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# Molecular Crystals and Liquid Crystals

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# A Comparative Study of UVand EB-Cured PDLC Films via Electro-Optical Properties

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# A COMPARATIVE STUDY OF UV- AND EB-CURED PDLC FILMS VIA ELECTRO-OPTICAL PROPERTIES

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Recent studies of electro-optical properties of Ultraviolet (UV) and Electron Beam (EB) cured Polymer Dispersed Liquid Crystals (PDLCs) show substantial advantages of EB systems. Threshold and saturation voltages are lower while the contrast ratio is higher for EB systems compared to UV counterparts. The present paper confirms this observation on new data of electro-optical response functions of systems made of Tripropyleneglycoldiacrylate (TPGDA) and E7. Effects of film thickness and conditions of preparation on electro-optical (EO) response functions are investigated. Both UV- and EB-curing techniques lead to transmission versus voltage curves that are highly reproducible.

Keywords: electron beam; liquid crystal; polymer; polymer dispersed liquid crystal; ultraviolet

## INTRODUCTION

PDLCs or polymer Dispersed Liquid Crystals are made of micron sized droplets dispersed in a solid polymer matrix [1–3]. They have a remarkable electro-optical behaviour since they can be switched from an opaque to a transparent state simply by application of an electric field. PDLCs are very useful in various applications including optical shutters, privacy windows,

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telecommunications and information displays. Preparation of these films is often based on the polymerization induced phase separation (PIPS) process combined with radiation curing using either Electron Beam (EB) or UV light. EB curing leads to high monomer conversion ratios without a need to use a photoinitiator. This has a strong impact on film performance and mechanical strength [4–8].

In this paper, systems made of TPGDA/E7 are used at a composition 30/70 by weight-% cured via PIPS process with UV and EB radiations. In the former case, a small amount of photoinitiator is added to initiate the polymerization/cross-linking processes. Analysis of the electro-optical parameters in terms of the film thickness indicates remarkable differences between UV- and EB-cured samples. The results are discussed by looking at the optimal transmissions and voltages applied.

### **EXPERIMENTAL PART**

## **Materials**

The nematic LC used in this work is the eutectic mixture E7 (Merck Ltd, Poole, GB) containing four cyanoparaphenylene derivatives. It exhibits a positive dielectric anisotropy at  $T=20^{\circ}\mathrm{C}$  and a nematic-isotropic transition temperature  $T_{\mathrm{NI}}=61^{\circ}\mathrm{C}$  [9]. The refractive indices of E7 at  $T=20^{\circ}\mathrm{C}$  are given as:  $n_{\mathrm{o}}=1.5183$ ,  $n_{\mathrm{e}}=1.7378$  ( $\lambda=632.8\,\mathrm{mm}$ ). The monomer is Tripropyleneglycoldiacrylate (TPGDA) (UCB, Belgium). In the case of UV cured systems, the photoinitiator used was Lucirin TPO (BASF).

# **Sample Preparation**

30 weight-percent (wt-%) of monomer and 70 wt-% of the LCs were mixed together at room temperature for several hours, and used as initial reactive mixtures for EB-curing. UV-cured samples were prepared from the same mixture containing 2wt-% (of the acrylate mixture) of a conventional photoinitiator (Lucirin TPO, BASF).

Samples for electro-optical studies were prepared by sandwiching the initial reactive mixture between a glass plate (Balzers, Liechtenstein) and a Polyethyleneterephthalate (PET) sheet (Renker, Germany), both coated with a thin transparent layer of conducting indium/tin oxide. A  $100\,\mu m$  thick PET-sheet has been used for the UV curing process. Our EB generator requires a thinner PET substrate of  $50\,\mu m$  to allow a uniform penetration of the applied dose in the depth of the sample.

The film thickness was measured by a micrometer calliper (Mitutoyo; uncertainity:  $\pm 1\,\mu\text{m}$ ). No temperature control during the irradiation processes has been performed.

# **Electron Beam Curing**

The generator used in our experiments was an Electrocurtain Model CB 150 (Energy Sciences Inc.) with an operating high voltage of  $175\,\mathrm{kV}$ . The samples prepared as mentioned above were placed in a tray which was passed under the Electroncurtain on a conveyor belt. The results presented here correspond to a dose of  $105\,\mathrm{kGy}$ .

# **Ultraviolet Curing**

The UV light source used was a Minicure Model MC4-300 (Primarc UV technology) equipped with a medium pressure mercury arc lamp rated  $80 \,\mathrm{W}$  per cm. The samples were placed on a conveyor belt. Results corresponding to  $52 \,\mathrm{and} \, 150 \,\mathrm{mJ/cm^2}$  are given.

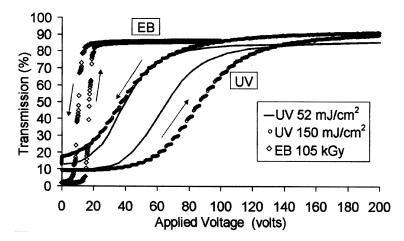
# **Electro-optical Measurements**

Electro-optical experiments were performed at room temperature by measuring the transmission of an unpolarized HeNe laser light at a wavelength of  $\lambda=632.8\,\mathrm{nm}$  orienting the films normal to the incident laser beam. The distance between the sample cell and the detector (silicon photodiode) is approximately 30 cm. The collection angle of the transmitted intensity is about  $\pm2^\circ$ , so that principally forward scattering is detected. The intensity of transmitted light is recorded on a micro-computer using an interface card (DAS 1600-2). Transmission measurements are corrected using appropriate calibration standards.

