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Complete List of Authors:	Pande, Gaurav; University of Colorado at Boulder, Chemical and Biological Engineering Radka, Brian; University of Colorado at Boulder, Chemical and Biological Engineering McCracken, Joselle; University of Colorado at Boulder, Chemical and Biological Engineering White, Tim; University of Colorado at Boulder, Chemical and Biological Engineering

ARTICLE

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Gaurav K. Pande[a], Brian P. Radka[a], Joselle M. McCracken[a], and Timothy J. White[a]*

The selective reflection inherent to the cholesteric liquid crystal (CLC) phase (in the planar orientation) can be tuned or broadened by the incorporation of polymer stabilizing networks (PSCLC). Prior reports detail that the mechanism is ion-mediated deformation of the polymer stabilizing network, which retains structural chirality. The structural chirality of the polymer stabilizing effectively overpowers the chirality of the CLC mixture. Here, we incorporate monomers containing groups known to trap cationic metal halide ions, into the polymer stabilizing network. Specifically, we incorporate monofunctional control monomers as well as monofunctional acrylate monomers 4-Acryloxyhexylterpyridine (TPy-Ac), 4-((6-(Acryloyloxy)hexyl)oxy)benzoic-15-crown-5 (OBA-15C5), and 4-((6-(Acryloyloxy)hexyl)oxy)benzoic-18-crown-6 (OBA-18C6). The electro-optic response is enhanced only by the inclusion of OBA-15C5 which previously has been shown to selectively trap and interact with sodium (Na^+) ions.

1. Introduction

Liquid crystals (LCs) self-assemble into achiral and chiral mesophases.[1] Due to their pervasive use in displays, nematic liquid crystals (NLCs) are widely available from commercial suppliers as single component or eutectic mixtures. Mixing a chiral dopant with NLCs can result in the formation of the cholesteric liquid crystal (CLC, sometimes referred to as the chiral nematic) typified by a periodic, helicoidal structure. In the planar orientation, CLCs inherently exhibit a selective reflection and accordingly, are compelling optical materials subject to decades of extensive research.[2, 3]

The wavelength of the selective reflection of the CLC is directly associated with the periodicity of the CLC phase, which is defined by the distance over which the nematic director completes a 360° rotation. The pitch length of the CLC phase is inversely proportional to the concentration ([C]) and the helical twisting power (HTP) of chiral dopant(s) as shown in eqn. (1):

$$P=1/(HTP+[C]) \tag{1}$$

When multiple chiral dopants are utilized, as in this work, the HTP value can be estimated as the mean of the HTP values of all chiral dopants.[4] The periodicity of the CLC phase produces a selective reflection of light defined by the Bragg equation when the wavelength of light is approximately equal to the pitch. Specifically, the center of the reflection wavelength is calculated from eqn. (2):

$$\lambda_C = n_{avg.} P_0 \tag{2}$$

where n_{avg} is the average refractive index of the liquid crystal, and P_0 is the initial pitch length. The breadth of the reflection is defined as the bandwidth of wavelengths that meet the Bragg condition ($\Delta\lambda$), and defined by eqn. (3):

$$\Delta\lambda = \Delta n\, P = (n_e - n_0)\, P_0 \tag{3}$$

where Δn is the birefringence of the liquid crystal media, n_e is the extraordinary refractive index, and n_0 is the ordinary refractive index.[5]

Extensive prior investigation has explored the electro-optic response of the CLC phase, which have been reviewed.[6] A promising approach to directly adjust the periodicity of the CLC phase (to affect the reflection wavelength or bandwidth) has been to stabilize CLC phase by the polymerization of a low concentration of a crosslinked polymer network to form polymer stabilized cholesteric liquid crystalline (PSCLC) materials. Polymer stabilization has been utilized to realize on-off switching of the reflection notch, by serving as a template for liquid crystalline molecules aligned into the homeotropic orientation to return into the hierarchically complex, helicoidal state.[7] In a series of examinations, we have detailed the utilization of polymer stabilization to form PSCLCs formulated with negative dielectric constant ($-\Delta\varepsilon$). Electro-optically, we have reported electrically induced bandwidth broadening,[8, 9] red and blue shift tuning,[10, 11] reflection notches switching and splitting,[12, 13] and transmission control.[14]

