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Modification of the Electro-Optical Properties of Liquid Crystal Devices in the Inverse Mode

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ABSTRACT: An optical device operating in the inverse mode for the transmission of light radiation has been developed, fabricated using a liquid crystal with positive dielectric anisotropy and characterized by a high transmittance. It has been established that improvement of the electro-optical properties, achieved by modifying the polymer–liquid crystal interface, leads to an increase in the optical transmittance of the films up to 80%. This effect is interpreted as a memory effect arising both from the mechanical influence of aligned polymer chains and from the reorientation of the liquid crystal director under the action of an internal built-in electric field. A new approach to the fabrication of polymer-dispersed liquid crystal (PDLC) systems operating in the inverse mode has been proposed. This approach is realized through the formation of a long-term, stable internal DC electric field in a PDLC initially operating in the normal mode and doped with metallic nanoparticles. The incorporation of nanoparticles enables the generation of strong internal DC electric fields within the PDLC and facilitates the formation of a uniformly aligned director configuration of the nematic liquid crystal 5CB, with an OFF-state transmittance of up to 75%. The combination of the internal DC electric field with an external electric field (E_{ext} = 1 V/μm) results in a randomization of the liquid crystal director, thereby switching the device into an opaque ON state. The proposed device overcomes several limitations inherent to inverse-mode PDLC systems based on liquid crystals with negative dielectric anisotropy ($\Delta\varepsilon < 0$), such as the lack of commercially available materials with the required physicochemical properties. The excellent electro-optical performance demonstrated by this device may be advantageous for the development of electrically switchable chromogenic materials and the fabrication of smart windows.

KEYWORDS: polymer, liquid crystal dielectric anisotropy, electric field, nanoparticle, film,

1.INTRODUCTION

Polymer-dispersed liquid crystals (PDLCs) constitute a class of soft matter with strong potential for applications in switchable windows and display technologies. They are capable of modulating light without the use of polarizers [1, 2], and their fabrication process is relatively simple and cost-effective. PDLCs are typically formed either by micron-sized liquid crystal droplets encapsulated within a polymer matrix (the so-called “Swiss cheese” morphology) or by liquid crystals filling the voids and interstices of a polymer network (the “polymer network” morphology), which imparts mechanical rigidity to the composite [3]. The physical operating principle of PDLCs is based on the electrically controlled reorientation of the liquid crystal director from an initially random distribution (typically associated with a highly opaque state) to a uniformly aligned configuration (corresponding to a highly transparent state) parallel to the applied electric field. The emergence of an internal electric field (E_{int}) can also induce reorientation of the liquid crystal director from a disordered state to a more or less ordered configuration (depending on the strength of the external electric field), thereby resulting in a transparent state of the device. Such transmittance values can be increased or decreased when the external electric field is applied parallel or antiparallel, respectively, to the internal DC electric field.

The mass ratio of the liquid crystal–polymer components significantly affects the electro-optical response of “charged” PDLC systems following exposure to a high-intensity electric field. In fact, a quasi-linear electro-optical response can be achieved only within a narrowly defined compositional range [4]. Reported transmittance values in the OFF state reach only about 25–30% [5], which is insufficient for practical applications such as smart windows.

In the present work, a fabrication approach for PDLC cells operating in the inverse mode is proposed, based on the creation of an internal DC electric field in PDLC systems initially functioning in the normal mode and doped with metallic nanoparticles. The incorporation of nanoparticle fillers enables efficient control of charged PDLC systems with a strong internal DC electric field and allows the achievement of OFF-state transmittance values of up to 75%. Such devices operate in the inverse mode, since the initially opaque state can be switched to a transparent state upon application of an DC electric field.

