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The Effect of Oligomer on Optical Transmissions and Applied Voltages of a Flexible Polymer Dispersed Liquid Crystal Formulation

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Abstract

We studied the effect of a di-acrylic oligomer on electro-optical performances of a flexible PDLC formulation by UV-curing phase separation technique. The study included the behavior of optical transmissions and switching voltages of PDLC as function of low concentration range of 2.5-25.5 weight percent of oligomer. The results showed improvements of all optical transmissions and saturation voltage, except the threshold voltage, at around 10% range of oligomer concentration. We discussed these results on the bases of different morphologies and surface anchoring originating from refractive index and photo-initiator effects of oligomer.

Keywords: Oligomer; Liquid crystal; Optical transmission; Contrast ratio; Haze; Applied voltage

Introduction

During the past few decades, the Polymer Dispersed Liquid Crystal (PDLC) technology has been the subject of enormous academic and industrial research and development, which have resulted to many scientific publications, as well as industrial-scale manufacturing and commercialization of various products worldwide. Although there all over twenty global producers of PDLC films and glass products, but the major scientific publications have been dominating by academic studies.

In addition to general PDLC review literature [1-11], the published studies have been carried out by most academic and few industrial research on the effect of different material and process conditions, including the effects of matrix and liquid crystal chemical structures [12-21], material compositions [22-32], curing process parameters [33-47], film thicknesses [48-50], nanoparticles [51-53] and dyes [54-58] on morphology and electro-optical properties of PDLC films. The correlation between materials and process conditions with electro-optical performances of PDLC is essential for industrial development and manufacturing of flexible PDLC products. Currently, the majority of industrial developments and productions of commercial products are based on UV-cured PDLC by Polymerization Induced Phase Separation (PIPS) method. Among other parameters, the chemical and physical parameters of materials influence on the kinetics of phase separation and dynamics of matrix polymerization, which determine the morphology and electro-optical properties of final PDLC products.

At the industrial scale, the relations between the material and process conditions are essential to establish direct quantitative correlations between process parameters and electro-optical properties on quality control and manufacturing of PDLC film products. In this respect, we have reported a series of industrial-scale studies on the effects of material and process parameters on morphology and electro-optics of flexible PDLC films. These studies have shown the effects of these factors on polymer matrix micro-structure; liquid crystal droplet size and number density; optical transmissions; switching voltages and response times in various flexible UV-cured and thermoset PDLC formulations [19,29-31,39,41,45,46,49,50]. Within an industrial production program, such studies aimed on developing empirical relations between formulation, process parameters, morphology and electro-optics for subsequent scale-up and manufacturing. Such relations are essential for direct quantitative correlations between process parameters and electro-optical properties during PDLC production.

Although the effect of chemical and physical parameters on the performances of PDLC have been published extensively, the choice of oligomer was based on our preliminary unpublished experimental works, which had indicated that by substituting the PDLC matrix with certain amount of an oligomer could improve the phase separation, polymerization kinetics, increase the resistivity of matrix and subsequently the electro-optical properties of PDLC formulations. To the best of our knowledge, the use of oligomer in PDLC has been reported in the literature only as photo-initiator [32] and surface modifying agent [59]. Consequently, in the present study we explored the effect of a photo-curable di-acrylate oligomer on the electro-optical performances of an industrial-scale PDLC formulation to determine the improvements of optical transmissions and switching voltages of UV-cured PDLC formulation as a function of oligomer up to 2.5-25.5% concentration range. The experimental results of this study are presented and discussed in the subsequent sections.

Experimental

Materials

The utilized materials in this study consist of commercial Q142 liquid crystal mixture obtained from Qingdao under the trade name of QYPDLC142 having the following optical and thermal properties:

a. birefringence: $\Delta n=0.251$;

b. ordinary refractive index: $n_o=1.525$;

c. nematic-crystal transition temperature: -20 °C;

d. nematic-isotropic transition temperature: +105 °C.