Prior studies indicate that the electro-optic coupling in these PSCLCs is primarily associated with ions interacting with the polymer stabilizing network. Despite aggressive purification, even commercial eutectic LC mixtures used in displays contain ionic impurities that negatively impact device

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[a] *Department of Chemical and Biological Engineering, University of Colorado, Campus Box 596, Boulder, CO 80309, USA. Email: tim.white@colorado.edu.*

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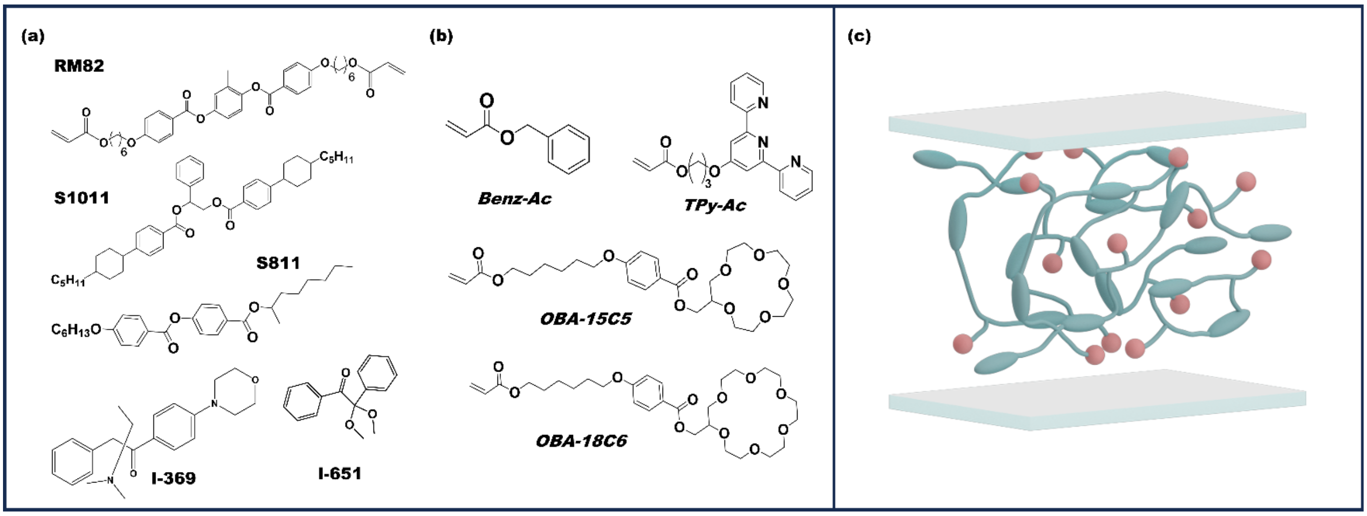

Figure 1. (a) Chemical structures of the monomer RM82, left-handed chiral dopants (S1011, S811), and radical photoinitiators (I-369, I-651) used in this study. (b) Chemical structures of the dopant monomers (c) Illustration of the loosely crosslinked stabilizing polymer network with the host CLC removed, the blue ellipsoids represent the difunctional liquid crystalline monomer units while the red spheres represent the monofunctional dopants.

performance. Examinations report typical LCs contain ionic impurities (10^9-10^{14} ions per cm^3) associated with synthetic and purification steps (salts, catalyst, moisture, and dust), alignment layers, and/or degradation of the LC molecules by UV light exposure.[15-18] Ions can reduce the switching speed of LC displays as well as other irregularities such as image sticking, image flickering, increase the voltage holding ratio, color staining, and image non-uniformity.[19-25] Many of these issues have been addressed both by improving materials to include movement towards hyper-fluorinated systems but also through device design, such as overdriving.

