Realisation of cholesteric liquid-crystalline materials reflecting both right- and left-circularly polarised light using the wash-out/refill technique

Jinbao Guo[a]; Fang Liu[b]; Fengjin Chen[a]; Jie Wei[a]; Huai Yang[b]

[a] College of Materials Science and Engineering, Beijing University of Chemical Technology, Beijing, People's Republic of China [b] Department of Materials Physics and Chemistry, School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing, China

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Jinbao Guo[a], Fang Liu[b], Fengjin Chen[a], Jie Wei[a]* and Huai Yang[b]

[a]*College of Materials Science and Engineering, Beijing University of Chemical Technology, Beijing 100029, People's Republic of China;* [b]*Department of Materials Physics and Chemistry, School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China*

In this study, a single-layer polymer-stabilised liquid crystal (PSLC) film reflecting both right- and left-circularly polarised light has been developed by a wash-out/refill method. A polymer network/liquid crystals (LCs) composite, in which the polymer network had a certain helical structure, was prepared by photo polymerisation of the photo-polymerisable LC monomers (LCMs) in a LCM/LC/photoinitiator mixture. Then, the LC molecules were extracted from the composite film and cholesteric LCs (Ch-LCs) with the opposite helical structure were refilled into the film. The film was constructed by the polymer network with a certain helical structure and Ch-LCs with the opposite helical structure could reflect both right- and left-circularly polarised light simultaneously. The reflection intensity of the film is close to 100% when the pitch lengths of the two opposite helical structures are the same, which goes beyond the 50% reflectance limit of Ch-LCs. It was demonstrated that the memory effect of the polymer network is an important mechanism for the resulting film properties.

Keywords: cholesteric liquid crystal; circularly polarised light; wash-out/refill; polymer network; memory effect

1. Introduction

Cholesteric liquid crystals (Ch-LCs) are an important class of biological and synthetic materials, in which the molecules are macroscopically arranged in a periodic helicoidal structure. A Ch-LC exhibits many remarkable optical properties due to the formation of a macroscopic helical structure. One of the great features of the Ch-LC is its unusual optical property of selective reflection of circularly polarised incident light [1]. Ch-LCs with a single pitch selectively reflect light of a wavelength between $\lambda_{\min} = Pn_o$ and $\lambda_{\max} = Pn_e$. Here n_o and n_e are the ordinary and extraordinary refractive indices of the locally uniaxial structure, respectively. The bandwidth of the selective reflection spectrum $\Delta\lambda$ is given by $\Delta\lambda = \lambda_{\max} - \lambda_{\min} = (n_e - n_o)P = \Delta n P$, where $\Delta n = n_e - n_o$ is the birefringence. The circularly polarised light with the same handedness as the helix is reflected within this reflection band, and the maximum reflectance of unpolarised light from the Ch-LC is never greater than 50%. Outside the reflection band both polarisation states are transmitted [1, 2]. Much attention has been focused on wideband reflection and its application with Ch-LC materials [3–24] . However, Ch-LC materials that reflect both the right- and left-circularly polarised light (R-CPL and L-CPL) also have important potential as tunable bandpass filters, reflectors or polarisers and temperature sensors [25–32].

The explanation of the reflection of both R-CPL and L-CPL can be found in biological structures. The wings of the rare kind of beetles *Plusiotis resplendens* have a layered structure: between layers with a cholesteric-like structure, possessing the same handedness, a layer with unidirectional orientation of fibrils, owning half-wave ($\lambda/2$) plate properties, is arranged. This type of structure reflects both R-CPL and L-CPL [25]. Man-made materials have been produced by imitating such a fabrication scheme; it has been noted that recently a similar structure comprising a dye-doped polymeric nematic defect layer sandwiched between polymeric Ch-LC films was demonstrated to realise the reflectance in the photonic band-gap region exceeding 50% [26, 27]. In addition, it is well known that two Ch-LC cells with the same mean reflection wavelength, but opposite helicity senses, may be associated to increase the reflected light intensity [28]. However, in view of practical application, there exist some drawbacks in stacking layers to add up their individual optical properties, such as the diffusion between soft layers in the fluid media, and optical defects and losses at the interfaces in optical filtering with a multilayer system.

