

Article

Preparation of Cholesteric Polymer Networks with Narrow-Bandwidth Reflection and Memory Effect

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Abstract: A polymer network with a memory effect based on a polymer-stabilized narrow-bandwidth cholesteric liquid crystal (CLC) was prepared using the washing-out/refill method. The effects of different polymerization conditions on the reflection properties of CLC films were investigated. Meanwhile, the selective reflection property and narrow-bandwidth reflection memory effect of the polymer network were proved, and the response mechanism was provided. Furthermore, different materials from liquid crystals, with an anisotropic refractive index, to toluene, with an isotropic refractive index, were refilled to polymer scaffolds with helical structures, which originated from the periodic arrangement of CLCs. It was confirmed that the reflection bandwidth of these films can be dramatically narrowed by the reduced birefringence (Δn) of the refilled materials. The narrowest bandwidth of 22.5 nm refilling toluene with an isotropic refractive index ($\Delta n = 0$) was obtained. These results may provide a novel idea for flexible reflective displays, color filters, printing, and colored cladding of a variety of objects.

Keywords: cholesteric liquid crystal; polymer network; memory effect; narrow-bandwidth



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1. Introduction

Cholesteric liquid crystals (CLCs) have a unique periodic spiral structure composed of the liquid crystal molecules with chiral structures or chiral nematic liquid crystals (N*-LCs). Owing to its unique periodic spiral structure, the CLC can selectively reflect certain wavelengths of light. The central reflection wavelength λ and bandwidth $\Delta\lambda$ can be given by:

$$\lambda = nP, \quad (1)$$

$$\Delta\lambda = \Delta nP, \quad (2)$$

$$P = (X_C \cdot \text{HTP})^{-1}, \quad (3)$$

where n , Δn , P , X_C , and HTP are the average refractive index, birefringence, helical pitch, concentration of chiral compounds, and helical twisting power of chiral compounds, respectively [1,2]. When circularly polarized light is incident, the light whose wavelength conforms to Equation (1) is completely reflected if the polarization direction is the same as the spiral direction of the liquid crystal molecule. The central reflection wavelength and bandwidth change under the action of the temperature, electromagnetic field, and stress. Therefore, CLCs have attracted significant interest in reflective liquid crystal displays [3–5], color filters [6–10],

smart windows [11–14], and liquid crystals lasers [15] owing to their unique periodic spiral structure. Polymer-stabilized liquid crystals (PSLCs) can be fabricated by adding an appropriate amount of polymerizable liquid crystal monomers into the CLC system; they possess the inherent reflection characteristics of CLCs as well as a stable structure owing to the polymer network [16,17]. For the reflective displays, highly saturated colors and narrow bandgaps are preferred, which lead to a wide color gamut [18,19]. Therefore, to achieve a wider color gamut, a CLC with a narrower reflection bandgap is highly suitable. However, the current reflection bandgap of CLCs, approximately 90 nm, is far from satisfactory and needs to be optimized for practical applications [20].

Many studies have been conducted on exceeding the reflectance limit of CLCs. Mitov et al. [21–23] fabricated a thermally induced helicity inversion CLC mixture with unusual reflectance. When the spiral became right-handed at elevated temperatures, the polymerizable monomer was cured by ultraviolet light. Owing to the memory effect of the polymer network, the reflectivity of the blend system exceeded 50% when the temperature was reduced to the same pitch, but left-handed as the cholesteric helix before the reaction of the polymer network. Washing out/refilling is another critical method for studying the selective reflection properties of N*-LC. Guo et al. [24,25] used a multi-step washout/refill method to create a hyper-reflective film with the good electro-optic performance of PSLCs. Some researchers are also interested in the LC films with narrower reflection bandgaps. Hu et al. [26] selected a series of CLC and hydrogen-bond chiral dopants as materials and filled them into plane-oriented cells to fabricate a novel type of thermally controllable reflective color paper. Yang et al. [27] put forward a facile method to fabricate freestanding, large-domain blue phase films based on self-assembly technology, which exhibit sharp photonic bandgaps with high reflectivity. Yan et al. [28] proposed a reflective display using polymer-stabilized blue phase liquid crystal comprising polarizer and color filters, a relatively narrow reflection band, submillisecond response time, and analogous greyscales by controlling the applied voltage. Xu et al. [29,30] experimentally demonstrated the fabrication of hyper-reflective, electrically switchable, fast response, and colorful reflective displays based on a multilayer blue phase liquid crystal film for the first time, and the effects of birefringence and the average refractive index of different refilled nematic liquid crystals on the reflection properties such as the reflectance, central wavelength, and linewidth of photonic band were investigated. Li et al. [31] prepared a novel flexible film with both super reflectivity and high stability to temperature and mechanical stress via a ‘washout–refill–assemble’ approach by refilling a polymer (optical adhesive) into a cholesteric film assembled using two cholesteric templates with opposite handednesses based on a liquid crystal/reactive mesogen mixture. Subsequently, the narrow-bandwidth templated CLC films were fabricated using the washing-out/refilling method. The reflection bandwidth of these films can be considerably reduced by the reduction in birefringence of the refilled materials [5]. However, the effects of the refractive index properties of the refilled liquid crystals and their effect on the narrow-bandwidth properties of CLC films are quite important [32–34] but have rarely been systematically investigated.