Specifically, prior reports indicate that ions including Na$^+$, NH$_4^+$, K$^+$, Mg^{2+}, Ca^{2+}, Zn^{2+}, Al^{3+}, F$^-$, Cl$^-$, NO$_2^-$, NO$_3^-$, Br$^-$, and SO$_4^{2-}$ can be present in LCs.[26] Further, LCs can degrade to UV light irradiation, elevated temperature, or strong electric fields, to create organic ionic species.[27] Multiple strategies have been reported to purify ionic contaminants from LCs ranging from using highly purified reagents during LC synthesis to classical methods such as multiple recrystallizations, extractions, vacuum distillation, vacuum sublimation, zone refining, chromatography, ion-exchange, and other chemical techniques have all been reported.[28] While effective, these approaches can be time-consuming and limit throughput. Another approach has been to incorporate ion-trapping agents.[29] A broad range of materials have demonstrated usefulness as ion-trapping agents. These include oxide nanoparticles, porous particles, ion trapping films, and metal-organic frameworks.[30-32] Ion-trapping agents (used for both temporal and permanent purification of LCs) are not widely used as typically their inclusion can distort LC alignment creating defects and leading to strong light scattering.[33]

These prior studies inform our current examination of PSCLCs. This and numerous prior studies have utilized a commercially available, $\Delta\varepsilon < 0$ nematic liquid crystal host of unknown composition (including ionic impurities). Further, while the utilization of ion-trapping agents is atypical in display applications, this approach could be readily adopted by incorporating comonomers into PSCLCs as ion trapping sites. For example, terpyridine moieties are known to form complexes through coordination with transition metal ions.[34] Specifically, 2,2′:6′2′′-terpyridine known (TPy) has three nitrogen coordination sites is one candidate. Further, crown ether molecules can capture cations such as Li$^+$, Na$^+$, K$^+$ and Cs$^+$ in the cage of the crown ether structure. Interestingly and of relevance to the work here, the ion-material interaction is dependent on cavity diameter and cation diameter.[35, 36] Of relevance, other reports use crown-ether molecules in LC devices for ion sensing applications.[37, 38]

Motivated by these prior studies, here we prepare PSCLCs with a variety of monofunctional comonomers to both explore the nature of the ion-polymer interaction as well as engineer enhanced responses. Specifically, we prepare control samples