Mitov and Dessaud [29, 30] have obtained a single-layer Ch-LC gel from photo-polymerisable monomer/Ch-LC composites whose optical characteristics go beyond the 50% reflectance limit, in which the Ch-LC has the characteristics of a thermally induced

*Corresponding author. Email: weijie-2008@hotmail.com; yanhuai@mater.ustb.edu.cn

inversion of the helicity sense. After the composites are cured by ultraviolet (UV) light when the helix is right-handed, the reflectance exceeds 50% when measured at the temperature assigned for a cholesteric helix with the same pitch but a left-handed sense.

The present work develops a single-layer polymer-stabilised liquid crystal (PSLC) film that can reflect both R-CPL and L-CPL simultaneously. Due to the permanent solid helical structure of the polymer network and the bulk liquid crystals (LCs) with the pitch length changing little with temperature, the prepared film can reflect both R-CPL and L-CPL in a wide temperature range. Thus, precise control of the measurement temperature is unnecessary for the Ch-LC gel mentioned, and the location of the reflection band can be controlled accurately through experimental design. This result was briefly reported by us in a previous letter intended for a broad readership [32]. The intention of the present paper is to give a detailed description of the preparation and the reflectance characteristics of Ch-LC films reflecting both R-CPL and L-CPL, and to widen the discussion of the memory effect in PSLCs.

2. Experiments

2.1 Materials

In this study, non-reactive LCs, SLC-1717 (Slichem Liquid Crystal Material Co. Ltd.); a photo-polymerisable nematic monomer, C6M; photo-polymerisable cholesteric monomer (PCM); chiral dopants, S811/R811 and R1011 (Merck Co. Ltd); and a photoinitiator, 2, 2-dimethoxy-2-phenyl-acetophenone (Irgacure 651, TCI Co. Ltd.), were used. C6M was synthesised

according to the method suggested by Broer *et al.* [33]. The PCM was prepared as described in our earlier paper [22]. Figure 1 shows the chemical structures of C6M, PCM, S811/R811, R1011 and Irgacure 651.

2.2 Experimental cells

In order to induce a planar orientation of the LC molecules, the inner surfaces of indium tin oxide (ITO)-coated glass cells were coated with a 3 wt% polyvinyl alcohol (PVA) aqueous solution. The deposited film was dried at 80°C for 30 min and subsequently rubbed with a textile cloth under a pressure of 2 g cm^{-2} in one direction. Polyethylene terephthalate (PET) films of 25 μm thickness were used as the cell spacers. The samples were filled into the cells by capillary action.

To induce photo polymerisation, the cells containing samples were irradiated for 0.5 h with UV light (0.87mW cm^{-2}, 365 nm). The compositions of the studied samples are listed in Table 1.

2.3 Measurements

The samples were observed with a polarising light microscope (POM) (Olympus BX51) with a heating stage (LinkamTHMS-600). The optical images were recorded using Linksys 2.43 software. The transmission spectra were obtained with a UV/visible (VIS)/near infrared (NIR) spectrophotometer (JASCO V-570) in transmission mode at normal incidence, while the transmittance of a blank cell was normalised as 100%. As usual, λ_M and $\Delta\lambda$ are measured from the spectrum by considering, respectively, the wavelength
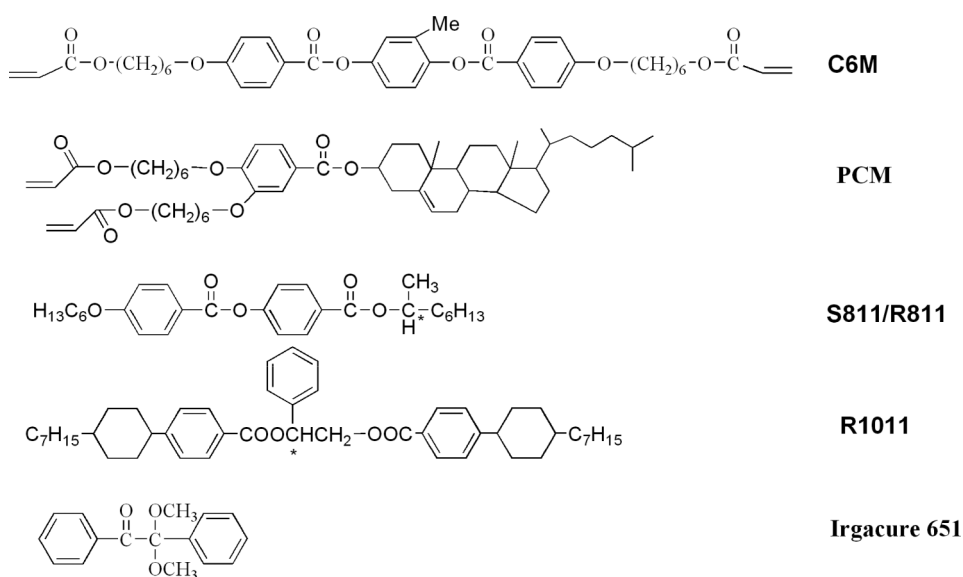

