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## Liquid Crystals

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### Characteristics of wide-band reflection of polymer-stabilised cholesteric liquid crystal cell prepared from an unsticking technique

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## Characteristics of wide-band reflection of polymer-stabilised cholesteric liquid crystal cell prepared from an unsticking technique

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An unsticking technique was applied for the fabrication of a polymer-stabilised cholesteric liquid crystal (PSCLC) cell. The template of polymer matrix in the planarly aligned cholesteric liquid crystal (Ch-LC) was perfectly preserved by lifting off the hydrophilic antisticking substrate. The desired PSCLC cell could then be obtained by injecting a third Ch-LC sample between two designed templates of PSCLC layer. By adjusting the pitch distribution in the two stacking templates, a reflectivity of nearly 50% incident light could be yielded and the bandwidth of the reflection spectrum could be controlled accurately in the PSCLC cell. Thus, a method to modulate the wide-band reflection of 50% visible light flux range was offered.

**Keywords:** chiral dopant; cholesteric liquid crystal; crosslink; monomer

### 1. Introduction

Polymer-stabilised cholesteric liquid crystals (PSCLCs) have attracted much attention in the past few years due to their extensive applicability to display devices and the fundamental insights they offer into liquid crystal/polymer composites (1–8). Cholesteric liquid crystals (Ch-LCs) possess a helical structure and exhibit two stable states at zero field: the focal conic texture and the planar texture. In the focal conic texture, they scatter light in forward directions, whereas in the planar texture, they reflect circularly polarised light with the same handedness as the helical axis. The reflection bandwidth,  $\Delta\lambda$ , is given by  $\Delta\lambda = \Delta n \times p$ , where  $p$  is the cholesteric pitch corresponding to length over which the director rotates  $360^\circ$ , and  $\Delta n = n_e - n_o$  is the birefringence of the LC. Since  $\Delta n$  value for colourless organic material is generally smaller than 0.3, the bandwidth of a single-pitch Ch-LC in the visible region is less than 100 nm. However, for some specific applications such as full-colour or white-or-black reflective displays, wide-band reflective polarisers are desired. Recently, much attention has focused on non-uniform pitch distribution in PSCLC to obtain a wide-band reflection effect (9–24).

The unsticking technique, having been used in fabricating dichroic sponge polymer dispersed liquid crystal (D-SPDLC) films (25), was introduced into the preparation of wide-band reflective cells in this study. The polymer matrix template was sandwiched between two different substrates: a hydrophilic antisticking

substrate and a hydrophobic sticking substrate. By lifting off the antisticking substrate, the polymer layer was left on the sticking substrate. The unsticking technique can be realised providing that the polymer network's adhesion strength on the antisticking substrate is weaker than that to the sticking substrate and also weaker than the network's mechanical strength.

The reflection intensity of the PSCLC cell decreases when Ch-LCs possess the focal conic texture, so a means of avoiding the focal conic texture in the PSCLC cell is important. Binet *et al.* (26) show that larger bandwidths are achieved by associating two layers of PSCLCs. The confusion of the molecular arrangement at the juncture of PSCLC layers may cause the decrease of transmitted light intensity. In this paper, the proposed unsticking technique could solve the above problem because it perfectly preserves the planarly aligned Ch-LC by lifting off the hydrophilic antisticking substrate. The prepared PSCLC cells have a wide-band reflection effect covering the visible spectrum. Moreover, this technology provides technical support for wide-band reflection applications such as brightness enhancement films.

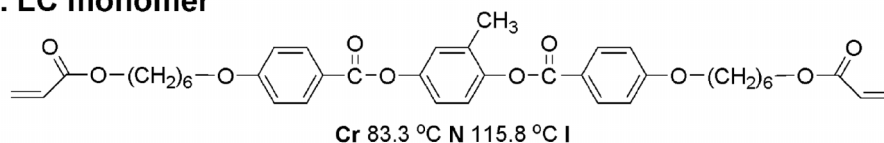
### 2. Experiments

#### 2.1 Materials

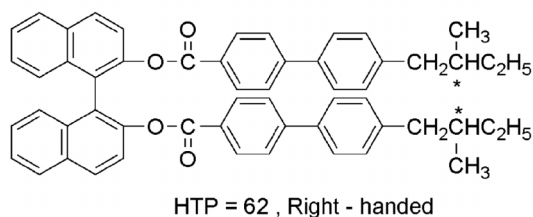
In this study, LC, (SLC-1717,  $n_o = 1.519$ ,  $n_e = 1.720$ ,  $\Delta n = n_e - n_o = 0.201$ ,  $T_{N-I} = 92^\circ\text{C}$ , Shijiazhuang Yongsheng Huatsing Liquid Crystal Co. Ltd.), LC

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### 1. LC monomer



### 2. Chiral dopant



### 3. Silane

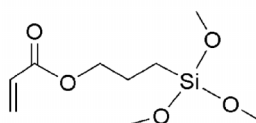


Figure 1. Chemical structure of LC monomer (C6M), chiral dopant and silane.

