Investigation of electro-optical responses of PDLC films via doping of two different dye materials

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Recently, color polymer dispersed liquid crystal (PDLC) display has been extensively investigated in the area of the switchable color window called the smart window. To use a window made of dye PDLC film, various electro-optical properties such as a high contrast ratio (CR) and a color band are essentially required. However in an alternative approach for colored smart windows production usually its CR is deteriorated. Also, color contrast is usually not achieved as much desired for the dye-PDLCs. We in this study, keeping the following problems, selected the two dyes, Sudan black B (SBB) and Rose Bengal (RB) combination and have employed them in the fabrication of PDLCs. In the present study dye-PDLCs at different dye contents and liquid crystals (LC) compositions have been investigated. From the obtained results, we observed that by doping the dye in PDLC the color band shifted to the new absorption area. The fabricated PDLC gave a good CR and color PDLC even at the small amount of both dye contents. Further, this study revealed that both V_{th} and V_{sat} also decreased for the prepared PDLC. Consequently, the following dye combinations can be possibly applied in dye PDLC industry.

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1. Introduction

Polymer dispersed liquid crystals (PDLCs) films are dispersions of liquid crystal (LC) droplets inside a polymer matrix. PDLC films have many potential device applications in displays and optical shutters due to their interesting electro-optical characteristics [1-3]. Furthermore, PDLC films provide many potential applications such as switchable windows, electro-optic switches, memories, gas flow sensors, optical sensors, infra-red shutters, angular-discriminating filters, thermo-optics, optical gratings etc., [4-8, 24].

By applying an external electric field, these films can be switched between the opaque state to the transparent states. Ever since under the influence of external electric field the LC directors align preferentially within the droplet. Consequently, with a suitable LC and polymer composition, the refractive indices of the droplet and the polymer matrix become matched, thus giving rise to a transparent state. On removal of the electric field, such refractive index matching is lost and light scattering appears, which is known as an opaque state [1-3, 8, 24]. The working principle of PDLC film can be seen in Fig. 1.

Among such displays films, dye-doped polymer dispersed liquid crystals (D-PDLCs) are known as colored devices in the LC display. Such D-PDLC devices provide higher reflectivity, wide viewing angle and low power consumption cost characteristics [9-18, 25-26]. Conversely, such D-PDLC films have some limitations which include low contrast ratio (CR) at higher dye contents, relatively high production cost due to higher consumption of LC and limited curing temperatures range [9-18, 25-26]. Essentially this field of study needed to be explored more deeply to overcome such shortcomings related to D-PDLC films.

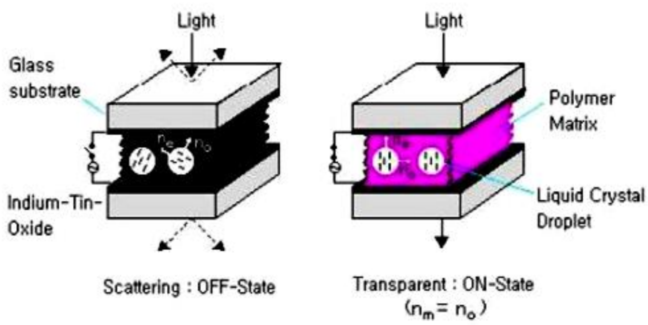


Fig. 1. The operational principle of PDLC film (color online)

For the purpose to control such conditions, we investigated doping of two dyes, Sudan black B (SBB) and Rose Bengal (RB) into the PDLC films for their improved electro-optical characteristics. Both of the dyes doped in PDLCs using the various composition of LC and polymers have been examined. The objectives of the study were to produce a high contrast ratio (CR), better absorbance, good dye contents ratio, and morphologies D-PDLC films.

All these characteristics of the prepared films have been studied and have been discussed in details in this work.