In this study, we demonstrated the memory effect of a polymer network of nematic LCs based on a small birefringence using the washout/refill method and investigated the influence of different factors on the optical properties of CLC systems. To fabricate CLC films with narrow bandwidths, S5011, with a larger helical twisting power, was selected as the chiral agent, a small birefractive index LC was selected as the main component of the CLC system, and C6M was added as the polymeric liquid crystal monomer and photoinitiator IRG651. Nematic LCs with an anisotropic refractive index and toluene with an isotropic refractive index were refilled into polymer scaffolds with helical structures that originated from the periodic arrangement of CLCs. The polymer networks obtained by washing out the narrow reflective CLC films had suitable pitch memory effects, which provided a new idea for the study of total reflection CLC films.

2. Materials and Methods

2.1. Materials

The nematic LC (HNG-720600, Jiangsu Hecheng Display Technology Co., Ltd., Jiangsu, China), the UV polymerizable monomer (C6M, Jiangsu Hecheng Display Technology Co., Ltd., Jiangsu, China), the chiral compound (S5011 Jiangsu Hecheng Display Technology Co., Ltd., Jiangsu, China), the chiral compound (R5011, Beijing Bayi Space LCD Technology Co., Ltd., Beijing, China), and the free radical photoinitiator (IRG651, TCI Co., Ltd., Shanghai, China) were used to fabricate the cholesteric liquid crystal templates in the study. Seven types of refilling materials, SLC-1717 ($n_e = 1.720$, $n_o = 1.519$, $\Delta n = 0.201$, $T_{N-I} = 92.0$ °C, Shijiazhuang Chengzhi Yonghua Display Material Co., Ltd., Shijiazhuang, China), HNG-704700 ($n_e = 1.556$, $n_o = 1.476$, $\Delta n = 0.080$, $T_{N-I} = 105.0$ °C, Nanjing Ningcui Optical Technology Co., Ltd., Nanjing, China), HNG-708200 ($n_e = 1.553$, $n_o = 1.475$, $\Delta n = 0.078$, $T_{N-I} = 107.0$ °C, Nanjing Ningcui Optical Technology Co., Ltd., Nanjing, China), HNG-720600 ($n_e = 1.547$, $n_o = 1.477$, $\Delta n = 0.070$, $T_{N-I} = 100.0$ °C, Nanjing Ningcui Optical Technology Co., Ltd., Nanjing, China), HNG-719200 ($n_e = 1.517$, $n_o = 1.474$, $\Delta n = 0.043$, $T_{N-I} = 104.0$ °C, Jiangsu Hecheng Display Technology Co., Ltd., Jiangsu, China), and HNG-717200 ($n_e = 1.513$, $n_o = 1.474$, $\Delta n = 0.039$, $T_{N-I} = 82.0$ °C, Jiangsu Hecheng Display Technology Co., Ltd., Jiangsu, China), were used to refill into the polymer network. The chemical structures of the above materials are shown in Figure 1.

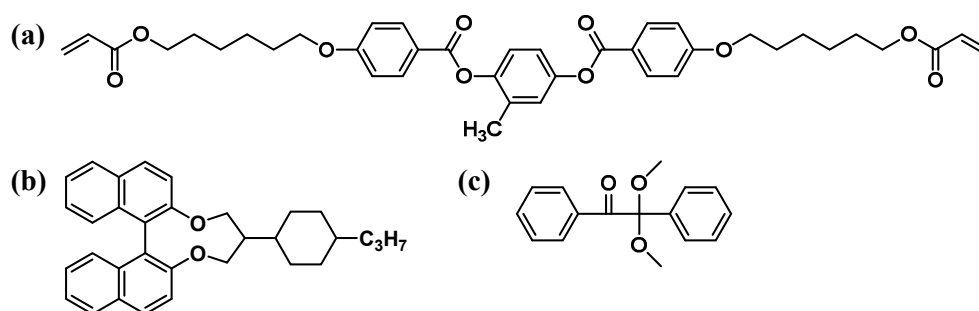


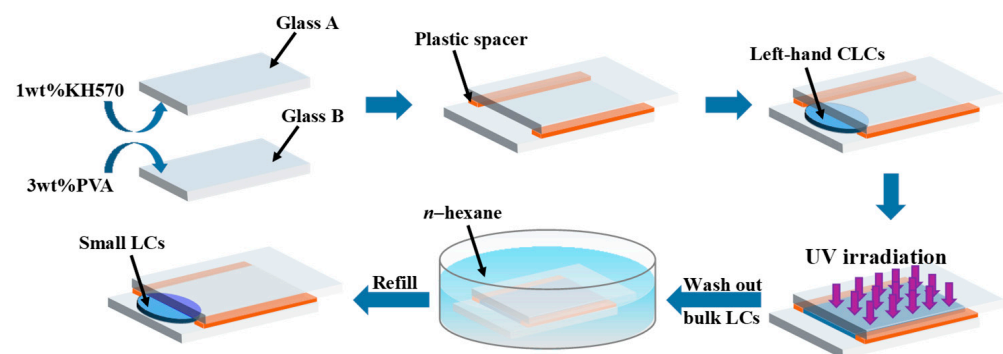
Figure 1. The chemical structures of (a) UV-polymerizable monomer C6M, (b) chiral compound 5011, (c) Photoinitiator IRG651.

