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Analysis of dichroic dye-doped polymer-dispersed liquid crystal materials for display devices

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ABSTRACT

Dichroic polymer-dispersed liquid crystals (DPDLCs) based on nematic liquid crystal materials with azo dye were investigated in detail for the application of display devices. Polarizing optical microscopy, differential scanning calorimeter and electro-optic experiments all have shown that the DPDLC containing low concentration of dyes modifies the basic properties of these materials like optical transmission, threshold voltage, contrast ratio and absorbance factor. A minimum amount of dye needs to be added to the liquid crystal with the polymer matrix for its effective phase separation and to minimize the transmittance in the OFF state and therefore gives rise to an overall improvement in contrast ratio of the devices. Molecular orientation and dynamics in droplet sizes are readily controlled in these DPDLC materials. These findings imply that the value of the threshold electric field E_{th} is approximately 8 V/µm for pure polymer-dispersed liquid crystal (without dye) where the threshold electric field E_{th} values are approximately 4.0 V/µm, 2.0 V/µm, 1.7 V/µm 1.0 V/µm, for 0.0625%, 0.125%, 0.25% and 0.5% with azo dye in DPDLCs, respectively. From the results we can also infer that the maximum contrast is approximately 2.55 times the minimum contrast observed in the experiment for DPDLCs. The results show that the DPDLC with proscribed dye concentration will be possibly suitable and promising functional electronic materials for green technology flexible liquid crystal display.

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1. Introduction

Molecular materials and polymeric systems have been considered to be promising optical materials for optimization and application in display devices [1–3]. Research advances in this field depend on the development of advanced materials with low threshold field and high contrast. Polymer-dispersed liquid crystal (PDLC) [1–6] materials, in which randomly orientated liquid crystal (LC) droplets are dispersed in a polymer matrix and polymer stabilized liquid crystal (PSLC) [7,8] have attracted a great deal of interest, especially in the fields of materials science and nanotechnology due to their manifold applications for instance in full color reflective displays [9], light shutters [10] and Frensnel lenses [11]. PDLCs are typically obtained by polymerization-induced phase separation [12] of the LC molecules from the polymer network. The rigidity, density and curing rate of the polymer govern the phase separation kinetics and the size of the LC

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E-mail addresses: pkumartiet@gmail.com (P. Kumar), swkang@jbnu.ac.kr (S.-W. Kang), lsh1@chonbuk.ac.kr (S.H. Lee), kkraina@gmail.com (K.K. Raina). droplet during the photo-polymerization. PDLCs show transparent and light-scattering states in the electric field ON and OFF states, respectively. As in the electric field on-state, the LC directors align themselves along the field, which results in a change from a highly scattered state to a transparent one.

However as a result of their low contrast ratio and switching behavior, the use of PDLCs for display devices is limited. Generally, to improve the contrast ratio, the use of dichroic dye-doped liquid crystal was proposed [13–15] and research of this phenomenon has been focused on the manufacture of photocontrollable devices which have a high contrast ratio [16–19]. Hsiao et al. [20] found that electrically switched distributed-feedback lasing action occurs in a dye-doped holographic polymer-dispersed liquid crystal (H-PDLC) transmission grating structure. Fuh et al. [21] fabricated electrically switchable spatial filters using dye-doped PDLC films. Petti et al. [22] observed the enhancement of the thermo-optical properties of PDLC upon doping with dye.

It is known that a change in the transmittance of a PDLC can take place by the photochromic reaction of photochromic molecules such as azobenzene derivatives [13–15]. The configuration of azobenzene derivatives can be transformed reversibly through the process of photoisomerization. The cis-azobenzene is formed using UV light around 366 nm, and transformed into the trans-azobenzene by visible

