

An Investigation into the Morphology and Electro-Optical Properties of 2-Hydroxy Ethyl Methacrylate Polymer Dispersed Liquid Crystals

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ABSTRACT: Polymer dispersed liquid crystal (PDLC) films are fabricated using E7 liquid crystals, tetraethylene glycol diacrylate (TeGDA) crosslinking agent, and 0–66.49 mol % 2-hydroxy ethyl methacrylate (HEMA). The effects of different levels of HEMA addition on the microstructure and electro-optical properties of the PDLC samples are examined using scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, and UV-Vis spectroscopy, respectively. The results show that the refractive index of the PDLC films is insensitive to the level of HEMA addition. However, an increasing HEMA content improves the degree of phase separation during the polymerization process and increases

the size and uniformity of the liquid crystal domain. As a result, the electro-optical properties of the PDLC films are significantly improved as the level of HEMA addition is increased. Overall, the results show that a PDLC comprising 40 wt % E7 liquid crystals, 33.51 mol % TeGDA and 66.49 mol % HEMA has a high contrast ratio (13 : 1) and a low driving voltage (10 V) and is therefore an ideal candidate for a wide variety of intelligent photoelectric applications. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 1349–1355, 2010

Key words: polymer dispersed liquid crystal (PDLC); morphology; phase separation

INTRODUCTION

As Hilsum¹ first demonstrated the effects of a fine dispersion of micrometer-scale glass particles on the optical penetration and light-scattering properties of a nematic liquid crystal solution, the feasibility of enhancing the optical properties of liquid crystals through the use of light-scattering particles has attracted intensive interest from manufacturers of liquid crystal displays (LCDs) and intelligent photoelectric applications. In 1982, Craighead et al.² infiltrated a micron-scale polymer matrix with nematic liquid crystals and examined the correlation between the light-scattering and optical transmittance properties of the resulting solution and the magnitude of the external electric field. A few years later, Ferguson and Drzaic³ fabricated a nematic curvilinear aligned phase (NCAP) film comprising a fine dispersion of liquid crystals uniformly distributed within a polymer matrix by applying an emulsion polymerization method to a solution of water-soluble polymers suspended in nematic liquid crystals. In 1986, Doane et al.⁴ demonstrated the fabrication of a new

class of materials known as polymer dispersed liquid crystals (PDLCs) by inducing the phase separation of a solution of polymer monomer and liquid crystals using a polymerization technique. Compared to the earlier proposals for composite polymer liquid crystal films, PDLC films have a number of fundamental advantages, including a lower cost, a more straightforward fabrication process, an improved applicability to large-scale display applications, and so forth. Furthermore, compared to traditional LC displays, PDLCs not only avoid the requirement to align the matrix, but also eliminate the need for additional polarizers and therefore improve both the transmittance and the viewing angle of the device.

PDLCs are fabricated using one of three different phase separation methods. In the first technique, known as polymerization induced phase separation (PIPS), the liquid crystal is dissolved into the monomer and a polymerization reaction is then induced either thermally or photochemically. In the second method, referred to as thermally induced phase separation (TIPS), a homogeneous mixture of liquid crystal and polymer is obtained at a temperature higher than the melting point of the polymer, and the mixture is then cooled at a carefully controlled rate to induce phase separation. In the third technique, known as solvent induced phase separation

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(SIPS), the liquid crystal and thermoplastic polymer are dissolved in a common solvent, which is then evaporated at a specified rate to prompt phase separation.⁵

The electro-optical properties of PDLC films depend fundamentally upon the droplet size within the liquid crystal domain and the amount of liquid crystal diffused in the polymer matrix. Consequently, the problems of optimizing the PDLC composition and identifying the processing conditions, which enhance the phase separation process have been extensively discussed in the literature. For example, Mormile et al.⁶ fabricated a PDLC film with a low driving voltage and a rapid reaction time by mixing unsaturated polyester resin and E7 liquid crystals in a weight ratio of 60 : 40. Meanwhile, Vaia et al.⁷ prepared PDLC films by mixing pentaacrylate and E7 liquid crystals in various weight ratios ranging from 0–50%, and examined their microstructures using scanning electron microscopy. The results showed that an ideal two-phase continuous structure of liquid crystal and polymer was formed for polymer additions in the range 35–40%. Abdoune et al.⁸ examined the effects of the UV exposure time and UV exposure dose on the electro-optical properties of a PDLC film fabricated using tripropylene glycol diacrylate (TpGDA) and E7 liquid crystals mixed in a weight ratio of 30 : 70. The experimental results revealed that for a given exposure time, a higher exposure dose resulted in a greater film thickness and a higher value of the threshold voltage (V_{th}). Benkhaled and Coqueret⁹ mixed TpGDA and E7 liquid crystals in a weight ratio of 30 : 70 and then induced a polymerization reaction using either an electron beam or a conventional UV exposure technique. It was shown that the PDLC fabricated using an electron beam polymerization method not only had a lower threshold voltage (V_{th}) and saturation voltage (V_{sat}) than the conventional film, but also showed no memory effect.