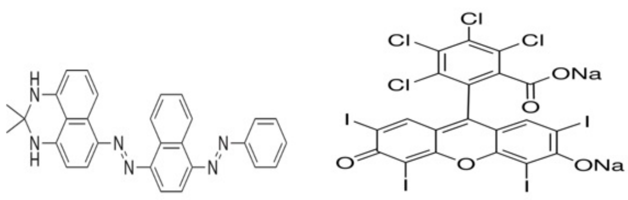
2. Experimental

2.1. Materials and methods

In this work, KU0016 (Konkuk Uni. KOREA) has been used as the liquid crystal, and NOA65 by NORLAND PRODUCTS INC) [19] was used as the polymer. The LC, KU 0016 has refractive indices (n_o=1.521, n_e=1.754) and it has a milky white liquid appearance and isotropic temperature ~ 90.3 ^oC. While NOA65 is a UV curable optical adhesive polymer which has a refractive index (n_p = 1.524). Two types of dye materials the Sudan Black B (SBB) (Sigma Aldrich)[20], and Rose Bengal (RB) (Sigma Aldrich) [21] were doped into PDLC sample films. Ever since in our earlier studies [10], doping of SBB in PDLC films exhibited improved electro-optical properties. Therefore in the present work, we have employed SBB along with the RB dye-doped mixture into the PDLC film to observe its electro-optical characteristics.

The electro-optical properties of each prepared film were measured, with both dyes (Sudan Black B, and Rose Bengal) contents mixed with the combination of liquid crystal and polymer composition. The chemical structures of the SBB dye and RB dye have been mentioned in Fig. 2.

The KU0016 liquid crystal and NOA65 polymer were mixed with both types of dyes in various ratios. For the purpose, the polymer and liquid crystal ratio is fixed at 1:1.5 (40 :60 wt%) and 1:1.2 (45: 55 wt%) wt ratio with the fixed Rose Bengal (RB) ratio at 0.2%. While in the sample composition, the Sudan Black B (SBB) ratio is varied at 0wt%, 0.02wt%, 0.05wt%, 0.2wt %, respectively.



(a) Sudan Black B (b) Rose Bangal

Fig. 2. The chemical structures of (a) Sudan Black B and (b) Rose Bangal dyes

2.2. Methodology

The composition mixture ratio for polymer and LC were taken from 1:1.5 and 1:1.2 wt ratios, respectively. In the prepared samples, both the dyes (SSB and RB) content composition was varied from 0.02 wt % to 0.2 wt%. Various mixture compositions of the fabricated sample PDLC cells have been mentioned in Table 1.

2.3. PDLC sample fabrication

Upon cutting the indium tin oxide (ITO) coated glass, such ITO glass was placed in distilled water and washed with an ultrasonic cleaner for 20 minutes. In the same way, upon washing it through isopropyl alcohol and acetone by three steps process, the ITO coating glass is dried for 2 hours at 60 °C to prevent possible stains. In the next step, the mixture of the prepared polymer material and LC is dropped and injected into the cell gap, by using the capillary action. To keep the cell gap thickness fixed we used 10 µm spacer.

Table 1. The ratio of the composition solution used for Dye-doped PDLC- film preparation

Sample No.	Polymer:LC Wt ratio	NOA 65	KU0016	Sudan Black B (SBB) (%)	Rose Bengal (RB) (%)
A1	NOA:KU0016 NOA (with dye) 1:1.5	1	1.5	0	0
A2		1	1.5	0	0.2
A3		1	1.5	0.02	0.2
A4		1	1.5	0.05	0.2
A5		1	1.5	0.2	0.2
B1	NOA: KU0016: (with dye) 1:1.2	1	1.2	0	0
B2		1	1.2	0	0.2
B3		1	1.2	0.02	0.2
B4		1	1.2	0.05	0.2
B5		1	1.2	0.2	0.2

A simple schematic view of PDLC fabrication method has been presented in Fig. 3. The prepared PDLC cell was irradiated with UV at a wavelength of 365 nm at an intensity of 1.8 mW/cm^2 for 5 minutes. The intensity of UV was measured using a UV light meter (Lutron Co.UV-340). In the present work, all the PDLC samples were prepared and investigated at room temperature conditions. So the both of the dye- doped PDLCs films were prepared by polymer induced phase separation (PIPS) method [10, 27-30]. The PIPS method is the the most simplest technique employed for the preparation of a PDLC film. The mixture of this method composed of reactive polymer precursor and LC. PIPS take place when a LC is mixed with a solution that has not yet undergone polymerization. Once a homogeneous solution is fabricated, the polymerization reaction continues to initiate. As the reaction undergoes further, the LC molecules come out of solution and begin to form droplets. Such droplets develop until the polymer binder becomes solid enough that the molecules are trapped and can no longer move easily [27-30].

