Bandwidth broadening induced by ionic interactions in polymer stabilized cholesteric liquid crystals

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Abstract: Cholesteric liquid crystals (CLCs) are selectively reflective materials that can exhibit a number of dynamic optical responses. We recently reported on electrically-induced, seven-fold increase in bandwidth in polymer stabilized CLCs (PSCLCs) subjected to DC electric fields. Here, the underlying mechanism of the electrically-controllable bandwidth broadening in PSCLCs is isolated by employing a variety of electro-optic experiments. We conclude that the mechanism is ionic charge trapping by the polymer network which subjects the material system to pitch expansion near the positive electrode and pitch compression near the negative electrode resulting in approximately linear pitch variation throughout the cell thickness.

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1. Introduction

The cholesteric liquid crystal (CLC) phase spontaneously organizes into a 1-D photonic bandgap represented by a helical variation in the director profile. Accordingly, CLCs maintain a selective reflection that is centered at the wavelength, $\lambda_o = n_{avg} p_o$, where n_{avg} is the average index of refraction of the liquid crystal, and p_0 is the cholesteric pitch length (defined as the distance for the LC nematic director to make one complete rotation about the helical axis). The bandwidth ($\Delta\lambda$) of the reflection notch is defined as,

$$\Delta \lambda \approx \Delta n \cdot p_o = (n_e - n_o) \cdot p_o \tag{1}$$

where Δn is the birefringence, n_e is the extraordinary refractive index, and n_e is the ordinary refractive index [1-3]. Typically the bandwidth for the reflection bandgap of CLCs in the visible spectrum formulated from commercially available nematic liquid crystal mixtures (with Δn of 0.2-0.3) is approximately 50-75 nm which is sufficiently large for applications such as narrow-band polarizers and optical filters. However, applications in reflective displays, dynamic polarizers, optical data storage, or switchable windows/mirrors necessitate larger and/or reconfigurable bandwidths [3–5]. While the commercial liquid crystal material suppliers continue to develop eutectic liquid crystal mixtures with increasingly larger birefringence values (now exceeding 0.3), it is unlikely that these values will ever reach values necessary to enable a broadband reflection spanning the entirety of the visible spectral range emanating from a CLC with a constant pitch.

A number of reports have demonstrated other approaches to increasing the bandwidth of CLCs. In the appropriate preparation conditions, polymer stabilization of CLCs (PSCLCs) can generate a pitch gradient across the cell gap that yields reflection bandwidths reaching 300 nm in the visible spectrum [6–8]. The bandwidth of the PSCLCs is switchable (on/off) but not adjustable. Recently, stimuli-driven variations in the magnitude the bandwidth of a CLC have been reported. Photoinduced, dynamic bandwidth broadening (photobroadening) to values as large as 1500 nm was recently reported in a CLC mixture composed with a high HTP bis(azo) 1,1'-binaphthyl chiral dopant [9]. The "photobroadening" effect is limited by speed and can be sensitive to environmental conditions. Only recently has the ability to electrically regulate the reflection bandwidth in PSCLCs been reported [10].

In this prior report, electrically-induced bandwidth broadening was readily observable when the following conditions were in place: mixtures formulated from negative dielectric anisotropy nematic liquid crystal hosts, polymer stabilization with low concentrations (< 10 wt%) of monomer, and application of DC electric fields [10]. The bandwidth broadening was completely reversible if the field was held beneath a critical damage threshold (typically < 5 V/μ m) and shown to exhibit a linear dependence on applied field after surpassing a threshold field (typically 0.5-1 V/ μ m). After the applied DC electric field was removed the reflection notch restored the original reflection bandwidth in the course of a few seconds. Variations in the magnitude of the dielectric anisotropy of the nematic liquid crystal host, the percentage of monomer (polymer) loading, and cell thickness all strongly affected the magnitude and threshold voltage of the bandwidth broadening [10]. Notably, the magnitude was shown to be independent of the birefringence of the nematic liquid crystal host. Several basic electro-

optical mechanisms [11–14] including ionic, polar, and polymeric interactions were considered in the context of these experimental observations.