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light around 433 nm. The cis-azobenzene destabilizes the LC phase, but on the contrary, trans-azobenzene is aligned with the LC molecules and enhances the LC phase and the mesophase of an azobenzene-doped LC can be controlled by light irradiation. The role of dyes in these systems is to allow the light absorption thus reducing remarkably the power necessary to induce non-linear optical effects. Dye doped LC can be highly absorbing as well as scattering in the unpowered state, while the powered state will be feebly absorbing and transparent [23–31]. The strong microscopic mutual interactions, among the dye and LC molecules, affect some parameters of the LC such as refractive index, dielectric constant, the orientational order, the phase transition temperature and molecular association [32-36]. When a dichroic dye with a geometric anisotropy is dissolved in a nematic LC, the dye molecules tend to arrange itself in such a way that its long molecular axis aligns along the LC director and a change of color intensity is obtained by controlling the direction of the LC molecules (Fig. 1b). A rod like, long molecular structure is favorable for a strong dichroic absorption. The orientation of the elongated dye molecule is governed by the nematic director configuration inside the droplet. Therefore, the dye absorbance is modulated by the orientation of the nematic director with an external field. In the OFF state, we assume random orientation from droplet to droplet of the symmetry axes of the bipolar droplets resulting in arbitrary orientation of the dye molecules (Fig. 1c). The extinction coefficient is therefore averaged over all droplets and is equal to the isotropic extinction coefficient of the dye. In the ON state, the droplet director and the dichroic dye molecules in the droplet are aligned normal to the surface of the film [Fig. 1(d)] and the dye extinction coefficient is equal to the perpendicular extinction coefficient. Dichroic dye that remains dissolved in the polymer binder will be unaffected by the external field and remain randomly oriented. Therefore, only the dye dissolved in the nematic droplet will exhibit dichroic properties and enhance the contrast of the film. In particular, absorption spectra and dichroic ratios of the dye in LCs are the dominant properties with regard to the performances of displays. In our previous research [37] we have observed that the controlled extent of dye is purposeful for improving the optical efficiency with reduced operating voltage and high contrast ratio of DPDLC. Consequently, in this paper the electro optics of the DPDLC materials has been investigated in detail using the azo disperse orange 3 dye.

2. Theory of DPDLC device

We consider a nematic liquid crystal (NLC) in which dye molecules has been homogeneously dispersed. The ordinary and extra-ordinary refractive indices of the birefringent NLC are n_0 and n_e , respectively. Outside these droplets is a cured polymer in the solid state with refractive index $n_p \approx n_o$. In the OFF state, the unintentional orientation of the symmetry axes of the droplets results in random orientation of the dye molecules. Therefore, when the film is thin or the scattering effect is not too much, the extinction coefficient is averaged over all droplets and approximates the isotropic extinction coefficient of the dye. In order to increase the brightness of the dichroic PDLC film, the dye concentration should be relatively low, and the film should be thin (We assume that there is no correlation between absorption and scattering). According to Beer's law [38], the OFF state transmittance can be written as

$$I_t = I_0 e^{-(\alpha + \beta)d} \tag{1}$$

where I_0 and I_t are the incident and transmitted light intensity and d is the sample thickness. α and β reflect the scattering and absorption coefficient of the film respectively. α is a function of the LC droplet size, curing temperature as well as mismatch in refractive indices between LC droplet and polymer matrix. Since polymer and LC molecules absorb nearly no visible light, thus the absorption of a PDLC film results from dye depending on extinction coefficient, concentration and distance *l* traveled by light to the film thickness (d) respectively. For normal incidence, l > 1 because of scattering, but for thin film $l \approx 1$ (the scattering effect is very small). In the ON state as shown in Fig. 1(b), the symmetry axes of the dye-doped NLC droplets reorient in only one direction, and thus the dye molecules absorb the light only slightly at normal direction because electric field vector of propagating light oscillates along the short axis of dye molecule. In addition, because of $n_p \approx n_o$, the scattering level is very low so that the largest portion of the incident light I₀ is transmitted through the dye-



Fig. 1. (a) UV–VIS absorbance of dichroic dye molecules, (b) alignment of dye molecules in a dichroic NLC droplet, (c) random orientation of the symmetry axes of the bipolar droplets (OFF state) and (d) alignment of the liquid crystal and dye molecules with the application of field (ON state) in DPDLC.

doped PDLC film. The path length of light is just the thickness of the film because there is no scattering.