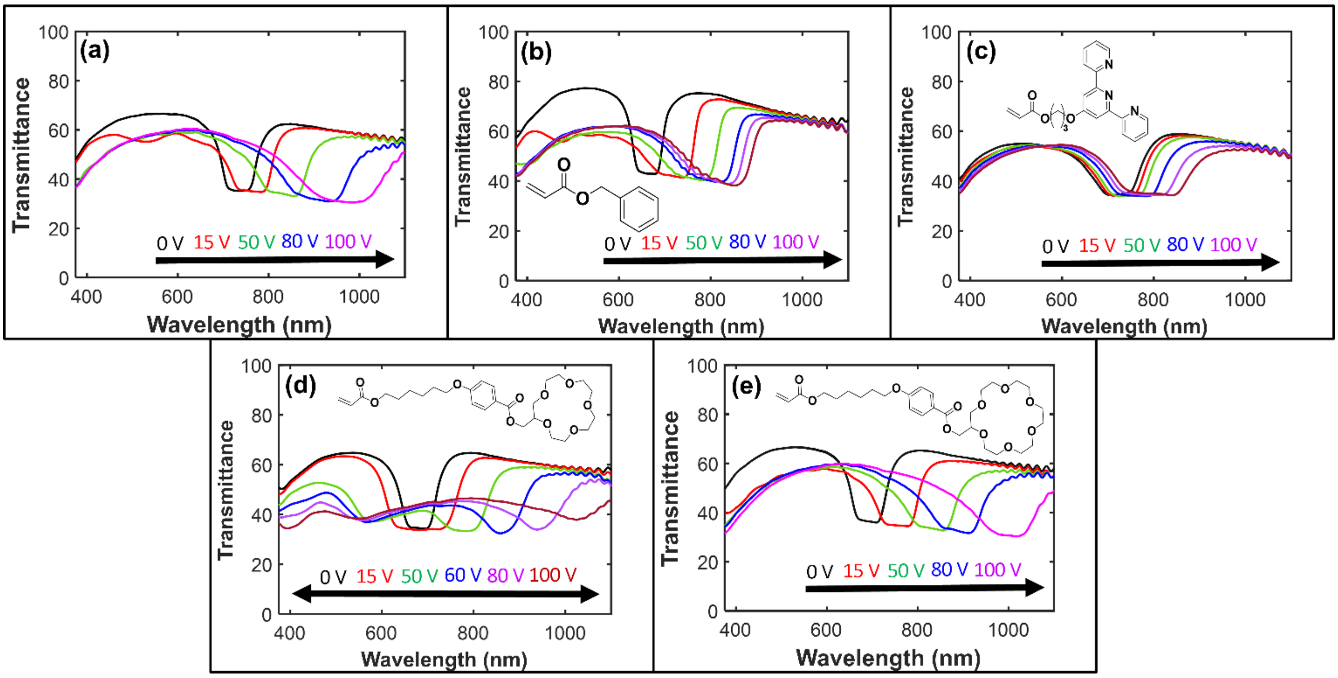

Figure 2. Transmission spectra were obtained as voltage was applied and increases for (a) PSCLC prepared only from 6 wt.% RM82, (b) a PSCLC prepared by copolymerization of 6wt% RM82 and 5 wt.% Benz-Ac, (c) a PSCLC prepared by copolymerization of 6 wt.% RM82 with 5 wt.% TPy-Ac, (d) a PSCLC prepared by copolymerization of 6 wt.% RM82 with 5 wt.% OBA-15C5, and (e) a PSCLC prepared by copolymerization of 6 wt.% RM82 with 5 wt.% OBA-18C6. PSCLCs formulations were based on mixture of the chiral dopants S1011, S811 (both 5 wt.%), photoinitiator I-369 (1 wt.%), and MLC-2079. Polymerization was initiated by UV light (50 mW/cm^2, 365 nm) for 10 min.

with benzyl acrylate (Benz-Ac) and contrast the electro-optic response and electrochemical properties of PSCLC prepared with 4-acryloxyhexylterpyridine (TPy-Ac), 4-((6-(acryloyloxy)hexyl)oxy)benzoic-15-crown-5 (OBA-15C5), and 4-((6-(acryloyloxy)hexyl)oxy)benzoic-18-crown-6 (OBA-18C6) as ion-trapping agents.

2. Experimental

2.1. Materials

Raw materials 2-(hydroxymethyl)-15-crown-5, 2-(hydroxymethyl)-18-crown-6, and 4'-chloro-2,2':6',2''-terpyridine were purchased from Ambeed and were used as received to synthesize monofunctional acrylate comonomers. Other chemicals such as dicyclohexylcarbodiimide, 4-dimethylaminopyridine, potassium hydroxide, triethylamine, 4-((6-(Acryloyloxy)hexyl)oxy)benzoic acid (6-OBA), and 1,3-propanediol were purchased from Sigma Aldrich. The comonomer benzyl acrylate was purchased from TCI Chemicals. The solvents dimethyl sulfoxide, dichloromethane, tetrahydrofuran (THF), methanol, acetone, and isopropyl alcohol were obtained commercially and used without further purification.

PSCLC formulations were based on the negative dielectric ($\Delta\varepsilon < 0$) nematic liquid crystal mixture (MLC-2079) purchased from Merck. Left-handed (S1011, S811; also, Merck) chiral dopants were mixed into MLC-2079 to form the CLC phase. The photopolymerization was initiated with either Irgacure 369 and Omnirad 651 (from IGM Resins). The LC monomer RM82 was purchased from Willshire Technologies and used as received.

2.2. Synthesis

Three comonomers were synthesized: 4-Acryloxypropyl-terpyridine (TPy-Ac), 4-((6-(Acryloyloxy)hexyl)oxy)benzoic-15-crown-5 (OBA-15C5), and 4-((6-(Acryloyloxy)hexyl)oxy)benzoic 18-crown-6 (OBA-18C6). 4-Acryloxypropyl-terpyridine (TPy-Ac) was synthesized following a previous report[34] with minor modifications (Scheme S1, Supplementary Data). Additionally, we synthesized OBA-15C5 and OBA-18C6 following a previous report [37] with minor modifications.