The goal of the work presented here is to further elucidate the mechanism that underlies the novel ability to electrically-regulate the magnitude of the bandwidth of a PSCLC. Towards this end, we employ electro-optic experiments specifically targeted at exploring the fundamental interaction of the materials with applied DC fields. Together, these results strongly indicate that the underlying mechanism is ionic in nature and the bandwidth broadening is attributable to charge trapping on the polymer network. Understanding the fundamental mechanism of this phenomenon is a key step to enable further enhancement and development by informing future research to target strategies that exploit this mechanism to improve the performance properties.

2. Experimental

The CLC mixtures were formulated from negative dielectric ($-\Delta\epsilon$) nematic liquid crystal (ZLI-2079 (EMD Chemical) or ZLI-4033 (EMD Chemical)), chiral dopant (S1011, EMD Chemical), LC monomer (RM 257, EMD Chemical), and photoinitiator (Irgacure 651, Ciba). The CLC mixtures were drawn into self-prepared LC alignment cells made from ITO-coated glass substrates separated by uniformly dispersed glass spheres of either 10 or 15 µm (as specified) thickness. Planar alignment was achieved by treating the ITO-coated substrates with polyimide (PI-2555, HD Microsystems) which was rubbed with a cloth prior to cell construction. After filling, photopolymerization was initiated with exposure to 365 nm light at an intensity of 28 mW/cm² for three minutes using an Argon ion laser as a light source. The cells were marked to indicate which side faced the exposing light source during polymerization. After UV exposure, the cells were subsequently post-cured under an incandescent lamp for 5 minutes to assure a very high degree of polymerization was reached.

To facilitate the elucidation of the underlying mechanism, this work also employs liquid crystal alignment cells in the twisted nematic (TN) orientation as well as with patterned (interdigitated) electrodes. The TN cells were prepared with 3.6 μ m spacing in ITO coated glass substrates coated with PI-2555 and rubbed for left-handed twist. Planarly aligned cells with interdigitated electrodes with spacing of 15 μ m (spacing between electrodes) x 15 μ m (electrode width) x 9 μ m (cell gap thickness) were purchased from Instec. The cells with interdigitated electrodes were filled with nematic liquid crystal mixtures and subsequently polymer stabilized with the identical concentrations (absent the chiral dopant) and methods as those described for PSCLC samples.

Optical characterization of the bandwidth broadening was derived from both reflection and transmission spectra that were collected simultaneously using a dual channel fiber optic spectrometer with a white light probe, a broadband beam splitter, and collection optics. The data collection rate was manually controlled with a minimum capture time of approximately 30 ms per spectra. Square electrical waveforms (amplified by as much as a factor of 46) were generated with a high voltage inverting AC/DC amplifier and applied across the cell. During spectroscopic experimentation, the cell was aligned such that the UV exposed side of the cell faced the incoming white light probe. Switching on times of the electrically-induced broadening were relatively slow (several seconds) with respect to the capture speed of the spectrometer.

Impedance spectroscopy was used to study the complex dielectric permittivity of the material composition as a function of frequency and DC voltage bias. A Nova Control impedance analyzer was used to apply up to 40V DC bias for frequency scans between 1Hz-1MHz using a 100 mV AC voltage. All LC cell thicknesses were measured optically to correctly account for the cell thickness in determining the empty cell capacitance ($C_o = \epsilon_o A/d$), where ϵ_o is the dielectric permittivity of free space, A is the electrode area (25 mm²), and d was the measured cell thickness. Impedance spectra were fit to the appropriate circuit model that included the capacitance of the liquid crystal (C_{CLC}), the series resistance of the ITO substrates (R_{ITO}), and the double layer capacitance (C_{DL}) and resistance (R_{DL}) in parallel that modeled the pinning of ionic contaminates along the electrodes.