For electro-optical measurements, an external electric field is applied across the PDLC film. The output of a frequency generator is amplified and used to drive the shutter device. Starting from the electrical off-state, the applied sinusoidal voltage of frequency 145 Hz was increased continuously up to desired maximum value  $V_{\rm max}$ . It is subsequently decreased in the same way. The whole scan up and down ramp takes 120 s with an additional measuring time 60 s allowing to follow the relaxation behaviour of the transmittance in the off-state. The same procedure is repeated several times using the appropriate maximum voltage.

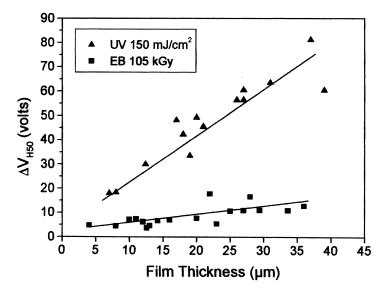
#### RESULTS AND DISCUSSION

Figure 1 represents three electro-optical curves, two of them give results of UV-cured systems with different doses while the third one corresponds to the case of an EB-cured film. Increasing the dose of UV radiation by nearly a factor of 3 (i.e., from 52 to 150 mJ/cm<sup>2</sup>) leads to minor changes in the



**FIGURE 1** Electro-optical curves for 3 systems made of TPGDA/E7 precursor mixtures under UV curing with  $52\,\mathrm{mJ/cm^2}$  and  $150\,\mathrm{mJ/cm^2}$  and EB-cured film with a dose of  $105\,\mathrm{kGy}$  ( $\lambda = 632.8\,\mathrm{nm}$ , sinusoidal voltage ramps of frequency  $145\,\mathrm{Hz}$  at room temperature).

electro-optic parameters. In the case of UV-curing, the plateau of transmission is hardly reached even at the highest applied voltage. The characteristic voltages  $V_{10}$ ,  $V_{90}$  and  $\Delta V_{50}$  increase slightly with the dose. Higher doses produce films that require higher voltages to be activated. Here, the crosslinking density of the network increases yielding smaller droplet sizes [10,11]. Note also that the light transmission is enhanced while the width of hysteresis broadens. Figure 1 exhibits also an electrooptic curve from an EB-cured system. For shortness, we limit ourselves to this single example leaving a more detailed discussion of UV and EBcured PDLCs to the near future. Nevertheless, a brief analysis of the curves in Figure 1 leads to the observation of a considerable improvement of electro-optic parameters in the case of EB-curing. One finds that  $V_{10}$  and  $V_{90}$ are lower,  $T_{\rm off}$  remains near zero before and after applying the electric field meaning that there are no or very weak memory effects. Moreover the maximum transmission  $T_{\rm ON}$  exceeds 85% and is already achieved at a voltage less than 20 V. The width of hysteresis  $\Delta V_{50}$  is also very narrow. These results confirm the conclusions made earlier by Gyselinck et al. [12] that EB-cured PDLC films have better electro-optical responses as compared to their UV counterparts. In Figure 2, the hysteresis width  $\Delta V_{\rm H50}$  is plotted against film thickness for the two systems obtained under EB- and UVcuring. Although data points are somewhat scattered, a linear increase of  $\Delta V_{\rm H50}$  versus thickness is found in both cases. Clearly, hysteresis is more pronounced for the UV-cured system. A more detailed analysis of those



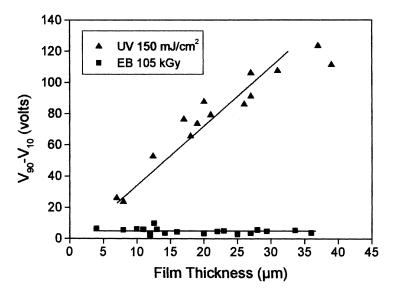
**FIGURE 2** Hysteresis width  $\Delta V_{\rm H50}$  versus film thickness for EB- and UV-cured samples. Doses are 105 kGy for the EB- and 150 mJ/cm<sup>2</sup> for the UV-system. The continuous lines represent guides for the eye.

data and in particular the slope of the V's versus film thickness can certainly give useful information on properties such as the elastic Frank constant and the dielectric anisotropy in terms of the method of preparation of the films. Figure 3 illustrates the variation of  $\Delta V = V_{90} - V_{10}$  as a function of film thickness for the same systems. This quantity is directly related to the increase of transmission versus voltage for both systems.  $\Delta V$  increases linearly with film thickness and the slope in the case of UV-cured sample is higher. Indeed the EB-cured blend yields a switching from off- to on-state within a much smaller voltage interval  $\Delta V$  as compared to the UV-cured samples.

Finally, note that the chemical stability is more easily achieved in the case of EB-curing. Further analysis of various PDLC materials is in progress to correlate between the method of preparation, morphology of the samples with the observed electro-optical properties.

### CONCLUSIONS

Electro-optical properties of composite materials made of TPGDA/E7 mixtures cured by UV and EB radiation techniques are investigated. Under all conditions, the electro-optical curves are characterized by a low



**FIGURE 3**  $\Delta V = V_{90} - V_{10}$  as a function of film thickness for UV-(150 mJ/cm<sup>2</sup>) and EB-cured (105 kGy) samples. The continuous lines represent guides for the eye.

transmission in the initial off-state and relatively high transmission in the ON-state. EB curing leads to films characterized by reduced thresold and saturation voltages as compared to analogous UV-cured systems hence a better electro-optical performance. The voltage width at 50% transmission that describes the electro-optical hysteresis increases linearly with the film thickness. The slope of the line is much higher in the case of UV cured systems. Similar observations are made when analyzing data for  $\Delta V = V_{90} - V_{10}$ .

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