2. MATERIALS AND EXPERIMENTAL METHODS

PDLCs were prepared via thermally induced phase separation (TIPS) following the procedure described in [2]. Polyvinyl alcohol (PVA, Aldrich) and the nematic liquid crystal 5CB (Merck) were used as the film components in various mass ratios (from 2:1 to 1:2) and doped with different concentrations (up to 0.5 wt%) of CdS metallic nanoparticles (NPs). This system was selected after a series of trials involving other nanoparticles, which either phase-separated with the liquid crystal or were combined with other liquid crystals (E7 and MBBA), and which produced only relatively weak internal DC electric fields (E_{int}). The PVA and 5CB components were dissolved in distilled water. After solvent evaporation, a small amount of the mixture was placed between two conductive substrates, heated to 85 °C, and then cooled to room temperature (20 °C) to induce phase separation of the liquid crystal and polymer matrix (PIPS). The sample thickness was set to approximately 25 μm using a teflon spacer. Microscopic droplets of 5CB exhibit a broad nematic temperature range—from 22 to 43.5 °C and a high dielectric anisotropy, $\Delta\varepsilon$ = 16.6 (measured at 1 kHz and 20 °C). These properties, together with other characteristics such as elastic and optical parameters (Table 1) [6], make 5CB attractive for both theoretical studies and experimental applications.

Table 1. Selected physicochemical properties of the PVA+5CB+CdS mixture at room temperature

Parameters	Indicators	Sources
Transition temperature N-Is	43,7°C	[3, 5]
$\Delta\varepsilon$ (ν=1κHz)	16.6	[1, 7, 8]
ε_{II} (ν=1κHz)	22.4	[2, 8]
$\mathcal{E}_{\perp}$ *(ν=1κHz)*	*5.4*	*[6, 5]*
Δn (λ=589 nm)	0.2610	[2, 7, 9]
n_0 (λ=589 nm)	1.5270	[6, 7, 5]
n_e (λ=589 nm)	1.7880	[2, 7]

The electro-optical properties of the samples were measured using an optical setup previously described in [1]. A He–Ne laser with a wavelength of λ = 632.8 nm and a maximum power of 2 mW served as the light source. The intensity of the incident laser beam, measured through an empty cell, was taken as the maximum (full) intensity. The response times for switching ON and OFF, denoted as τ_{on} and τ_{off}, were defined as the time required for the transmittance to decrease to 10% of its maximum value upon application of the external electric field, and the time needed to reach 90% of the optical response after removing the external field, respectively.

Measurements were performed by monitoring the driving signal (frequency ν = 1 kHz, field amplitude E_{pp} = 1.3 V/μm) and the photodiode response using a TOUPCAM digital video camera connected to a PC and a memory oscilloscope. Glass transition temperatures were determined by differential scanning calorimetry (DSC) using a Netzsch instrument (model VK-DSC 3000 L) at a heating rate of 10 °C/min.

3. RESULTS AND DISCUSSION

Previous studies [1–6] have shown considerable interest in improving the electro-optical properties of PDLC films during the composite fabrication process. In particular, it is of interest to investigate the changes induced when PDLCs operate in the normal mode under the application of high-intensity electric fields (the "charging" process) over a period of 10 minutes. It was found that lower reorientation fields and sharper transitions between the ON and OFF states can characterize films during the charging process, as this may induce changes in the morphology, dielectric properties, and surface anchoring energy of the PDLC [7–9]. Accordingly, memory states can arise either from the mechanical fixation of the liquid crystal director, which becomes more aligned during the cooling process, or from the generation of an internal DC electric field (E_{int}) at the interfaces of the liquid crystal droplets. This internal DC electric field originates from ionic impurities in the sample components, which segregate at the droplet boundaries
during the charging process. This phenomenon is also known as the Maxwell–Wagner effect [10].

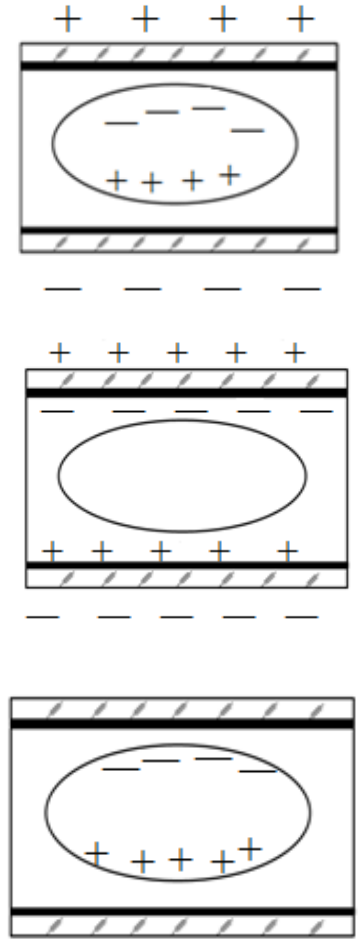

Fig. 1. Liquid crystal droplets in a DC electric field: (a) $\sigma_p \ll \sigma_{lc}$ under a vertically applied field E; (b) $\sigma_p \gg \sigma_{lc}$ under a horizontally applied field E; (c) $\sigma_p \ll \sigma_{lc}$ in the absence of an applied field E

