

# Structural Effect of Photoinitiators on Electro-optical Properties of Polymer-Dispersed Liquid Crystal Composite Films

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**ABSTRACT:** Two types of photoinitiators were synthesized: (1) a  $\alpha,\omega$ -telechelic oligomeric photoinitiator, by the reaction of poly(propylene glycol) diglycidylether (PPGDGE) and 2-hydroxy-2-methyl-1-phenyl-propan-1-one (Darocur 1173), and (2) a polymeric photoinitiator, by copolymerization of a monomer that had a liquid crystalline property, 4-[ $\omega$ -(2-methylpropenoxy)decanoxy]-4'-cyanobiphenyl, with a vinyl monomer that had a photosensitive group. For comparison, low-molecular-weight (low-MW) photoinitiator (Darocur 1173) also was used. Attention was directed to the structural effect of the photoinitiators on the electro-optical properties of polymer-dispersed liquid crystal (PDLC) film in which the LC phase occupied a major volume (80 wt % of the composite film). For the preparation of PDLC films by the polymerization-induced phase separation method, the optimum UV-curing temperature was observed at 50°C, a temperature slightly higher than the cloud temperature ( $T_{\text{cloud}}$ ) of the low-MW LC/matrix-form-

ing material mixture. It was found that the electro-optical performance of the PDLC cell fabricated with the oligomeric or polymeric photoinitiator was better than that of the PDLC cell made with a low-MW photoinitiator (Darocur 1173), exhibiting lower driving voltage ( $V_{90}$ ) and higher contrast ratio under identical formulation conditions. Oligomeric photoinitiators allowed premature phase separation between the LC and matrix phases, resulting in relatively pure LC-rich phases. For the polymeric photoinitiator, incorporation of mesogenic moieties into the photoinitiator resulted in not only a well-defined LC/matrix morphology but also in low driving voltage ( $V_{90}$ ) because of reduced friction at the LC/matrix interfaces. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 99: 162–169, 2006

**Key words:** composites; phase separation; photopolymerization; dispersions

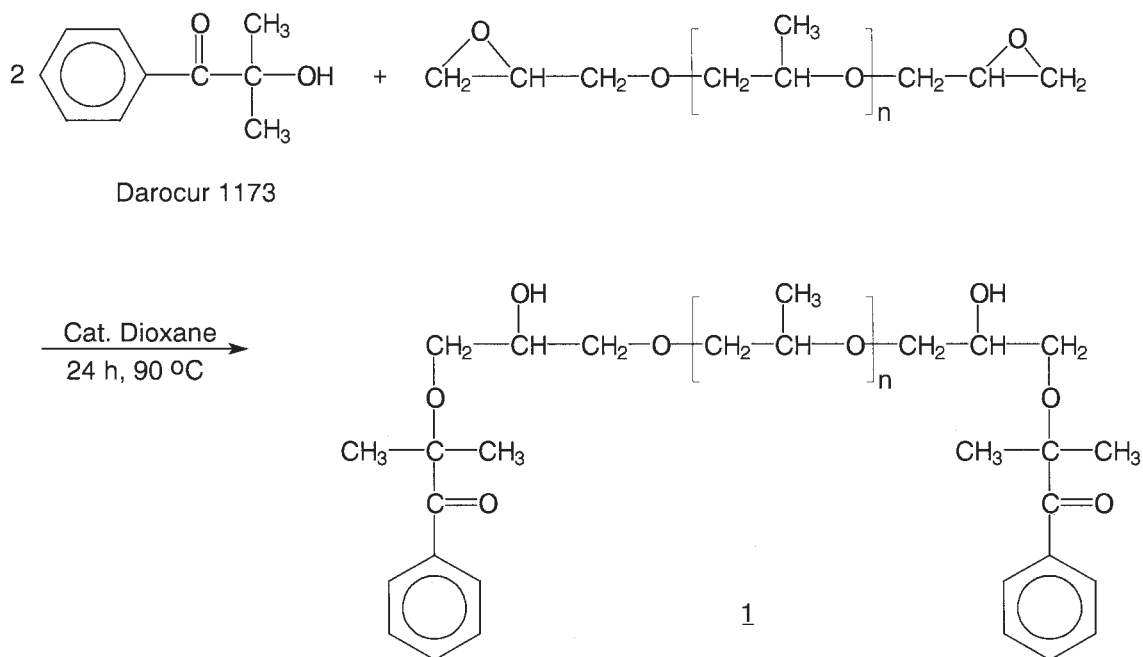
## INTRODUCTION

Polymer-dispersed liquid crystal (PDLC) films, which have great potential for use in electro-optical devices such as large-area displays, open-curtains, and light valves, are heterogeneous systems composed of dispersed or continuous LC phases in various polymer matrices.<sup>1–5</sup> Since Ferguson<sup>6</sup> and Doane et al.<sup>7</sup> introduced the practical use of PDLC films, there has been remarkable interest in these films, with a focus on both applied and basic research<sup>8–10</sup> into developing PDLC devices to control the electro-optical properties of PDLC films. Various methods<sup>11–15</sup> such as thermally induced phase separation (TIPS), solvent-induced phase separation (SIPS), polymerization-induced

phase separation (PIPS), and encapsulation can be used to prepare PDLC films. Among these, PIPS with the aid of UV light is one of the most commonly used methods for preparing PDLC films in which polymerization is induced by a photoinitiator in a mixture of low-molecular-weight (low-MW) LC, UV-polymerizable oligomers, and multifunctional monomers. From an initially homogeneous single phase, the formation of a heterogeneous composite film composed of LC- and polymer-rich phases is induced by the increased immiscibility between the LC and matrix-forming material as polymerization proceeds. Because the performance of the PDLC cell is proportional to the volume fraction of the LC phases, the LC concentration should be taken to the maximum allowed by the specific matrix. To do this, it is required that processing variables such as the kind of LCs, monomers/functional oligomers, UV-light intensity, polymerization temperature, and photoinitiator concentration be optimized. Photoinitiator concentration, for example, needs to be optimized in order to obtain a uniformly phase-separated PDLC film. An excess amount of a photoinitiator

