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# Enhanced reflection band broadening in polymer stabilized cholesteric liquid crystals with negative dielectric anisotropy

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## Highlights

- Reflection band of polymer stabilized cholesteric liquid crystal can be greatly broadened.
- Ions are trapped on polymer networks.
- Polymer network moves under applied DC electric fields.
- Broadening of reflection bandwidth is enhanced by doping guest materials.

## Abstract

The <u>photonic</u> band gap of cholesteric <u>liquid crystal</u> with negative <u>dielectric</u> anisotropies can be broadened under DC electric voltages by polymer stabilization. The broadening i FEEDBACK  $\square$ 

broadening is strongly dependent on the <u>ion concentration</u> in the polymer stabilized cholesteric liquid crystal. We observed a linear relation between the broadening and the ion density. By using the optimal material formulation, we achieve 3 folds broadening to cover the entire visible light region with low voltages.



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## 1. Introduction

Cholesteric <u>liquid crystals</u> (CLCs) consist of chiral elongated molecules (or achiral elongated molecules with chiral dopants) whose long molecular axis twist in space around an orthogonal helical axis (called the helical axis) [1]. They are self assembled 1-D <u>photonic crystals</u> and are well known to exhibit selective reflection at the central wavelength  $\lambda = [(n_e + n_o)/2]P$  with the bandwidth  $\Delta \lambda = (n_e - n_o)P[1,2]$ , where *P* is the pitch,  $n_e$  and  $n_o$  are the extraordinary and ordinary <u>refractive</u> <u>indices</u> of the liquid crystal. Their unique feature of reflection is made used in many applications such as reflective displays [3,4], tunable color filters [5,6] and mirror-less lasers [7,8].

In applications, CLCs are usually sandwiched between two parallel substrates coated transparent conductive <u>electrode</u>. When a voltage is applied across the cell, the produced electric field is perpendicular to the substrate. When the liquid crystals are in the planar state, where the helical axis is perpendicular to the cell substrate and the molecules are parallel to the substrate, they exhibit the selective reflection. The applications of CLCs can be greatly expanded if their reflection can be electrically tuned. Although there have been many efforts [[9], [10], [11], [12]], it is very challenging to achieve electrical tuning of the reflection of CLCs composed from only low molecular weight materials. If the CLC has a negative <u>dielectric</u> anisotropy, the molecules remain parallel to the cell substrate and the reflection remains unchanged when a voltage is applied across the cell. If the CLC has a positive dielectric anisotropy, when a voltage is applied across the cell, the liquid crystal molecules reorient and either the helical axis tilted to parallel to the cell substrate or the <u>helical structure</u> is unwound, and then the reflection disappears [1,2]. One method to avoid the tilting of the helical axis is to use in-plane-switching (IPS) interdigitated electrodes, which are on the same substrate, where the generated electric field is parallel to the cell substrate. The drawback of this method is that there are dead areas (on top the electrodes), where the color is not tuned, and the driving voltage is high. Another method is to use electrostatic force to change the cell thickness, which has the problem of narrow tuning range and high driving voltage [9].

positive dielectric anisotropy, the liquid crystal molecules tend to align parallel to the electric field and thus perpendicular to the substrate when a voltage is applied across the cell. As a result of the competition between the aligning effects of the electric field and the polymer network, the liquid crystal form wrinkled cholesteric layer structure where the helical axis (which is perpendicular to the cholesteric layer) in some regions is tilted by an angle  $\theta$  away from the cell normal direction. For normal incident light with respect to the cell substrate, the angle between the incident light and the helical axis is increased to  $\theta$ , and the reflected color of the CLC is shifted to shorter wavelength according to the Bragg law  $\lambda = [(n_e + n_o)/2]P \cos \theta$ . This approach, however, has the drawback that the reflectance will decrease when the helical axis is tilted. Recently a more promising method is developed [19], which use CLCs with negative dielectric anisotropies and polymer stabilization. When a DC voltage is applied across the cell, the liquid crystal molecules remain parallel to the substrate while the dispersed polymer network moves, probably due to trapped ions on the polymer network. The polymer network has a strong aligning effect ('structural' chirality)on the liquid crystal. When the polymer network moves, it produces a pitch gradient such that the reflection band canbe broadened from 50 nm to more than 300 nm.

