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Electro-optic properties of thiol-ene polymer stabilized ferroelectric liquid crystals

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Electro-optic properties of polymer stabilized ferroelectric liquid crystal (PSFLC) systems are examined as a function of varying concentrations of either a linear or crosslinked thiol-ene polymer. The thiol-ene method of polymer stabilization is a drastic change from previous studies designed to avert the problem of polymer phase separation. FLC rise time and tilt angle measurements were used to determine the effects of the polymer network on the optical properties of the system. The addition of monomer impurities to both systems demonstrated a reduction in tilt angle, which translated into decreased switching speeds in both systems prior to polymerization. The crosslinked thiol-ene system showed increased switching times due to the creation of polymer in the interlayer spacing of the FLC, but exhibited minimal increase in the rotational viscosity of the system. In addition, the crosslinked polymer systems resulted in an increase in the liquid crystalline order, which produced an increase in the contrast ratio of the system. The linear polymer system showed drastically different results as compared with the crosslinked system. The rise time and tilt angle measurements decreased upon polymerization of the linear thiol-ene and the rotational viscosity and contrast ratio values also decreased. We suggest that the linear thiol-ene polymer phase separation from the interlayer spacing leads to a microscopic misalignment of the FLC molecules, causing a decrease in the optical properties of the LC.

1. Introduction

Liquid crystals (LCs) are materials that flow like liquids and are characterized by anisotropic optical properties, as well as positional and orientational molecular order. These materials exhibit unique properties that enable them to be used for a variety of applications, including displays [1–3], telecommunication switching [4, 5], and optical storage [6, 7]. In particular, due to the increasing market for high definition displays, liquid crystal displays (LCDs) have become a prominent area of research in the LC field, as they hold the potential for enabling displays with higher resolution, lighter weight, and reduced thickness.

Currently, nematic LCs are used extensively for LCD applications. However, these nematic LCs have several drawbacks, which limit their commercial viability for high definition displays. Ferroelectric LCs (FLCs) have enhanced electro-optical properties relative to conventional nematic LCs, which makes them highly desirable

for a range of applications. One of the primary benefits of FLC materials is associated with switching speeds, where FLCs switch approximately 100 times faster than nematics. One of the major reasons FLCs have not achieved significant commercial practicality is due to the mechanical susceptibility of these molecules, in which the alignment of the FLCs is often destroyed by even subtle mechanical shock. To evade the mechanical shock problems, extensive research has been undertaken in the area of polymer dispersed liquid crystals (PDLCs) [8–12] and polymer stabilized liquid crystals (PSLCs) [13–20]. PSLCs show the greatest promise to reduce the susceptibility to mechanical shock without compromising optical properties.

In PSLC systems, a small amount of polymer (< 5%) is introduced into the LC to stabilize the system, which is accomplished by *in situ* polymerization of dissolved monomer with UV irradiation. In this method, the dissolved monomers are aligned in the LC prior to UV irradiation. Previous work has shown that certain types of acrylic polymer phase separate upon polymerization of the monomer, thus drastically limiting the amount of

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stabilization possible [21]. It is believed that this phase separation occurs because of the rapid, non-homogeneous molecular mass evolution of the acrylate polymer systems used, thus rendering the polymer insoluble in the LC. These polymer systems polymerize via a chain growth mechanism, where high molecular mass molecules are formed even at very low conversions. To circumvent this phase separation problem, McCormick *et al.* showed that linear fluorinated acrylate polymer systems could be retained in the interlayer spacing of the LC, but that crosslinked fluorinated acrylate systems phase separated from the LC [22].

In recent work, we completely changed the polymerization mechanism to avoid polymer phase separation by utilizing a thiol-ene system. Thiol-enes polymerize via a radical step growth mechanism [23]. A step growth polymerization results in a more homogeneous growth mechanism, where large molecular masses are not formed until high functional group conversion of the monomer. The growing polymer chains very quickly transform from low molecular mass chains to a cross-linked network, preventing phase separation from the interlayer spacing of the LC. The homogeneous growth mechanism allows the thiol-ene polymers to remain soluble in the LC, but a crosslinked network is required to lock the polymer in the interlayer spacing to avoid phase separation.