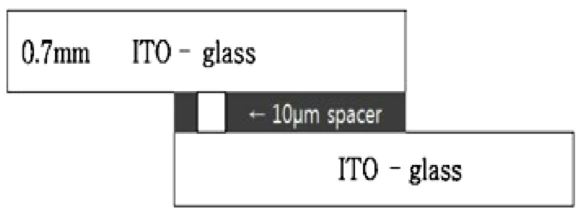


Fig. 3. A view of the PDLC film fabrication method

2.4. PDLC analysis technique

For the evaluation of the intermolecular interactions, free volume space among polymer matrix, the dye contents, and LC molecules, the conventional and latest analysis techniques were utilized [22].

2.4.1. EO properties

The absorption of both dye at different dye mixture composition and polarized parallel absorption spectra of the LC-polymer mixtures in the visible spectral following region (360-700 nm) at zero was measured using Spectrophotometer (MINOLTA, CM-3500d, Japan) [10].

Further for the transmittance measurement of the prepared PDLC samples according to the doping of both dye contents, a spectrophotometer (MINOLTA, CM-3500d, Japan) was utilized. The transmittance at max: 50V was measured which was stepwise achieved to increase by 5 Volt and was initiated from 0V, OFF state condition. The electro-optical performance parameters of the dye-doped PDLC films consist of light transmittance both at the off-state (T_{min}) and on-state (T_{max}), the threshold voltage (V_{th}) and the saturation voltage (V_{sat}), the contrast ratio (CR). Among these parameters, the V_{th} and V_{sat} are defined as the electric voltage required for the transmittance to reach 10% and 90% of the T_{max}, respectively.

2.4.2. POM analysis

A polarizing optical microscope (POM) was utilized to investigate the surface morphology of the dye composition contents and phase transition of the LC domains in the polymer matrices of the PDLC films. This technique was adopted (using Olympus Model BX-60) at 10X magnification fitted with the charge coupling device attached with a digital camera which was interfaced with a computer [22]. In the second step, the electro-optical properties of the PDLC films were observed by the UV-vis spectrophotometer Minolta (Japan) (model UV-3500d) and by a voltmeter, which is used to control the applied voltage.

2.4.3. SEM analysis

For the purpose to understand the droplet size and extant of phase separation, the fabricated PDLC films were observed by SEM analysis technique, scanning electron microscope (Japan, JOEL Co. JSM5200) at a 500 x magnification [22]. For the observation of SEM micrographs, initially, the LCs were extracted out from the prepared films by dipping them in the ethanol/isopropyl 1:1 mixture for 10 days [23]. Afterwards, these films were dried to evaporate all solvent at vacuum 60^{o}C temperature. Lastly, the surface images from SEM were taken for small pieces of the prepared films [23].

3. Results and discussion

PDLC Visual Characteristics (ON/OFF States)

The visual images of the fabricated dye-doped PDLCs at 0/40 V are presented in Fig. 4 (A3-A4, B3–B4). A close look to this figure shows that at OFF state, the cell scatters light strongly. Thus PDLC showed a white and opaque state. However, upon applying the external electric field to the sample, a transparent state (ON) can be observed with a clear background. This situation appeared due to the LC molecules became parallel to the electric field and the directors were arranged parallel to the electric field in each droplet. At the same time, the refractive index of the LC and the refractive index of the polymer became equal, that enabled the incident light pass through the PDLC sample, so the PDLC became into a transparent state (as can be seen in Fig. 4).

Interestingly for attaining the color images we used the 0.02:0.2% and 0.05:0.2% SB:RB dye contents for 1:1.5 and 1: 1.2 wt ratios of LCs, the PDLC films turned blue-black color for 0.2:0.2 wt % of SBB: RB for 1: 1.2 LC contents (Fig. 4 (A3-A4, B3–B4)). This situation can be also confirmed from the absorbance data. The absorption spectra showed that for SBB dye, the maximum absorbance peak shifted to left and a broadband spectrum has been seen from 500 nm to 550 nm. This clear shift of spectrum and turning blue- black PDLC can have industrial importance that can be adopted in LCD windows.