The applied DC electric field, arising from either bound or mobile charges, induces reorientation of the nematic molecules

within the PDLC film [3]. In fact, polarization charges in the investigated cell can either decrease or enhance the effective electric field acting on the nematic director. If the dielectric conductivity of the nematic liquid (σ_{lc}) is higher (i.e., the nematic is more conductive) than that of the polymer matrix (σ_p), polarization charges at the droplet surface will reduce the effective electric field at the droplet interface (Fig. 1a). Ion segregation within the nematic droplet can persist during solvent evaporation, as the increased viscosity of the polymer matrix inhibits ionic mobility.

When the condition $\sigma_p \gg \sigma_{lc}$ is satisfied, the polymer matrix, characterized by higher dielectric permittivity or conductivity, can enhance the electric field inside the droplet (Fig. 1b). This regime promotes the transition to a memory state, in which the induction of an applied DC electric field enables the transformation of the conventional quadratic electro-optical response of 5CB with polymer dispersion into a quasi-linear response [9]. This approach allows control over the optical transmittance of the device. In the absence of an external field, the ions become "frozen" at the liquid crystal–polymer interface (Fig. 1c).

PDLC films fabricated using PVA and the nematic liquid crystal 5CB are typically characterized by high threshold fields of 1.0–2.0 V/μm and slow transitions from the ON to the OFF state. Figure 2 shows the differences in electro-optical characteristics between a control PDLC sample (i.e., without CdS, curve *a*) and a PDLC sample doped with 0.5 mass % CdS (curve *b*). It can be observed that the doped samples exhibit lower driving (switching) fields (less than 0.4 V/μm for curve *b*) and steeper slopes in the transmittance (T) versus electric field (E) dependence, which is attributed to the increased conductivity of the cell due to the addition of CdS nanoparticles.

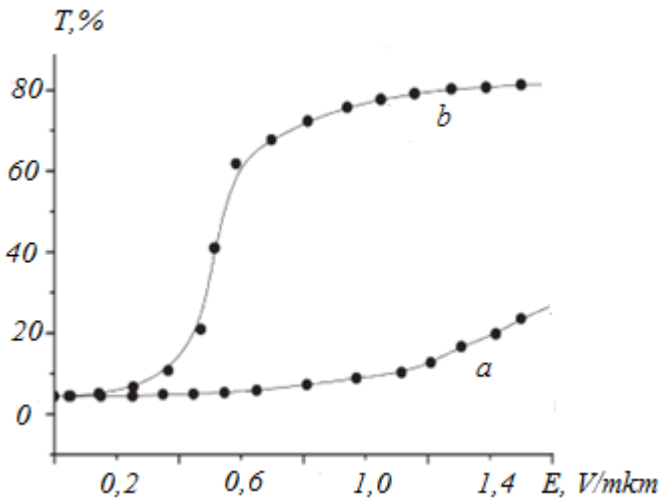

Fig. 2. Electro-optical characteristics of PDLC samples: control PDLC without nanoparticles (curve *a*) and PDLC doped with 0.5 mass % CdS (curve *b*)