The matrix N65 pre-polymer under the trade-mark NOA65 with refractive index $n_p=1.524$ was procured from Norland Optical Adhesives. The oligomer B744 under the trade-mark Bomar® BR-744P, which is an aliphatic polyester urethane di-acrylate and a di-functional oligomer with refractive index $n_B=1.474$ was purchased from Bomar. B744 has the ability to undergo free radical polymerization, improves adhesion and is used to formulate adhesives, inks, coatings, and a variety of other products. The Irga-Cure 819 (819) and Irga-Cure (814) photo-initiators were obtained from Ciba. The UV absorber Tinuvin (TV400) was procured from BASF. The 25mm plastic micro spacer NM was obtained from Suzhou Nanomicro and Acrylic Acid (AA) was procured from Kaitai. All materials were utilized as-such without further purification.

Preparation and methods

The flexible PDLC samples were prepared with Polymerization Induced Phase Separation (PIPS) technique by UV radiation of homogeneous mixtures of liquid crystal Q142, pre-polymer N65 and B744 oligomer and other materials. The material compositions (weight %) of the base PDLC formulation were as follows:

Q142=40% / N65=51% / AA=4% / TV400=4% / 819=0.5% / 184=1.0% / NM=0.6%.

The B744 oligomer was systematically substituted for N65 in the PDLC formulation within 2.5-25.5 weight percent concentration range. The final PDLC formulations with various substituted compositions of B744 oligomer are tabulated in Table 1, where all weight percent concentrations of utilized materials in base formulation and those with B744 are highlighted in bold cases. The uncured PDLC formulations of Table 1 were pre-heated at 50 °C for 10 minutes and then, as presented in Figure 1, were poured between the vertical gap of two support ITO-PET film rolls on a custom-made coater/lamination system (Sigma Sivo) and under the coating rolls the uncured PDLC sandwich were passed through a pressure roll to insure the uniformity of PDLC film. The thickness homogeneity of PDLC layers were insured by 25mm plastic NM micro-spacers.

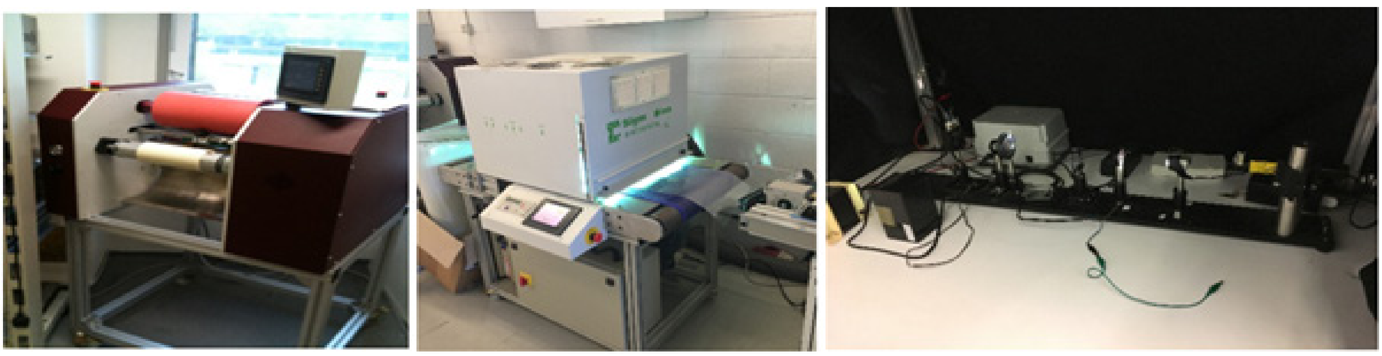

Figure 1: Lab-scale coater/laminator(left), UV-IR curing conveyor (middle) and electro-optical system (right).

Table 1: The base PDLC formulation and those with various concentrations of B744 oligomer.