monomer, 1, 4-di-[4-(6-acryloyloxy) hexyloxy benzoxy]-2-methyl benzene (C6M), chiral dopant, (+)-1, 1'-binaphthyl-2, 2'-diyl bis(4'-(2-methylbutyl)biphenyl-4-carboxylate), silane surface coupling agent, 3-(acryloyloxy) propyltrimethoxysilane (TCI Co. Ltd.) and photoinitiator, 2, 2-dimethoxy-1, 2-diphenyl-ethanone (IRG651, TCI Co. Ltd.) were used. Figure 1 shows the chemical structures of C6M, the chiral dopant and silane surface coupling agent, respectively. The above chiral dopant was laboratory synthesized. C6M was synthesized according to the method suggested by Broer *et al.* (27). To prepare the desired compounds, methylene chloride ( $\text{CH}_2\text{Cl}_2$ ) was heated over calcium hydride and then distilled. All other

chemicals were commercially available and used without further purification. Figure 2 shows typical synthetic routes of the chiral dopant.

#### 2.2 Synthesis of the chiral dopant

##### 2.2.1 (+)-4'-(2-methylbutyl) biphenyl-4-carboxylic acid (a)

A mixture of (+)-4'-(2-methylbutyl)biphenyl-4-carbonitrile (12.5 g, 0.05 mol), NaOH (16.0 g, 0.4 mol) and 80 ml  $\text{H}_2\text{O}$  was dissolved to 400 ml diethylene glycol in a 1000 ml two-necked flask and heated under  $180^\circ\text{C}$  for 48 h. After the

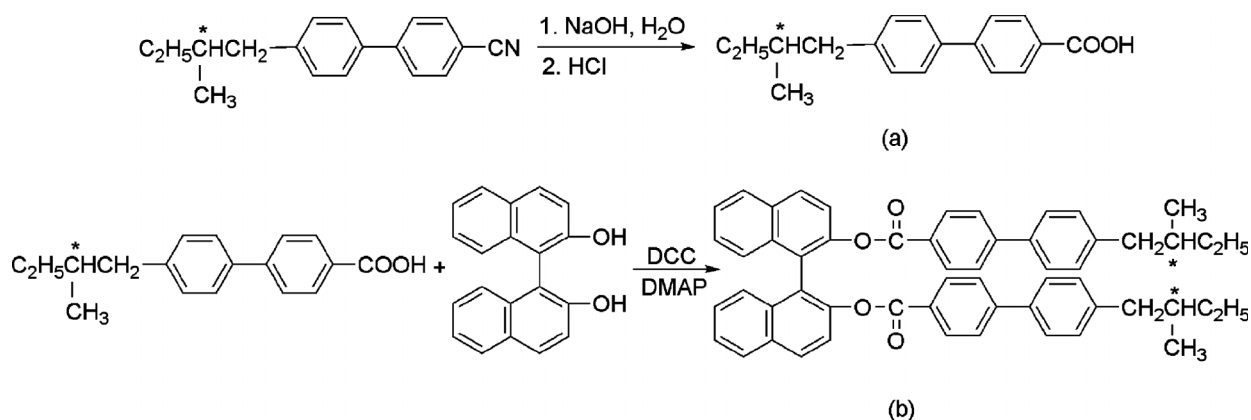


Figure 2. The synthetic routes of the chiral dopant.

reaction, crude product was precipitated when the mixture was acidified to pH 3.0 with hydrochloric acid. The crude product obtained was then washed twice with 300 ml of water and recrystallised from 95% alcohol.

Characterisation Data: White crystal; yield 11.2 g (83.2%), IR (KBr,  $\text{cm}^{-1}$ ): 2531–3022 (acidic -OH stretching), 1678 ( $\text{C}=\text{O}$  stretching).  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 8.19–8.17 (2H, d), 7.71–7.70 (2H, d), 7.57–7.56 (2H, d), 7.27–7.26 (2H, d), 2.72–2.67 (1H, q), 2.46–2.41 (1H, q), 1.71–1.69 (1H, m), 1.43–1.42 (1H, m), 1.23–1.20 (1H, m), 0.96–0.89 (6H, m).

### 2.2.2 (+)-1, 1'-binaphthyl-2, 2'-diyl bis (4'-(2-methylbutyl) biphenyl-4-carboxylate) (b)

(+)-1, 1'-binaphthyl-2, 2'-diol (2.9 g, 0.01 mol), (+)-4'-(2-methylbutyl) biphenyl-4-carboxylic acid (5.9 g, 0.022 mol), N, N'-dicyclohexyl carbodiimide (DCC) (4.5 g, 0.022 mol) and 4-pyrrolidino-pyridine (DMAP) (0.12 g, 0.001 mol) were dissolved in 200 ml dry  $\text{CH}_2\text{Cl}_2$  at room temperature and heated to 40°C under magnetic stirring for 48 h. After the resulting solution was evaporated, the crude product was obtained and then purified by column chromatography (silica gel, ethyl acetate/hexane = 1/3) and recrystallisation with ethanol (EtOH).