The samples were subjected to a DC electric field of varying strength (up to ~30 V/μm) for specified durations (up to ~20 minutes) at a controlled temperature range of 20–40 °C, in order to establish the maximum achievable internal DC electric field (E_{int}) capable of orienting the liquid crystal director, according to the scheme shown in Fig. 1 (c). The applied parameters (E_{charge}, Δt_{charge}, T_{charge}) promote an increase in the number of ions segregated at the liquid crystal–polymer interface, thereby enhancing the internal DC electric field. It is crucial to properly adjust these parameters to avoid mechanical immobilization of the droplet directors at the polymer–liquid crystal interface, which would render them insensitive to electric fields, and, conversely, to achieve alignment of the droplet directors through the internal DC electric field, which can be further modulated by applying an external electric field. The applied E_{charge} values and the resulting E_{int} values do not exhibit a strict one-to-one correlation. It was experimentally determined that E_{charge} values below 20 V/μm result in low internal DC fields (E_{int}), whereas E_{charge} values above 30 V/μm cause dielectric breakdown of the samples. Therefore, the following experimental conditions were selected: $E_{charge} \approx 25$ V/μm; $t_{charge} \sim 10$ min; $T_{charge} = 40.0 \pm 0.1$ °C; PVA:5CB = 2:3 (mass ratio).

During the charging process, the sample was first heated to the target temperature T_{charge}. The electric field was then applied for the specified charging time, during which ion segregation occurred at the droplet interfaces, resulting in the formation of an internal DC electric field. Afterward, the device was cooled to room temperature, at which the internal DC field remained stabilized at the liquid crystal–polymer interface, and finally, the external field was switched off. To prevent dielectric breakdown, the applied DC electric field was varied gradually. It is evident that ion segregation, and consequently the internal DC electric field responsible for orienting the liquid crystal directors, is closely related to the glass transition temperature (T_g) of the polymer matrix. Softer polymer matrices are unable to effectively retain the induced internal DC fields, whereas stiffer matrices cannot adequately hold the segregated ions. A straightforward approach to maintain high internal DC fields is to perform the charging process while heating the samples to temperatures near the polymer's glass transition (35–40 °C for PVA films, as determined by differential scanning calorimetry), followed by cooling to room temperature before switching off the applied charging field. The charging temperature (T_{charge}) plays a crucial role, as it softens the polymer matrix and facilitates ion segregation. Generally, the higher the charging temperature, the greater the magnitude of the internal DC electric field, and consequently, the higher the OFF-state transmittance. In our experiments, the charging temperature (T_{charge}) was varied from 20 to 40 °C. Charging at temperatures below 20 °C or above 40 °C did not allow the establishment of a long-term stable E_{int}.

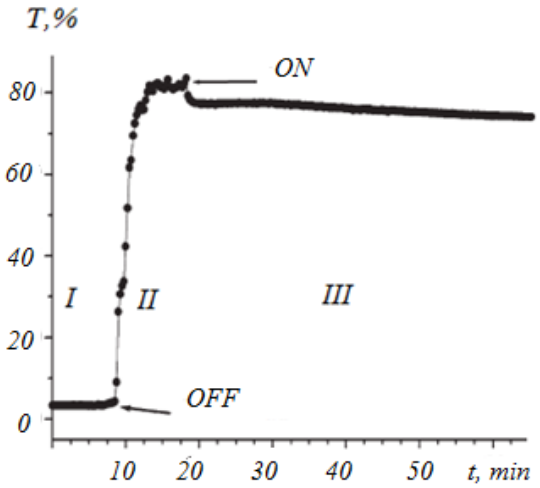

Fig. 3. Time dependence of the optical transmittance for a sample doped with 0.5 mas % CdS nanoparticles: before (I), during (II), and after (III) the charging process

Figure 3 shows the temporal evolution of the optical transmittance for a sample doped with 0.5 mas % CdS nanoparticles before (I), during (II), and after (III) the charging process. It can be seen that the sample's transmittance initially increases from its OFF-state value to a maximum exceeding 80% upon application of the charging electric field, then remains nearly constant throughout the charging period, and finally decreases to approximately 75% within 10 minutes after the external field is switched off. Such a high OFF-state transmittance can be interpreted as a memory effect, arising both from the mechanical influence of aligned polymer chains and from the reorientation of the liquid crystal directors under the action of the internal DC electric field, E_{int}. The magnitude of the internal DC field remains stable over extended periods, provided the samples are stored at temperatures below the polymer's glass transition temperature (approximately 40 °C).