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Scheme 1

used in the polymerizing mixture may remain as impurities either in the matrix or in LC droplets because of inefficient initiation, causing long-term instability of the resulting PDLC film.

In the present work, we investigated the structural effect of photoinitiators on the electro-optical properties of the resulting PDLC cells. First, a new oligomeric photoinitiator was synthesized. Because the molecular weight of the oligomeric photoinitiator was similar to that of UV oligomers, the oligomeric photoinitiator was effectively phase-separated from low-MW LCs in the matrix-forming domains, resulting in improved electro-optical performance of PDLCs because of less contamination of the LC phases. Second, we synthesized a polymeric photoinitiator with mesogenic groups in the main chain in order to increase the chemical affinity to LC molecules at the LC/matrix interface and to reduce the concentration of unreacted fragments of photoinitiators inside the LC phases. This could reduce the response time of the LC phases and, hence, improve the performance of the PDLC cells.

## EXPERIMENTAL

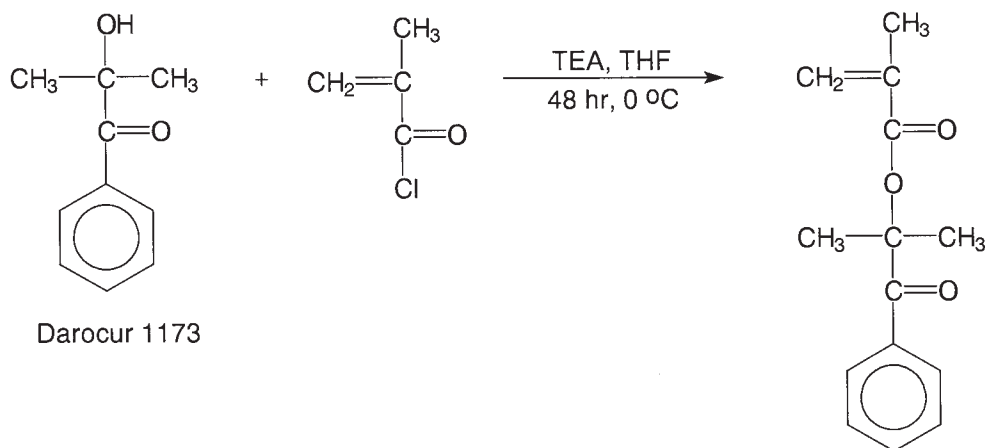
### Materials

BL-002, a eutectic LC mixture (BDH Co., Poole, UK;  $\Delta n = 0.246$ ,  $T_{\text{NI}} = 72^\circ\text{C}$ ,  $\Delta\varepsilon = 15.6$ ) and 7CB, a single LC (Merck Co., Whitehouse Station, NJ;  $\Delta n = 0.178$ ,  $T_{\text{KN}} = 30^\circ\text{C}$ ,  $T_{\text{NI}} = 42^\circ\text{C}$ ,  $\Delta\varepsilon = 11.5$ ), were used as received. 2-Hydroxy-2-methyl-1-phenyl-propan-1-one (Darocur 1173, Merck Co.), and poly(propylene glycol) diglycidylether ( $M_w = 380$  g/mol; PPGDGE, Aldrich Chem.

Co., Milwaukee, WI) were used to synthesize a new type of  $\alpha,\omega$ -telechelic oligomeric photoinitiator (1). Poly(ethylene glycol) dimethacrylate (PEGDM) ( $M_w = 400$  g/mol) and 1,1,1-trimethylol propanetriacrylate (TMPTA) from Aldrich Chem. Co. were used as difunctional oligomer and trifunctional monomers, respectively. Methacryloyl chloride, triethylamine, 4-cyano-4'-hydroxybiphenyl, and 10-bromo-1-decanol were purchased from Aldrich Chem. Co. and used as received. Tetrahydrofuran (Aldrich Chem. Co.) was distilled over sodium prior to use. Other solvents and chemicals were of reagent grade and used without further purification.

### Synthesis of oligomeric photoinitiator (1)

Oligomeric photoinitiator (1) was synthesized as shown in Scheme 1.<sup>16</sup> In a nitrogen atmosphere in a dark room, 0.5 g of Darocur 1173 was added to 10 g of dioxane solution in which 0.586 g of PPGDGE had been dissolved. The reaction of the Darocur 1173 photoinitiator with the epoxy groups at both ends of PPGDGE was performed with 100 ppm of acid catalyst ( $\text{H}_2\text{SO}_4$ ) diluted in dioxane for 24 h at  $90^\circ\text{C}$ . After the reaction the resulting solution was neutralized with 0.1N NaOH solution, and dioxane was removed with a rotary evaporator. Salt formed by adding ethyl ether to the resulting product was removed by washing several times with distilled water. The remaining moisture was removed by stirring with anhydrous magnesium sulfate, followed by filtration. The  $\alpha,\omega$ -telechelic oligomeric photoinitiator (1) was purified by using a column chromatography filled with silica gel



Scheme 2

and a mixture of ethyl acetate/*n*-hexane [1 : 5 (v/v)] as an eluent.