2.2. Preparation of Samples and Cells

The water solution of 3% mass ratio of polyvinyl alcohol (PVA) was coated on clean indium tin oxide (ITO) glass using a homogenizer at 2.5 K rpm, and then put into the oven at 100 °C for 1 h. After removal, it was rubbed with clean flannelette 25 times in parallel directions, and finally the parallel orientation glass was prepared. KH-570, water, and isopropyl alcohol were mixed in the weight ratio of 1:49:50, and the resulting mixture was coated on clean ITO glass with a homogenizer at 2 K rpm and then baked in an oven at 100 °C for 20 min to prepare glass B with an oil-friendly treatment layer. Polyethylene terephthalate (PET) films with the thickness of 36 μm were adopted as the spacing pads to separate the two glass substrates, and finally 502 glue was used to seal the two sides of the gaskets. Table 1 lists the different percentage compositions of HNG-720600, S5011, C6M, IRG651, where A0–A5 and B1–B6 represent samples with different C6M and chiral dopant contents, respectively. The mixtures A0–A5 and B1–B6 were filled into the liquid crystal cells by capillary siphon effect at 60 °C. The prepared sample cells were irradiated with UV light (3 mW/cm², 365 nm) from the side of glass A for 150 s to obtain the CLC films. The polymerized sample was immersed in *n*-hexane solution for 7 days, the bulk LCs were removed and dried at room temperature for 12 h to obtain the polymer network with memory effect, and then the nematic liquid crystal small molecules were re-injected. The refilling process was maintained for 12 h in a 50 °C vacuum oven with a vacuum of 0.08 kPa. The complete process is shown in Figure 2.

Table 1. The composition of samples.

Sample	720600/S5011/C6M/I651
A0	97.60/2.20/0.00/0.20
A1	92.60/2.20/5.00/0.20
A2	87.60/2.20/10.00/0.20
A3	82.60/2.20/15.00/0.20
A4	77.60/2.20/20.00/0.20
A5	72.60/2.20/25.00/0.20
B1	78.04/1.76/20.00/0.20
B2	77.88/1.92/20.00/0.20
B3	77.72/2.08/20.00/0.20
B4	77.56/2.24/20.00/0.20
B5	77.40/2.40/20.00/0.20
B6	77.24/2.56/20.00/0.20

**Figure 2.** The schematic of CLC films fabricated by the washing-out/refilling method.

2.3. Apparatus

The morphology of the CLCs was studied by scanning electron microscope (SEM, SU8020, Hitachi, Japan). The optical texture of the samples was measured by a polarizing optical microscope (POM, LEICA DM2700 M, Wetzlar, Germany). The characteristic peaks of the material were measured by a Fourier transform infrared spectrometer (FT-IR, Nicolet IS10, Madison, WI, USA). The reflectance analyses were measured using an fiber optic spectrometer (AvaSpec-2048 FT-SPU, Beijing, China).

3. Results and Discussion

3.1. Effects of the Content of Each Component on the Reflection Properties of CLC Films

The experiments with Groups A and B were conducted to explore the effects of the UV polymerizable monomer and chiral compound on the reflection bandwidth of CLC films. Figure 3 shows the optical texture of samples A0–A5 before and after the UV irradiation and after washing out the bulk LCs. The samples of Group A maintained a good plane texture before and after polymerization; however, after the removal of bulk LCs, the plane texture of the A1–A3 samples with low C6M content broke, owing to the destruction of the polymer network during shrinkage. Excessive C6M led to phase separation, which destroyed the plane texture of the CLC film, causing a significant decrease in the transmittance of the CLC film and nearly no selective reflection.

The reflection spectra of the samples with different C6M and chiral dopant contents before and after polymerization are shown in Figure 4. When the UV light was on, the polymerizable monomer polymerized rapidly on the side close to the UV light, whereas on the side away from the UV light, it diffused in the direction of the UV light. As shown in Figure 4a,c, the reflection bandwidths of samples A0–A5 showed a decreasing trend initially; subsequently, the reflection bandwidths increased owing to the competition between the polymerization of the polymerizable monomer and diffusion of the molecules [35]. Considering that reflectance is an essential property of CLC films in some applications,

although sample A2 had the narrowest reflection bandwidth, its reflectance was much lower than that of sample A4 (20 wt%); therefore, the C6M content of Group B was fixed at 20 wt% in this study.