3. Results and discussion

The bandwidth broadening is illustrated at applied DC fields ranging from 1 to 4 V/ μ m in Fig. 1(a) in a PSCLC based on a mixture of S1011 with ZLI-2079 stabilized by photopolymerization of 5 wt% RM257. Importantly for the work reported here, the shapes of reflection spectra are asymmetric and dependent on the polarity of the applied DC bias [Fig. 1(b)]. Extensive experimentation and analysis reported in our prior publication ruled out electrohydrodynamic instabilities, electromechanical, and electrophoretic effects as the underlying mechanism for the bandwidth broadening reported in Fig. 1(a) and 1(b) [8,10]. Subsequent experimentation, not detailed in this report, has excluded flexoelectric mechanisms due to the indistinguishable electro-optic response of samples intentionally prepared with large concentrations of defects. Thus, the experiments in this examination are primarily focused on delineating between the current-based mechanism referred to as the electric Lehmann effect [15,16] and ionic mechanisms. Notably, the electric Lehmann effect requires the simultaneous presence of ionic current and molecular chirality to generate molecular rotations under applied electric fields.



Fig. 1. a) Transmission spectra of a PSCLC cell subjected to DC fields ranging from 0 to 4 V/ μ m. b) Reflection spectra of a PSCLC cell with zero (–), forward (–), and reverse (–) DC bias applied.

The impedance spectra for both unstabilized and PSCLC cells measured the real (ε) and imaginary (ε ["]) dielectric response at applied DC voltages ranging from 0 to 34 V as a function of frequency [Fig. 2(a)]. Representative dielectric spectra collected from an unstabilized CLC sample are plotted in Fig. 2(a) at 0 V and 34 V DC bias. The equivalent circuit model used to fit the data is inset into Fig. 2(a). The real dielectric response (ε) of the sample at 0 and 34 V DC bias are indistinguishable. This is expected for CLCs prepared with negative dielectric nematic liquid hosts subjected to DC fields, as the applied field does not change the order or orientation. However, the measured imaginary component (ε) is strongly affected at frequencies below 1 kHz. The influence of the application of DC bias at low frequencies is attributable to the diffusion of ionic contaminants. It is well known that liquid crystals, alignment layers, and chiral dopants contain ionic impurities which can influence the resulting electro-optic properties of a device [17–20]. When the samples are subjected to an applied DC bias (in this case 34 V), the positively and negatively charged ionic contaminants within the liquid crystalline mixture are attracted and diffuse to the oppositely charged electrodes to form a double layer (charge screening). Since the onset of double layer dielectric relaxation occurs well below the frequencies examined here, an approximate dielectric relaxation model can be used to fit the imaginary dielectric response (ε) at low frequencies with the following relationship, $\varepsilon^{"} = \sigma_{\rm eff}/(\varepsilon_0/2\pi f)$, where $\sigma_{\rm eff}$ is the effective ionic conductivity of the film, ε_0 is the permittivity of free space, and f is the applied frequency. The calculated values of σ_{eff} are plotted in Fig. 2(b) for both unstabilized CLC and PSCLC samples. Evident in Fig. 2(b), polymer stabilization reduces the magnitude of the effective ionic conductivity. During the impedance analysis, a fiber optic spectrometer simultaneously measured the optical properties of the sample, plotted in Fig. 2(b) as the relative change in the pitch $(\Delta p/p_0)$.

At a threshold field (>0.1 V/µm), the effective ionic conductivity (σ_{eff}) of the sample decreases with increasing DC bias, while the notch bandwidth begins to increase. The decrease in conductivity is once again attributable to formation of a double layer of ionic impurities at the LC/electrode interface [18,19]. At fields above 1 V/µm, the effective conductivity of the samples levels off as the double layer is fully developed. The nonzero conductivities for the samples above this threshold field strength depend mainly on the concentration of ions that cannot be trapped by the electric field and the intrinsic conductivity of the liquid crystal host. The lack of current flow at higher biases seemingly eliminates the electric Lehmann effect [15,16] as a potential mechanism.