### Synthesis of polymeric photoinitiator with mesogen moieties

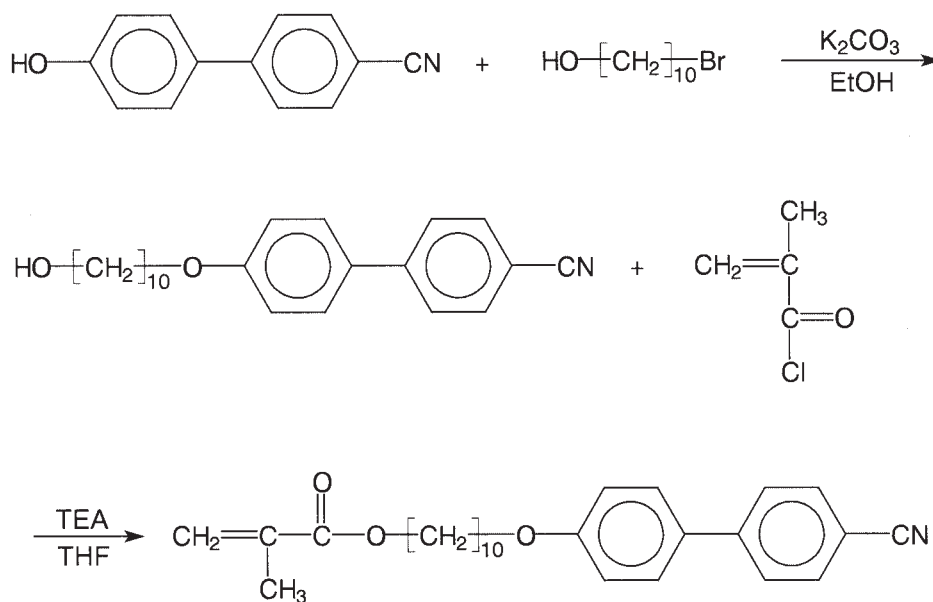
Vinyl monomer with a photoinitiating group (2)

The mixture of 2.5 g of methacryloyl chloride, 3.0 g of Darocur 1173, and 2.8 g of triethylamine (TEA) in 15 mL of dried THF was reacted in a dark room at 0°C for 24 h, as shown in Scheme 2. The reaction mixture was filtered in order to remove the precipitated salt, then concentrated by evaporating the THF using a rotary evaporator. The final product, **2**, was purified by using a column filled with silica gel and a 1 : 6 (v/v) mixture of ethyl acetate/*n*-hexane as an eluent. <sup>1</sup>H-NMR (CDCl<sub>3</sub>) δ: 7.35–8.03 (m, 5H, Ar *H*), 5.55–6.20 (d, 2H, —CH<sub>2</sub>), 1.82 (s, 3H,

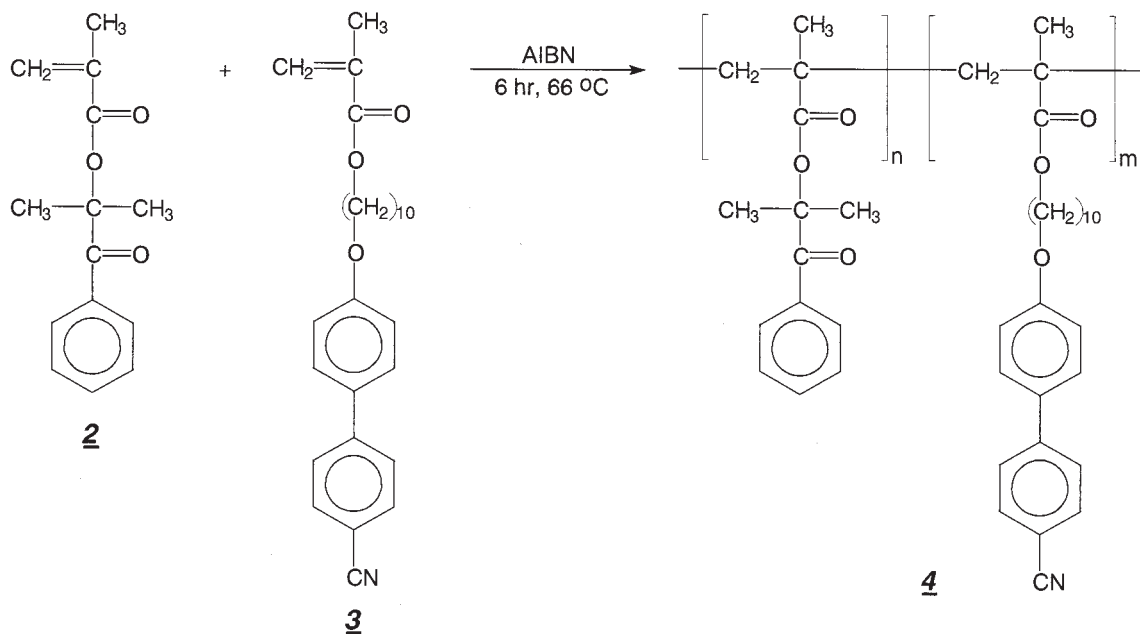
—CH<sub>3</sub>), 1.80 (s, 6H, —CH<sub>3</sub>). FTIR (KBr) cm<sup>-1</sup>: 1690 (C=C), 1000–1200 (C—O—C), hydroxyl peak (3600 cm<sup>-1</sup>) from Darocur 1173 disappeared.