Figure 1.   The chemical structures of the materials used.

Table 1. The compositions of the Ch-LC mixtures in this study.

Cell[a]	Mixture (type)	Weight ratio/wt%[b]
	LHHS	
A (I/V)/B (I/VI)	I	82.0 (SLC-1717)/15.0 (PCM)/3.0 (C6M)
C (II/VII)	II	70.0 (SLC-1717)/15.0 (S811)/10.0 (PCM)/5.0 (C6M)
	III	83.0 (SLC-1717)/17.0 (S811)
	RHHS	
D (IV/III)	IV	65.0 (SLC-1717)/20.0 (R811)/15.0 (C6M)
	V	95.91 (SLC-1717)/4.09 (R1011)
	VI	97.45 (SLC-1717)/2.55 (R1011)
	VII	75.0 (SLC-1717)/25.0 (R811)

[a]Cell A (I/V) means that cell A was obtained by infiltrating mixture V into the cell containing mixture I after polymerisation and extraction of the non-reactive LCs thereafter.
[b]An amount of 2 wt% (compared to C6M and PCM) of photoinitiator Irgacure 651 is added for mixtures I, II and IV.

for the minimum of transmitted light inside the peak and the peak bandwidth at half-height. Scanning electron microscopy (SEM) investigations of the polymer network morphologies were carried out following removal of the LCs from the sample. The cell was plunged in liquid nitrogen and broken in a direction perpendicular to the plates. One of the two pieces was immersed in cyclohexane for 24 h, and later in tetrahydrofuran (THF) for 20 min to remove all of the residue LCs and to leave the bare network architecture. After the solvent removal, the polymer network was coated with a thin film of gold (40 nm) and investigated using a scanning electron microscope (Cambridge S360).

3. Results and discussion

To prepare PSLCs film reflecting both R-CPL and L-CPL simultaneously, mixtures I–VII were prepared as listed in Table 1. Herein, the polymer network was formed mainly by photo polymerisation of C6M or C6M/PCM in the non-reactive LCs, in which the PCM is a cholesteryl compound with a left-handed helix (Figure 1). Among the samples, mixtures I–III

with left-handed helical structures (LHHSs) contained the PCM or S811, while mixtures IV–VII with right-handed helical structures (RHHSs) were added to right-handed R1011 or R811. The position of the reflection band can be adjusted by choosing an appropriate concentration of the PCM and the chiral dopant.