3. Experimental details

DPDLC films were prepared by polymer induced phase separation method [39] using the materials, NLC E7 (E. Merck, UK), UV curable optical adhesive NOA-65 (NORLAND, NJ) and disperse orange 3 azo dye (Sigma Aldrich). The molecular structure and absorption spectrum of the dye molecule are shown in Fig. 2. The NLC shows a wide temperature range of nematic phase in which the dye molecules were dissolved in low concentration (0.0625%, 0.125%, 0.25%, 0.50% and 1.0% wt./wt). First, dye was doped with the NLC and then dispersed in the polymer matrix in the 1:1 wt./wt. ratio. In order to ensure proper mixing, this homogenous mixture was heated to isotropic temperature (60 °C) and simultaneously shaken in a vacuum chamber. The mixture was then filled in between indium tin oxide (ITO) coated glass substrates by capillary action. The sample cells with 10 µm gap (cell thickness) were sealed by optical adhesive and then cured under UV light (intensity~2 mW/cm²) for an hour at room temperature. The cell assembly was placed in a programmable temperature controller coupled to hot stage (Linkam Model TP 94 and THMS 600) and then cooled down to room temperature at the rate of 0.1 °C per min. Uniform dispersion of dichroic LC droplets in the polymer matrix was viewed under crossed polarizers at a magnification of 100× through polarizing optical microscope (POM) (Olympus Model BX-51P) fitted with charge coupling device (CCD) digital camera interfaced to a computer. The phase transition temperatures observed in POM study were confirmed with calorimetric measurements using a differential scanning calorimeter (DSC), (Lineseis DSC L63). Further, the electro-optical properties (optical switching and transmittance) of the cells, positioned between a pair of cross polarizers with a direction of 45° to the transmission axes of the polarizers were evaluated and compared with a PDLC cell that fabricated without using dye. The electro-optic responses were studied using white light spectra under an applied electric field to the sample with a function generator (Philips FG-8002). The output responses were recorded and detected on digital storage Oscilloscope



Fig. 2. (a) Molecular structure and (b) absorption spectrum of disperse orange3 azo dye.

(Tektronix Model TDS 2024) using a photo-multiplier tube (Model RCA 931-A) for analyses.

4. Results and discussion

4.1. Transition temperature

After dye addition in different concentrations, we noticed a significant change in nematic to isotropic (T_{N-I}) transition temperature for NLC in DPDLC films through POM study. DSC measurements of the materials were made to confirm this phase sequence and allow the quotation of accurate phase transition temperatures as well as temperature dependence electro-optic characteristics of DPDLC systems. We peeled DPDLC films from glass plates and 3.0 mg sample films were placed into an aluminum standard sample crucible and crimped shut, plus deliberated with DSC for a temperature scan at a heating rate of 5 °C/min. The sample was heated in the range from room temperature to 65 °C. Fig. 3(a) presents the DSC curves obtained during heating of the sample. The endothermic peaks at 61 °C, 60 °C, 58.9 °C, 57 °C and 54.4 °C are where the material undergoes a nematic to isotropic liquid transition for 0%, 0.0625%, 0.125%, 0.25%, and 0.5% DPDLCs, respectively. In Fig. 3 (b), we summarize the transition temperatures as obtained with POM and DSC. Comparing the results, there is a good agreement in the transition temperature and was



b) Variation in T_{N-I} with dye



Fig. 3. Differential scanning thermograms of (a) nematic liquid crystal E7, PDLC, and DPDLCs (up to .5% dye), (b) comparison of the phase transition temperature (T_{N-I}) measured in the DSC and POM experiment. Polymer (NOA 65) and liquid crystal (Nematic E7) contents were in 1:1 (wt/wt ratio) in all samples.

found similar within ± 1 °C, indicating that the phase transition of the LC droplets was decreased in the DPDLC with the addition of dye. It is noticed that at higher dye concentration, the T_{N-1} shows large variation as compared to the low dye concentration. As we reported earlier, one factor to this difference in the transition temperature is due to larger concentration of some ionic impurities arising from dye and monomer molecules when dissolved in LCs [37].