Vinyl monomer with a mesogenic group (3)

4-[ω-(2-Methylpropenoxy)decanoxy]-4'-cyanobiphenyl (**3**) was obtained as shown in Scheme 3. In the first step, 7.0 g of 4-cyano-4'-hydroxybiphenyl was dissolved in 150 mL of ethanol with slow heating, then stirred for 30 min after adding 7.0 g of K<sub>2</sub>CO<sub>3</sub>. The solution of 7.0 g of 10-bromo-1-decanol in 25 mL of DMF was slowly added to the previously prepared solution, followed by reflux for 24 h. After removing KBr, byproduct, through hot filtration, the filtrate was poured into distilled water. The precipitate was recovered by filtration and dried at 40°C for 24 h. Then the



Scheme 3



Scheme 4

4-(hydroxydecanoxy)-4'-cyanobiphenyl intermediate was recrystallized in benzene (yield 46%).

A solution of 7.0 g of 4-(hydroxydecanoxy)-4'-cyanobiphenyl and 4.0 g of methacryloyl chloride in 100 mL of THF was prepared with stirring for 30 min under a  $N_2$  atmosphere and then reacted for 24 h after adding a solution of 4.0 g of TEA in 100 mL of THF at 0°C. The resulting product (3) was recovered and purified the same way as was the preparation of vinyl monomer (2) (yield 45%).  $^1H$ -NMR ( $CDCl_3$ )  $\delta$ : 6.9–7.7 (m, 8H, Ar H), 5.5–6.1 (d, 2H,  $-CH_2-$ ), 4.0–4.2 (d, 4H,  $-O-CH_2-$ ), 2.0 (s, 3H,  $=C-CH_3$ ), 1.2–1.9 [m, 16H,  $-(CH_2)_8-$ ]. FTIR (KBr)  $cm^{-1}$ : 2200 ( $-CN$ ), 1700 ( $C=C$ ), hydroxyl peak ( $3600\ cm^{-1}$ ) from 4-(hydroxydecaoxy)-4'-cyanobiphenyl disappeared.

#### Polymeric photoinitiator (4)

As can be seen in Scheme 4, the polymeric photoinitiator (4) was prepared by polymerizing 2 and 3 monomers in THF solvent using 2,2'-azobisisobutyronitrile (AIBN) (0.5 mol%) in a dark room under  $N_2$  atmosphere for 6 h at 60°C. The feed ratios of 3/2 were 100 : 0, 90 : 10, and 80 : 20 mol %. After polymerization, the polymeric photoinitiator (4) was purified by the repeated dissolution/precipitation method and were dried for 48 h at 40°C.

#### PDLC film preparation

Formulations in the initial homogeneous mixture was fixed at an 80 : 20 (w/w) ratio of LC to matrix-forming material. The eutectic mixture, BL-002, and matrix-

forming materials including a difunctional oligomer (PEGDM), a multifunctional monomer (TMPTA), and an oligomeric photoinitiator (1) were thoroughly mixed and filled into a PDLC cell fabricated with two ITO glasses (gap 11.7  $\mu m$ ) by capillary force. The PDLC cell was placed on a hot stage with a variable temperature controller and irradiated by UV light (ORC Polymer Print, 12  $mW/cm^2$  at 312 nm) for 3 min at a certain temperature.

#### Instrumental analysis

The  $^1H$ -NMR and FTIR spectra of the synthesized compounds were taken on a Bruker AM 300 (300 MHz) using  $CDCl_3$  as a solvent and on a Midac FTIR spectrometer, respectively. Molecular weight was measured using a Waters gel permeation chromatograph (GPC) equipped with a 410 differential refractometer, a 510 HPLC pump, and Shodex column (AT 805/T), using THF as an eluent against polystyrene standards at room temperature. UV-visible absorption spectra were obtained with a Shimadzu UV-2100. An elemental analyzer (Carlo Erba Model 1106) was used to calculate the compositional ratio of polymeric photoinitiator.

A polarized optical microscope (POM, Zeiss Jenalab) equipped with a hot stage whose camera had been replaced by a photosensor connected to a Hameg 408 digital oscilloscope was used to observe the change in transmittance with an applied voltage. An HP 8904A function generator combined with a Kepco BOP500M power amplifier was used to apply voltage to the PDLC film or switch it on or off. The on/off time of the PDLC cell at a given voltage was measured with an

TABLE I  
Characterization Data of the Polymeric photoinitiator (4)

No.	Monomer feed ratio [M <sub>1</sub> ]/[M <sub>2</sub> ] <sup>a</sup>	Elemental analysis (wt %)				Copolymer composition <sup>b</sup> [M <sub>1</sub> ]/[M <sub>2</sub> ]	Yield (%)	$\bar{M}_n$ ( $\times 10^4$ )	$\bar{M}_w/\bar{M}_n$
		C	H	N	O <sup>c</sup>				
Poly(3) <sup>e</sup>	0 : 100	77.33 <sup>d</sup>	7.87 <sup>d</sup>	3.34 <sup>d</sup>	11.46 <sup>d</sup>	0 : 100	70.2	2.1	2.24
4(a)	10 : 90	74.37	9.31	3.41	12.92	22.5 : 77.5	64.2	1.9	4.22
4(b)	30 : 70	72.89	8.88	2.67	15.57	43.1 : 56.9	60.3	0.82	5.45

<sup>a</sup> [M<sub>1</sub>] = 2; [M<sub>2</sub>] = 3

<sup>b</sup> Determined by elemental analysis.