We also found that the samples can withstand numerous ON/OFF cycles (up to 100) without significant reduction of E_{int} and without dielectric breakdown. The selected charging temperature (T_{charge} = 40 °C) enables charging of samples with highly plasticized droplet surfaces, promoting efficient ion segregation and, consequently, the formation of stronger internal DC electric fields.

Figure 4 compares the electro-optical response of an "uncharged" PDLC (curve *a*) and a "charged" PDLC (with an internal DC electric field of 1 V/μm, curve *b*) as a function of an external DC electric field, E_{ext}, applied antiparallel to the internal field. Prior to the charging process, the electro-optical response resembles that of a conventional PDLC in the normal mode, with an opaque OFF state that can be switched to a transparent state at E_{ext} = 1 V/μm.

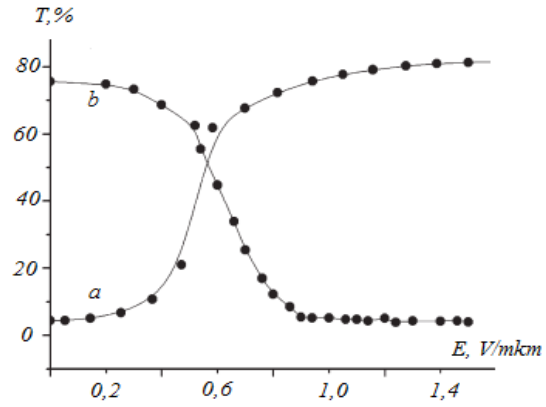

Fig. 4. Electro-optical response of uncharged (curve *a*) and charged PDLC (curve *b*) as a function of the external DC electric field, E_{ext}, applied antiparallel to the internal DC electric field

After the charging process, a transparent OFF state is observed (with a transmittance of approximately 75%), which decreases to a minimum value of around 4–5% at E_{ext} = 1.0 V/μm. It is noteworthy that the OFF-state transmittance of the uncharged sample (Fig. 4,*a* at 0 V/μm) is comparable to the ON-state transmittance of the charged sample (Fig. 4,*b* at 1 V/μm). However, a quasi-linear electro-optical response can be achieved only within a narrowly defined compositional range of the composite, since in the OFF state the transmittance reaches merely 25–30%, which is insufficient for practical smart window applications..

The reduction in transmittance observed in curve *b* of Fig. 4 is due to the decrease of the effective electric field acting on the liquid crystal director: $E_{eff} = E_{int} - E_{ext}$. The minimum transmittance is reached when the external electric field completely "cancels out" the internal field, i.e., when $E_{eff} = 0$. At this point, the director in the liquid crystal droplets adopts a random orientation. The minimum transmittance observed in curve *b* of Fig. 4 demonstrates that mechanical memory, which could be induced during cooling, does not affect the OFF state. Thus, the generation of a high-intensity internal DC electric field in a PDLC operating in the normal mode enables it to function effectively as a medium operating in the inverse mode.

The OFF-state transmittance (T%) achieved after the charging process depends on the magnitude of the internal DC electric field, E_{int}, that can be induced. It is assumed that the strength of the internal DC field may depend on the number of ions present in the samples, which segregate during charging and become "frozen" at the droplet interfaces (Fig. 1c). Therefore, an evaluation of the induced internal DC electric field was carried out, and the electro-optical behavior of samples doped with different concentrations of CdS nanoparticles (1, 3, and 5 mas %) was investigated. As shown in Fig. 5, after the charging process, the OFF-state transmittance increases (to approximately 30%, 60%, and 75%, respectively) with increasing CdS nanoparticle content, and this enhancement correlates with the magnitude of the internal DC electric field. In fact, the internal DC field values were 0.80, 0.86, and 1.0 V/μm for CdS concentrations of 1, 3, and 5 mass %,

respectively. Increasing the concentration of metallic nanoparticles in the PDLC enhances the Maxwell–Wagner effect [11, 12], which serves as the source of the internal DC electric field. In other words, the system contains a greater number of ionic impurities that can segregate and accumulate at the liquid crystal droplet interfaces during the charging process.