The transfer of energy from dye molecules to LC by means of the absorption of electromagnetic radiation at higher concentration also contributes to lowering the T_{N-I}. Thermal radiation, which increases with increasing temperature, is also a direct result of the movements of molecules in the material and reduced the order parameter of the LC mixture in DPDLC sample. At the same time, in the experiment the sample is constantly illuminated by electromagnetic radiation from light source therefore a net transition from higher isotropic temperature to lower temperature results. The other possible reason to lowering the T_{N-I} may be that the azo molecule which shows a rodlike shape when in the trans form, but is bent and banana-like in the cis form acts like an impurity and therefore destabilizes the liquid crystalline phase. However in our experiment the cis molecules' contribution to lowering the phase transition temperature may be up to some extent and is not the key aspect. As shown in Fig. 4 there is a sharp absorbance peak at around 340 nm indicating that the trans-cis photoisomerization has taken place.

4.2. Morphological characteristics of droplets

Fig. 5 shows the droplet morphology of DPDLC with different dye concentration at room temperature. It is seen that LC droplets distributed in DPDLC films form spheroid-like bipolar droplet configuration [Fig. 5 (a-f)] in a continuous matrix phase. In our experimental results we find that the bipolar droplet configurations are stable with respect to dye concentration. Microscopic observations as shown in Fig. 5 (f) revealed the presence of spherical bipolar LC droplets even when a relatively large amount of dye concentration was introduced. The structure of a droplet mainly depends on the elastic properties of LC and polymer. It is characterized by the spatial dependence of the director field and orientational order parameter. Director configuration of the nematics droplets depends essentially on the type of molecular anchoring at the interface. When the LC molecules are strongly anchored parallel to the surface and the elastic constant for the splay, twist, and bend deformation do not differ considerably, the bipolar structure [40] results with two point of defect on the axis of cylindrical symmetry. In Fig. 5 (f) the point defect of the droplets is not always on the axis of cylindrical symmetry,



Fig. 4. UV–VIS absorbance of dichroic liquid crystal mixtures with different dye concentration, measured at room temperature without any applied electric field.

some of them have a point defect located at the position between the surface and axis of the droplets and form a twisted bipolar configuration.

It is generally believed that the director configuration of LC droplets is a result of the competition of the surface anchoring strength and the Frank elastic constant of the LC in the droplets. Earlier theoretical calculation including the splay K_{11} , twist K_{22} , and bend K_{33} elastic constants suggests a uniform configuration for smaller LC droplets. This suggests that there is a difference in the surface anchoring energy between the lower and higher dye concentration droplets. Further it is known that the translational diffusion of a bipolar droplet can cause the change of its apparent optical texture. A bipolar LC droplet can show an apparent cross texture when its optical symmetry axis is oriented parallel to the path of the light (i.e., perpendicular to the crossed polarizers). As shown in Fig. 6(b, d), the bipolar droplets apparent optical texture was changed and the LC droplets exhibit a well defined maltese type crosses under an applied electric field 30 V_{P-P}, at room temperature.

Furthermore, the droplet size is relatively smaller and rather more uniform [Fig. 5 (b–d)] at lower dye concentration ($\leq 0.25\%$) than higher dye concentration. The average maximum diameters of LC droplets were 4.20, 4.50, 6.83, 9.90, and 19.60 µm for 0.0625, 0.125, 0.25, 0.5 and 1.0% DPDLCs respectively. These results are supported well with our previous research with anthraquinone dye and smaller size of the droplets may be due to the less free volume available for the dispersed low dye concentration droplets in the polymer matrix [37]. Further, as shown in Fig. 4 it is clear from the UV–VIS absorbance, which was measured at room temperature without applying any electric field, the azo dye in the films absorbs UV light and absorbance increases with the increase of dye concentration, which reduced the UV curing intensity results in an increase of the droplet size [41]. The lower UV cure rate results in a slower freezing of the PDLC once phase separation is induced, allowing more time for the LC to form larger droplets. It is reasonable to assert that the degree of polymerization, when the drops first form, is an important determiner of drop growth and eventual film morphology. Drop growth requires diffusion of LC material towards and diffusion of monomer and polymer away from the drop, and movement of the drop boundary in to the polymer rich region. Further with increasing the dye concentration the DPDLC mixture moves closer to the phase boundary resulting in less developed polymer matrix when phase separation occurs and more time to LC to form large droplets.