<sup>c</sup> Determined by calculation; [100 - (C + H + N)] (wt %)

<sup>d</sup> Theoretical value.

<sup>e</sup> Homopolymer of 3.

oscilloscope, and the data were manipulated with a personal computer.

## RESULTS AND DISCUSSION

### Characterization of polymeric photoinitiator (4)

Two polymeric photoinitiators with different compositions, 4(a) and 4(b), were prepared from the copolymerization of 2 and 3. For comparison, homopolymer poly(3) also was prepared. The composition of the polymeric photoinitiators (4) was calculated by elemental analysis using the eq. (1)

$$\frac{W_H}{W_N} = \frac{1 \times 18X + (1 - X)33}{1 \times 14(1 - X)} \quad (1)$$

where  $W_H$  and  $W_N$  are the weight fractions of the hydrogen and nitrogen elements, respectively, measured by elemental analysis, and  $X$  is the mole fraction of 2 in the copolymer. The results are summarized in Table I, which shows that 4(a) and 4(b) were composed of 2 : 3 in ratios of 22.5 : 77.5 and 43.1 : 56.9 (mol %), respectively. Molecular weight was measured as 21,000 g/mol for poly(3), 19,000 g/mol for 4(a), and 8200 g/mol for 4(b). It appeared that the molecular weight of the polymers decreased with an increasing content of 2 in the feed, accompanied by an increased molecular weight distribution. This might have originated from steric hindrance of 2, which had bulky side groups.

Samples with varying content of mesogenic monomer 3 in the synthesized polymers showed clear differences in LC behavior. Optical microscopy pictures of three samples are presented in Figure 1. Poly(3) and 4(a) (77.5 mol % of 3) showed LC transitions at 83°C and 65°C, respectively. However, 4(b) (56.9 mol% of 3) showed no LC transition. This indicated that the molecular orientation of the mesogenic units 3 in the matrix was hampered by an increased content of 2 in the synthesized polymers.

### Effect of temperature on PDLC fabrication

It is well known that the electro-optical properties of PDLC cells strongly depend on the phase separation process and the final morphology of LC/matrix polymer composite film. Because a relatively large amount of LC (ca. 80 wt %) was used in the fabrication of PDLC cells in our experiments, the preparation conditions of the PDLC film by the PIPS method were studied carefully. In the PDLC films with the oligomeric photoinitiator (1), the formulation of the LC and matrix-forming materials was set as follows; BL-002 (LC) : 1 : PEGDM (difunctional oligomer):TMPTA (trifunctional monomer) = 80 : 7 : 12 : 1 (wt %). Then this mixture was UV-irradiated at various temperatures. The change in transmittance of the resulting PDLC films as a function of the applied voltage is shown in Figure 2. It was observed that the PDLC samples photopolymerized at temperatures below or above 50°C resulted in a high initial transmittance. This phe-

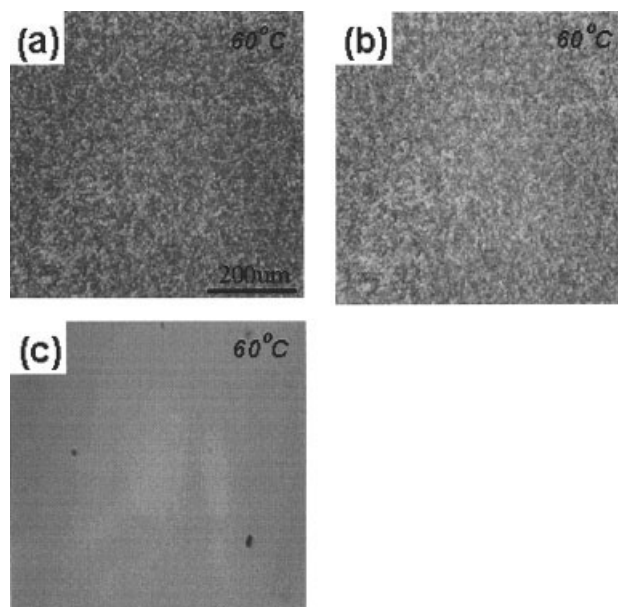
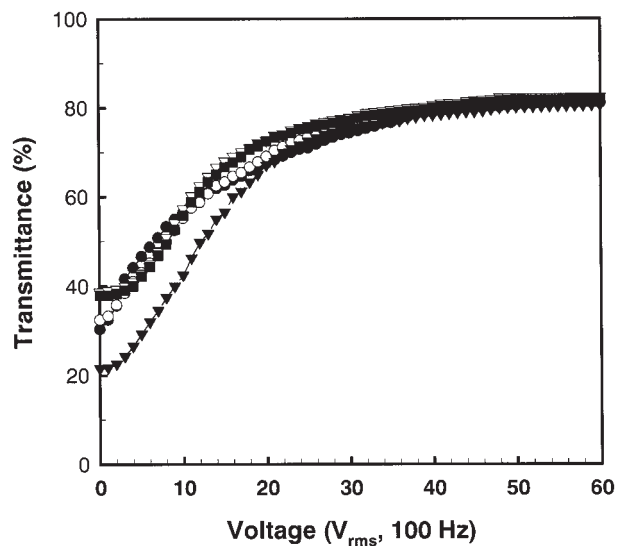


Figure 1 Polarized optical microphotographs of (a) poly(3), (b) 4(a), and (c) 4(b). Scale bar = 200  $\mu$ m.