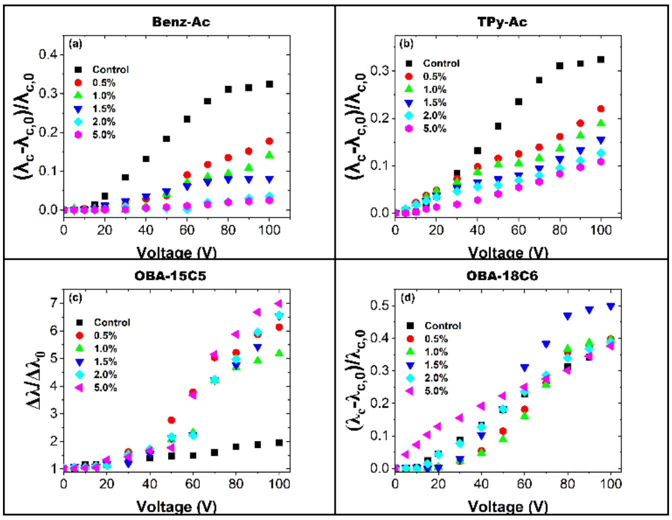

Figure 3. Influence of increase weight percent loading of different comonomer's on original notch position change as a function of voltage. (a) Benz-Ac) (b) TPy-Ac (c) OBA-15C5 and (d) OBA-18C6. PSCLC's samples were prepared using RM82 (6 wt.%), chiral dopants S1011, S811 (5 wt.%), photo initiators I-369 (1 wt.%), and different weight percent loading of comonomer's in MLC-2079. Polymerization was initiated by UV light (50 mW/cm^2, 365 nm) for 10 min.

(Scheme S2, Supplementary Data). The structure and purity of these comonomers was confirmed by ^{1}H NMR and Fourier-transform infrared spectroscopy (Fig. S1 and S2, Supplementary Data).

2.3. Sample preparation

The liquid crystal mixture MLC-2079 was mixed with left-handed (S1011, S811) chiral dopants, myriad monomer compositions, and either the photoinitiator Irgacure 369 or Omnirad 651. All polymer-stabilized networks were contained 6 wt.% RM82. Monofunctional comonomers Benz-Ac, TPy-Ac, OBA-15C5, and OBA-18C6 were added in specified amounts. The mixtures were melt-mixed and drawn by capillary action into both commercial and self-prepared alignment cells on a hot plate above their isotropic temperature, then held at that temperature for 5 min before slowly cooling to room temperature. Manufactured alignment cells were obtained from Instec Inc. (S type, ITO (100Ω/sq.), 20 μm cell gap, active area 1 cm^2). After cooling, the samples were polymerized at room temperature by exposure to UV irradiation (365 nm, Intensity 50 mW/cm^2, for 10 min).

2.4. Property characterization

Transmission spectra were collected with STS-VIS and STS-NIR spectrometers (Ocean Insight) operating concurrently. Spectra were taken with OceanView software while an electric field was applied. Dielectric permittivity was measured using a potentiostat from GAMRY Instrument (Reference 600+). Fourier-transform spectra was collected utilizing a Nicolet, Is50 FTIR.