Fig. 6 shows the influence of external field on the droplet morphology at room temperature. It can be seen that dichroic LC droplets have a bipolar configuration and their bipolar axes are randomly distributed inside polymer matrix relatively at low applied voltage 5 V_{P-P} for all the DPDLC films [Fig. 6 (a, c)]. However, at higher voltage (~30 V_{P-P}) the LC droplets exhibit a well defined maltese type crosses for low concentration (\leq 0.25%) of azo dye [Fig. 6 (b)]. The LC droplets containing 0.5 and 1.0% azo dye [Fig. 6 (d)] showing the nematic texture, is expected due the much higher droplet size and lower transition temperature from nematic to isotropic phase for these concentrations.

4.3. Electro-optical properties: effects of dye concentration

The temperature dependence light transmission as a function of applied voltage of DPDLC film with 0.625% dye concentration is shown in Fig. 7(a). We found that as temperature increases, transmission decreases that can be seen clearly in inset at saturation level. One possible reason for this remarkable behavior may be attributed to the decrease in dichroic ratio with temperature due to lower order parameter by thermal fluctuation and consequently stronger absorption by dye molecules leading to a lower transmission and moreover the variation of refractive index of the materials as a function of temperature. The transmission increases with applied electric field up



Fig. 5. Droplet morphology of dichroic PDLCs with different dye concentration at room temperature with varying dye contents (a) 0.0625% (b) 0.125% (c) 0.25% (d) 0.5% and (e) 1.0%, at a magnification of 100× at 20 °C without any applied field. Polymer (NOA 65) and liquid crystal (Nematic E7) contents were in 1:1 (wt/wt ratio) in all samples.



Fig. 6. Droplet orientation of dichroic PDLCs with 0.125% and 1.0% azo dye under electric field (a, c) 5 V_{P-P} , and (b, d) 30 V_{P-P} , at a magnification of 100× at 30 °C.



Fig. 7. (a) Temperature dependent with 0.0625% dye content and (b) different dye concentration dependent, transmission characteristics of dichroic PDLC as a function of the applied voltage at fixed frequency 50 Hz. Polymer (NOA 65) and liquid crystal (Nematic E7) contents were in 1:1 (wt/wt ratio) in all samples.

to a fixed voltage, beyond which it is independent on the applied electric field for all the DPDLC films, indicating that the director orientation is completed at that electric field and transmission reaches its maximum.

Fig. 7 (b) illustrates the influence of transmission as a function of electric field for various dye concentrations in DPDLCs. In comparing the transmission of DPDLC samples we found that the maximum transmission shifted to lower values with the increase of dye concentration because the ordinary refractive index of the LC matches more closely with that of the polymer (1.5028) at low dye concentration at room temperature (results not shown here). These results may also be related to the fact that higher absorbance of the visible light at higher dye concentration relatively results in lower transmission, as shown in Fig. 4. It is noticed here that the DPDLC film containing 0.0625% dye concentration shows almost no absorption peak in visible region, it hints that the dye molecules with this small quantity reduce the scattering with the alignment of the LC molecules along the director and enhance transmission in the ON state. Further, the LC in these films being in near to isotropic phase can explain the electro-optic characteristic of higher dye concentration PDLC films not responding to an electric field at higher temperature.

Fig. 8(a) shows that the driving voltages from minimum (T_{min}) to maximum transmission (T_{max}) for these DPDLC films are different. This finding implies that the value of threshold electric field (E_{th}) is





Fig. 8. Dye concentration dependent (a) threshold electric field and (b) absorption coefficient (β), of dichroic PDLCs at temperature 30 °C. The β straight line is least squares fit to the experimental data (squares).