It is inevitable that some changes in electro-optic properties occur because of the polymer's presence. Nonetheless, it has been observed that the addition of polymer to the LC system actually enhances some of the electro-optic properties [14, 18, 22, 24, 25]. The polymer relegates the LC into domains, whose size is dependent on the polymerization conditions and monomer type. The polymer network is essential in maintaining LC order past the clearing point and increasing the total LC order in the system. The former effect allows the LC device to be operated at elevated temperatures, while the latter translates directly into improved contrast ratios and enhanced electro-optical properties. In this work, we analyse the effect of two thiol-ene monomer and polymer systems on the electro-optic properties of a FLC. Optical microscopy and contrast ratio measurements are used to ascertain the effect of the polymer host structure on the electro-optic properties of the FLC, to assist in the understanding of these systems.

2. Experimental

Two commercially available thiol-ene systems were analysed in this research, a linear system and a crosslinking system. The monomers that form the linear system were 1,6-hexanedithiol and trimethylolpropane diallyl ether. The monomers that form the crosslinked system

were pentaerythritol tetrakis(3-mercapto-propionate) and triallyl-1,3,5-triazine-2,4,6-(1H, 3H, 5H)-trione. All photopolymerizations were initiated with benzophenone. All chemical materials were purchased from Aldrich, Co. (Milwaukee, WI), and used without further purification. Each of the monomers utilized in this study is commercially available. There are a limited number of commercially available thiol-ene monomers, which limits the number of chemical structures available for use. In particular, there are no commercially available series of monomers with similar chemistry and systematically varied functionality. The FLC utilized was a 1:1 by weight mixture of W7 and W82 which undergoes the following transitions, isotropic \rightarrow 58°C \rightarrow smectic A \rightarrow 48°C \rightarrow smectic C* \rightarrow 13°C \rightarrow more ordered smectic phase. The synthesis of W7 and W82 is described elsewhere [26, 27]. Chemical structures of the monomers and liquid crystals are shown in figure 1. Polymerization of the monomer systems was accomplished using ultraviolet (UV) light with an intensity of 10 mW cm⁻² at a peak wavelength of 365 nm (Blak Ray Inc. Model B 100 AP, San Gabrielle, CA).

Electro-optic properties were obtained using a polarizing microscope (Nikon Optiphot 2-pol) with a rotating hot stage (built in-house, controlled via Instec Inc., Boulder, CO, controller and heating unit), connected to a digitizing oscilloscope (Tektronix Inc. TDS 340, Beaverton, OR). Sample cells were prepared from indium tin oxide (ITO) coated glass plates with a mechanically rubbed nylon alignment layer. Cells were glued together using 2 μ m spherical spacers dissolved in optical adhesive (Norland Optical Adhesive 61, Norland Ind., Cranbury, NJ).

Rise time response of the samples was measured using a 22 V μ m⁻¹ square wave applied across the cell at a frequency of 100 Hz generated from a variable function generator (Tektronix Inc. CFG 250, Beaverton, OR) and amplified via a custom-built voltage amplifier. Rise time response of the sample was read directly from the digitizing oscilloscope, and was measured precisely from the optical signal. Tilt angle was measured using a half angle analysis [28].