The PSLC film reflecting both R-CPL and L-CPL was prepared by carrying out the following procedure. It should be noted that the definition of the handedness of polarised light provided by Born is used in this study. That is, a left- (right-) handed helix selectively reflects L(R)-CPL; herein, the sample with a left-handed helix was representative as shown in Figure 2(a). At first, the cells containing mixtures with a certain helical structure were irradiated with UV light (365 nm, 0.87 mW cm^{-2}) for 30 min for polymerisation purposes. Following that, the cells were immersed in cyclohexane for about 48 h and later in THF for 20 min to remove the non-reactive LCs. After that the cells were kept in vacuum chamber at 60°C for about 3 h. Thus, the polymer network with a LHHS was obtained as shown in Figure 2(b). Finally, the cells containing the polymer network were refilled with the sample with an opposite helical structure (the RHHS) by a vacuum-filling
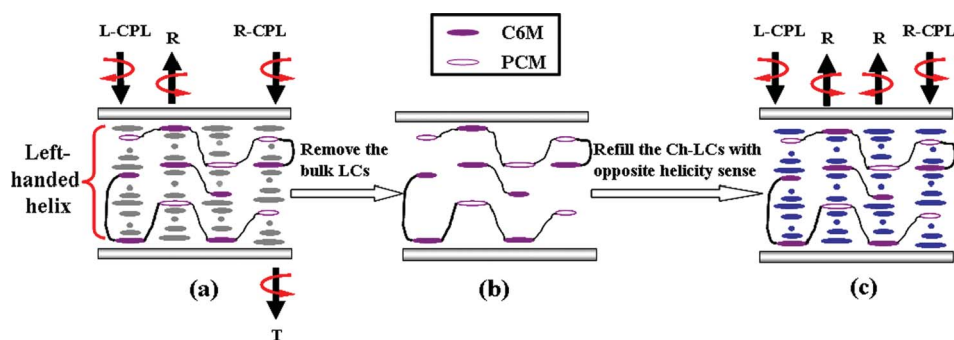

Figure 2. The schematic representation of the method to prepare the expected PSLC film. R – reflection; T – transmission
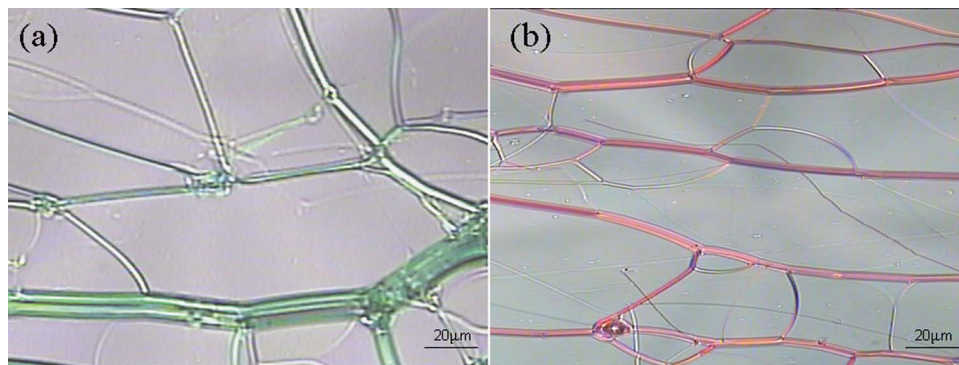
Figure 3.   The POM photographs of (a) cell A and (b) cell D at room temperature.

process as shown in Figure 2(c). After the above process, as indicated in Table 1, cell A (I/V) means that cell A was obtained by infiltrating mixture V into the cell containing mixture I after the polymerisation and extraction of non-reactive LCs thereafter.

Figure 3(a) and (b) shows polarising light microscopy photographs of cells A and D at room temperature, respectively. It can be seen that the cells refilled with Ch-LCs exhibited an appropriate Grandjean planar texture when observed under the POM. This should be attributed to the surface anchoring of the inner surfaces of the cells and the bulk anchoring of the polymer network on the LC molecules. When the polymer network is prefabricated with a certain helical structure and then refilled with Ch-LCs with the opposite helical structure into the network the polymer network, the cells still remained in the planar texture,

as shown in Figure 3, which demonstrated that the above film preparation process did not disrupt the orientation of the LC molecules.