Q142/N65/AA/TV400/I819/I184/ NM: (40/51/4/4/0.5/1/0.6)
Q142/N65/B744/AA/TV400/819/184/NM: (40/48.5/2.5/4/4/0.5/1/0.6)
Q142/N65/B744/AA/TV400/819/184/NM: (40/45.9/5.1/4/4/0.5/1/0.6)
Q142/N65/B744/AA/TV400/819/184/NM: (40/40.8/10.2/4/4/0.5/1/0.6)
Q142/N65/B744/AA/TV400/819/184/NM: (40/35.7/15.3/4/4/0.5/1/0.6)
Q142/N65/B744/AA/TV400/819/184/NM: (40/30.6/20.4/4/4/0.5/1/0.6)
Q142/N65/B744/AA/TV400/819/184/NM: (40/25.5/25.5/4/4/0.5/1/0.6)

As presented in Figure 1, The uncured PDLC film samples were then cured on a custom-made UV-IR conveyor belt machine (Sigma Sivo) equipped with a high-pressure UV mercury lamp and infrared heater. The curing was accomplished by PIPS phase separation technique at UV intensity of $72mW/cm^2$, line speed of 0.3 meter/min and 40 °C cure temperature. The experiments were carried out on three PDLC samples for each formulation and the reported electro-optical results are the average values of three samples. Also as presented in Figure 1, the electro-optical properties of PDLC samples were carried out on a specially constructed bench-top photometric system mounted on an optical rail consisting of white light source, sample holder, photometer, amplifier, function-generator and electronic data acquisition network. The optical transmissions and switching voltages of PDLC films were measured at room temperature through transmission-voltage curves with VAC square-wave at 100Hz frequency. The on-state haze of PDLC samples was measured by BYK-Gardner Haze-Guard with a white light source.

Result and Discussion

The experimental results of optical transmissions including opacity (T_{off}), transparency (T_{on}), contrast ratio ($CR=T_{on}/T_{off}$) and on-state haze (H_{on}), as well as the switching voltages including threshold voltage (V_{10}) and saturation voltage (V_{90}) of PDLC film formulations at corresponding concentrations of B744 oligomer are tabulated in Table 2. The optical transmissions were determined from transmission-voltage curves, where T_{off} and T_{on} were the optical transmissions in the absence and at the maximum applied voltages, respectively. The switching voltages were also measured from transmission-voltage curves, where V_{10} and V_{90} were measured at 10% and 90% of optical transmission values, respectively. The details of these experimental measurements are discussed in the following sections.

Table 2: The electro-optical properties of PDLC films at various B744 oligomer concentrations.

B744 (wt%)	T_{off} (%)	T_{on} (%)	CR	H_{on} (%)	V_{10} (volt)	V_{90} (volt)
0	2.8	76.5	30.8	5.6	18.4	38.8
2.5	2.5	75.2	30.1	6.4	19.3	37.7
5.1	2.3	74.9	30.7	6.9	21.2	37.0
10.2	2.1	76.9	36.6	4.9	23.9	38.2
15.3	2.9	75.6	26.1	5.3	20.3	37.0
20.4	4.4	75.5	17.2	6.1	16.8	38.9
25.5	6.6	75.6	11.5	7.1	16.4	41.8

Effect of oligomer on optical transmissions

From the data of Table 2 and Figure 2, we present the T_{off} (opacity) and T_{on} (transparency) of PDLC film as a function of oligomer concentration. The T_{off} initially exhibits a decreasing trend below 10% and rapid increase above 10% B744 oligomer concentration ranges. In particular, we observe a distinct minimum value of T_{off}=2.1% at 10.2% oligomer concentration. Also, in Figure 2, we observe a sharp decline trend of T_{on} up to 5% of oligomer concentration range followed by an increasing trend up to a maximum value of T_{on}=76.9% at 10.2% oligomer concentration. However, above this concentration range, Ton begins to decrease again and reaches a plateau value of around 75.5%. Consequently, both optical transmissions T_{off} and T_{on} of PDLC film formulations exhibit improvements with minimum T_{off} (maximum opacity) and maximum T_{on} (maximum transparency) at around 10% concentration range of B744 oligomer. In the absence of other parameters, such as morphology, we could only explain the effect of oligomer on the improvement of optical transmissions should be due to index matching effect of liquid crystal droplets and matrix at 10% oligomer concentration range.