To determine the spontaneous polarization, a triangular wave at 500 Hz and 80 V peak-to-peak was applied to the sample cell and the current response was measured on a digitizing oscilloscope [29]. The spontaneous polarization of specific samples was normalized to that of neat W7/W82. From this polarization data, the rotational viscosity of the system was determined. Equation (1) was used to determine the normalized rotational viscosity:

$$\gamma_n^* = \mathbf{P}^* \tau^* / (\sin^2 \theta) \quad (1)$$

where \mathbf{P}^* is the normalized spontaneous polarization,

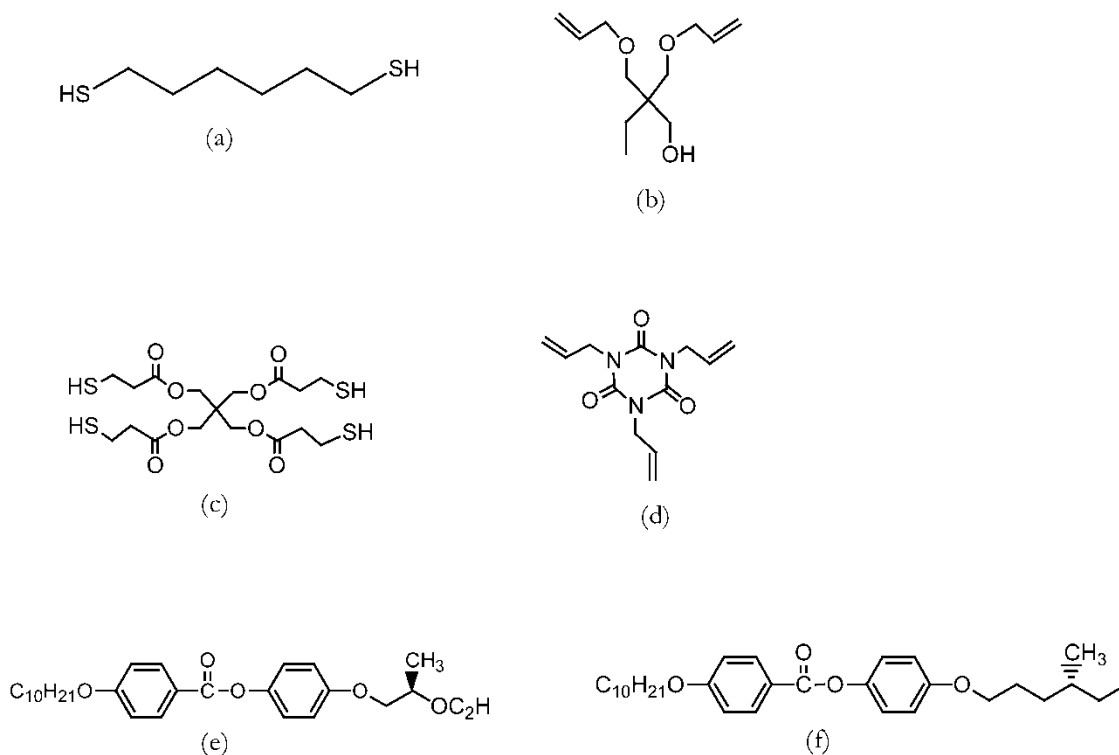


Figure 1. Chemical structures of the thiol-ene monomers, liquid crystalline monomer and ferroelectric liquid crystals utilized in this study. Shown are (a) 1,6-hexanedithiol, (b) trimethylolpropanediallyl ether, (c) pentaerythritol tetrakis(3-mercaptopropionate), (d) triallyl-1,3,5-triazine-2,4,6-(1H, 3H, 5H)-trione, (e) W7, and (f) W82.

τ^* is the normalized rise time, $(\sin^2 \theta)^*$ is the normalized sine squared of the tilt angle, and γ_n^* is the normalized rotational viscosity of the LC with the rotation of the director about an axis normal to the director and the spontaneous polarization [30].

Contrast ratio experiments were performed using the following technique. Polarized light from a HeNe laser (Uniphase Model 1508P-0, Manteca, CA) ($\lambda = 633$ nm, power = 0.95 mW) was passed incidently through the sample with an area of 25 mm², followed by a cross polarizer and a photodiode detector that collected the transmitted light from the sample. The corresponding electrical signal was recorded on a digitizing oscilloscope, and these electrical values were used to determine the contrast ratio, C ,

$$C = \langle I_2 \rangle / \langle I_1 \rangle \quad (2)$$

where I_2 is the intensity of the transmitted light in the 'bright' state, and I_1 is the intensity of the transmitted light in the 'dark' state [31]. The complete absence of light was set to zero transmission, so that the absolute intensity of light transmission was being monitored.