Figure 4 shows the time dependence of the transmission spectra of cell A after mixture V was refilled into the cell with mixture I. We can observe that the position of the reflection band of cell A had a great change with increasing time. At first, the reflection band is ambiguous when Ch-LC molecules with RHHSs (mixture V) were refilled into the cell with mixture I at the beginning. However, the location of the reflection band gradually became obvious and remained at all times with time increasing. This suggested that the anchoring effect of the polymer network on the LC molecules plays an important role in determining the reflectance properties of the Ch-LC as described previous investigations [29–31].
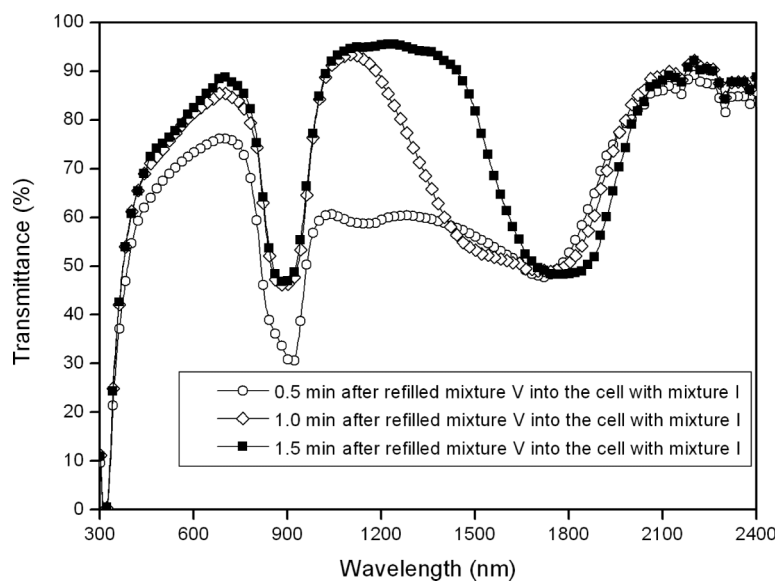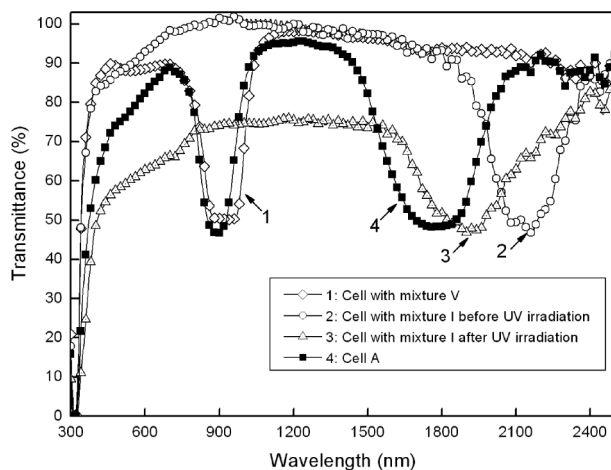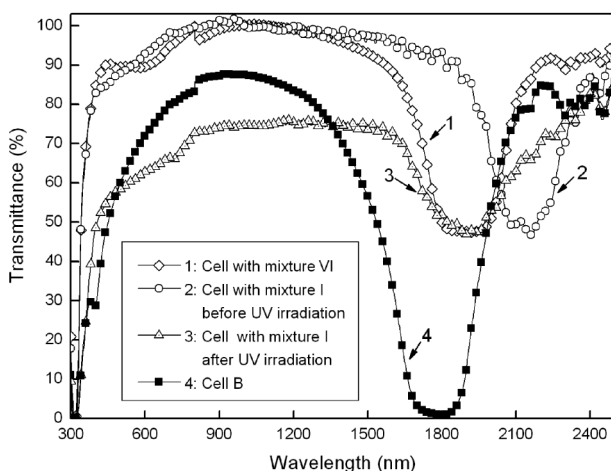

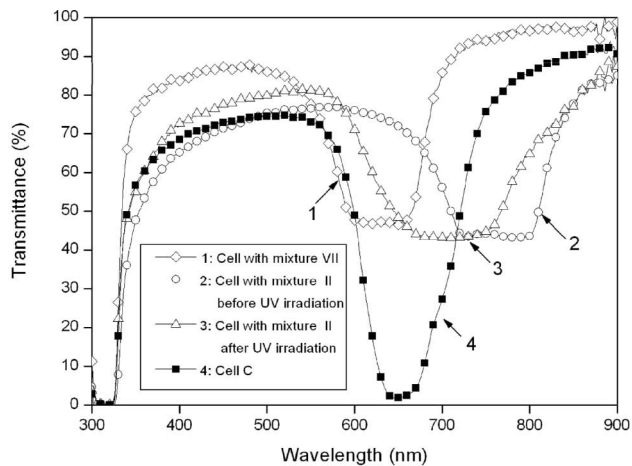Figure 4.   The time dependence of the transmission spectra of cell A.