Characterisation Data: White crystal; yield 5.2 g (66.2%), IR (KBr,  $\text{cm}^{-1}$ ): 1736 ( $\text{C}=\text{O}$  stretching),  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 8.00–7.98 (2H, d), 7.92–7.90 (2H, d), 7.70–7.69 (4H, d), 7.60–7.58 (2H, d), 7.48–7.36 (12H, m), 7.35–7.32 (2H, t), 7.25–7.19 (4H, d), 2.68–2.64 (2H, q), 2.42–2.37 (2H, q), 1.69–1.64 (2H, m), 1.44–1.38 (2H, m), 1.22–1.15 (2H, m), 0.94–0.79 (12H, m).

### 2.3 Characterisation and measurements of samples

The synthesised compounds were characterised by FT-IR (Perkin Elmer Spectrum One) with wave number ranging from 400 to 4000  $\text{cm}^{-1}$ ,  $^1\text{H-NMR}$  (Bruker DMX-400 spectrometer) with  $\text{CDCl}_3$  as the solvent, polarising optical microscopy (POM) (Olympus BX51) with a hot stage calibrated to an accuracy of  $\pm 0.1^\circ\text{C}$  (LinkamTHMS-600), and differential scanning calorimetry (DSC), (Perkin Elmer Pyris 6) with heating rate of  $10^\circ\text{C}/\text{min}$  and cooling rate of  $10^\circ\text{C}/\text{min}$ . The reflection spectra was obtained by UV/VIS/NIR spectrophotometry (JASCO V-570) at normal incidence. The morphology of the polymer network was observed by scanning electron microscopy (SEM) (Leicas 440I). The water contact angle was measured on a contact angle system OCA20 (DataPhysics, Germany).

### 2.4 Preparation of samples

The hydrophilic antisticking substrate was obtained by dipping the substrate in potassium dichromate solution for 24 h and then cleaning it thoroughly using deionised water. The hydrophobic sticking substrate was treated with silane surface coupling agent and as detailed in the following treatment: the typical deposition procedure is to clean the substrate thoroughly so as to remove all the organic and inorganic residues, dip the substrate in a dilute solution of the silane surface coupling agent (0.5% by volume in water), and agitate at room temperature for about 10 min; the substrate is then rinsed in deionised water to remove excess silane; then, excess water is blown free with  $\text{N}_2$ ; finally, the silane coating is further cured at  $110^\circ\text{C}$  for 1 h.

The compositions of the studied samples are listed in Table 1. The LC, C6M, chiral dopant and photo-initiator system was mixed by solvent cast method from methylene chloride solutions and then filled into the cells by capillarity. The phase transition temperatures of samples 1–4 were measured by DSC. The composite exhibited a planar texture when it was filled into cells under homogeneous boundary conditions. PET (polyethylene terephthalate) films (10  $\mu\text{m}$  thick) were used as cell spacers. The samples were prepared by irradiating with UV light (1  $\text{mW}/\text{cm}^2$ , 365 nm) for 1 h at  $25^\circ\text{C}$ , which results in the polymerisation of C6M and silane coupling agent.

Figure 3 shows the mechanism of preparing the desired cell. The hydrophilic antisticking substrate was slowly separated from the PSCLC layer (Figure 3(a)). Another PSCLC layer, with a different chiral dopant concentration, was fabricated in the same way. The two sticking substrates with the PSCLC layer were combined to make a new cell of 30  $\mu\text{m}$  thickness (Figure 3(b)). A third sample with a chiral dopant concentration intervention of that in the PSCLC layers on the two sticking substrates was filled into the resulted cell by capillary action (Figure 3(c)). The cell was then irradiated for 1 h with UV light (1  $\text{mW}/\text{cm}^2$ , 365 nm) (Figure 3(d)). Thus a non-uniform pitch distribution in the cholesteric phase was realised due to the diversity of chiral dopant concentration from one substrate to the other, and was then fixed by the photopolymerisation of LC monomers.

Table 1. The compositions and the Ch-I phase transition temperatures ( $T_{\text{Ch-I}}$ ) of samples 1–4.