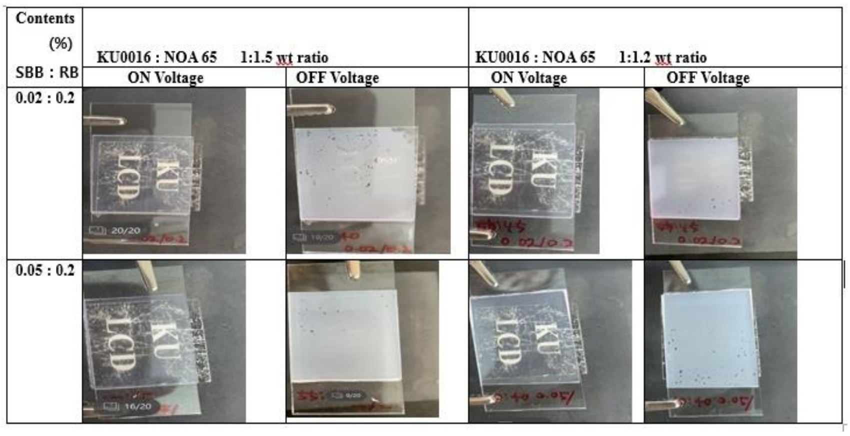


Fig. 4. The electro-optical response of the prepared PDLC cell (for LC and polymer composition of 1:1.5 wt ratio to 1:1.2 wt ratio) for both dye-contents (SBB: RB 0.02: 0.2% and 0.05: 0.2%) at voltage ON, and at voltage OFF conditions (color online)

Electro-optical properties

a. Dye Absorbance measurement

To have the absorbance spectra of two (SBB and RB) dye contents using Polymer/LC 1:1.5 and 1:1.2 wt ratio in the ranges of 380 -700 nm wavelengths at OFF state voltages has been determined. The obtained spectra for both the dyes (only with Polymer/LC 1:1.2 wt ratio) in the visible range have been plotted in Fig. 5 (b). The absorbance results for SBB reveals that this dye showed maximum absorbance peak in the region of 580-590 nm in the KULC0016 LC. Further, as RB dye is mixed in with SBB the absorption spectra have been taken. It can be seen that both dyes showed some interesting behavior. Which can be confirmed by the shift of the maximum peak to the left of the spectra and it showed a maximum peak at 550-560 nm at 0.2/0.2 % contents of both dyes.

Interestingly when both dyes have been fabricated in PDLC film, it enriched the PDLC's blue-black shade. Which is a basic requirement as industrial window design. This interesting behavior of mixtures of these two dyes provoked us to further studies its properties in PDLC. For the purpose, we have studied its electro-optical properties in details which are presented in the following figures.

A close look to the Fig. 5(b) predicts that both of the dyes showed maximum absorption peaks at two different regions for 0.05:0.2% and 0.2:0.2% dye contents (sample B4 and B5). The higher absorption band was observed from 540-580 nm with maximum absorbance at 550 nm. The largest absorption peak for both dyes 0.2:0.2% at 550 nm was observed as 0.146%. Moreover, such spectra showed an increase in absorption intensities with the addition of dye contents. The maximum absorption of both dyes was observed for 0.2:0.2% PDLC-dye film.

For the dye SBB's maximum solubility test in 1:1.2 wt ratio LC contents, we used the different dye concentration in PDLC samples mixture, in the following studied PDLC mixture (Fig 5(a)). We observed that the same dye contents in 1:1.5 LC contents which showed the same absorption peak at different absorbance intensity (not shown here). Based on the following results, we selected RB dye in SBB dye as the dopant to further check its absorption pattern. This absorption technique is generally applied to check the dye solubility in the media and the color band. According to Fig. 5(b), the color band was observed in the range of 550 nm.

The shift in the color band of 580 nm (SBB dye) to 550 nm (SBB:RB) revealed that the two dyes have intermolecular interactions. Such intermolecular interactions may have appeared due to the de-localized electrons on SBB and RB molecules. The projection of the conjugation of the de-localized electrons and shifting color band, and the solubility effect of the dye at different dye contents can be also verified in Fig. 4.

