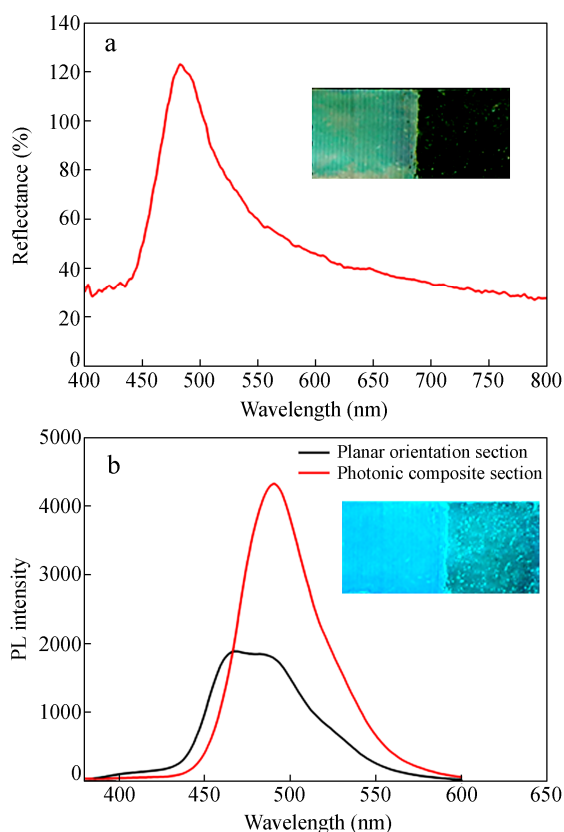


Fig. 5 (a) Reflectance spectrum of the LCN inverse opal film; (b) Fluorescence spectra of the LCN inverse opal film and LCN film without inverse opal structure (Inset pictures are half photonic composite film taken under reflectance mode and fluorescence mode; the inverse opal composite part is in the left side, and the right side is the plane orientation part.)

Fig. 5(a), the photonic composite film was prepared, wherein half of the composite film (left, the photonic film section) exhibited reflectance color and the other half (right, ordinary LC film section) had no reflectance. As a result, it is clearly seen that the fluorescence intensity of the photonic film section was much stronger than that of the ordinary LC film section. According to the fluorescence spectrum (Fig. 5b), it shows an around 50% increase in fluorescence intensity. The observed fluorescent enhancement may be attributed to most overlapping between emission band and photonic stopband. Many previous studies have focused on the enhancement of the luminescence emission in the PCs/luminescent nanoparticle system, such as quantum dot, upconversion nanoparticles and so on^[34–40]. Here the typical enhancement effect was found in a LCN-based photonic system. We believe that the enhancement in luminescence emission of dicyanodistyrylbenzene-based LCN could be used to construct some novel photonic patterns for sensor and security applications.

We further investigate the fluorescence and reflectance spectra of the photonic composite LC film upon UV light irradiation. As shown in Fig. 6(a), the fluorescence intensity increased in the first 10 min and then decreased as the film underwent UV light radiation for next 10 min. The luminescence emission behavior demonstrated here is

different from the dicyanostyrylbenzenic monomer in solution-state, which may be ascribed to the crosslinked structure of the LCN. The formed polymer networks after visible-light photopolymerization constrained the dicyanostyrylbenzenic units. Moreover, *Z/E* isomerization of dicyanostyrylbenzenic units made LC units more intensive under UV light radiation. Thus, the fluorescence intensity increased due to the AIEE effect of the dicyanostyrylbenzenic monomer. As the UV light irradiation continued, the fluorescence intensity decreased. This result may have the following two explanations. First, UV light-induced reform in configuration (*Z,E* isomer further transforms to *E,E* isomer) resulted in a further decrease of LC order. Meanwhile, *E,E* configuration has the twisted configuration because of its rigid structure, leading to larger steric interference. Thereby it may prevent the molecules packing well and thus reduce its effective π -conjugation length. Secondly, it is seen in Fig. 6(b) that the reflectance intensity of the photonic film decreased accompanied with a slight blue shift from 500 nm to 470 nm after 30 min UV light irradiation. The consequent offset in the wavelength between photonic stopband and fluorescence band led to a decrease in fluorescence intensity. Thus, we believe that these two aspects resulted in the decreased fluorescence intensity. All the above results also indicated the reflection color as well as the fluorescence behavior of the photonic film could be effectively controlled and modulated by 365 nm UV irradiation via photoisomerization of dicyanostyrylbenzenic monomer.

Furthermore, we demonstrated a reflection/fluorescence two-mode microscopic array images in the photonic film. Figure 6(c) is the schematic diagram of the micropattern preparation. The patterned images were achieved upon UV light radiation for 20 min with a negative photomask placed on top of the photonic film. Figures 6(d) and 6(e) are the POM images of the LC photonic film of the reflective mode under ambient conditions and the emissive mode under a UV lamp, respectively. In the case of reflection mode, grid structure with alternate blue and blue-green color is seen in Fig. 6(d). Here we note that the exposure area shows blue color, while non-exposure region exhibits blue-green color. This result is well in accordance with the reflection spectra as shown in Fig. 6(b). Let us discuss why the reflection color has a blue shift. When exposed to UV light radiation for 20 min, the closely packed air hole structures of the upper inverse opal structure may become disordered due to adequate *Z/E* photo-isomerization of the dicyanostyrylbenzene moiety as mentioned above. Thus the reflected color underwent blue-shift accompanied by the decrease of the reflection intensity. With respect to the fluorescence mode, the emission color in exposure parts is weaker than that of non-exposure parts, which also agrees with the spectrum result.