3. Results and discussion

The electro-optic properties of a LC system are one of the most important characteristics for determining

the viability of the system for various applications. If the enhanced optical characteristics of FLCs are to be utilized, the beneficial mechanical effects of polymer stabilization must not significantly hinder the switching speed or LC alignment. Thus, the effect of polymer on the electro-optical properties of the FLC mixture was investigated with both a crosslinked and a linear thiol-ene system.

The variation in molecular mass evolution of thiol-ene polymerizations allows the polymer to remain soluble in the FLC until it is locked into place as the system very rapidly transforms from low to high molecular mass (linear polymer) or a gel (crosslinked polymer). In the absence of phase separation, the rotational viscosity of the FLC solution is increased due to the creation of polymer in the interlayer spacing. The optical response is also hindered, depending on the type and amount of polymer in the interlayer spacing of the FLC. The effect of crosslinked polymer stabilization on the FLC properties was first examined by varying the concentration of crosslinked thiol-ene polymer in the system. To determine the impact of the polymer on the FLC properties, a baseline of the characteristics of the monomer were evaluated (see figure 2).

The rise time and tilt angle of the FLC decrease as the monomer concentration is increased, which is caused by

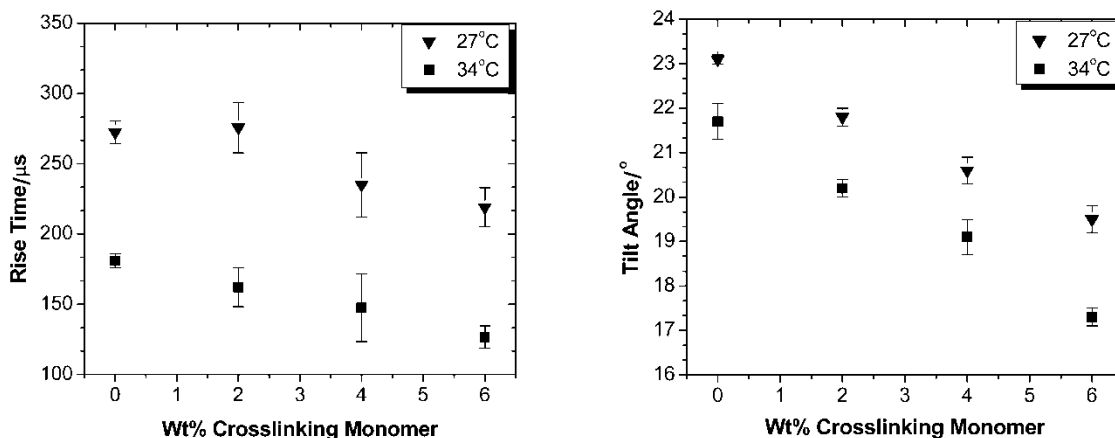


Figure 2. Baseline optical response characteristics for the crosslinking monomer solution. The decreasing trend in both rise time and tilt angle is due to the monomer acting as an impurity.