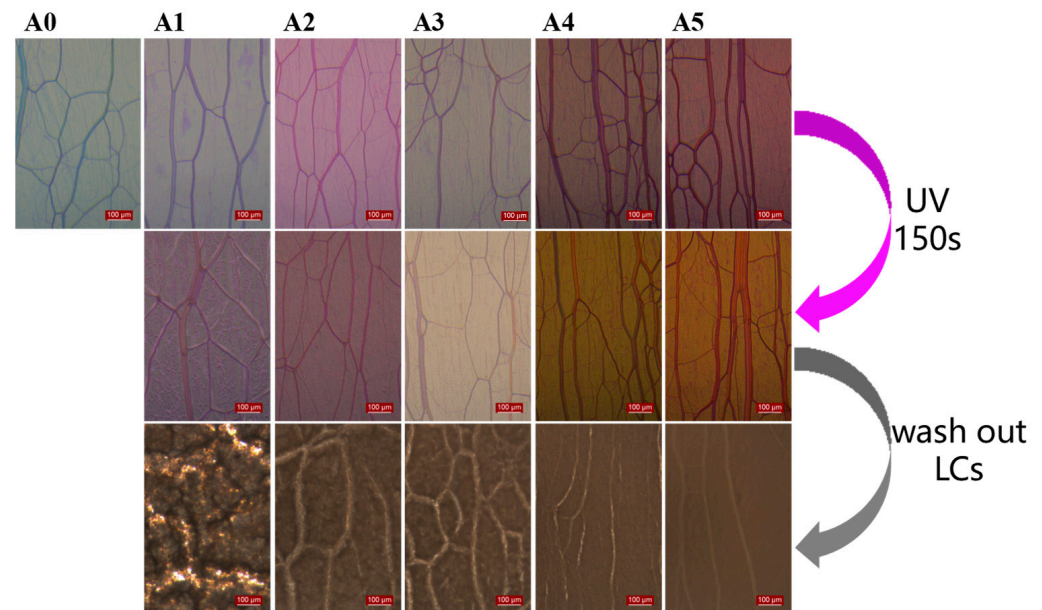


Figure 3. POM images of samples A0–A5 before and after UV irradiation and after washing out the bulk LCs.

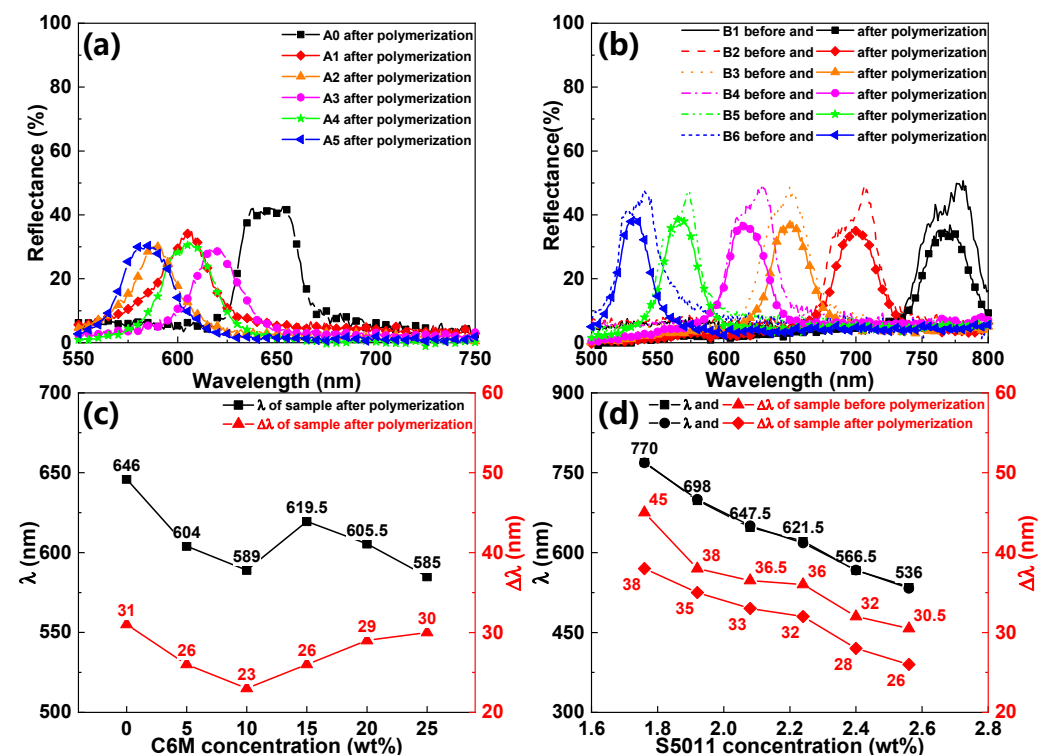


Figure 4. (a) The reflection spectra and (c) reflection bandwidth of samples A0–A6 after polymerization; (b) the reflection spectra and (d) reflection bandwidth of samples B1–B6 before and after polymerization.