Fig. 2. (a) Representative real (\blacksquare , \bullet) and imaginary (\square , O) dielectric spectra at 0 V (red, circles) and 34 V (black, squares) DC bias as a function of frequency. Best fit theoretical curves were calculated from the equivalent circuit model (inset). (b) The effective conductivity (σ_{eff}) was calculated from the dielectric spectra and plotted as a function of applied DC field for an unstabilized CLC (\bullet) and PSCLC (\blacksquare). Optical spectra were simultaneously collected during dielectric analysis and used to calculate the measured percent change in pitch ($\Delta p/p_o$, %) as a function of electric field for the PSCLC sample.

We hypothesize that trapped ionic charges could displace the polymer network across the cell gap which correspondingly adjusts the local pitch length (twist angle). To influence the bandwidth of the reflection notch in a PSCLC, the distortion of the polymer network must directly affect the local pitch length (twist angle). To confirm this hypothesis, we examined the electro-optic response of polymer stabilized nematic mixtures composed of negative dielectric anisotropy nematic liquid crystal aligned in the twisted alignment. In this experiment, the twist is induced purely by the boundary conditions of the cell. The twisted nematic liquid crystal samples were sandwiched between crossed polarizer and analyzer. The transmission axis of the polarizer is parallel to the liquid crystal on the entrance plane. The cell thickness was chosen such that the optical retardation was near the 1st minimum and the transmission was close to zero at the measured wavelength of 543 nm. When the twist angle deviates from linearity, it can strongly impact the optical transmittance. If the application of a DC bias voltage induces no change in the transmission of a twisted nematic sample, it is a strong indication that the twist angle is unchanged throughout the sample thickness. However, if the transmission is subject to large changes, this can serve as a strong indication that the local twist orientation of the nematic is being deformed by the applied DC bias. The $3.6 \,\mu m$ test cell was composed from a nematic mixture was made from the negative dielectric anisotropy nematic liquid crystal ZLI-4330, 5 wt% monomer RM257, and 0.5 wt% of photoinitiator Irgacure 651. An unstabilized mixture in the twisted nematic cell does not exhibit any change in transmittance with applied DC voltage [Fig. 3]. However, upon polymer stabilization, the cell shows a clear increase in transmission as a function of applied DC voltage [Fig. 3]. Thus, the data in Fig. 3 are strong indication that the twist in the nematic director of the LC during the application of an applied DC bias voltage is disrupted only in the presence of the polymer network. The lack of molecular chirality in the twisted nematic

samples further confirms the exclusion of the electric Lehmann effect as a possible mechanism.



Fig. 3. Transmission of unstabilized (\blacksquare) and polymer stabilized (\bullet) twisted nematic cells under DC fields from 0 to 5.5 V/µm.

To visualize the polarity dependence and polymer network displacement inherent to the proposed mechanism, we prepared samples in cells with patterned electrodes. The electrode spacing is evident in the bright field transmission micrograph shown in Fig. 4-i. The micrographs presented in Fig. 4-ii and 4-iii (for a polymer stabilized nematic sample after photopolymerization of a mixture of photoinitiator, 5 wt% RM257, and ZLI-2079) as well as 4-iv and 4-v (for an unstabilized nematic mixture containing 5 wt% RM257 and 95 wt% ZLI-2079) were collected between crossed polarizers and are taken with the same magnification, resolution, and sizing as Fig. 4-i to allow for direct comparison of the images. As evident in Fig. 4-iv, application of + 15 V DC induces only fringe effects at the edge of each electrode (period of 2d). However, the presence of the polymer network results in large, visually observable changes in the transmission of light (with a period of 2d) on the negative electrodes during application of + 25 V DC and -25 V DC fields [Fig. 4-iv and 4-v]. The field induced transmission is attributable to the increase in concentration of the polymer network near the negative electrode, which disrupts the local director profile and also generates defects. The real-time response of the samples under the application of the DC fields can be observed in Media 1 (unstabilized nematic cell) and Media 2 (polymer stabilized nematic cell). Evident in Media 2, the electrically induced effect is nearly immediately observed upon application of the field and almost immediately disappears upon removal of the field with nearly identical time scales as the broadening effects reported in Fig. 1 and 2.