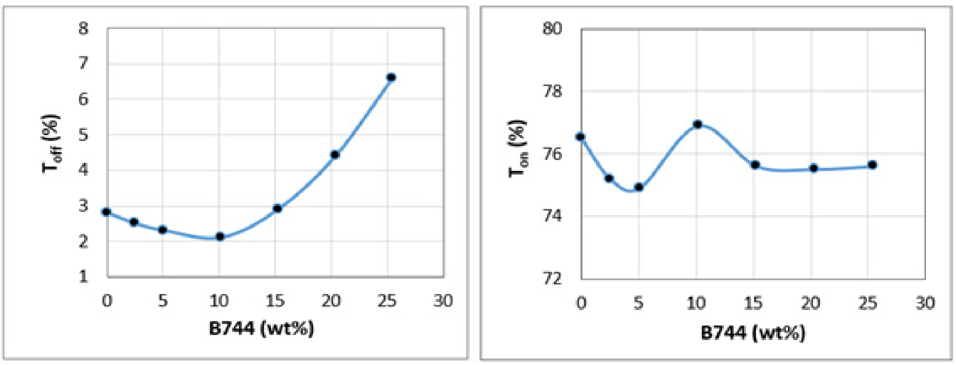

Figure 2: The T_{off} (left) and T_{on} (right) values of PDLC film as a function of B744 oligomer concentration.

It is a common knowledge that, during the phase separation and matrix polymerization, some uncured pre-polymers are trapped in the liquid crystal droplets and the matrix is plasticized by certain amount, which could significantly affect the optical transmissions of PDLC film. In the present PDLC formulation, although the initial refractive indices of Q142 (n_o=1.525) and N65 (n_p=1.524) are initially equal, but due to the residual presence of B744 (n_B=1.474) in both liquid crystal droplets and matrix, one expects a more significant changes in the index miss-matching of PDLC film due to composition variations of B744 oligomer. Therefore, one expects that, except at around 10% B744 concentration range, which exhibit minimum T_{off} and maximum Ton values, the index miss-matching is responsible for the behaviour of these optical transmissions at other concentrations below and above 10% concentrations. Such results have been also reported in the literature, where the effect of refractive index provided increase transparency and decrease opacity in PDLC [60,61].

Also, according to Figure 2, the trends Toff shows gradual decrease below 10% and exponential increase above 10% B744 concentration ranges. Such trends could be also explained due to variations in droplet morphology and scattering, which could be described according to the following known Beers-Lambert relationship [62].

$$T_{off} = T_o \cdot \exp^{-(\eta.\delta.d)} \quad (1)$$

Where T_{off} is the light transmission in off-state, T_o is the incident light intensity, η is the droplet number density, δ is the "scattering cross section" of a single droplet and d is the sample thickness. Therefore, in addition to the effect of refractive index, the minimum value of T_{off} at 10% B744 concentration should also be a result of highest number density and scattering cross-section of PDLC morphology. It should be also noted that, the di-acrylic photo-initiator nature of B744 oligomer is another parameter that should contribute to the phase separation, polymerization kinetic, morphology and electro-optical properties of PDLC. However, due to the lack of such experimental data, it is not possible to further elaborate on the photo-initiator effect of B744 oligomer in the present study. In addition, according to Table 2 and Figure 3, we also present the effect of B744 oligomer on the contrast ratio (CR=T_{on}/T_{off}) and on-state haze (H_{on}) of PDLC films. In respect, we also observe that similar to T_{on} and T_{off} trends, the contrast ratio exhibits a maximum value of CR=36.6 at 10% oligomer concentration range. Also due to the effect of refractive index, the on-state haze shows a minimum value of H_{on}=4.9% at around 10% B744 concentration. Subsequently, whereas at above 10% oligomer concentration range, the CR decreases but Hon increases sharply, their trends optical are reversed below 10% oligomer range.