According to Equations (1)–(3), with an increase in the chiral dopant content, the central reflection wavelength of the samples showed a blue shift, and the reflected bandwidth gradually narrowed. For samples B1–B6, the bandwidth gradually decreased because

the larger the concentration of chiral compounds, the smaller the gradient formed in the process of molecular migration, as shown in Figure 4b,d. The central wavelength of sample B3 was 647.5 nm, which was close to the central wavelength of red light. So, sample B3 was taken as the research object to study the influence of different conditions on the reflection performance of CLC films.

3.2. Effects of UV Intensity and Irradiation Time on the Reflection Properties of CLC Films

Sample B3 was set to investigate the effects of UV intensity and irradiation time on the reflection bandwidth of CLC films. It was poured into several cells and polymerized for 150 s under different UV intensities at 60 °C. The reflection bandwidths of the samples first increased and subsequently decreased with increasing UV intensity, as shown in Figure S1 (see supplementary materials). The increase in the reflection bandwidths of 3 mW/cm² was caused by the low density polymer network, which was not enough to fix the pitch at low light intensity. When the light intensity exceeded 3 mW/cm², the polymerization rate of the monomer became faster, and the monomer diffusion was no longer dominant [36]. Subsequently, the pitch gradient of the liquid crystal film was reduced, and the reflection bandwidth became narrower with the light intensity.

Sample B3 was poured into the LC cell and irradiated with 3 mW/cm² UV light at 60 °C, and the reflection spectra were measured every 10 s, as shown in Figure 5. With the extension of the irradiation time, the reflection bandwidth gradually narrowed. The reflection bandwidths at polymerization times of 120 and 150 s were the same, which proved that the polymerizable monomer was completely polymerized. Furthermore, the reflection bandwidth did not narrow down, reaching the narrowest reflection bandwidth of approximately 30 nm. The $\Delta\lambda$ in all reflection spectra was calculated by the difference of the reflection wavelengths at $(R_{\max} + R_{\min})/2$. $R_{\max}$ and $R_{\min}$ are the maximum reflectivity and minimum reflectivity of the sample, respectively.

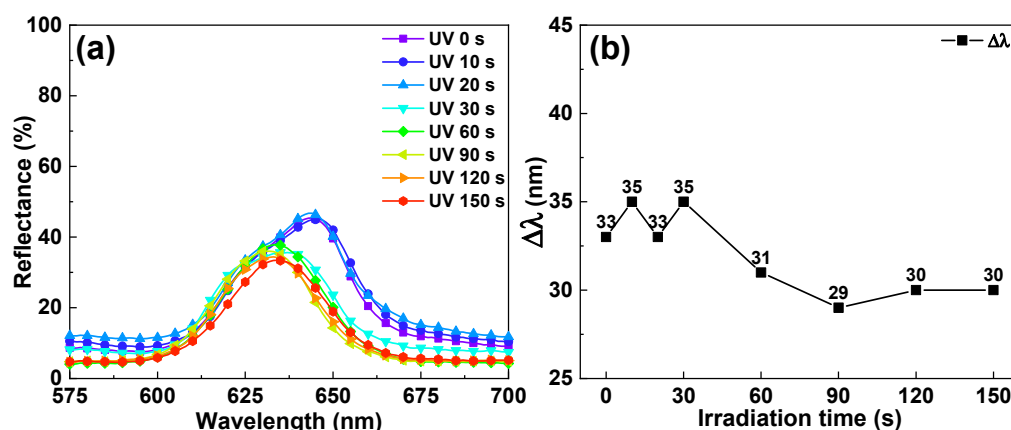


Figure 5. The (a) reflection spectra and (b) reflection bandwidth of sample B3 irradiated for different times.

3.3. Effects of the Temperature on the Reflection Properties of CLCs

Sample B3 was added to the LC cell, and six identical samples were irradiated by 3 mW/cm² UV light at different temperatures from 67 to 40 °C for 150 s. As shown in Figure 6, the pitch gradient of sample B3 was larger and the reflection bandwidth increased from 27 to 35 nm as the polymerization temperature decreased from 65 to 40 °C, respectively. The diffusion and polymerization rates of polymerizable monomers decreased with the decrease in the temperature. Because of unilateral illumination, the polymerization rate on both sides of the liquid crystal cell was not uniform, leading to a diffusion of the monomer. The pitch gradient of the polymerized system was larger than that of the non-polymerized system, resulting in a wider reflection bandwidth.