**Figure 2** Effect of fabrication temperatures on electro-optical properties of PDLC cells: 38°C (●), 45°C (○), 50°C (▼), 55°C (▽), 60°C (■).

nomenon may be explained by the cloud temperature ( $T_{\text{cloud}}$ ) of the initial mixture of LC/matrix-forming material. In this work, the  $T_{\text{cloud}}$  of the initial formulation was determined by POM to be 49°C. The PDLC samples that photopolymerized at temperatures lower than  $T_{\text{cloud}}$  might have an uneven distribution of LC-rich domains in size because of a pre-phase-separated state of the initial mixture. With photopolymerization at temperatures higher than  $T_{\text{cloud}}$ , in contrast, LC-rich phases containing the oligomeric photoinitiator (**1**) could be formed because of enhanced solubility between the LC and matrix-forming material at elevated temperatures. Therefore, the oligomeric photoinitiators (**1**) that remained inside LC droplets may not have effectively initiated the photopolymerization, and then these unreacted **1** photoinitiators may have acted as impurities after fabrication of PDLC cells. Moreover, the enhanced solubility of the LC with matrix-forming material may have caused fewer refractive index differences between two phases, resulting in high initial transmittance of the PDLC cells. POM photographs of PDLC films taken at different temperatures before photopolymerization, shown in Figure 3, provide evidence of these contentions. It is essential, therefore, that photopolymerization be carried out just above the  $T_{\text{cloud}}$  of the initial mixture at which the mixture could have a slightly phase-separated status consisting of an LC-rich phase and a **1**: PEGDM: TMPTA-rich phase. Subsequent UV irradiation can lead to polymerization-induced phase separations of this state, resulting in the formation of a relatively pure LC-rich phase in the PDLC cells.

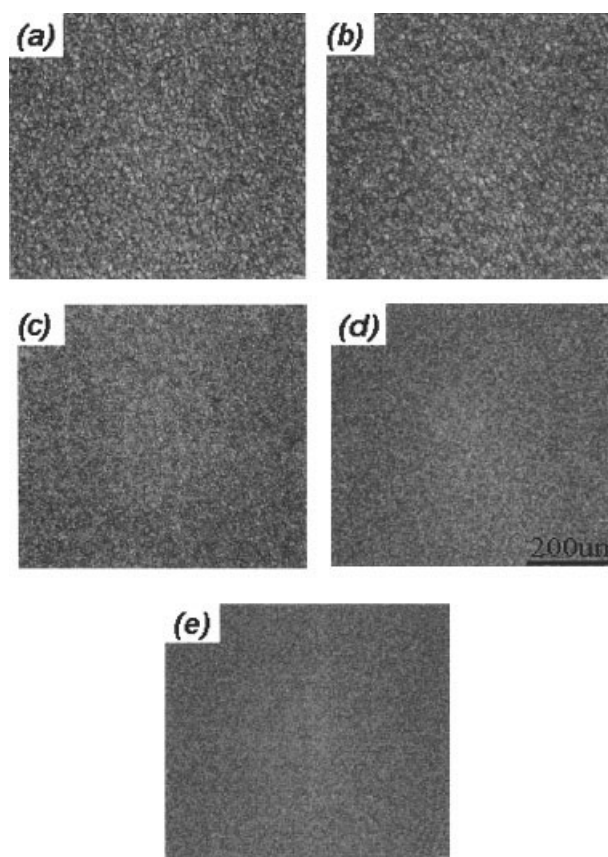
With the polymeric photoinitiator (**4**), the composition was set to 7CB (LC 80 wt %) and matrix-forming components [**4(b)**: PEGDM: MMA = 6:6:8 wt %]. The  $T_{\text{cloud}}$  of LC and matrix-forming components also

was measured at about 49°C using POM equipped with a hot stage. As shown in Figure 4, the domain size of the PDLC films decreased as the UV irradiation temperature was increased from 37°C to 50°C.