Fig. 4. Transmission micrographs of cells prepared with patterned (interdigitated) electrodes. (i) Bright field transmission micrograph of a cell with 15 μ m (spacing between electrodes) x 15 μ m (electrode width) x 9 μ m (cell gap thickness). (ii) Polarized optical micrograph taken between crossed polarizers of a polymer stabilized nematic when subjected to a + 25 V DC field. See Media 2. (iii) Polarized optical micrograph taken between crossed polarizers of a polymer stabilized nematic when subjected to a - 25 V DC field. (iv) Polarized optical micrograph taken between crossed polarizers of an unstabilized nematic when subjected to a 15 V DC field. See Media 1. (Inset) Images (ii-iv) were collected with the nematic director aligned to the polarizer and analyzer as shown. Images (i-iv) are collected with the magnification, resolution, and sizing to enable direct comparison.

We propose that these observations can be explained by ionic charge trapping onto the polymer network. Figure 5 illustrates the proposed mechanism. Before application of the DC field, the pitch is uniformly distributed across the cell gap and the PSCLC maintains the expected bandwidth proportional to pitch length and birefringence. Upon application of a DC field, the ionic contaminants within the mixture are pulled to the boundary layers [illustrated in Fig. 5(b)], with cations migrating to the negatively charged substrate and anions migrating to the positively charged substrate. After the free charges are pulled to the electrodes, trapped cations on the polymer network are subjected to an electromechanical force that redistributes the polymer network across the cell gap. Evidence for this process is reported in Fig. 3, wherein the displacement of the local polymer network strongly effects the distribution of the twist angle across the sample thickness. The movement is visually observed in Fig. 4 evident in the local distortion of the LC director orientation which allows for transmission between the cross polarizers only above the negative electrodes. The field-induced transmission near the negative electrodes indicates a local variation (increase) in the polymer density in this region. A recent study has reported that cations may bind more strongly than anions to polymer networks within PSCLCs [21]. The four ester groups in the monomer RM257 are known to be effective charge trapping sites for cations [22]. Upon ion trapping, the polymer is subjected to a force that is dependent on the magnitude of the field as well as the polarity of the DC bias [Fig. 1(b)] resulting in the polymer network being "attracted" to the negative electrode. From these results, it is clear that the mechanism is underpinned by the interaction between the concentration (and structure) of the crosslinked polymer network and the fluidic CLC. The resulting distortion of the polymer across the cell gap compresses the pitch on one side of the cell [blue side, Fig. 1b] and simultaneously lengthens the pitch on the other side [red side, Fig. 1(b)]. This leads to a nearly linear variation of the pitch across the cell gap, observable as symmetric broadening of the bandwidth of PSCLCs employing negative dielectric anisotropy nematic liquid crystal hosts and subjected to applied DC fields.



Fig. 5. Illustration of the ionic charge trapping mechanism proposed here. a) Before application of the DC bias the CLC director configuration (gray) is uniform across the cell gap. The polymer network (blue lines), anionic (-) and cationic (+) contaminants, and bound cations (\oplus) on the polymer network are illustrated. b) After applying a DC bias above the voltage threshold, a fully developed ionic double layer is formed resulting in an electric field (E_{DL}) that opposes the applied electric field (E_{AP}). However E_{DL} is insufficient to effectively screen E_{AP} from the bulk PSCLC. This results in an interaction between the net electric field (E) with the bound cations, deforming the polymer network, and a deformation of the CLC director configuration.

4. Conclusions

The mechanism for electrically induced reflection broadening in polymer stabilized CLCs of negative dielectric anisotropy is elucidated. Electro-optic studies strongly support an ionic charge trapping mechanism, resulting in the movement and distortion of the polymer network

under the influence of DC field. Impedance spectroscopy suggests that double layer formation occurs prior to the observed broadening mechanism. Once the double layer is fully formed, the applied DC field interacts with the cationic charges trapped within the polymer network which subsequently displaces the polymer network and distorts the cholesteric pitch. The association of the presence of the polymer network and the displacement in the twist angle is demonstrated in polymer stabilized nematic mixtures in twisted nematic boundary conditions. The large and reversible notch broadening observed here has potential applications for full-color or polarizer free displays, broadband polarizers, waveplates, and optical data storage media.

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