CONCLUSIONS

In summary, we have fabricated a reflection-fluorescence dual-mode photonic composite LC film based on dicyanostyrylbenzenic-based liquid crystal polymer network. Irreversible *Z/E* photo-isomerization of dicyanostyrylbenzenic units led to tunable reflectance and luminescence intensity of

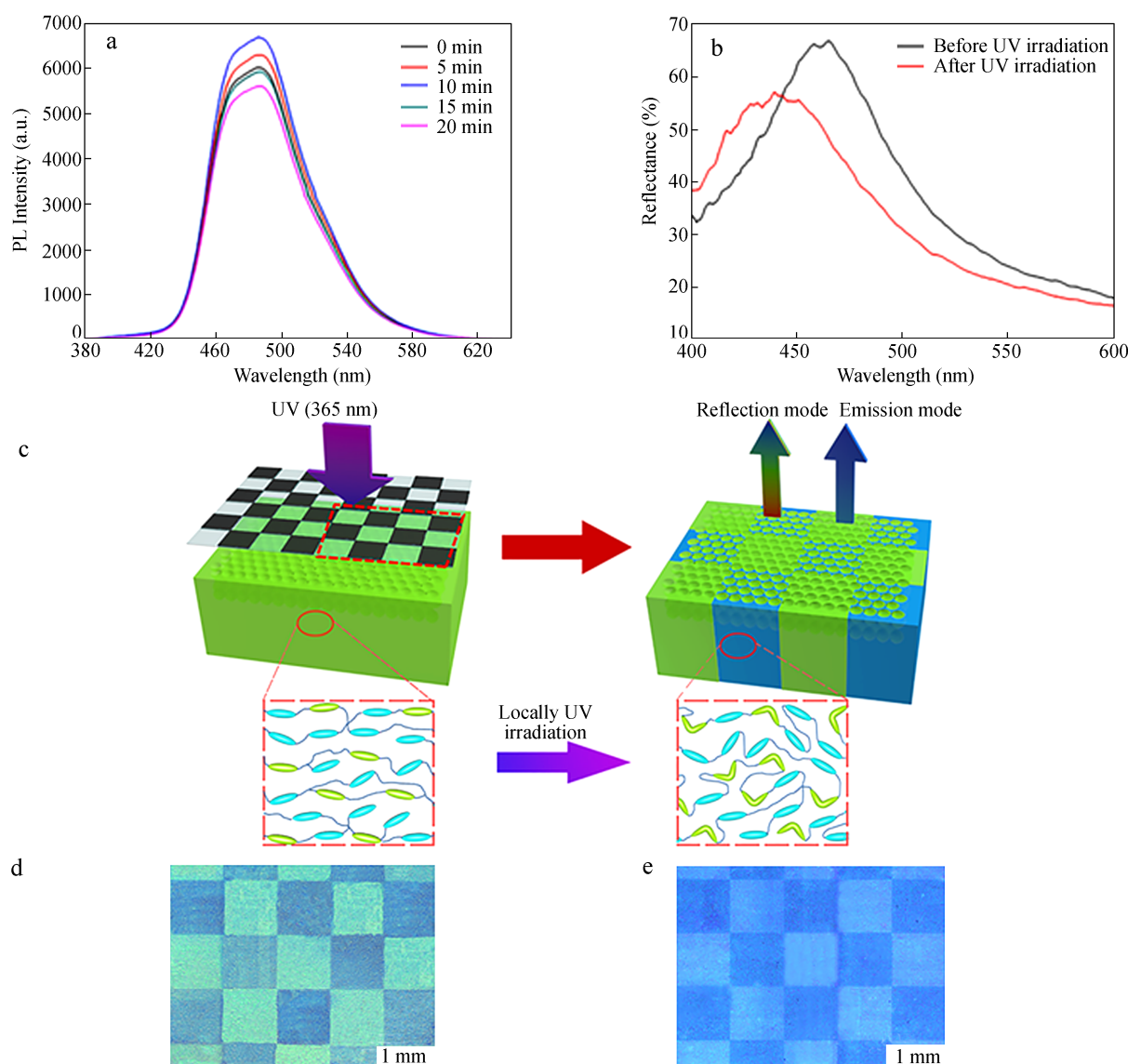


Fig. 6 (a) Fluorescence spectra of the LCN inverse opal film under UV irradiation for 0, 5, 10, 15, 20 min; (b) Reflectance spectra of the LCN inverse opal film before and after UV irradiation; (c) Schematic diagram of the micropattern preparation; (d) POM image of checkered pattern in reflection mode, where the blue parts underwent UV radiation; (e) POM image of checkered pattern in emission mode, where the dark blue parts underwent UV radiation

the photonic film under UV light irradiation. Based on these observations, dual-mode micro-patterns with considerable contrast were achieved through regional UV light irradiation. Our approach demonstrates the effective photo-tuning both of reflection and emission in photonic composite films and paves the way toward their use in photonic applications.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at <http://dx.doi.org/10.1007/s10118-018-2072-4>.

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