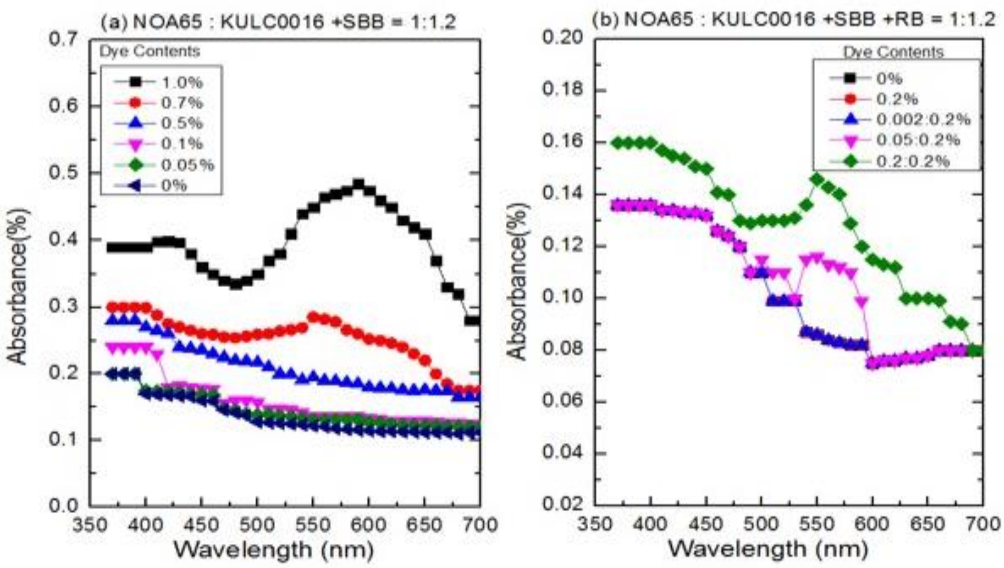


Fig. 5. Absorbance spectra of (SBB and RB) dyes with two LC and polymer compositions (a) PDLC with (SBB only) 1:1.2 wt ratio (b) PDLC with (SBB:RB) 1:1.2 wt ratio (color online)

b. Voltage-transmittance(V-T) characteristic

The transmittance of dye-doped PDLC films as a function of an applied voltage can be seen in Fig. 6 (sample A1 and A5). Here the Fig. 6(a) shows the transmittance curves of dye-doped PDLC prepared with NOA 65: LC as 1: 1.5 wt % ratio, and it consists of dye contents (SBB:RB) as 0:0 wt% and 0.2:0.2 wt%, respectively.

With the increase in applied voltage, the transmittance of both dye contents PDLC increased, particularly with 0.2:0.2wt% dye contents such behavior is dominant.

Further, according to this scenario, the maximum transmittance is increased with the addition of both dye contents. It can be concluded that higher dye-doped PDLC contents displayed the higher transmission than the lower dye-doped PDLC at voltage ON state. Ever since the LC ratio is decreased in the composition mixture (1:1.2) the electro-optical properties (V_{th}, V_{sat}, T_{ON}, T_{OFF}) were found greatly improved. Such situations were also observed for the contrast ratio (can be seen in Fig. 7 (a, b)).

Similarly, Fig. 6(b) presented the transmittance results for the dye-doped PDLC fabricated with NOA 65: LC as 1: 1.12 wt % ratio (sample B1-B5). It is composed of both dye contents (SBB:RB) varying in the ranges of 0:0 wt% to 0.2:0.2wt % respectively. This figure also validates that the maximum transmittance is achieved with the addition of both dye contents.