In this paper we report an experimental study of the effects of alignment layer, moisture and ionic additives on the polymer stabilized CLCs. Our results show that the reflection band broadening depends on the electric <u>resistivity</u> of the material: the broadening is enhanced when the resistivity is decreased. This verifies that the broadening is caused by the movement of the polymer network, because when the resistivity is lower, more ions are trapped on the polymer network and the electrostatic force on the polymer network is larger and thus the movement of the polymer network is larger under a given voltage. By using the optimal alignment layer and guest material, we are able to greatly enhance the broadening.

## 2. Experiment and results

The CLC used was a mixture consisting of 74.9% nematic host MLC-2079, 2.8% chiral dopant R1011, 15.9% chiral dopant R811, 5.3% <u>monomer</u> RM257 (all from Merck) and 0.7% <u>photo-initiator Benzoin</u> Methyl Ether (from Polyscience). The nematic host has the <u>dielectric</u> anisotropy of  $\Delta \varepsilon \approx -6.7$  and <u>birefringence</u> of 0.15. The mixture was filled into cells consisting of two parallel <u>glass substrates</u> coated with <u>indium-tin-oxide</u> (ITO) <u>electrodes</u>. A solution of <u>polyimide</u> PI2555 (Nissan Chemical) and thinning solvent was spin coated on top of the ITO and rubbed for homogeneous alignment of the <u>liquid crystal</u>. The concentration of PI2555 was 10% except otherwise specified. The cell thickness controlled by 10 µm spacers. The size of the cell was  $3.0 \times 2.5$  cm<sup>2</sup>. The cells were cured under UV light with intensity of 3 mW/cm<sup>2</sup> for 45 min to



Some liquid crystal devices are sensitive to moisture inside the cells. We explored the effects of moisture on the reflection band broadening of the polymer stabilized CLC. We made empty cells in the same way and then divided them into three groups. For the first group, within a few houses after the fabrication, cells were filled with the liquid crystal/monomer mixture and cured under UV light. For the second group, the cells were kept in a dry oven for a few days, and then were filled and cured. For the third group, the cells were kept in a humidity chamber, which contained a cup of water and was kept at 60 °C, for a few days. During this process, moisture was absorbed into the cells. The cells were then filled and cured. The broadening of the cells from the first and second groups were the same and is shown in Fig. 1(a). The reflection bandwidth was increased from 50 nm to 120 nm when 35 V was applied. The broadening of the cell from the third group was quite different and is shown in Fig. 1(b). The reflection bandwidth was only increased slightly when the same 35 V voltage was applied. According to the model that the broadening is caused by the movement of the dispersed polymer network, the possible factors responsible for different broadening are the polymer network and ion density in the material. We found that the polymer network was not affected by the moisture by SEM study (which will be discussed in details later in this paper). We then checked the ion density by measuring the resistivity spectra of the filled cells. 0.3 V was applied in the measurement and the frequency was varied. The results are shown in Fig. 2. The resistivity of the cell kept in the dry oven was lower and thus had more ions. The polymer network moved more under a given applied voltage, resulting in a wider broadening. How the absorbed moisture change the resistivity is, however, not clear at this moment. A probable reason is that ions originally in the liquid crystal diffuse into the absorbed moisture which phase separates from the liquid crystal. In future experiments, the empty cells were freshly made and thus do not absorb moisture.

(2) Effect of alignment layer thickness





Fig. 1. Reflection spectra of the polymer stabilized CLC cells at 0 V and 35 V. (a) empty cell stored in dry oven, (b) empty cell stored in humidity chamber.