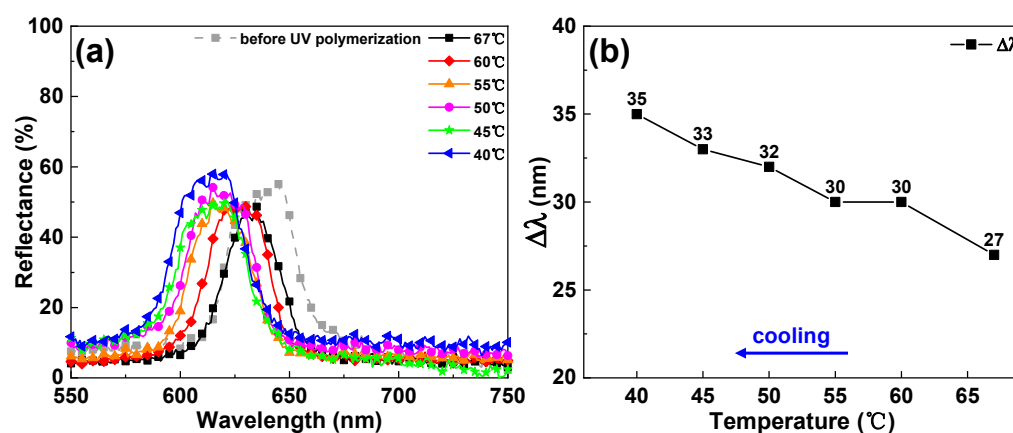


Figure 6. The (a) reflection spectra and (b) reflection bandwidth of sample B3 at different temperatures.

3.4. Selective Reflection Properties and Memory Effect of Polymer Networks

To demonstrate the formation of polymer networks and complete washing out of LCs in the polymer networks, the polymerization and washing-out processes of sample B3 with *n*-hexane for 7 days were analyzed by FT-IR and SEM. As shown in Figure S2a, the absorption peaks of acrylate groups at 1635 and 1411 cm^{-1} disappeared after polymerization, indicating that the sample was completely polymerized. After washing out, the characteristic peak (2233 cm^{-1}) of the cyano group in HNG-720600 disappeared, (see Figure S2b), indicating that the unreacted N*-LCs in this system were completely washed out.

SEM photographs of the polymer networks with different monomer concentrations after immersion in *n*-hexane for 7 days are shown in Figure 7. Smooth polymer networks can be observed for samples containing less than 5 wt% C6M; this was attributed to the fact that the monomers dissolved well in this system, and the free radical chain polymerization occurred. When the C6M content reached approximately 10 wt%, precipitation polymerization occurred, owing to the dissolution limit of C6M in the system, forming a rice-grain-like network. As the C6M was liquid crystalline, the molecular arrangement before polymerization was a self-organizing helical structure interspersed between the N-LC molecules. The helical structure was fixed rather than damaged during the polymerization process and retained after washing out the N-LCs. Furthermore, the higher the monomer concentration, the denser the polymer network and smaller the pores [25,37].

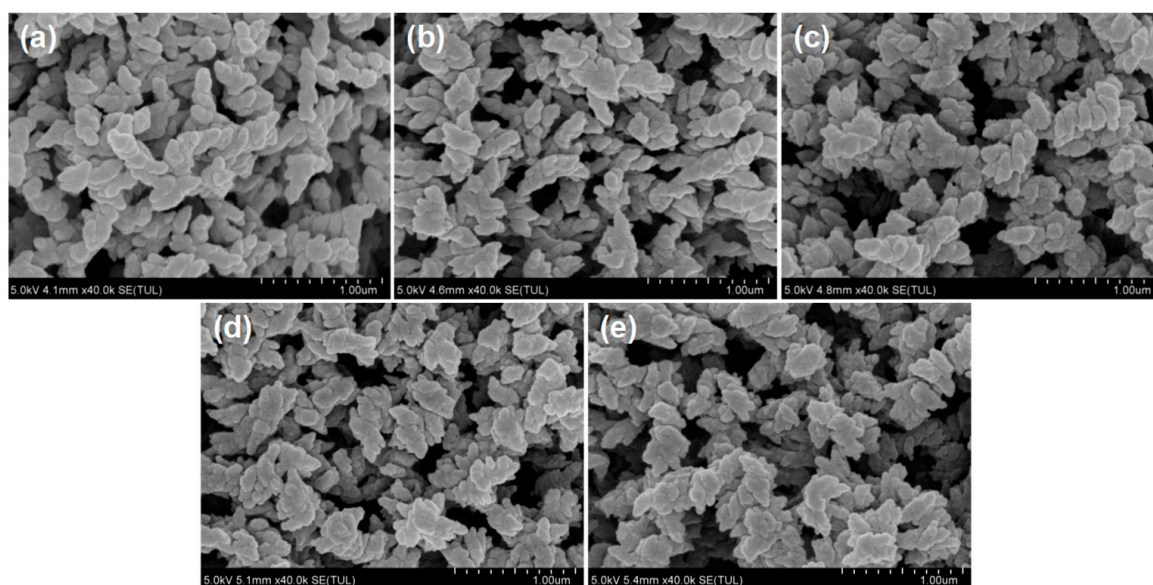


Figure 7. (a–e) SEM photographs of samples A1–A5 after immersion in *n*-hexane for 7 days.