Sample	Weight ratio <sup>a)</sup>	$T_{\text{Ch-I}}$ ( $^\circ\text{C}$ ) <sup>b)</sup>
1	77.97 / 6.0 / 6.0 / 0.03	83.9
2	78.97 / 5.0 / 6.0 / 0.03	85.9
3	79.97 / 4.0 / 6.0 / 0.03	87.1
4	80.97 / 3.0 / 6.0 / 0.03	88.1

Note: <sup>a)</sup> Weight ratio: SLC-1717/chiral dopant/ C6M/IRG651

<sup>b)</sup>  $T_{\text{Ch-I}}$ : clearing temperature from cholesteric to isotropic phase

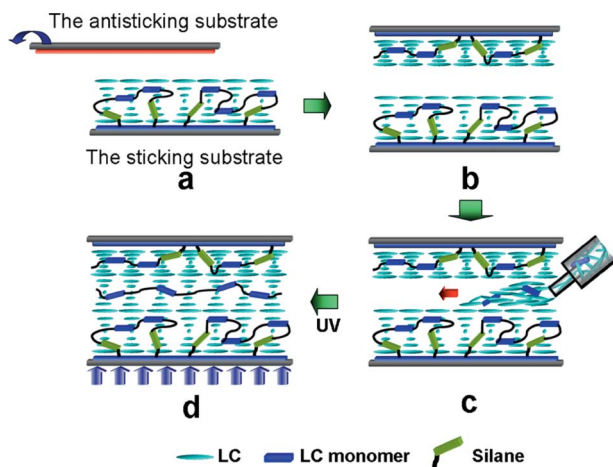


Figure 3. Schematic illustration of fabrication of a PSCLC cell by the unsticking technique. (a) The PSCLC layer was sandwiched by two substrates: antisticking substrate and sticking substrate. The antisticking substrate was lifted off. (b) Two sticking substrates with the preserved PSCLC layer were combined into a new cell. (c) The third sample consisting of LC, LC monomer, chiral dopant and photoinitiator was injected into the new cell. (d) A wide-band reflection effect was obtained.

### 3. Results and discussion

#### 3.1 Characterisation and helical twisting property of the chiral dopant

To enhance the effect of the chiral dopant diffusion, a chiral dopant with a large helical twisting power (HTP) value was synthesised. The synthesised chiral dopant was identified using FT-IR and  $^1\text{H-NMR}$ . Figure 4 shows the FT-IR spectrum of 4'-(2-methylbutyl) biphenyl-4-carboxylic acid (a) and 1, 1'-binaphthyl-2, 2'-diyl bis (4'-(2-methylbutyl) biphenyl-4-carboxylate) (b). The specific split absorptions around  $1736\text{ cm}^{-1}$  may be due to the existence of the two carbonyl groups. Figure 5 shows the  $^1\text{H-NMR}$  spectrum of the chiral dopant. Peaks for the protons of the molecule were all identified.

The HTP value of the chiral dopant can be calculated from the relationship between Ch-LC reflection locations and weight concentrations of the chiral dopant. As can be seen from Figure 6, the transmission/reflection peaks locate at 660, 470, 390, 360 and 345 nm when the weight concentrations of the chiral dopant are 4%, 5%, 6%, 7% and 8%, respectively. The reflectances and the transmittances of curves within the bandwidth are both close to 50%. With the increasing concentration of chiral dopant in the LC host of SLC-1717, the regions of reflectance and transmission shift to shorter wavelengths. Thus the HTP value of the chiral dopant can be calculated according to Equations (1) and (2):

$$p = [(HTP) \cdot X_c]^{-1} \quad (1)$$

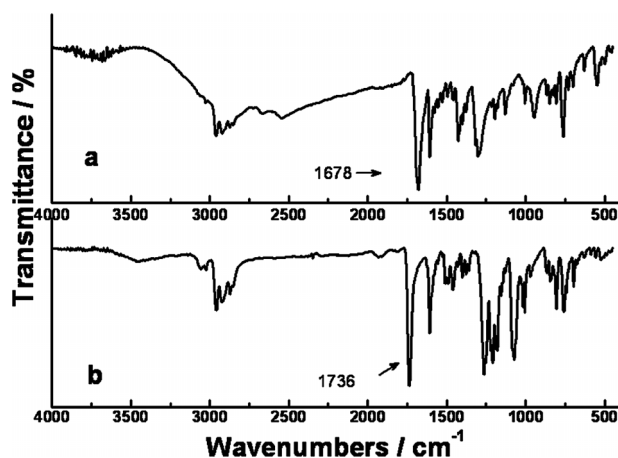


Figure 4. The FT-IR of 4'-(2-methylbutyl) biphenyl-4-carboxylic acid (a) and 1, 1'-binaphthyl-2, 2'-diyl bis (4'-(2-methylbutyl) biphenyl-4-carboxylate) (b).

$$\lambda = n \times p \quad (2)$$

Here,  $p$  is the pitch of Ch-LC,  $X_c$  is the weight concentration of chiral dopant,  $\lambda$  is the reflection bandwidth,  $n$  is the average birefringence of the LC. So the average HTP value calculated is about  $62\ \mu\text{m}^{-1}$ .

#### 3.2 UV-curing dependence on the substrate treated with silane

The usual method for producing a uniformly oriented thin film of LC samples is to generate substrate interfaces which have an orienting action on the LC molecules. These interfaces have been generated by a variety of means including chemical cleaning, rubbing, mechanical surface scribing or deformation, physical adsorption and deposition of organic substances. Silane surface coupling agents have been used to generate chemically stable interfaces which can be simply and reproducibly deposited on a wide variety of conducting and insulating substrates (28–30). Here, various substrates were prepared using the unsticking technique. The hydrophilic antisticking substrate treated with a potassium dichromate solution resulted in a contact angle (CA) of about  $12 \pm 2^\circ$ , as shown in Figure 7(a), and the hydrophobic sticking substrate treated by silane resulted in a CA of about  $94 \pm 2^\circ$  as shown in Figure 7(b).