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Fig. 2. Impedance spectra of the CLC cells. (a) empty cell stored in dry oven, (b) empty cell stored in humidity chamber.



with a thinning solvent. We prepared three solutions with the PI2555 concentrations: 0%, 10% and 34%. The solutions were spin coated on the surface of the glass substrates and then baked and rubbed to generate the homogeneous alignment of the liquid crystal. The substrates were assembled into empty cells. It is well known that the higher the concentration of PI2555, the thicker the alignment layer is [20,21]. When the PI2555 concentration was 10%, the alignment layer thickness was measured to about 20 nm. When the PI2555 concentration was 34%, the alignment layer thickness was measured to about 220 nm, which is still much thinner than the cell thickness. Under a given voltage, the broadening increased with decreasing PI2555 concentration as shown in Fig. 3. For the cell without PI2555, the broadening is very large. When 10 V was applied, the reflection band covered the entire visible light spectrum. Note that the reflection at short wavelength side is decreased, which was due to the fact that the pitch gradient is so large that for each pitch there is not sufficient number of layers to produce the saturated reflection. The photographs of the cell under 3 different voltages are shown in Fig. 4. In the absence of applied voltage, the cell reflected green light. When a voltage higher than 10 V was applied, the color changed to white. It should be noticed that even without PI2555, the rubbed ITO still can produce a weak homogeneous alignment of the liquid crystal. In addition, the CLC had a negative dielectric anisotropy and was aligned well into the planar state by applying voltages before UV curing. The reflection bandwidths of the three cells as a function of applied voltage are shown in Fig. 5. The bandwidths increased approximately linearly with the applied voltage. Note that the alignment layer thicknesses were in sub-micron region, much smaller than the liquid crystal layer thickness.





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Fig. 3. Reflection spectra of the polymer stabilized CLC cells with alignment layers made from different PI2555 concentrations under various voltages.





Fig. 5. Normalized reflection bandwidth of the polymer stabilized CLC cells under vs. the applied DC voltage.







Fig. 4. Photographs of the polymer stabilized CLC cell without PI2555 under three different voltages.





Fig. 6. Impedance of the CLC cells with different alignment layer thicknesses as a function of voltage frequency.



Fig. 7. Chemical structures of <u>monomer</u> RM257 and guest materials.





Fig. 8. Reflection spectra of the four CLC cells with the guest materials under various DC voltages.





Fig. 9. The normalized reflection bandwidth of the polymer stabilized CLC cells doped with guest materials as a function of applied DC voltage.







 Fig. 10. Impedance of the polymer stabilized CLC cells doped with guest materials as a function of voltage frequency.

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Fig. 11. Ratio of normalized reflection bandwidth broadening vs. ratio of conductance.





Fig. 12. SEM pictures of the <u>polymer network</u> formed in the polymer stabilized CLC cells doped with guest materials. (a) No guest material, (b) <u>Succinic acid</u>, (c) <u>Phenylacetic acid</u>, (d) 1-Phenylhexane.

We also check the resistivity spectrum of the cells with the different alignment layer thicknesses. The resistances of the three cells are shown in Fig. 6. The results show clearly that the resistance (impedance) increased with the PI alignment layer film thickness. This also support with the idea that ions are necessary for the DC voltage induced reflection band broadening in the polymer stabilized CLCs. A possible reason for the change of the resistance change with the alignment layer thickness is that the alignment layer also acts as an insulation layer. When there is no alignment layer, ions in the ITO conducting film can migrate into the liquid crystal and thus increases the ion density and enhances the reflection band broadening.

(3) Effect of ionic additives

The <u>ion concentration</u> depends on the host material which is the mixture of liquid crystal and monomer [22,23]. The structure of the monomer RM257 is shown in Fig. 7. It has phenyl rings and <u>ester</u> group. The structure of the nematic liquid crystal host MLC-2079 is unknown, but is believed to have a rigid core consisting of phenyl and <u>cyclohexane rings</u> and a <u>hydrocarbon</u> flexible tail. In order to investigate the effect of the phenyl ring and ester group on the trap of ions, we add three guest materials, whose chemical structures are shown in Fig. 7, into the host material. <u>Succinic acid</u> has only ester group; <u>Phenylacetic acid</u> has phenyl ring while 1-Phenylhexane has both. We made three mixtures: each had one of the guest materials. The concentration of Succinic acid was 0.5%. The concentration of Phenylacetic acid was 0.8%. The concentration of 1-Phenyhexane acid was 0.27%. The mixtures were filled into three cells, respectively.