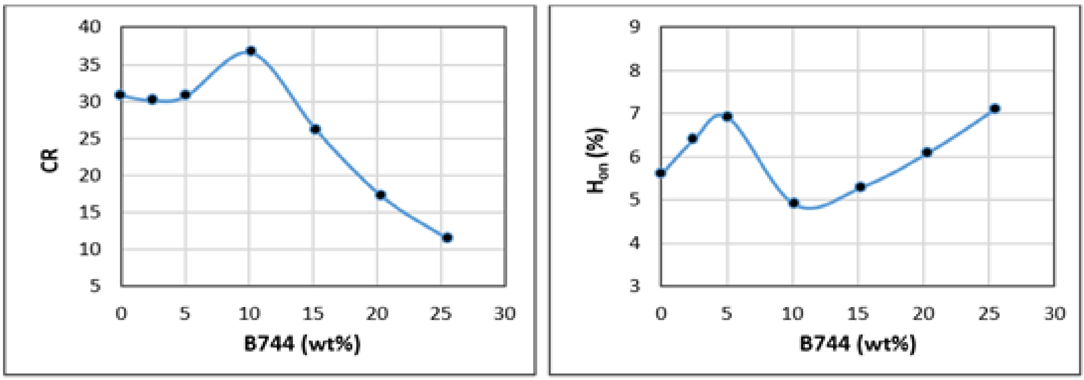

Figure 3: The effect of B744 oligomer concentration on CR (left) and H_{on} (right) values of PDLC film.

Therefore, in agreement with T_{on} and T_{off} behaviour, the CR and H_{on} of studied PDLC formulations also exhibit improvements at around 10% concentration range of B744 oligomer. Although in the present study we did not study the contributions of refractive index and B744 photo-initiator effects on morphology and electro-optical performances, one could only speculate that improvements in the optical transmissions within 10% B744 concentration range should also originate from the complex relations between the refractive index, curing conditions and morphology in the studied PDLC formulation.

Effect of oligomer on switching voltages

The variations of threshold voltage (V_{10}) and saturation voltage (V_{90}) of studied PDLC film formulations as a function of B744 oligomer composition that are tabulated in Table 2 are presented in Figure 4. Accordingly, in contrast to optical transmissions, the overall trends of switching voltages with oligomer concentration are rather different in the studied PDLC formulations. Namely in the case of V_{10}, we observe a consistent increasing trend below and decreasing trend above 10% B744 concentration ranges. Furthermore, the result clearly indicates that V_{10} exhibits a typical maximum value at 10% B744 range. On the other hand, in contrast to V_{10} behaviour, V_{90} exhibits a decreasing trend below and increasing trend above 10% B744, where V_{90} exhibits a small maximum value at this concentration range.

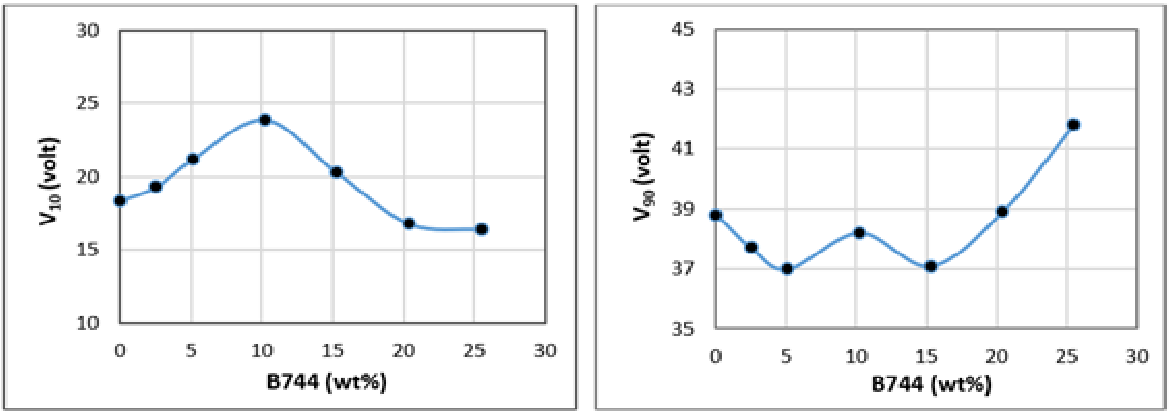

Figure 4: The effect of B744 oligomer concentration on V_{10} (left) and V_{90} (right) values of PDLC film.