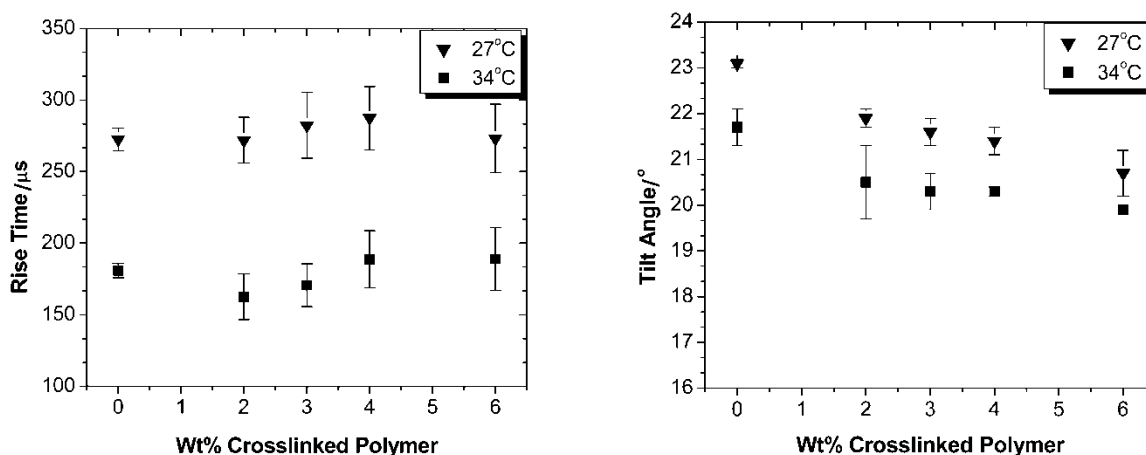


Figure 3. Optical characteristics of the crosslinked polymer system at varying concentrations of polymer. Due to the increased viscosity of the crosslinked polymer network, the rise time remains constant over the specified concentration range.

the increasing level of impurities in the system. Impurities, such as monomer and polymer, lower the phase transition temperatures, which directly correlates to a reduction in the tilt angle in the smectic at a fixed temperature. The rotational viscosity of the FLC molecules in the monomer system remains low, as shown in table 1, where the normalized rotational viscosity for the 6 wt% crosslinking monomer system is within experimental error of unity, showing no increase in rotational viscosity as compared with the neat FLC. Thus, with the retention of high mobility and a reduction in the tilt angle, the FLC rotates a shorter distance during switching, yielding shorter rise times.

The rise time and rotational viscosity show that the addition of the crosslinking monomer to the FLC does not significantly hinder the rotational ability of LC molecules. Upon polymerization of the crosslinking monomer mixture, the rise times of the polymer systems

increase over their baseline monomer characteristics, as seen in figure 3. This increase in rise time is due in part to the formation of a crosslinked network that confines the FLCs to domains. However, as seen in table 1, the rotational viscosity of the 6 wt% polymer system shows a minimal increase upon polymerization and creation of these FLC domains.

The rise time remains constant within experimental error over the polymer concentration range studied. As stated, the LC molecules become confined to domains

Table 1. Electro-optic properties of 6 wt% crosslinking thiolene before and after polymerization.

	Normalized polarization	Normalized rotational viscosity
Monomer	0.87 ± 0.03	0.97 ± 0.13
Polymer	0.91 ± 0.03	1.12 ± 0.12

and this domain confinement slightly increases the rotational resistance of the LC molecules. Thus, with the domain confinement, the time required for the change of optical state from 'dark' to 'bright,' or *vice versa*, is minimally increased. As the polymer concentration is increased, domain confinement additionally increases, which leads to small increases in the FLC rotational viscosity. The constant rise time is a consequence of the equivalence of the minimal rotational mobility restrictions and the reduction in cone rotation distance with the decrease in tilt angle.

In addition to the rise time increase, the tilt angle is shown to increase at each concentration over the respective baseline monomer characteristics (see figures 2 and 3). These higher tilt angles suggest that the polymer has an orientational effect on the FLC, promoting a higher degree of FLC order. This organizational effect has been documented by Hikmet and Michielsen [18] for systems utilizing an acrylate polymer network, where uniaxially oriented bulk FLC molecules remained uniaxially oriented throughout the nematic and ferroelectric ranges. In addition, they found that the birefringence of the FLC network remained constant with increasing temperature before rapidly decreasing at a temperature above the clearing point of the FLC. These characteristics of the FLC network show that the polymer gel has a significant impact on the FLC molecules not chemically bonded to the gel. In our system, the increase in the tilt angle of the polymer systems over the baseline monomer systems shows that there is an interaction between the polymer and the FLC molecules. Preliminary indications of this polymer ordering effect were shown in previous work [23]. Crosslinking monomer dichroism studies accomplished by Cramer *et al.* showed that the crosslinking monomer segregates in the interlayer spacing of the FLC [23]. Polymerization of this aligned monomer creates an aligned polymer in the interlayer spacing and relegates the LC molecules to domains, thus increasing the organization of the LC molecules. Thus, the polymer directly influences the increased order of the FLC molecules.