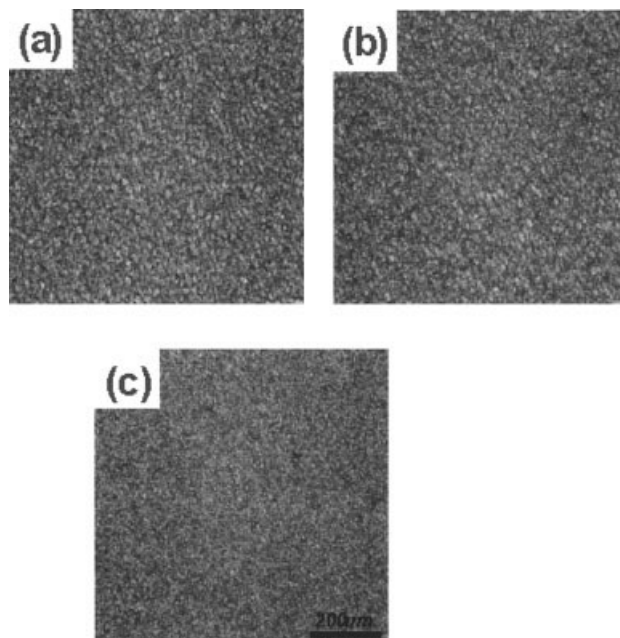
### Electro-optical properties of PDLC films

To investigate the effect of photoinitiator performance on the electro-optical properties of the PDLC cells, three types of photoinitiators were used: low-MW photoinitiator (Darocur 1173),  $\alpha,\omega$ -telechelic oligomeric photoinitiator (**1**), and polymeric photoinitiator (**4**) with mesogenic side groups. The formulations for the fabrication of PDLC cells are shown in Table II. The concentration of photoinitiators was based on the same number of net photoinitiating groups as determined by UV-vis spectroscopy. When the polymeric photoinitiator (**4**) was employed, MMA was added to reduce the viscosity of the initial mixture.

Figure 5 shows the transmittance versus voltage curves of the PDLC cells. It appeared that the PDLC cell fabricated with composition **B** using oligomeric photoinitiator (**1**) exhibited a much higher contrast ratio than did the cell from composition **A** using low-

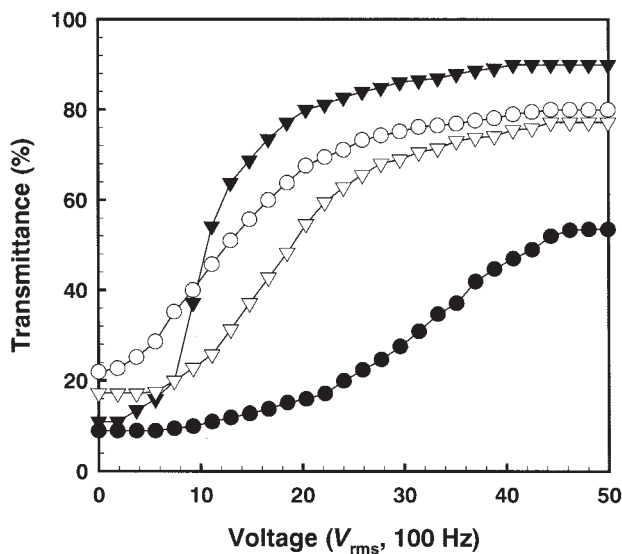


**Figure 3** Polarized optical microphotographs of PDLC cells fabricated using oligomeric photoinitiator (**1**) at different temperatures: (a) 38°C, (b) 45°C, (c) 50°C, (d) 55°C, (e) 60°C. Scale bar = 200  $\mu\text{m}$ .



**Figure 4** Polarized optical microphotographs of PDLC cells fabricated using polymeric photoinitiator (**4**) at different temperatures: (a) 37°C, (b) 43°C, (c) 50°C. Scale bar = 200  $\mu\text{m}$ .

MW photoinitiator (Darocur 1173). This may be attributed to the low miscibility of **1** with LC molecules, which rendered the oligomeric photoinitiator (**1**) to separate prematurely into the matrix-forming mixture, as presented in Figure 6(b). As a result, relatively pure LC-rich domains could be obtained in the fully phase-separated state of the PDLC cells. In contrast, the low-MW photoinitiator (Darocur 1173) moiety may remain in the LC phase even after photopolymerization-induced phase separation, as schematically presented in Figure 6(a). Unreacted fragments of low-MW photoinitiator could act as impurities, detrimentally affecting the electro-optical properties, especially the long-term stability of PDLC cells.



**Figure 5** Transmittance versus voltage plots of PDLC cells fabricated of different compositions: (A) (●), (B) (○), (C) (▽), and (D) (▼).

As can be seen in Figure 5, PDLC cells prepared using composition **D** with the polymeric photoinitiator [**4(b)**] showed the highest contrast ratio and a lower  $V_{90}$  value (ca. 30 V), a voltage required for 90% of saturated transmittance of the PDLC film. This was considered to have occurred because of the dual function of the polymeric photoinitiator in the PDLC. As a result of chemical affinity and size consideration, the polymeric photoinitiator had low miscibility with low-MW LC and hence yielded well-defined phase separation with LC phases during the fabrication of PDLC cells. Moreover, the incorporation of mesogenic side groups, which had a structure similar to LC, in the polymeric photoinitiator could have led to reduced friction in the movement of molecules at the LC/matrix interface, as can be seen in Figure 6(c).

**TABLE II**  
Formulations with Different Types of Photoinitiators Employed for Fabrication of PDLCs

Composition	LC (wt %)	Photoinitiator (wt %)	PEGDM <sup>f</sup> (wt %)	MMA <sup>g</sup> (wt %)	TMPTA <sup>h</sup> (wt %)	MM <sup>i</sup> (wt %)
A	80.0 <sup>a</sup>	3.2 <sup>c</sup>	15.8	—	1.0	—
B	80.0 <sup>a</sup>	7.0 <sup>d</sup>	12.0	—	1.0	—
C	80.0 <sup>b</sup>	2.6 <sup>c</sup>	6.0	8.0	—	3.4
D	80.0 <sup>b</sup>	6.0 <sup>e</sup>	6.0	8.0	—	—

<sup>a</sup> BL-002.