Figure 8 shows a schematic illustration of photopolymerisation between molecules of C6M and the silane on the sticking substrate. Before polymerisation, the Ch-LC molecules exhibited a planer alignment while the LC monomers also aligned parallel to the substrates. The photopolymerisable C=C group

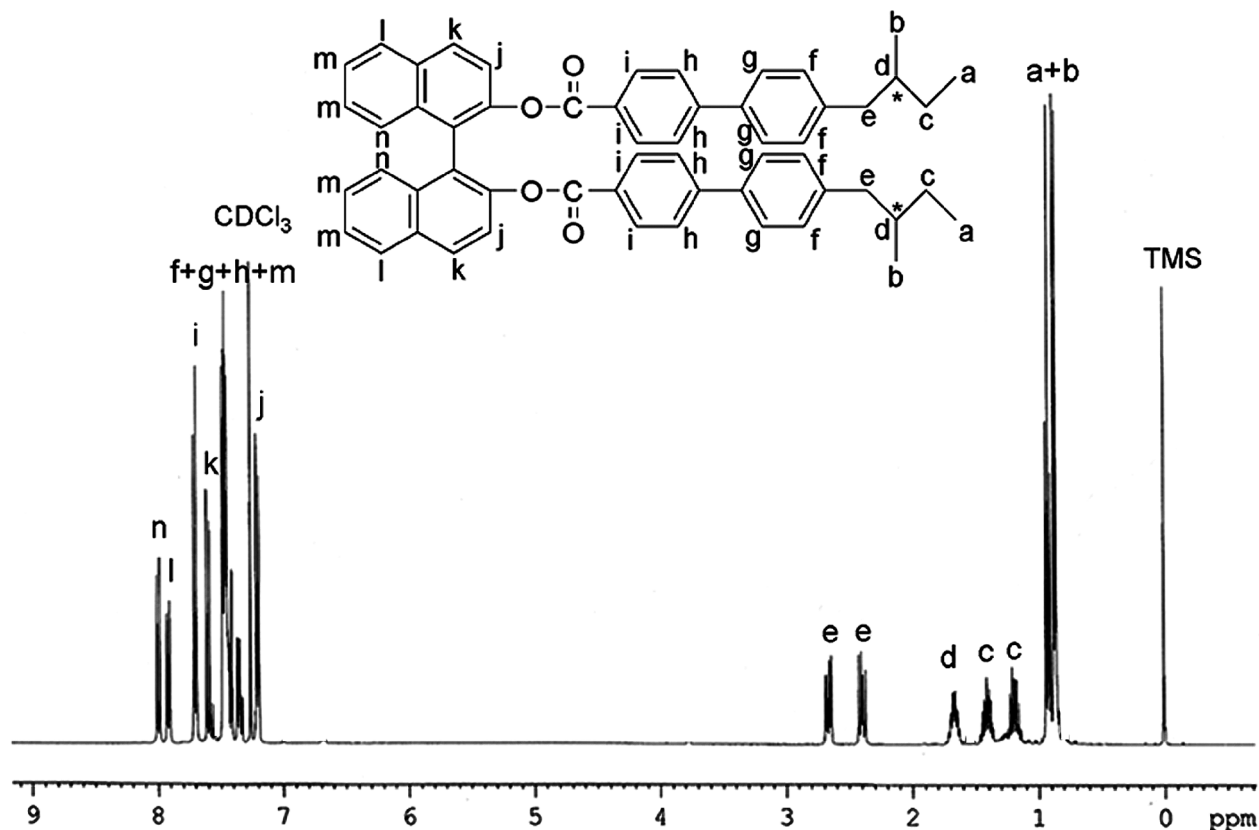
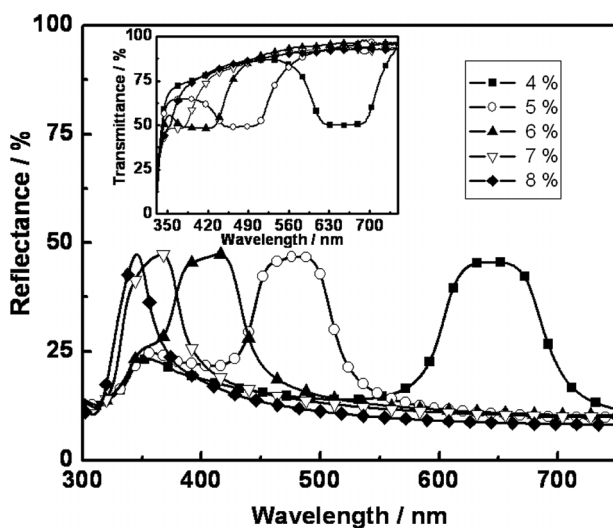
Figure 5. The  $^1\text{H}$ -NMR of the chiral dopant.