A second indication of an organizational increase with the addition of a polymer gel is found from the contrast ratio measurements. Table 2 shows the contrast ratio for the pure W7/W82 FLC system and the 6% crosslinked polymer FLC systems. The studies show

Table 2. Contrast ratio comparisons of neat FLC and two differing polymer samples.

Sample	Contrast ratio
Pure W7/W82	12.9 ± 2.3
6 wt% crosslinked polymer	17.3 ± 1.6
6 wt% linear polymer	8.4 ± 1.9

that there is an increase in the contrast ratio of approximately 30% from the pure W7/W82 system to the 6 wt% crosslinked system. This increased organization allows for an enhanced dark state, as the aligned LC molecules allow no light leakage. A similar increase in contrast ratio was previously demonstrated by Li *et al.* using a LC diacrylate stabilizing network; they showed that the contrast ratio of a conventional surface stabilized FLC cell could be increased approximately two-fold by the addition of 2 wt% LC diacrylate [25]. This increase in contrast ratio was directly associated with an increase in alignment due to the presence of the diacrylate polymer network.

Presently, as utilized in the research described above, the most commonly used technique for forming a crosslinked network that is able to remain in the FLC is through the use of LC diacrylates [13, 14, 18, 25, 32]. As stated earlier, previous research utilizing aliphatic diacrylates confirmed that the diacrylate would phase separate from the FLC upon polymerization [22], thus leaving the LC diacrylate as the predominant form of crosslinked stabilization. Unfortunately, Hikmet and Boots [33] determined that the maximum scattering for these anisotropic gels decreases with increasing polymer concentration. Consequently, as the stabilizing polymer is increased, the optical properties of the system are reduced, and there is a definite break-even point for the amount of polymer that can be introduced while still maintaining the enhanced properties of the FLC. However, as we have shown, utilization of thiol-enes to create a crosslinked network that remains in the FLC does not appear to encounter the negative drawbacks of the LC diacrylate systems. We are able to maintain fast switching speeds with little or no effect on the rotational viscosity of the system. More importantly, we can increase the contrast ratio of our FLC system upon polymerization of the crosslinked thiol-ene system.

In addition to the crosslinking system, we analysed the effect of a linear system on the electro-optic behavior of the FLC. Due to the lack of a network structure, the linear system exhibits different electro-optic performance as compared with the crosslinked system. By utilizing these two systems, we are able to compare the relative impact of rotational viscosity on the optical properties of the FLC. The optical properties of the linear monomer system are very similar to those of the crosslinking monomer system (see figure 4), implying that neither the linear nor crosslinking thiol-ene monomer systems have a significant effect on the switching ability of the FLC. The high mobility of the linear system is exemplified in table 3, where the 6 wt% linear monomer system also demonstrates a normalized rotational viscosity of approximately unity. The molar