3. Results and discussion

Here, we are concerned with exploring the fundamental nature of ion-polymer interactions in PSCLCs with the goal of optimizing the electro-optic response. Accordingly, we prepared a series of PSCLC formulations based on the compounds drawn in Fig. 1a. Monofunctional comonomers known to act as ion traps (or a control) are illustrated in Fig. 1b. Mixtures were drawn into alignment cells and subject to photopolymerization to prepare crosslinked polymer stabilizing networks with pendant sites associated with monofunctional comonomers (Fig. 1c). To scope this study, we first examined the electro-optic response of PSCLCs prepared to exhibit red-shifting tuning based on a mixture of the chiral dopants S1011, S811 (5 wt.%) and the -$\Delta\varepsilon$ nematic LC mixture MLC-2079. Five PSCLCs were prepared, all containing 6 wt.% RM82 and initiated with 1 wt.% of Irgacure 369. A typical PSCLC formulation and associated electro-optic response is presented in Fig. 2a, based on the homopolymerization of RM82. The initial selective reflection is red-shifted with application of a DC field of 0-100V. A second control PSCLC sample is illustrated in Fig. 2b, prepared by copolymerization 5 wt.% Benz-Ac with 6 wt.% RM82. The copolymerization of benzyl acrylate into the polymer stabilizing network is not expected to introduce distinctive ion-polymer interactions and simply dilutes the overall crosslink density of the network. As evident in Fig. 2b, the electro-optic response of the PSCLC prepared with Benz-Ac is decreased compared to the PSCLC prepared with only RM82. This is like prior studies that detail the importance of concentration and crosslink density.[39]

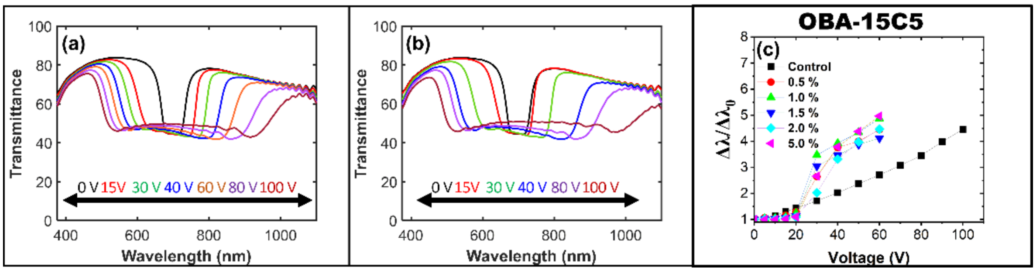

Figure 4. Transmission spectra were obtained for (a) conventional PSCLC (b) OBA-15C5 (5.0%) and (c) summarized original notch position change as a function of voltage with increase concentration of OBA-15C5 compared with conventional PSCLC sample. PSCLC's samples were prepared using RM82 (6 wt.%), chiral dopants S1011, S811 (5 wt.%), photo initiators I-651 (1 wt.%) in MLC-2079. Polymerization was initiated by UV light (50 mW/cm^2, 365 nm) for 10 min.

From these data, it can be concluded that simply adding in a monofunctional comonomer, such as Benz-Ac, may decrease the electro-optic response.

These samples are compared to PSCLC prepared with comonomers previously identified as potential ion traps. PSCLCs were prepared by the copolymerization of 5 wt.% of either TPy, OBA-15C5, or OBA-18C6. Like Benz-Ac, the inclusion of TPy mutes the electro-optic response of the PSCLC with its inclusion. However, the incorporation of the crown ether moieties does show distinct electro-optic response (Fig. 2d, 2e). Evident in the spectral data for PSCLC prepared with OBA-15C5, the sample undergoes bandwidth broadening but at lower voltages. PSCLC prepared with OBA-18C6 red-shift with a response similar to that of the control PSCLC with RM82. Together, these data indicate that i) copolymerization of monofunctional monomers generally decrease the electro-optic response (Benz-Ac), ii) copolymerization of TPy, known to interact with transition metal ions, does not enhance electro-optic coupling, and iii) both crown ether comonomers enhance or at least retain the electro-optic response of a typical PSCLC.To assess any concentration dependence in the PSCLC, we prepared samples copolymerized with 0.5-5.0 wt% of Benz-Ac (Fig. 3a), TPy-Ac (Fig. 3b), OBA-15C6 (Fig. 3c), and OBA-18C6 (Fig. 3d). To allow comparison, the data are plotted as referenced to their starting reflection wavelength (Benz-Ac, TPy-Ac, OBA-18C6) or to their starting bandwidth (OBA-15C5). Again, regardless of the concentration, the incorporation of any amount of Benz-Ac or TPy-Ac into the PSCLCs reduces the electro-optic coupling. Conversely, the incorporation of OBA-15C5, even at 0.5 wt.%, contributes to an exceptional enhancement. The incorporation of OBA-18C6 is more nuanced, with some evidence of enhancement that could be muted by the nascent contribution of crosslink density. However, from these data, it seems clear that ion-polymer interactions generally seem to be associated with alkali metals such as Li^+, Na^+, or Ca^{2+} rather than transition metals (e.g., those that would pair with TPy-Ac). Notably, the inclusion of OBA-15C5 results in a four order of magnitude decrease in ion density compared to the other PSCLCs. This is indicative that the comonomer is trapping ions upon mixing and arresting them into the polymer stabilizing network. The ion density was measured with the current method, which examines free ion mobility (Fig. S3, Supplementary Data).