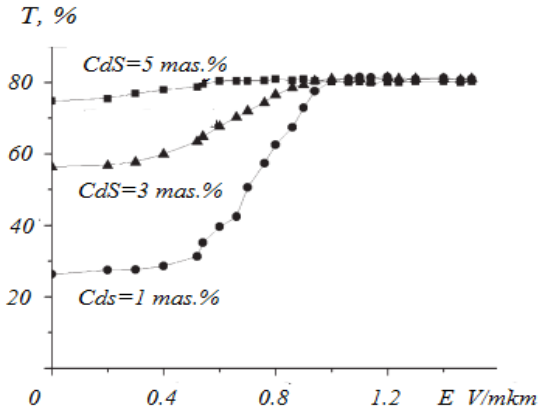

Fig. 5. Optical transmittance (T) as a function of the external DC electric field (E_{ext}) applied parallel to the internal DC electric field (E_{int}) for PDLC samples with increasing CdS nanoparticle concentrations of 1, 3 and 5 mass %, respectively

When an external DC electric field is applied parallel to the internal DC field (Fig. 5), the transmittance of these samples increases to approximately 80%, exhibiting behavior similar to that of conventional PDLC operating in the normal mode, but with a higher OFF-state transmittance. The samples experience an effective electric field, which is the sum of the internal DC field and the external field: $E_{eff} = E_{int} + E_{ext}$. Such a field promotes more efficient reorientation of the liquid crystal director. It should be noted that the transmittance of the sample doped with 5 mas % CdS remains nearly unchanged and can be considered almost insensitive to the applied external electric field.

When the external DC electric field is applied antiparallel to the internal DC field (Fig. 6), the transmittance of these samples decreases to 4–5%, exhibiting behavior analogous to a PDLC operating in the inverse mode.

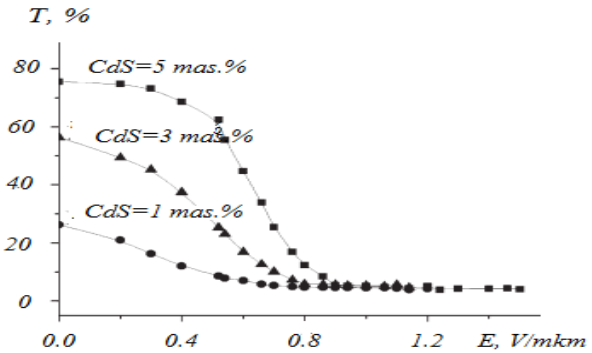

Fig. 6. Optical transmittance as a function of the external DC electric field applied antiparallel to the internal DC electric field for PDLC samples containing 1, 3, and 5 mas. % CdS nanoparticles, respectively

In this case, the samples experience an effective field given by the difference between the internal DC field and the external field: $E_{eff} = E_{int} - E_{ext}$, which ensures a random orientation of the liquid crystal director when $E_{eff} = 0$. It should be noted that the OFF-state transmittance for the sample doped with 5 mas. % CdS is approximately 75%, representing the baseline transmittance for an ideal PDLC operating in the inverse mode. The response times for the ON and OFF states, denoted as τ_{on} and τ_{off}, are on the order of a few milliseconds, comparable to values observed in other inverse-mode PDLCs doped with conductive molecules [13].

In summary, these results support a new approach for producing PDLCs operating in the inverse mode, achieved by generating a long-lasting, stable internal DC electric field in a PDLC initially operating in the normal mode and doped with metallic nanoparticles. The use of nanoparticles enables the formation of strong internal DC fields and allows the 5CB liquid crystal directors to adopt a uniform oriented configuration, yielding an OFF-state transmittance of up to 75%. Combining the internal DC electric field with an external field ($E_{ext} = 1$ V/μm) induces a random orientation of the liquid crystal directors, switching the device into an opaque ON state. Devices fabricated using liquid crystals with positive dielectric anisotropy ($\Delta\varepsilon > 0$) overcome certain limitations typical of inverse-mode PDLCs based on liquid crystals with negative dielectric anisotropy ($\Delta\varepsilon < 0$), such as the lack of commercially available liquid crystals with specific physicochemical properties. The excellent electro-optical performance demonstrated by these devices may prove valuable for the development of electrically switchable chromogenic materials and the fabrication of smart windows.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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