Figure 5. (a) The transmission spectra of a cell with mixture V, a cell with mixture I before and after UV irradiation and cell A. (b) The transmission spectra of a cell with mixture VI, a cell with mixture I before and after UV irradiation and cell B.
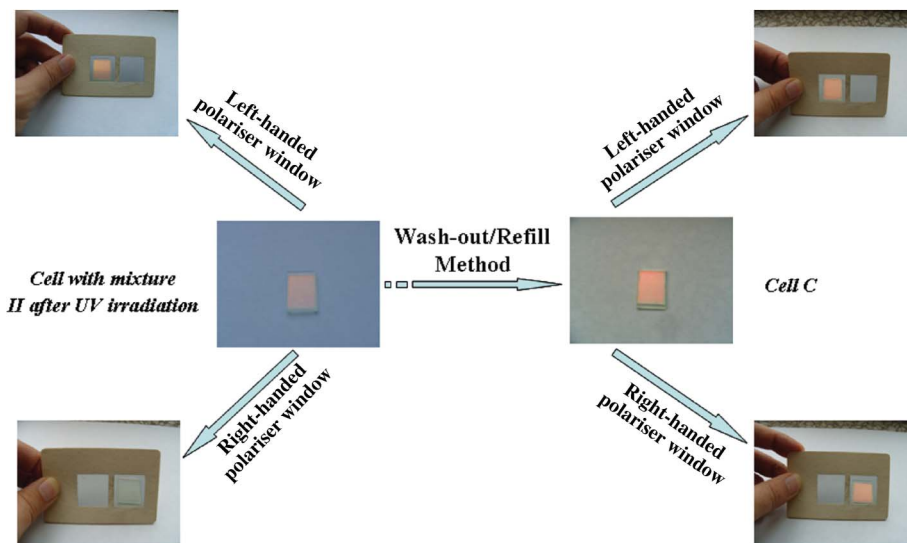
Figure 5(a) shows the transmission spectra of a cell with mixture V, a cell with mixture I before and after UV irradiation and cell A. For the cell with mixture V with 4.09 wt% R1011, the reflection wavelength is 910 nm and the maximum reflection intensity is close to 50% (curve 1). The cell with mixture I also exhibits 50% reflectance before and after UV irradiation (curves 2 and 3), and its reflection wavelength shifts from 2155 (curve 2) to 1905 nm (curve 3) as a result of the formation of the polymer network in the bulk LCs after UV irradiation due to the crosslinking-induced volume shrinkage [29, 30]. After SLC-1717 was washed off and refilled with mixture V, we were able to observe two reflection bands in the spectra of cell A (curve 4) occurring due to the difference in the pitch lengths of the polymer network and the bulk LCs (mixture V).