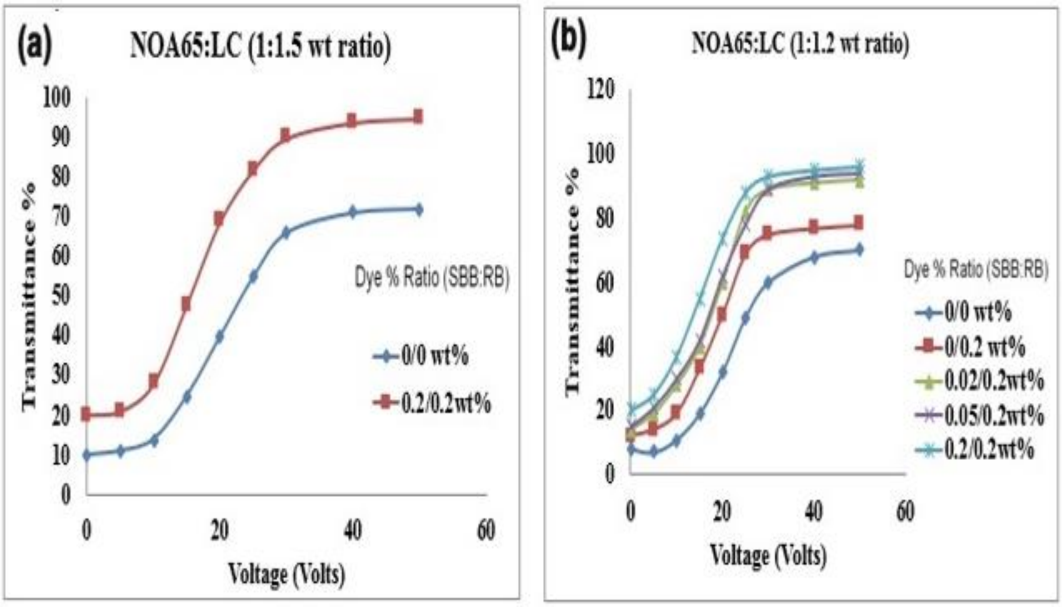


Fig. 6. (a) Transmittance as a function of applied voltage for NOA 65: LC (1: 1.5 wt % ratio), (b) Transmittance as a function of applied voltage for NOA 65 : LC (1: 1.12 wt % ratio) (color online)

The contrast ratio (CR) [10] is the ratio between the two transmittances at min voltage and at max voltage and expressed as CR= T/T_{ON}. The contrast ratio of dye-doped PDLC films as a function of an applied voltage can be seen in Fig. 7 (sample A1 and A5). Here the Fig. 7(a), give a projection of CR of two dye-doped PDLCs upto 20 volts and exhibited that it increases with applied voltage for 0:0 wt% of any dye or maxim wt % of dye.

Nevertheless, there is a clear difference in the area of the small voltage between the two curves. Particularly the dye-doped PDLC with max dye contents of both dyes predicted the higher CR values. However, the Fig. 7 (b), the contrast ratio (observed with NOA65: LC as 1:1.2 wt% dye-based PDLCs) showed that CR increases gradually with the increase of SBB:RB dye contents and later at higher voltage all the dye ratios showed the maximum 1 CR. The larger difference at the low voltage of CR with the different dye contents verifies the usefulness of these two dyes in PDLCs applications. Furthermore, it is clear from the figure that CR increases with the SBB:RB contents.

We believed that for the 1:1.2 wt% of NOA65: LC, the higher CR could be obtained even with higher contents of both dyes, which can be verified from the absorbance data that the solubility of dyes increasing with the increase in dye contents.

Table 2 shows the two important parameters of electro-optical properties V_{th} and V_{sat} of 1:1.5 & 1:1.2 LC ratios at zero and at highest dye contents. The table shows that V_{th} and V_{sat} for both LCs contents decreased as dyes added. Moreover, it is observed that the 1:1.2 LC ratio PDLCs mixture showed good electro-optical response as compared to 1:1.5 wt ratio. The following data has been calculated from the V-T graphs by inserting the linear trend lines.