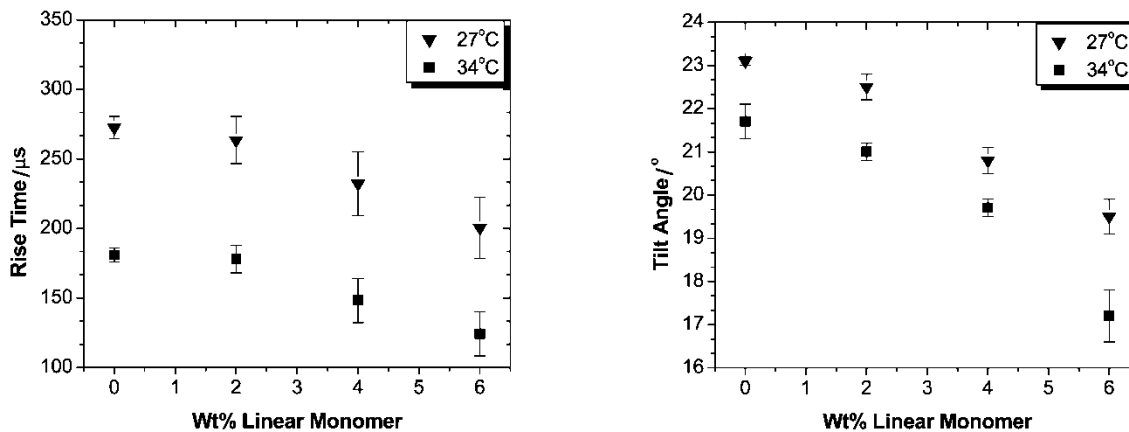


Figure 4. Optical characteristics of the linear thiol-ene systems before polymerization. The decreasing trends in rise time and tilt angle are due to the increasing levels of impurities in the system.

Table 3. Electro-optic properties of 6 wt% linear thiol-ene before and after polymerization.

Sample	Normalized Polarization	Normalized rotational viscosity
Monomer	0.91 ± 0.07	0.93 ± 0.14
Polymer	0.61 ± 0.12	0.75 ± 0.15

concentration of monomer in both systems is of the same order, and at the small concentrations of monomer utilized, the effect on the optical properties is approximately equivalent. The system independence described above does not hold subsequent to polymerization, as the rotational viscosity is not equivalent for the cross-linked and linear polymer systems. Optical properties for this linear polymer system are shown in figure 5.

In contrast to the crosslinked polymer systems, where

the polymer shows an increase in rise time with polymerization, the rise time for all linear polymer concentrations is lower than that of the neat FLC. Moreover, as seen from figures 4 and 5, the rise time for the polymer samples is not significantly different from the corresponding baseline monomer samples. The optical response supports the phase separation findings from the previous SAXS and phase transition data [23]. First, if the linear polymer remained in the interlayer spacing, it should have a similar organizational effect to that of the crosslinked polymer. However, the linear polymer data actually show a reverse trend in this respect, with an apparent decrease in tilt angle and contrast ratio following polymerization. With the apparent reduction in tilt angle, it appears that the alignment of the FLC is actually being negatively impacted. The lowered contrast ratio indicates that there are many