Unfortunately, as evident in Fig. 2a, the control PSCLC sample redshifts while the compositions incorporating OBA-15C5 broaden. To assure that the appearance of enhancement is not due to a byproduct of the difference in the stimuli-response, we prepared PSCLC compositions known to exhibit bandwidth broadening and incorporated in OBA-15C5. The transmission spectra of the control PSCLC is shown in Fig. 4a and contrasted to a PSCLC prepared with 5 wt.% OBA-15C5 in Fig. 4b. As summarized in Fig. 4c, any concentration of OBA-15C5 again enhances the electro-optic coupling, reaffirming the influence indicated in the data presented hereto.

Electrochemical impedance spectroscopy measurements were taken for both unstabilized CLC and PSCLC. Dielectric constants as a function of frequency are represented as a complex number in the form shown in Equation 4.

$$\varepsilon(\omega) = \varepsilon'(\omega) - i\varepsilon''(\omega) \qquad (4)$$

The real part of the dielectric constant (ε') is related to the ability of the system to store energy (capacitance) while the imaginary part (ε'') is related to the energy loss in the system through charge movement (conductivity).[40] We employ the calculated effective conductivity (σ_{eff}, Equation 5) to study the effect of ion-trapping by the comonomers.

$$\sigma_{eff} = \omega\, \varepsilon'' \qquad (5)$$

Where ω is the angular frequency and the calculation is completed by fitting the data to the linear region at lower frequencies in the log-log plot.[41] Selected dielectric plots and calculated σ_{eff} for CLCs and PSCLCs are shown in Fig. 5(a-f). Changes in conductivity can be attributed to two factors: i) the concentration of ionic species present and ii) the mobility of those ions.

The five PSCLC compositions were examined before and after polymerization. Generally, the inclusion of the monofunctional comonomers increase the conductivity even before polymerization. This is explainable in that the synthesis of these materials, while pure, inevitably have ionic contaminants. Upon polymerization, the conductivity of the PSCLC increases in the control sample but generally decreases in the other four compositions. Most notably, the composition prepared with OBA-15C5 has a dramatic, two order of magnitude decrease in effective conductivity. This suggests that the OBA-15C5, upon polymerization, is incorporating ionic species into the polymer stabilized network and may be reducing the effective ion concentration in the liquid crystalline bulk.

These results explain the distinguished contribution of OBA-15C5 to the electro-optic response. Prior examinations indicate an appreciably high Na^+ presence. Comparatively, the selectivity of OBA-15C5 versus OBA-18C6 indicates the coupling to Na^+. While the other comonomers also affect the dielectric constant and effective conductivity, their contribution is comparable small. Reasons for this could include limited coupling with ionic groups in the mixtures.

Conclusion

PSCLC elements were prepared with various monofunctional acrylate monomer, some known to interact with particular ionic species. From this examination, it seems clear that the ion-polymer interaction is associated with alkali metal ions such as Li^+, Na^+, and Ca^{2+} rather than transition metals. Functionally, the rational incorporation of comonomers with moieties such as crown ether complexes that interact with these ionic impurities considerably enhance the electro-optic coupling of PSCLC. Impedance spectroscopy suggests that rational incorporation of crown ether complex effectively interact with the ionic impurities which can be seen as decrease in conductivity. This decrease in effective conductivity indirectly indicates charge retention in the polymer stabilizing network. Accordingly, the network has stronger electromechanical coupling which reduces the voltage requirements and enhances the response time.