Figure 6. The reflectance and transmittance spectra of Ch-LCs with different concentrations of the chiral dopant as a function of wavelength.

of the silane surface coupling agent could crosslink with the LC monomer (C6M) under UV irradiation. The FT-IR technique gives complementary information about the crosslink reaction. The decay of the absorption bands of silane and C6M double band at  $1635\text{ cm}^{-1}$  was followed as a function (Figure 9). Thus

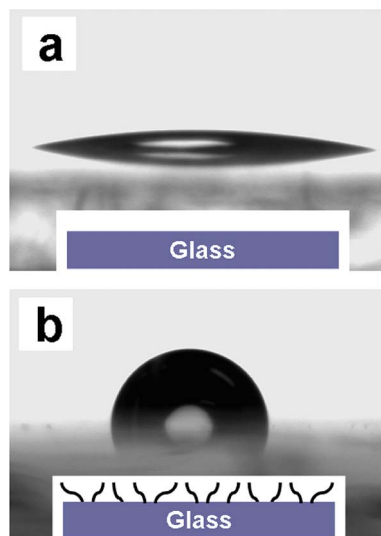


Figure 7. Photographs of water droplet shape on (a) the antisticking substrate and (b) sticking substrate.

the composite was tightly stuck to the sticking substrate and the planer texture was preserved by the network as well. By lifting off the hydrophilic antisticking substrate, the PSCLC layer was left on the hydrophobic sticking one.

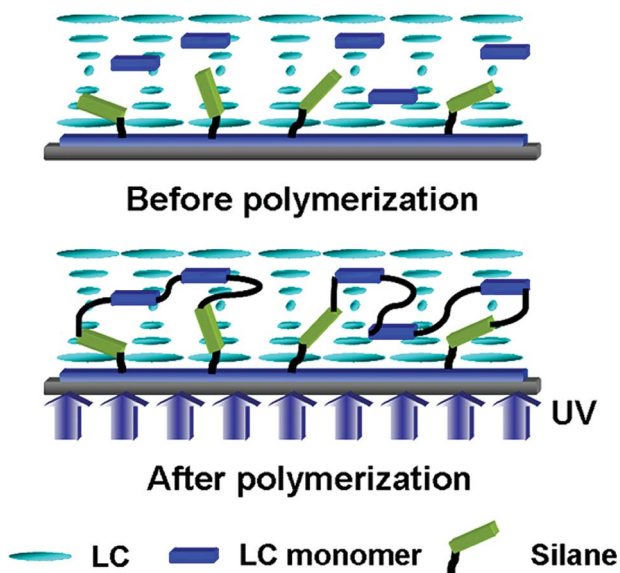


Figure 8. Schematic illustration of photopolymerisation between molecules of C6M and the silane on the sticking substrate.

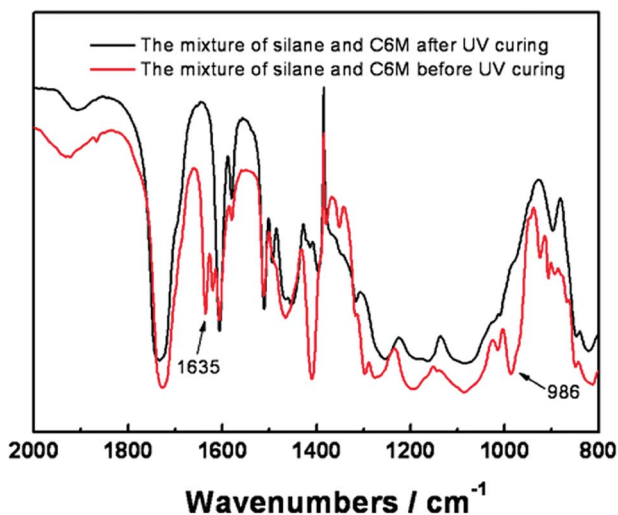


Figure 9. Decay of the stretching vibration bands of C=C double bond at  $1635\text{ cm}^{-1}$  after UV exposure ( $365\text{ nm}$ ,  $1\text{ mW/cm}^2$ ) for 10 min. The investigated mixture was composed of silane/C6M = 0.50/0.50 mol%.