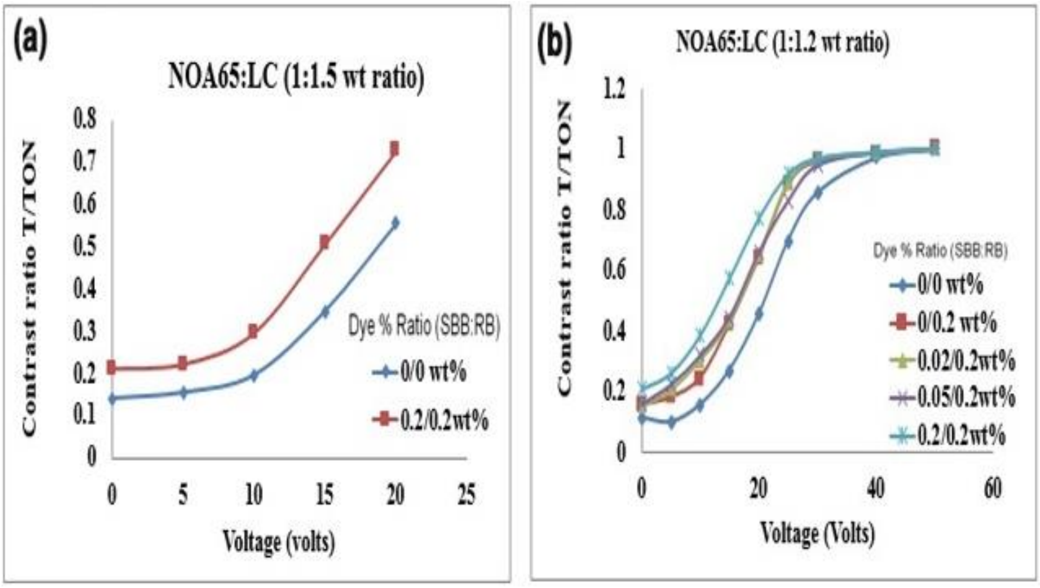


Fig. 7. (a) Contrast ratio as a function of applied voltage for NOA 65 : LC (1: 1.5 wt % ratio) (b) Contrast ratio as a function of applied voltage for NOA 65 : LC (1: 1.12 wt % ratio) (color online)

Table 2. Electro optical properties of the two dyes (SBB: RB) contents doped in the PDLCs

Electro- optical properties	Polymer : LC wt%	
	1:1.5 wt%	1:1.2 wt%
V_{th}-volts(0-SBB/0-RB wt%)	10	13
V_{th}-volts (0.2-SBB /0.2-RB wt%)	8	7
V_{sat}-volts (0-SBB /0-RB wt%)	30	27
V_{sat} -volts (0.2-SBB /0.2-RB wt%)	28	25

c. Morphology observation

After the fabrication of the dye-doped PDLC, the characteristics of such prepared films have been investigated in detail. Dye-doped PDLCs morphologies were considered by the conventional method by employing polarized optical microscopy (POM) at 10x lens magnification with 90 angle of polarizer film at zero applied voltage [22]. Such PDLC film images can be observed in Fig. 8, taken with various dye (SBB: RB) contents. Both dye contents have been varied according to the PDLC films compositions (for LC and polymer composition of 1:1.5 wt ratio to 1:1.2 wt ratio) as mentioned in Table 1.

The following Fig. 8 (A1- A4) showed two states; dark/colored state under the POM. Such states of the POM images predicted the random orientation of LCs with the polymer matrix. A close look to the POM images particularly revealed that sample A5 and sample B5 with 0.2:0.2 % (SBB: RB) dyes showed the largest droplet size.

Further from the POM images, it was observed that the structure of the rest of the sample films showed a lot of small droplets, with the smaller proportion of both dye contents, the lower the light transmittance appeared and the darker the sample films were seen. It can be seen from such images, that these structural characteristics affect the electro-optical properties of the prepared dye-doped PDLC films.

d. SEM observation

In order to further investigate the dye-doped PDLC films, the SEM analysis technique was performed. The surface morphology of the samples was evaluated to understand the effects of dye contents variations, the droplet formations and droplets sizes in the PDLC films.

Moreover, the SEM image technique can be helpful to find out the variations in the intermolecular interactions with the various dye compositions as well. Fig. 9 presents the SEM photographs of samples (A1–A5) taken with the LC: polymer composition of 1:1.5 wt ratio by employing the two dye contents.

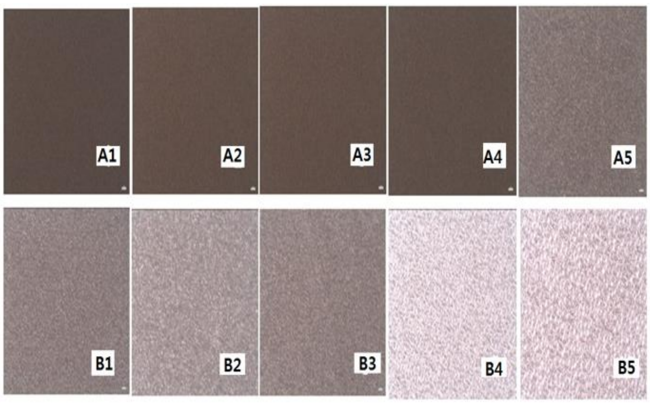


Fig. 8. POM droplet morphology of the D-PDLC with the both dye contents in the samples (A1 – B5)

It can be seen from Fig. 9 that differences in the molecular structures, dye doping compositions and with the functionality of the monomers resulted in random droplet morphologies such as random polymer balls, and porous polymer matrix. Particularly sample A1-A3 were composed of typical porous polymer networks with smaller droplet sizes oriented randomly. However, sample A4-A5 microstructures were different from larger porous polymer networks and dropet sizes. Further, it clarifies as the dye-doping contents were increased, which resulted in the enhancement of the wide polymer networks and larger droplet sizes (sample A4-A5). Nevertheless in all samples, the size of the droplets, their shape sequence were of almost the same trend, which demonstrated the droplet growing size with both dyes (SBB:RB) compositions.