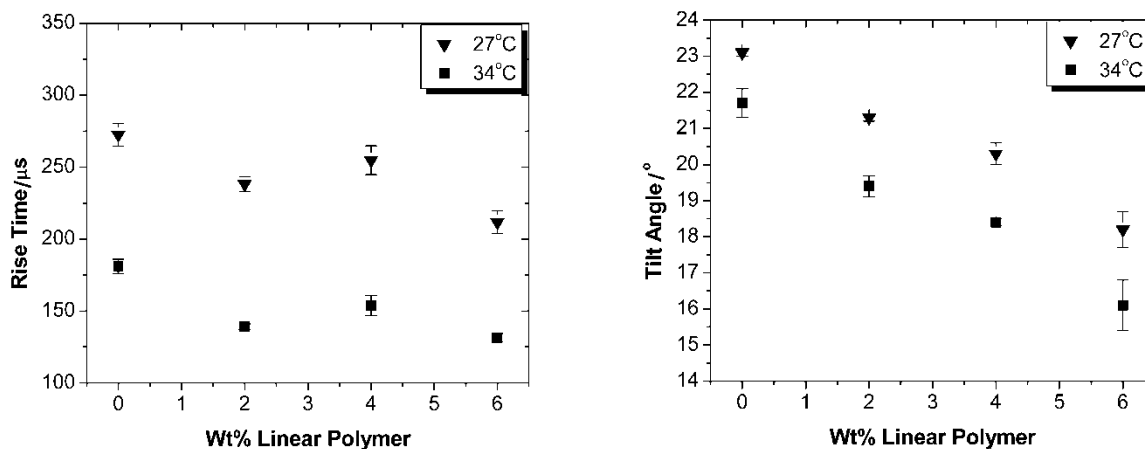


Figure 5. Optical properties of the linear thiol-ene polymer system. Rise times for the varying concentrations of polymer are equivalent or lower than those for the neat FLC. Tilt angles decrease with increasing polymer concentration and all polymer concentrations exhibit a lower tilt angle as compared with their corresponding monomer concentration.

small FLC domains and numerous defects, which lead to varying degrees of transmission and scattering as light passes through the sample.

The second indication of polymer separation is the apparent rotational viscosity of the linear systems. With the addition of polymer to the system, the rotational viscosity should increase if the linear polymer remains in the interlayer spacing. This increase in rotational viscosity should translate into an increase in rise time for all polymer concentrations, with the higher concentrations showing a more negative effect on the ability of the FLC to switch. However, the rise time for all concentrations of polymer is lower than that for the neat FLC, and the increasing effect on the rise time for higher polymer concentrations is not evident. Additionally, as presented in table 3, rotational viscosity measurements on the 6wt% linear polymer sample provide further evidence of the rotational viscosity decrease and FLC misalignment.

The rotational viscosity presented in table 3 seems to be contradictory to the physical effects of an introduced polymer. Even in the presence of phase separation, the introduced polymer should not act as a lubricant to rotation, as indicated by the rotational viscosity. However, as reported by Chandani *et al.* [34], there is a correlation between the LC alignment and the quality of the collected polarization data. A reduction in alignment quality drastically reduces the accuracy of the polarization data that can be acquired. The data presented strongly indicates that the FLC is becoming misaligned under the influence of the phase separating linear polymer. Thus, without proper alignment in the post-polymerized state, polarization measurements are prone to significant error.

The results of the experiments described above show that there is a drastic difference in the phase separation tendencies of the crosslinking and linear polymer systems. It was shown that the crosslinking polymer system remains in the interlayer spacing, but the linear polymer system phase separates from the interlayer spacing. One possible explanation for this occurrence is the chemical differences between the monomer systems used. For instance, as seen in figure 1, the linear-ene monomer has a polar hydroxyl group, while the crosslinking monomers lack significant polarity. However, previous work using SAXS experimentation demonstrates that the chemical differences do not have a large impact on the overall phase separation tendencies of the monomers [23]. The SAXS experiments confirm that the two differing monomer systems both swell the layer spacing of the LC in a similar manner, illustrating that each is segregated prior to polymerization. Only upon polymerization does the linear system show phase separation, and this phase

separation is suggested to be due to the lack of a crosslinked network capable of confining the polymer to the interlayer spacing.

4. Conclusions

The optical properties of several PSFLC systems are described and analysed. The optical properties of the PSFLC vary dramatically depending on the type of polymer system utilized. The crosslinked polymer showed improved alignment due to the formation of domains caused by the polymer, as demonstrated by an increase in the contrast ratio relative to the neat FLC. In addition, crosslinked polymer systems showed a slight increase in the rise time with only a minimal increase in rotational viscosity. More importantly, LC domains are formed without the use of LC monomers, commonly used for anisotropic gels. However, the linear thiol-ene polymer demonstrated signs of phase separation from the interlayer spacing. The rotational viscosity of the linear system appeared to decrease significantly, and the switching speeds remained consistent with the monomer-FLC system. The reduction in rotational viscosity is an indication that the phase separating polymer reduced the FLC alignment, as demonstrated by the reduction in contrast ratio over the neat FLC.

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