After washing out, only the polymer scaffold remained. The reflection spectra of samples B1–B6, refilled with 720600 and opposite CLC B1'–B6', and the reflection spectra of sample B3, refilled with different LCs and isotropic liquid toluene ($n = 1.490$, 633 nm, 25 °C) were measured, as shown in Figure 8. After refilling with the small LC molecules of 720600, the polymer network showed a memory effect that samples B1–B6 reproduced on the reflected crest before washing out, as shown in Figure 8a. Samples B1'–B6' were designed, and the configuration of the anti-chiral CLCs had allocation ratios of 720600/S5011 = 98.17/1.83, 97.97/2.03, 97.84/2.16, 97.71/2.29, 97.52/2.48, and 97.37/2.63, respectively. However, when the opposite chiral CLCs were refilled, the permeability was not as high as that of pure nematic LCs. The effect of the total reflection CLC films was not as expected, which was attributed to the network being too rigid and dense for the opposite chiral CLCs with good stability and relatively poor fluidity to pass through.

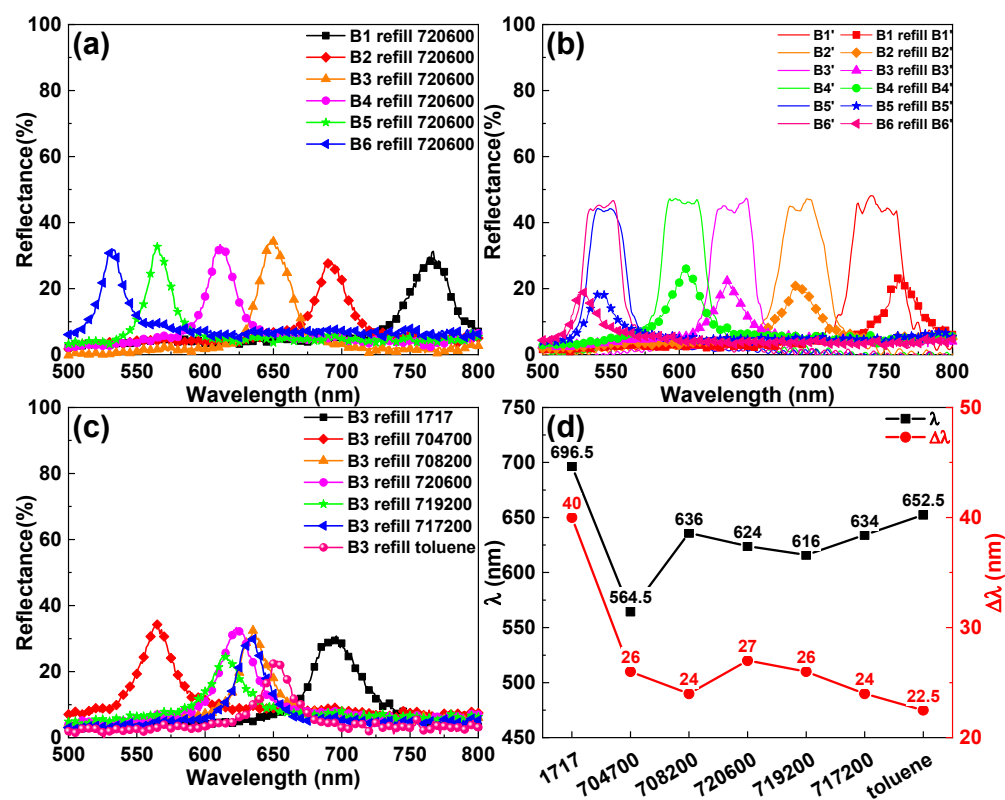


Figure 8. The reflection spectra of samples B1–B6 after refilling (a) 720600, (b) 720600 with opposite chiral labeled by B1'–B6', (c) the reflection spectra and (d) reflection bandwidth of sample B3 after refilling different LCs and isotropic liquid, toluene.

Figure 8c shows the normalized reflectance of CLC films refilled with different LCs. The Δn values of the refilled LCs are listed in Table 2. Figure 8c indicates that refilling different nematic LCs and toluene reproduced the reflective bands before washing out, which demonstrated the memory effect of the polymer network prepared in this study. As shown in Figure 8d, the λ of the CLC films refilled by 1717, 704700, 708200, 720600, 719200, and 717200 were centered at 696.5, 564.5, 636, 624, 616, and 634 nm, and the bandwidths were $\Delta\lambda_{1717} = 40$ nm (716.5–676.5 nm), $\Delta\lambda_{704700} = 26$ nm (577.5–551.5 nm), $\Delta\lambda_{708200} = 24$ nm (648–624 nm), $\Delta\lambda_{720600} = 27$ nm (637–610 nm), $\Delta\lambda_{719200} = 26$ nm (629–603 nm), and $\Delta\lambda_{717200} = 24$ nm (646–622 nm), respectively. In contrast, for the CLC film refilled by isotropic liquid, such as toluene, the λ values of the sample were centered at 652.5 nm, and the bandwidth was as narrow as 22.5 nm (663.5–641 nm); this was the narrowest reflection bandwidth ever reported for a CLC film. These results indicated that with the decrease in the birefringence (Δn) of the refilled material, the corresponding bandwidth of the CLC film was considerably reduced. The experimental results were consistent with the theoretical

expectation, where the bandwidth in the reflection spectrum of the CLC film is proportional to the birefringence of the refilled materials: $\Delta n \cdot p = (n_e - n_o) \cdot p$, where n_o and n_e are the ordinary and extraordinary refractive indices of the refilled material, respectively. For the same color with a similar pitch (p), the smaller the birefringence (Δn) of the refilled material used, the narrower the bandgap obtained.