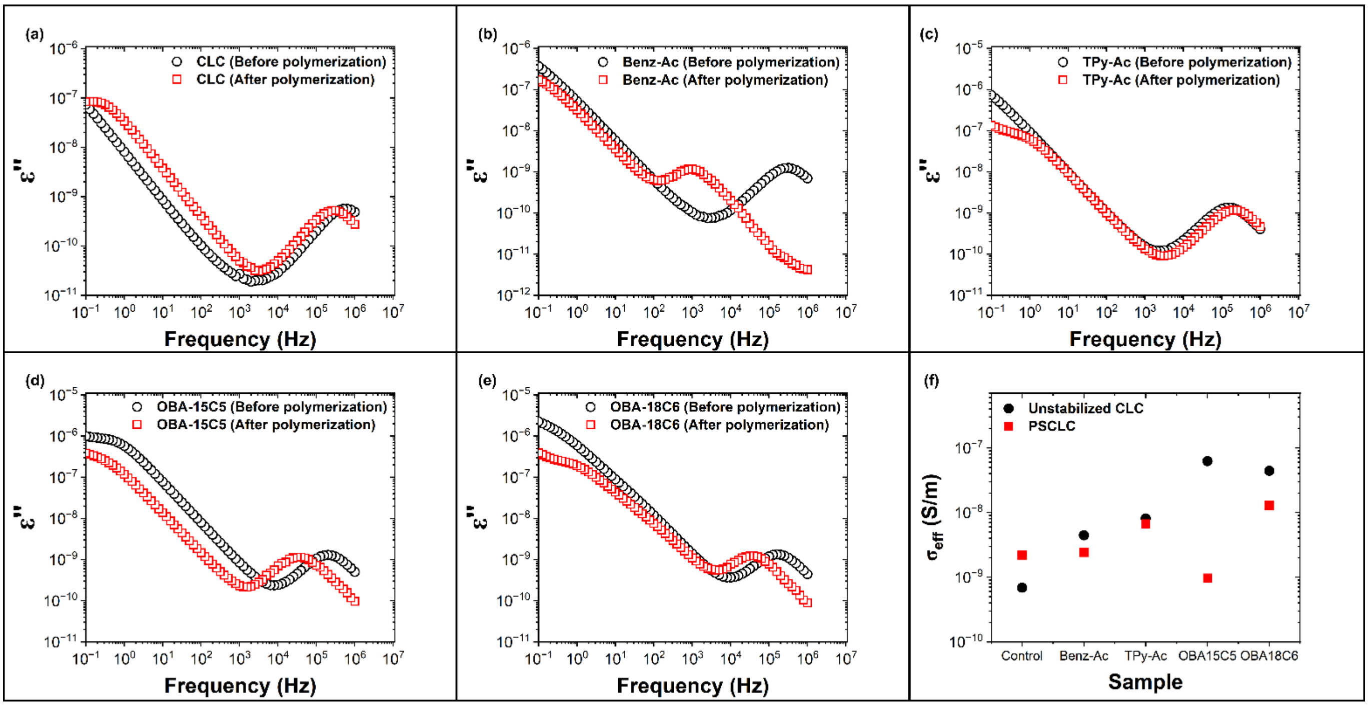

Figure 5. Dielectric spectra as a function of frequency obtained before and after polymerization for (a) Conventional PSCLC (b) Benz-Ac (c) TPy-Ac (d) OBA-15C5, and (e) OBA-18C6 samples. The effective conductivity (σ_{eff}) was calculated from dielectric spectra and plotted in (e). All samples were prepared using RM82 (6 wt.%), chiral dopants S1011, S811 (5 wt.%), photo initiators I-369 (1 wt.%), comonomer's (1 wt.%) in MLC-2079. Polymerization was initiated by UV light (50 mW/cm^2, 365 nm) for 10 min.

Conflicts of interest

There are no conflicts to declare.

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