The reflection spectra of the cells with the guest materials under various applied DC voltages are shown in Fig. 8. The reflection spectra of the cell without guess material is also shown as a reference in the figure. Under a given voltage, the order of broadening from wide to narrow is (a) 1- Phenylhexane, (b) Phenylacetic acid, (c) no guest material and (d) Succinic acid. In order to clearly show the effect of the guest materials on the broadening, the normalized reflection bandwidths of the four cells as functions of the applied DC voltage are shown in Fig. 9. 1-Phenylhexane enhanced greatly the broadening while Succinic acid suppressed the broadening. This suggests that the phenyl ring is responsible for the ion trapping, but not the ester group.

We also carried out a study of the resistivity of the cells doped with the guest materials. The impendence of the cells as a function of the frequency of the applied voltage is sh

trapped, the more the polymer network moves under DC voltages, and the wider the reflection band is broadened. The mechanism how the additives affect the resistivity of the liquid crystal host is not well understood and more researches are needed.

(4) Relation between reflection band broadening and impedance

Experiments reported in the previous sections show that the reflection band broadening is related to the impedance of the polymer stabilized CLC cells: the lower the impedance, the larger the broadening is. We tempted to investigate the quantitative relation between these parameters. In each set of experiment, the size of the cells was the same, but in different sets of experiments, the size of the cells was different (the same cell thickness but different area). The best way to compare the results is to consider the ratio between the normalized bandwidth increments and the ratio between the inversed impedances (namely, the ratio between the conductances) in each set of experiment. The normalized band broadening is defined by

$$NBB = \frac{\Delta\lambda - \Delta\lambda_o}{\Delta\lambda_o},\tag{1}$$

where  $\Delta \lambda_o$  is the reflection bandwidth at 0 V, and  $\Delta \lambda$  is the bandwidth when a voltage is applied. The impedance is  $\rho$  and the inversed impedance (the conductance) is  $\sigma = 1/\rho$ .

In the set of experiment studying the effect of moisture, for the cell without moisture (the benchmark cell),  $\sigma_1 = 1/(3.2 \text{ M}\Omega)$ ,  $NBB_1 = 1.26$ at 35 V; for the cell with moisture,  $\sigma_2 = 1/(4.6 \text{ M}\Omega)$ ,  $NBB_2 = 0.35$ at 35 V.  $\sigma_2/\sigma_1 = 0.706$  and  $NBB_2/NBB_1 = 0.275$ .

In the set of experiment studying the effect of alignment layer, for the cell with the alignment layer made from 10% PI2555 (the benchmark cell),  $\sigma_1 = 1/(23 \text{ M}\Omega)$ ,  $NBB_1 = 0.3$ at 10 V; for the cell without alignment layer,  $\sigma_2 = 1/(3 \text{ M}\Omega)$ ,  $NBB_2 = 1.5$ at 10 V.  $\sigma_2/\sigma_1 = 7.66$  and  $NBB_2/NBB_1 = 5.0$ . For the cell with the alignment layer made from 34% PI2555,  $\sigma_3 = 1/(52 \text{ M}\Omega)$ ,  $NBB_3 = 0.0$ at 10 V.  $\sigma_3/\sigma_1 = 0.44$  and  $NBB_2/NBB_1 = 0.0$ .