Table 2. The refraction index of refilling LCs.

LC	1717	704700	708200	720600	719200	717200
n_e	1.720	1.556	1.553	1.547	1.517	1.513
n_o	1.519	1.476	1.475	1.477	1.474	1.474
Δn	0.201	0.080	0.078	0.070	0.043	0.039

POM images and reflection spectra of sample B3 are shown in Figure 9. The planar texture of the sample was intact before and after polymerization, washing out, and refilling, as shown in Figure 9a–d. The central reflection wavelength of sample B3 was 647.5 nm before polymerization and 649 nm after polymerization and refilling, as shown in Figure 9e. The central reflection wavelength was nearly constant, which proved that the polymer network was well preserved.

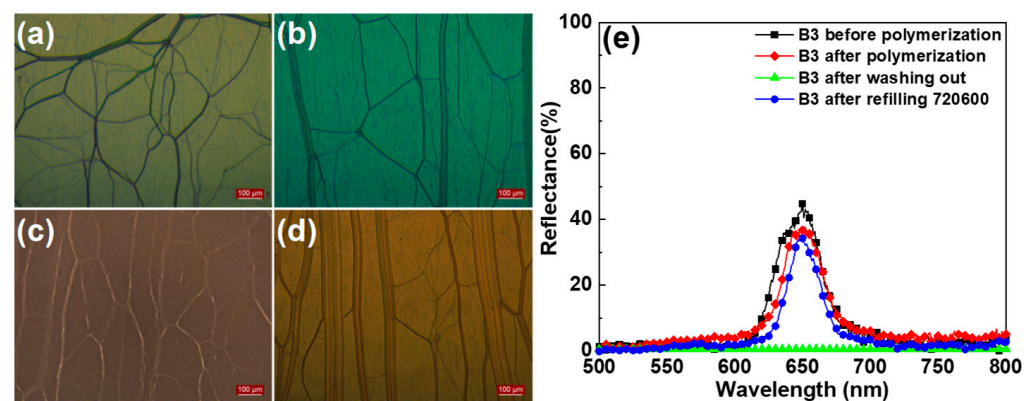


Figure 9. (a) POM images of sample B3 with planar texture before the polymerization, (b) after the polymerization, (c) after washing out, and (d) after refilling. (e) The reflection spectra of sample B3 during these processes.

4. Conclusions

In summary, a narrow-bandwidth CLC film with memory effect was prepared using the wash out/refilling method, and the effects of each component content, intensity of UV light, polymerization time, and polymerization temperature on the reflection bandwidth of the CLC system were studied. During the washing-out/refilling process, the selective reflection property was reproduced in the washed polymer network because of the memory effect. Furthermore, different materials, from liquid crystals with an anisotropic refractive index to toluene with an isotropic refractive index, were refilled into polymer scaffolds with helical structures that originated from the periodic arrangement of CLCs. The bandwidth was further narrowed to 24, 26, 27, 24, 26, and 40 nm by refilling small molecules LC 717200, 719200, 720600, 708200, 704700, and 1717, respectively, with a birefringence index ($\Delta n = 0.039, 0.043, 0.045, 0.070, 0.078, 0.080$, and 0.201). The narrowest bandwidth of 22.5 nm was obtained by refilling toluene with an isotropic refractive index ($\Delta n = 0$). This study provides a new approach for the preparation of narrower reflective films for applications in flexible reflective displays, color filters, printing, and colored cladding of a variety of objects.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cryst13050787/s1>, Figure S1: The (a) reflection spectra and (b) reflection bandwidth of sample B3 under different polymerization light intensities; Figure S2: FT-IR spectroscopy of sample B3 (a) before and after polymerization, (b) before and after washing out.

Author Contributions: Conceptualization, Z.X. and W.Z.; methodology, Z.X. and Y.C. (Yinjie Chen); software, W.Z., Y.C. (Yuchen Cui) and Y.G.; validation, Y.C. (Yifan Chang) and B.L.; formal analysis, Y.C. (Yinjie Chen) and Z.Z.; investigation, Z.X. and L.X.; resources, Z.X. and Y.C. (Yinjie Chen); data curation, Y.C. (Yifan Chang) and B.L.; writing—original draft preparation, L.X.; writing—review and editing, Z.X. and L.X.; visualization, Y.C. (Yuchen Cui) and Y.G.; supervision, Y.C. (Yinjie Chen) and W.Z.; project administration, Y.C. (Yinjie Chen), Z.Z. and L.L.; funding acquisition, Y.C. (Yinjie Chen) and L.L. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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