approximately 8 V/µm for pure PDLC (without dye) [37], where as the threshold electric fields are approximately 4.0 V/µm, 2.0 V/µm, 1.7 V/µm 1.0 V/µm, for 0.0625%, 0.125%, 0.25% and 0.5% with azo dye DPDLCs, respectively. It indicates that as dye concentration increases, the threshold voltage decreases whereas the transmission is up to more than 2 times than pure PDLC. It is expected that a small amount of azo dye dissolved in NLC makes it light absorbing and the photoexcited azo dye molecules induced a positive torque that can greatly enhance the orientation parameter of the material [42]. We also presume that the threshold voltage in the DPDLC is also affected by the droplet size. Because the surface effect is greater for smaller DPDLC droplets therefore anchoring force from the surfaces is greater for the low concentration of dye. A simple theoretical estimation of the threshold voltage (E_{th}) [43] for isolated LC droplets with radius R is given by

$$E_{\rm th} = \frac{1}{3R} \left[\frac{\sigma_{\rm LC}}{\sigma_{\rm Pol}} + 2 \right] \left[\frac{K \left(l^2 - 1 \right)}{\Delta \varepsilon} \right]^{1/2}$$
(2)

where σ_{pol} , σ_{lc} , K, l, and $\Delta \varepsilon$ are the conductivities of the polymer and LC, elastic constant, shape anisotropy (ratio of major to minor radius), and dielectric anisotropy, respectively. In the present case, the threshold voltage is mainly related to the size of the LC droplet, because σ_{pol} , σ_{lc} , l, and $\Delta \varepsilon$ are almost constant or similar. In this study,

Table 1	
Variation in contrast ratio as a function of dye concentration.	

Dye concentration (wt.%)	T _{ON} (%)	T _{OF} (%)	Contrast ratio
0.0625	90.00	0.850	105.88
0.125	86.33	0.825	104.64
0.25	81.793	0.816	100.23
0.5	51.024	0.694	73.52
1.0	15.024	0.368	40.82

as the droplet size of the LC is increased, the area between the LC and the polymer. Therefore, the threshold voltage of the DPDLC film with 1% dichroic dye having the largest droplet size is expected to be the lowest, and the DPDLC film with and without dye having the smallest size to be the highest, and thus V_{th} also behaves in the same manner as predicted by the theory with the above equation.

To investigate the effect of dye concentration on the contrast we measure the contrast ratio (T_{ON}/T_{OFF}) . As shown in Table 1, dye concentration increases from 0.0625% to 0.125%, the $T_{\rm ON}$ decreases slightly but T_{OFF} decreases much more, which give the maximum contrast relatively to all concentrations of dye ranging from 0.0625 to 1.0%. From the results we can infer that the maximum contrast is about 2.55 times higher than the minimum contrast observed in the experiment. The absorption coefficient is obtained by measuring the transmittances of PDLC films containing different dye concentration. From the measured transmittances of the films, the dye extinction coefficient can be obtained using Eq. (4). By least square fitting of the experimental data, we found that the straight line is least squares fit to the experimental data as shown in Fig. 8 (b). The theoretical results entirely hold up our experimental data. Table 1 shows the variation in contrast ratio as a function of dye concentration.

5. Conclusions

A detailed study of morphological and electro-optical properties of DPDLC with different concentration of disperse orange 3 azo dye has been carried out. The use of azo dye can improve their electro-optical properties significantly. It is found that under a constant UV intensity the dye concentration can be optimized for uniform droplets size, higher transmittance and higher contrast. The absorption coefficient and dye extinction coefficient were obtained by measuring the transmittances of DPDLC films using Beer's Eq. (1). By least square fitting of the experimental data, the straight line is least squares fit and satisfies the theoretical results. Our results illustrate that the LC droplets in these DPDLC films are predominantly in the bipolar configuration. Effect of electric field on these films has been found to strongly influence the LC droplet orientation at low dye concentrations. Low dye concentration DPDLC shows higher transmission and higher contrast ratio. Higher dye content results a lower value of clearing temperature and in a lower transmittance due to lower order parameter by thermal fluctuation. The threshold electric field (E_{th}) for switching the material from a scattered to highly transparent state is approximately $8 V/\mu m$ for pure PDLC (without dye), where as the threshold electric fields are approximately 4.0 V/µm, 2.0 V/µm and 1.7 V/µm for 0.0625%, 0.125%, 0.25% and 0.5% (wt/wt) dve in DPDLCs, respectively. We confirm that fine droplet size is a significant factor to control the opto-electric properties of these DPDLC systems. We believe that DPDLC materials with controlled dye concentration will possibly be suitable and promising functional electronic materials for green technology flexible liquid crystal display.

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