In the set of experiment studying the effect of ionic additives, for the cell without additive (the benchmark cell),  $\sigma_1 = 1/(31 \text{ M}\Omega)$ ,  $NBB_1 = 1.3$ at 20 V; for the cell with Succinic acid,  $\sigma_2 = 1/(38 \text{ M}\Omega)$ ,  $NBB_2 = 0.5$ at 20 V.  $\sigma_2/\sigma_1 = 0.82$  and  $NBB_2/NBB_1 = 0.38$ . For the cell with Phenylacetic acid,  $\sigma_3 = 1/(17 \text{ M}\Omega)$ ,  $NBB_3 = 2.6$ at 20 V.  $\sigma_3/\sigma_1 = 1.82$  and  $NBB_3/NBB_1 = 2.0$ . For the cell with 1-Phenylhexane,  $\sigma_4 = 1/(12 \text{ M}\Omega)$ ,  $NBB_4 = 3.3$ at 20 V.  $\sigma_4/\sigma_1 = 2.58$  and  $NBB_4/NBB_1 = 2.53$ .

The ratio between the conductances  $\sigma_i/\sigma_1$  vs. the ratio between the normalized band broadenings  $NBB_i/NBB_1$  is plotted in Fig. 11. It can be seen that the normalized band broadening is approximately linearly proportional to the conductance, which is in turn proportional to the ion density.

because presence of ions may affects the <u>polymerization</u> and produce different polymer networks. We used SEM to examine the polymer networks after the liquid crystal was extracted [[24], [25], [26], [27]]. The SEM pictures of the polymer networks are shown in Fig. 12. No significant change was observed. Therefore the guest materials mainly changed the ions density in the polymer stabilized cholesteric liquid crystal.

## 3. Discussion and conclusion

For Cholesteric <u>liquid crystals</u> with negative <u>dielectric</u> anisotropies, the molecules tend to align perpendicular to the applied electric field. When an electric field is applied across the cell, the CLC in the planar state is stable, where the liquid crystal molecules are already perpendicular to the field, and no change in its <u>optical property</u> is expected. Fantastically, it was discovered that the reflection band is broadened under DC electric fields if a <u>polymer network</u> is dispersed in the CLC. In the beginning of the project, there were two hypotheses for the broadening: Lehmann effect model and polymer network movement model [19,[28], [29], [30], [31]].

In the Lehmann effect model, when a DC voltage is applied across the cholesteric cell, charge carriers move through the liquid crystal. When the charge carriers collide with the chiral molecules, they exert a <u>torque</u> on the liquid crystal, which forces the liquid crystal to twist and thus change the helical pitch. This model was ruled out by the following experimental evidences. First Lehmann effect should exist without polymer networks. Experiments show, however, that polymer networks are necessary to produce the reflection band broadening. Secondly, chiral molecules are required into to have the Lehmann effect. Experiments show, however, that the optical property of twisted nematic cell (without chiral dopants) can be modified by applying DC voltage [32].

In the polymer network movement model, when a DC voltage is applied across the Cholesteric liquid crystal/polymer network system, charges are trapped in the polymer networks, and thus the polymer networks move under the <u>electrostatic force</u>. The polymer network is anisotropic and has a strong aligning effect which tends to keep the nearby liquid crystal parallel to the network. Because of the boundary condition that the polymer network is attached to the surface of the cell substrates, the movement of the polymer network is not uniform: the movement is small near the cell surface but large in the middle of the cell. Therefore in the top side of the cell (where the polymer network moves toward), the polymer network density becomes higher and thus the pitch is compressed, while in the bottom side, the polymer network density becomes lower and thus the pitch is stretched.

The broadening is only observed under DC voltages due to the fact that the movement of the polymer network is slow. We measured the response time of the polymer stabilize FEEDBACK  $\Box$ 

We studied a variety of factors affecting the reflection band broadening of polymer stabilized cholesteric liquid crystals. We found a correlation between the broadening and the <u>electrical</u> <u>resistivity</u>. The lower the electric resistivity is, the higher the ion density is and the wider the broadening becomes. By controlling moisture, reducing alignment layer thickness and adding guest materials, we achieved wide reflection band to cover the entire visible region under low voltages.

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