Fig. 10 shows the SEM photographs of samples (B1–B5) taken with the LC: polymer composition of 55:45 % by using the two dye contents. This figure almost showed the same trend of random droplet morphologies with a porous polymer matrix, in all the samples. When both dye (SBB:RB) doing contents were relatively higher, the wide polymer networks with larger droplet sizes (sample B4-B5) were observed. It was observed that the size of the droplets in all PDLC samples (A1-B5) remained in the range between 100 nm to 500 nm.

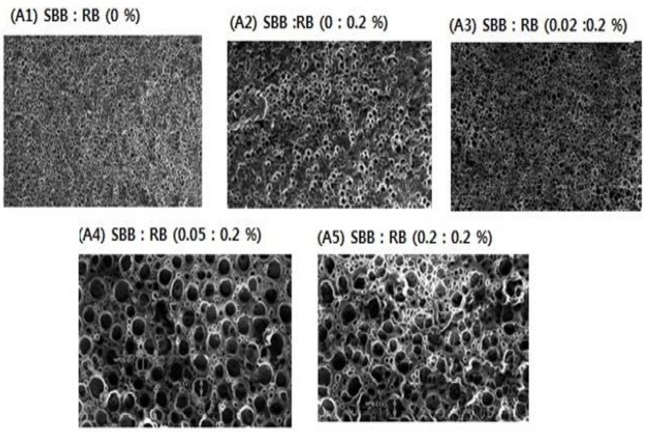


Fig. 9. SEM images of the D-PDLC samples (polymer:LC: 1:1.5 wt ratio) with two dye compositions (X 500 times resolution)

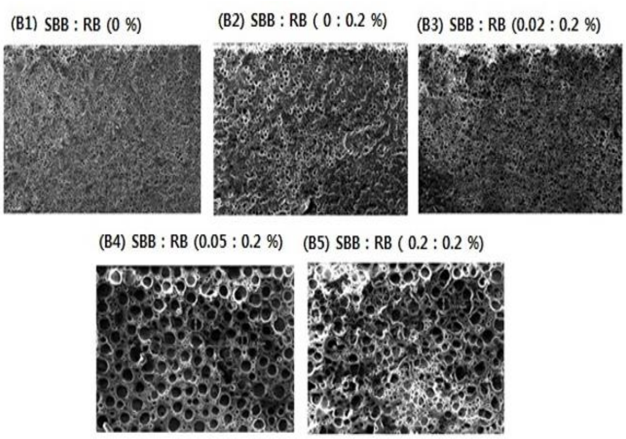


Fig. 10. SEM images of the D-PDLC samples (polymer: LC 1:1.2 wt ratio) with two dye compositions (X 500 times resolution)

4. Conclusions

In order to investigate the electro-optical properties of the two dyes doped PDLCs, the Sudan black B (max: Absorbance nm 580) and Red Blue (max: Absorbance nm 550) has been studied. Upon mixing the different ratios of both the dyes in the 1:1.5 and 1:1.2 wt ratios of monomer NOA65 and nematic LC, we observed an interesting phenomena on shifting of color band. Both of the dyes showed maximum absorption at (560 nm) and upon the fabrication of PDLC it exhibited a good color contrast. The electro-optical properties showed that both dyes with all their compositions demonstrated a decrease in V_{th} and V_{sat} and increase in TON, which consequently lead to an increase in the CR. The absorption band and CR are important parameters to reveal the dye-doped PDLCs characteristics.

Further, the morphology study (POM, SEM) prove that phenomenon, and it showed an increase in the droplet size for 1:1.5 and 1:1.2 wt ratio LCs. However, an error which was observed in the previous studies, the irregular droplet size of LCs was also remained controlled in the following LC/polymer/dye mixtures, which is essential to make a good electro-optical property for the dye-doped PDLC. Thus our findings may be taken as useful for the industrial purpose fabrication of dye-PDLCs.

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