<sup>b</sup> 7CB.

<sup>c</sup> Low-MW photoinitiator (Darocur 1173).

<sup>d</sup> Oligomeric photoinitiator (**1**).

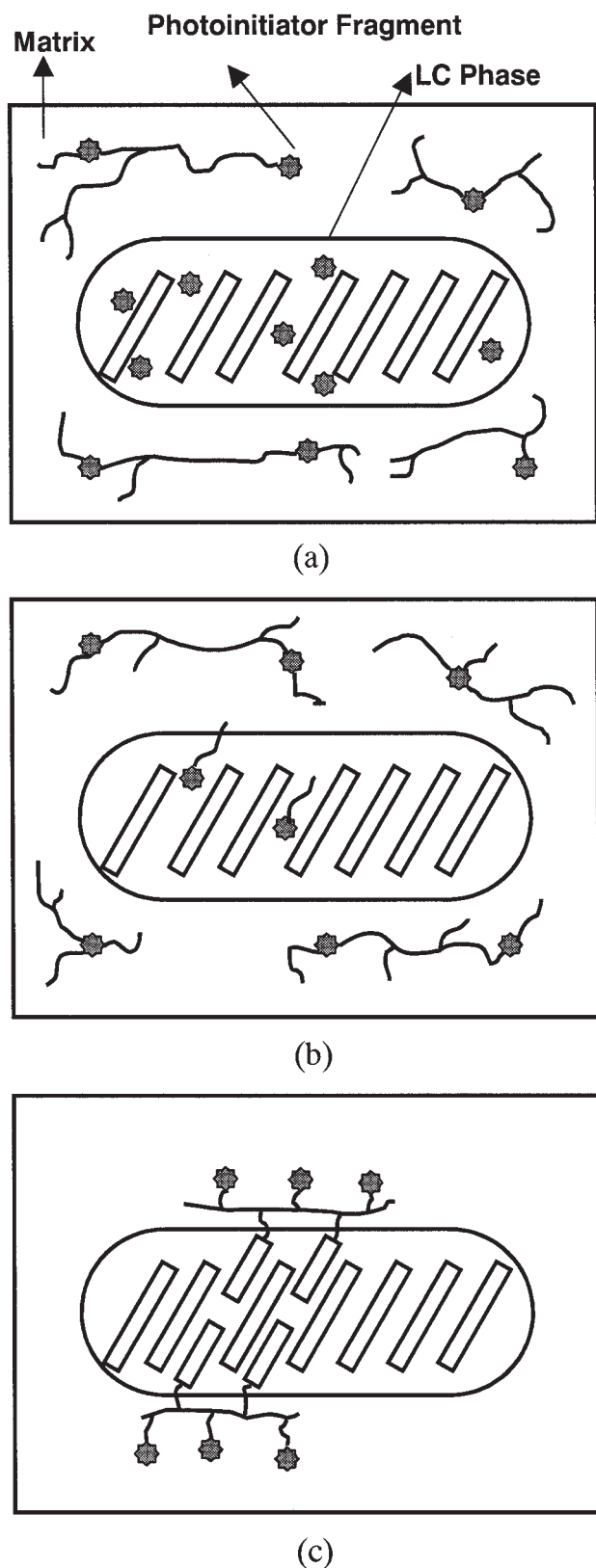
<sup>e</sup> Polymeric photoinitiator [**4(b)**].

<sup>f</sup> Difunctional oligomer.

<sup>g</sup> Methyl methacrylate.

<sup>h</sup> Trifunctional monomer.

<sup>i</sup> Mesogenic monomer (**3**).



**Figure 6** Schematic illustration of PDLC cells fabricated with different types of photoinitiators: (a) low-MW photoinitiator (Darocur 1173), (b) oligomeric photoinitiator (**1**), (c) polymeric photoinitiator (**4**) with mesogenic moieties.

## CONCLUSIONS

The structural effect of photoinitiators on the electro-optical properties of PDLC cells was investigated. The  $\alpha,\omega$ -telechelic oligomeric photoinitiator (**1**) was synthesized by the reaction of poly(propylene glycol) diglycidylether and a low-MW photoinitiator (Darocur 1173). Another type of photoinitiator synthesized was a polymeric photoinitiator (**4**) having mesogenic moieties that were structurally similar to LC molecules.

The optimum fabrication temperature of the PDLC cells with the oligomeric photoinitiator (**1**) or the polymeric photoinitiator (**2**) was found to be 50°C, slightly above the  $T_{\text{cloud}}$  of the initial LC/matrix-forming material mixture. Studies of the electro-optical properties revealed that PDLC cells fabricated with the oligomeric photoinitiator (**1**) exhibited higher transmittance under optimum fabrication conditions because of the increased immiscibility of the oligomeric photoinitiator (**1**) with the LC phase. It was observed that with the polymeric photoinitiator (**4**) possessing structurally similar mesogenic moieties to LC molecules, the electro-optical properties of the PDLC cells were further improved, resulting in low driving voltage and a high contrast ratio. Consequently, these results indicated that the electro-optical performance of PDLC cells could be improved by designing the structure and molecular weight of photoinitiators used in the formulation of LC/matrix-forming materials.

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