The controversial reverse trends between V_{10} and V_{90}, in particular their respective maximum values within the 10% B744 oligomer concentration range, could be partially explained by reference to the general theoretical relation between switching voltage and PDLC parameters according to the following relation [63]:

$$V = \frac{d}{<R>} \cdot \left(\rho_p + {}^{2}\!/_{\rho_{lc}}\right) \cdot \left\{ {}^{(K-1)}\!/_{(\varepsilon_{11} \cdot \Delta\varepsilon)} \right\}^{1/2} \quad (2)$$

where d is the film thickness, <R> is the droplet radius, r_p and r_{lc} are the matrix and LC resistivity, K is the average elastic constant, e_{11} and Δε are parallel and anisotropy of dielectric constant. The Equation-2 shows that the switching voltage is inversely proportional to droplet dimension. By referring only to this relation, we could speculate that, the liquid crystal droplet dimension decrease at 10% B744 concentration range. However, this effect could partly explain the opposite trends of V_{10} and V_{90} within the total concentration range of B744 oligomer. At this point, as has been also reported elsewhere [60], we can only hypothesize that in the present PDLC formulation, due to the presence of oligomer, the relation between switching voltages and morphology could be also depend on nonlinear dependencies of switching voltages with resistivity, elastic and dielectric parameters of liquid crystal and matrix. We also found an agreement with a recent study on the improvement of electro-optical performances of PDLC with a photo-initiator oligomer [32], which provided lower saturation voltage (V_{90}) and higher Contrast Ratio (CR) than those of identical PDLC formulation with monomer photo-initiator. In addition, this study has shown that, the photo-initiator oligomer allowed premature phase separation between the liquid crystal and matrix phases, resulting in relatively pure LC-rich phase, which resulted, not only a well-defined LC/matrix morphology, but also a low driving voltage (V_{90}) as a result of reduction in surface anchoring at the LC/matrix interfaces.

However, such speculation does not explain the different trends between V_{10} and V_{90} as function of B744 oligomer concentration. Considering the surface anchoring effect within liquid crystal droplets, one could expect that, only at 10% B744 concentration range V_{10} threshold voltage and V_{90} saturation voltage experience similar high surface anchoring. Such explanation suggests that, in the present studied PDLC formulation the surface anchoring factor seems to dominate over morphology only at 10% oligomer concentration range. However, other physical and chemical phenomena of oligomer and liquid crystal, such as viscosity, molecular polarities or other factors, should be responsible for different V_{10} and V_{90} trends within the overall studied concentration range of B744 oligomer.

Last but not the least, as it was mentioned in optical transmissions section, the refractive-index and photo-initiator effects of di-acrylic B744 oligomer are other factors that influence the switching voltages. Namely in the present PDLC formulation, as B744 was gradually is substituted for N65 up to 25.5%, it has not only reduced the overall refractive index of the matrix, but also by increasing the photo-curing function, it affected the morphology and electro-optical performances of PDLC films within the B744 concentration range. Except at 10% oligomer concentration range, a combination of B744 refractive-index and photo-initiator effects seems to be responsible for different trends of switching voltages contributing to different surface anchoring at V_{10} and V_{90}. However, it should be noted that due to the lack of more detailed experimental data regarding the effects of utilized oligomer on curing parameters and morphology, it is not possible to make further elaboration on contrasting trends of V_{10} and V_{90} in the present study.

Conclusion

In the present work, we studied the effect of di-acrylic B744 oligomer on the optical transmissions and switching voltages of a PDLC formulation. The study was part of our industrial R&D program, aiming at continuous improvements of electro-optical properties of flexible PDLC products for eventual scale-up and manufacturing. We found that optical transmissions T_{off}, T_{on}, H_{on} and CR of a selected lab-scale PDLC formulation were improved within the 10% concentration range of B744. The most reasonable explanation for these results were argued on the improved index-matching and morphology of PDLC at this concentration range. With respect to the switching voltages the results were different, where although the V_{10} exhibited an increasing trend, but V_{90} showed partial improvement due to a partial increase within B744 10% concentration range. Due to the lack of other experimental data, we could only speculate that in contrast to V_{10}, the partial improvement of V_{90} could be due to the surface anchoring differences of liquid crystal droplets between the threshold and saturation voltages.

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