Figure 5(b) shows the transmission spectra of a cell with mixture VI, a cell with mixture I before and after UV irradiation and cell B. The reflection wavelength of the cell containing mixture VI is 1905 nm (curve 1), which is identical to that of the cell with mixture I after UV irradiation (curve 3). As for cell B, equal pitch lengths of the polymer network and the bulk LC (mixture VI) combined with the opposite helicity sense cause the reflection bands of the polymer network and the bulk LCs to overlap, and the reflection intensity of cell B to approach 100% (curve 4). That is, both R-CPL and L-CPL had been reflected within the same reflection band by cell B. The film characteristic that reflects both R-CPL and L-CPL is attributed to the Ch-LC's composite being divided into two distinct environments: the bulk LCs and the strongly network-dominated regions. As shown in Figure 2(c), the PSLC film contains the polymer network with LHHSs and the Ch-LCs with RHHSs. Then, for the regions farthest from the polymer network, the enclosed LC should behave much the same as in the bulk, leading to Ch structures with a right-handed sense participating in the reflection of R-CPL. At the closest neighbourhood of the polymer network, due to the anchoring effect of the polymer network on the LC molecules, the LC should template the Ch order of the network, thus the helicity sense of these regions is left-handed, which participated in the reflection of L-CPL. We believe that the association of both behaviours within a single-layer film has led to both R-CPL and L-CPL being reflected simultaneously. It is important to note that the main function of the polymer network is to provide a stable internal memory of helicity sense.
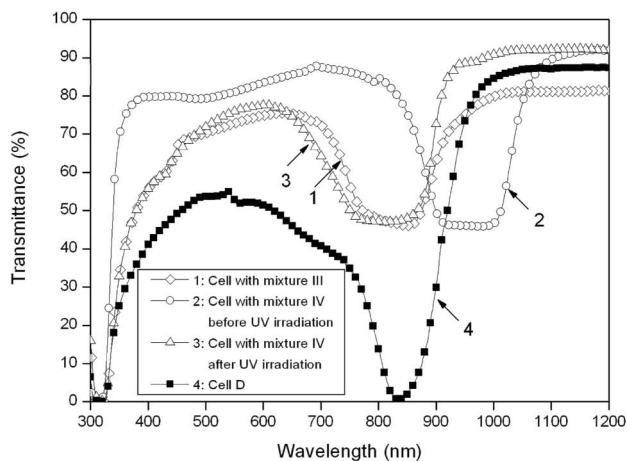
Figure 6(a) shows the transmission spectra of a cell with mixture VII, a cell with mixture II before and after UV irradiation and cell C. As shown in Table 1, mixture II contains 10% PCM and 15% S811, so the reflection band of the cell with mixture II after UV irradiation shifted to about 690 nm in the visible spectrum as, indicated in Figure 6(a) (curve 3). For cell C, owing to the overlap of the reflection band between the polymer network and that of the bulk LCs (curve 1), the reflection intensity of the cell also approaches about 100% (curve 4). As mentioned above, the cell with mixture II after UV irradiation and cell C both reflect red light in the visible spectrum. The images of the cell with mixture II after UV irradiation and cell C under a polariser are shown in Figure 6(b). Herein, the polariser has two windows for both left-handed polarised light and right-handed polarised light, which is fabricated by stacking multi-layer PVA films on triacetylcellulose (TAC) substrate, followed by dyeing and the uniaxial tension process. It can be observed that the cell with mixture II after UV irradiation exhibits selective reflection of light only within the

(a)

(b)

(c)

Figure 6.   (a) The transmission spectra of a cell with mixture VII, a cell with mixture II before and after UV irradiation and cell C. (b) The image photographs of a cell with mixture II after UV irradiation and cell C when observed by a polariser. (c) The transmission spectra of a cell with mixture III, a cell with mixture IV before and after UV irradiation and cell D.

left-handed polariser window. However, cell C after the above wash-out/refill process exhibits selective reflection of light within both the left-handed polariser window and the right-handed polariser window, as indicated in Figure 6(b), which further indicates that both R-CPL and L-CPL have been reflected by cell C within the same reflection band.

In order to further investigate the helical structure of the polymer network on the reflectance properties of the system, mixture IV with a right-handed helix was used to prepare the initial polymer network, which owns an opposite helical structure compared to that of the former sample. Figure 6(c) shows the transmission spectra of a cell with mixture III, a cell with mixture IV before and after UV irradiation and cell D. As regards cell D, due to the equal pitch lengths of the polymer network with RHHSs and the bulk LC with LHHSs, the reflection bands of the polymer network and that of the bulk LC overlap and the reflection intensity of cell D also approaches about 100% (curve 4) as shown in Figure 6(c). From the above result, we can confirm that the polymer network has a characteristic of memorising the Ch-LC helix structure of the system whether the initial helical structure of the polymer network in the PSLCs is left-handed or right-handed.