### 3.3 Morphology of the PSCLC layer on the substrate

In order to further investigate the morphology of the PSCLC layer on the sticking substrate, structural information was obtained by both POM (Figure 10 (a), (b), (c) and (d)) and SEM (Figure 10(e)). Figure 10(a) shows the typical planar texture of sample 1 after polymerisation, while Figure 10(b) shows the PSCLC layers of sample 1 on the sticking substrate. Comparing Figures 10(a) and 10(b), it can be inferred that the unsticking technique preserved the planar texture of PSCLC layer

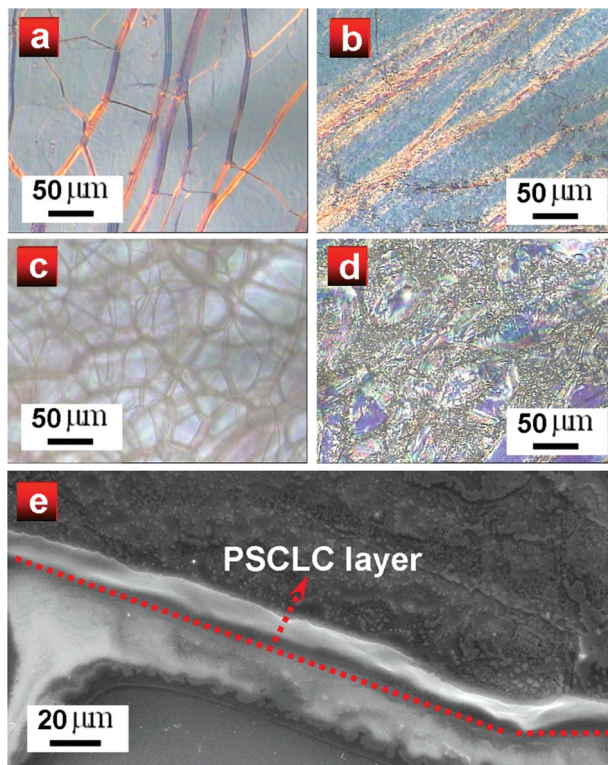


Figure 10. (a) Planar texture of PSCLC layer in sample 1 before lift-off. (b) Preserved planar texture of PSCLC layer in sample 1 after lift-off. (c) Planar texture of prepared PSCLC cell, combination A. (d) Broken planar texture of PSCLC layer in sample 1 after split method. (e) SEM photo of PSCLC layer with unsticking technique.

well. In the perfect planar texture, the Ch-LC helical axis is perpendicular to the cell surface. Thus the reflectance of incident light was hardly affected (3). When the third Ch-LC sample was injected, the perfect planar texture was retained owing to the crosslinking of monomers in the PSCLC layer, as shown in Figure 10(c). In contrast with the unsticking technique, the planar texture could be easily broken by splitting one substrate directly. It can be seen from Figure 10(d) that the planar texture is immingled with focal conic texture. Figure 10(e) shows the PSCLC layer morphology on the sticking substrate obtained by the unsticking technique. The surface of the PSCLC layer is smooth in the photo. The results show that the unsticking technique produced homogeneous lift-off without breaking the polymer network matrix, which results in uniform droplet size and distribution in the resultant PSCLC layer. PSCLC layers show a planar texture and focal conic texture prepared by the unsticking technique and splitting technique, respectively, as shown in Figure 11. In a planar texture, all the helical axes are arranged in the direction perpendicular to the substrate surfaces. If the pitch length is much larger or smaller than the wavelength of visible light, the cell will be transparent. In a focal conic texture, the helical axes

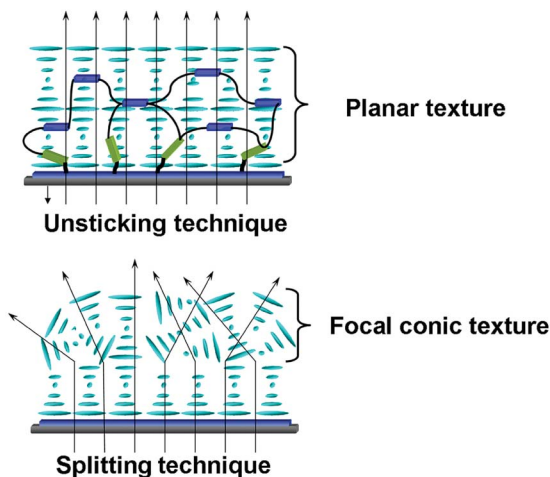


Figure 11. Schematic illustration of the unsticking technique and splitting technique.

are randomly arranged. This texture shows strong light scattering because of the discontinuous spatial change of the refractive indices at the domain boundaries, and it will decrease the reflective intensity of PSCLCs.

### 3.4 Reflection spectra of the PSCLC cell

A wide-band reflective PSCLC cell was prepared using the unsticking technique. Figure 12(a) shows the dependence of the reflection spectra of polymerised samples 1–4 on chiral dopant concentration. It was found that the bandwidths of samples 1–4 were located in the wavelength ranges of 381–435, 438–509, 531–609 and 728–832 nm, respectively, and the maximum reflection intensity is close to 50%. After the antisticking substrates in samples 1 and 3 were lifted off, the remaining PSCLC layers on sticking substrates were named as sample 1' (curve 1) and sample 3' (curve 2) as shown in Figure 12(b). The reflectance locations are about 371–442 and 521–595 nm, respectively. It should be noted that there was little change in reflectance spectra in Figures 12(a) and 12(b), indicating the reflection locations and bandwidth were little influenced by the unsticking technology. Samples 1' and 3' were then combined to make a new cell (curve 3), in which the reflectance location and bandwidth corresponded to those of the superposed sample 1' and sample 3'. Air bubbles emerging from the simply overlapped two samples could be avoided by siphoning the mixture (sample 2) into the new cell (called combination A here). The concentration of the chiral dopant in sample 2 was intervention of that in the PSCLC layers on the two sticking substrates. A wide-band reflectance cell was achieved via the diffusion of chiral dopant between sample 1' and sample 2 as well as sample 2 and sample 3' in combination A, as shown in curve 4. The reflective

wavelength ranges from 400 to 520 nm, which covers the reflective wavelength of samples 1, 2 and 3 with the reflection intensity of nearly 50%. Figure 12(c) shows the reflectance spectra of another cell (called combination B here) prepared with samples 3, 1' and 4' with the same procedure mentioned above. The reflective wavelength ranges from 400 nm to 740 nm, as shown in curve

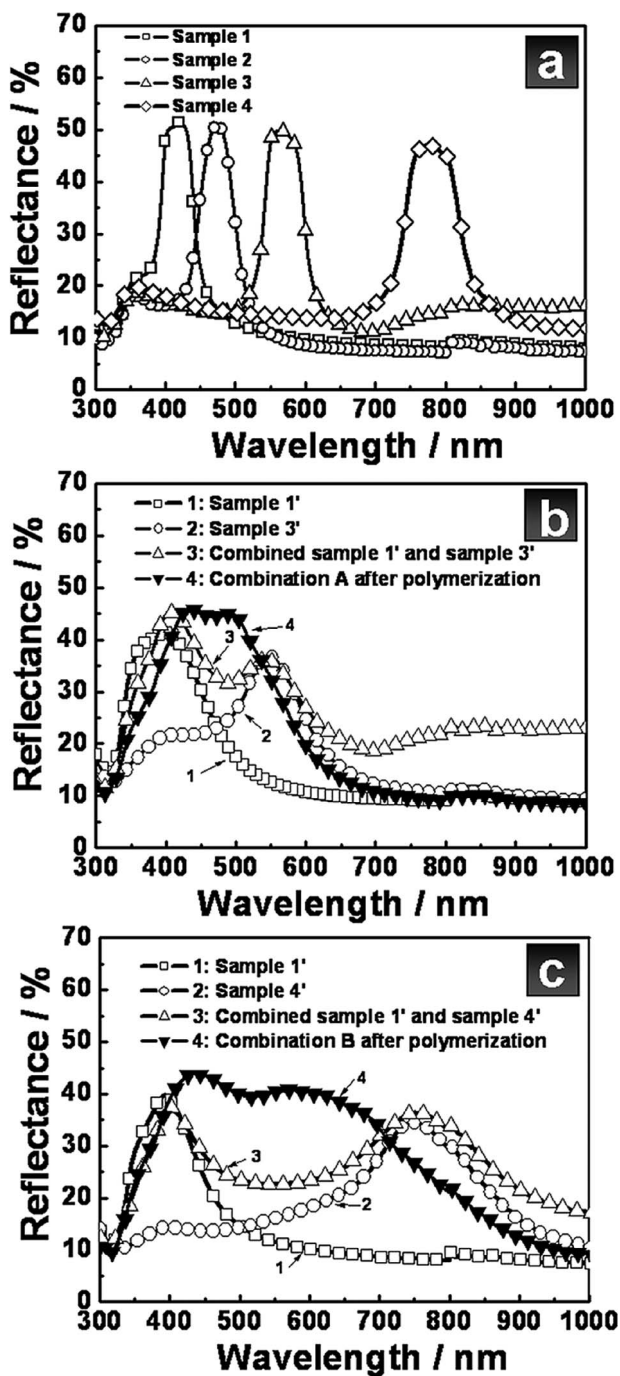


Figure 12. The reflection spectra of (a) samples 1–4 before lift-off. (b) Sample 1' and 3' after lift-off, cell combined by sample 1' and 3', combination A. (c) Sample 1' and 4' after lift-off, cell combined by sample 1' and 4', combination B.



4. Comparing Figures 12(b) and (c), it can be concluded that by combining the unsticking PSCLC layer with different chiral dopant concentrations, a stacking PSCLC cell with different reflection locations and bandwidths can be achieved.

#### 4. Conclusions

In summary, a novel chiral dopant with a large HTP value was synthesised and characterised. The unsticking technique dealing with hydrophilic antisticking substrates and hydrophobic sticking substrates was developed in preparing the air bubble-free and uniform PSCLC cell. The results show that the PSCLC layer on the sticking substrate exhibits a well-aligned Grandjean planar texture, and wide band reflection effect could be optimised via unsticking technology. By combining substrates with various PSCLC layers in which the concentration of the chiral dopant could be easily adjusted, accurate and controllable wide-band reflection cell was obtained.

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