The morphology of the polymer network in the cell with mixture I after irradiation, as investigated by SEM and viewed as parallel to the surface of the glass plate, exhibits smooth polymer strands (Figure 7); this indicates good solubility of the components within the LCs, which may be understood in the context of the Flory–Huggins model of polymer solubility [34]. The helical superstructure of the polymer network formed in the Ch phase can clearly be imaged, as shown in Figure 7. The SEM investigations undoubtedly suggest the transfer of the organisation of the mesophase onto
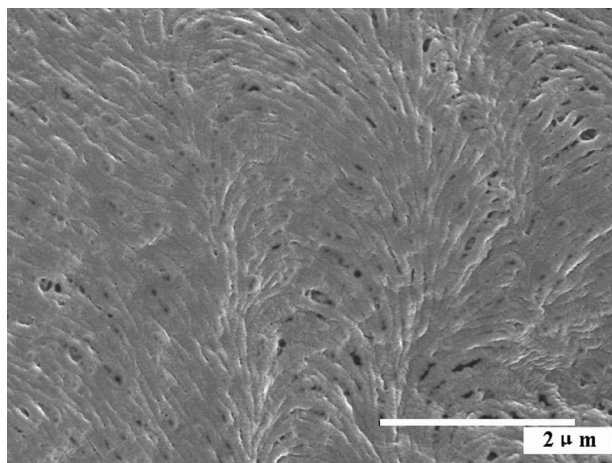

Figure 7.   SEM image of the polymer network formed in the cell with mixture I after UV irradiation.
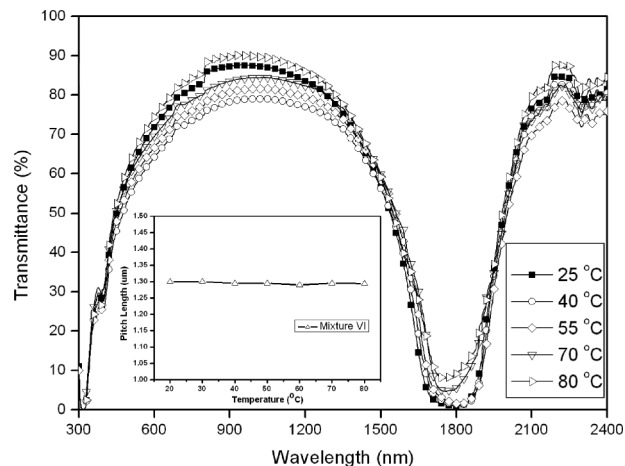

Figure 8.   The temperature dependence of the transmission spectra of cell B. The inset is the temperature dependence of the helical pitch length of mixture VI.

the structure of the network [35, 36], which offers strong evidence for the above explanation.

Figure 8 shows the temperature dependence of the transmission spectra of cell B, in which cell B was obtained by infiltrating mixture VI into the cell containing mixture I after polymerisation and extraction of non-reactive LCs thereafter. The inset in Figure 8 is the temperature dependence of the helical pitch length of mixture VI, which is determined by a Cano-rings method taken by a POM (Olympus BX51) at different temperatures. It is obvious that the helical pitch length of mixture VI changes little with the temperature from 20 to 80°C. Correspondingly, as its operating temperature was raised from 25 to 80°C, it can be seen that the reflection band of cell B has slight temperature dependence, and the reflection intensity is reduced by about 8% over the range in Figure 8, that is to say, the variation of reflection intensity with temperature is also soft. This indicates that the prepared PSLC material can reflect both R-CPL and L-CPL in a wide temperature range. This behaviour is the result of the permanent solid helical structure of the polymer network and the pitch length of mixture VI changing little with temperature as mentioned above, which we believe is due to the nearly temperature-independent helical twisting power of R1011.

4.   Conclusions

In summary, we have achieved a single-layer PSLC film that could reflect both R-CPL and L-CPL by adjusting the handedness of the helical structures of the polymer network and the bulk LCs. The reflection intensity is close to 100% with the same pitch of two opposite helical structures mentioned above in the

composite film. The results strongly demonstrated that the memory effect of the polymer network is an important mechanism for the resulting material property. Furthermore, the reflection band can be located in the range from NIR to visible by adjusting the concentration of the PCM and the chiral dopant. Therefore, due to the wavelength and the polarisation-selective reflection, they are applicable for both counterfeit deterrence in banknotes and secure documents, and for the authentication processes of brand and product protection. In addition, we demonstrated that the prepared PSLC film can reflect both R-CPL and L-CPL in a wide temperature range, which we believe is a key factor for ensuring their potential applications in many fields, such as tunable bandpass filters, polariser-free reflective displays without a backlight requirement, polarisers or colour filters, mirrorless lasing and smart, switchable reflective windows for the dynamic control of solar light.

Acknowledgement

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