Henry et al.¹⁰ manufactured a PDLC film comprising E8 liquid crystal, CN135 (monofunctional acrylic oligomer) and SR295 (tetrafunctional crosslinker), and investigated the respective effects of the level of SR295 addition, the exposure temperature and the exposure dose on the electro-optical properties of the film. The results showed that the magnitude of the inflection point voltage (V_{inf}) was directly related to the level of SR295 addition. Furthermore, it was shown that when the crosslinking agent was omitted, i.e. the PDLC was fabricated using E8 crystals and CN135 only, the inflection point voltage decreased with an increasing UV exposure dose, but decreased with a decreasing exposure temperature. Malik and Raina¹¹ manufactured PDLCs using E8 liquid crystals and NOA65 (Norland) and investigated the effects of the applied voltage and tempera-

ture on the morphology of the liquid crystals and the electro-optical properties of the PDLC film. The results showed that both the threshold voltage (V_{th}) and the optical transmittance increased with an increasing exposure temperature. Aleksander and Klosowicz¹² showed that when fabricating PDLC films using the PIPS technique, the liquid crystal domain decreased with an increasing exposure energy, but increased with an increasing liquid crystal content. In 2005, Henry et al.¹³ fabricated PDLC films using SR295 crosslinking agent and E8 liquid crystals and found that the diameter of the liquid crystal droplets increased as the relative content of the liquid crystals in the prepolymer mixture was increased. Whitehead and Gill¹⁴ manufactured PDLCs at a temperature higher than the phase separation temperature of the liquid crystal prepolymer mixture and showed that the droplet size reduced as the exposure temperature was increased, but increased as the liquid crystal content in the prepolymer mixture was increased. Furthermore, it was shown that the light-scattering and contrast ratio properties of the film were enhanced when the droplets had a diameter of approximately 1 μm .

In developing PDLC films for intelligent photoelectric applications, the objective is to produce a PDLC with a high contrast ratio, a low threshold voltage and a rapid reaction. In general, the electro-optical properties of PDLCs are dependent upon the choice of polymerization process, the exposure conditions, the degree of phase separation, the choice of monomer, the relative contents of the polymer and liquid crystals in the prepolymer mixture, the size of the liquid crystals following phase separation, and so forth. This study investigates the correlation between the morphology and electro-optical properties of PDLCs fabricated using E7 liquid crystals, tetraethylene glycol diacrylate (TeGDA) crosslinking agent, and 0–66.49 mol % 2-hydroxy ethyl methacrylate (HEMA). Utilizing scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy and UV-Vis spectroscopy, the correlation between the morphology and electro-optical properties of the various PDLC samples is systematically examined. The experimental results are then analyzed to identify the level of HEMA addition, which enhances the contrast ratio of the PDLC film whilst simultaneously reducing the threshold voltage.

EXPERIMENT

Materials

2-Hydroxy ethyl methacrylate (HEMA) and tetraethylene glycol diacrylate (TeGDA) were purchased from TCI Co. (Japan), while Benzoin and E7 liquid crystals were purchased from Acros Organics (UK)

TABLE I
Composition Ratios and Electro-optical Properties of Current PDLC Samples for Illumination Wavelength of 550 nm

Sample	Composition of polymer matrix		Refractive index of polymer matrix	T_{\max}^a (%)	T_{\min}^b (%)	CR ^c	V_{sat} (volt)	V_{th} (volt)
	TeGDA (mol %)	HEMA (mol %)						
Te40LC	100	0	1.5054	93.59	73.37	1.28	110	13
2Te1H40LC	66.23	33.77	1.5073	23.91	11.94	2.01	110	32
1Te1H40LC	50	50	1.5098	50.88	4.1	12.75	78	16
1Te2H40LC	33.51	66.49	1.5083	65.3	5.03	13	85	10

^a T_{\max} : Maximum transmittance (%).

^b T_{\min} : Minimum transmittance (%).

^c CR: Contrast ratio = T_{\max}/T_{\min} .

and E. Merck, respectively. The E7 liquid crystals had a clearing point of $T_{\text{NI}} = 58^\circ\text{C}$ and a composition of 51% *n*-pentylcyanobiphenyl (5CB), 25% *n*-heptylcyanobiphenyl (7CB), 16% *n*-octyloxycyanobiphenyl (8OCB), and 8% *n*-penylcyanoterphenyl (5CT). All the materials were used in an as-received condition without any further purification.

Sample preparation

The PDLC samples were prepared using the PIPS method with a photoinitiated polymerization technique. In the preparation process, the prepolymer mixtures were obtained by mixing 40 wt % E7 liquid crystal with 1 wt % Benzoin photoinitiator and then adding TeGDA and HEMA in the molar ratios shown in Table I. To ensure a homogenous mixture, the components were thoroughly mixed in a shaker mill at a temperature of 65°C for 15 min. The various PDLC samples were sandwiched between clean glass slides separated by Mylar spacers and were then cured using a UV light source (Spectronics Corp. UV-Crosslinker series, model XLE-1000A) with a wavelength of $\lambda = 365$ nm and an exposure intensity of $70,000 \mu\text{J}/\text{cm}^2$.

Measurements

The electro-optical properties of the various PDLC samples, namely the threshold voltage V_{th} , the saturation voltage V_{sat} , the minimum transmittance, the maximum transmittance, and the contrast ratio, were evaluated using a UV-Vis spectrometer (VARIAN, Cary-100). Note that V_{th} and V_{sat} were defined as the voltages required to obtain transmittances of 10 and 90%, respectively, where "100% transmittance" was defined as the intensity of the light beam following its passage through an empty LC cell. The refractive indices of the precursors and cured PDLC films were measured using an Abbe refractometer (Atago DR-M2) at a temperature of 25°C . Samples for SEM observations (Jeol, JSM-6360/LV) were prepared by soaking the PDLC films in methanol for 24

h to extract the low-molecular-weight LC content. The degree of phase separation and the curing kinetics of each sample were examined by casting PDLC films on a KBr tablet and then utilizing a real-time FTIR spectroscopy technique (Nicolet, 320). In the experiments, the degree of phase separation was quantified by measuring the fraction of liquid crystal within each cured film using the method described in Refs. 15 and 16. In general, the absorbance of the nitrile stretching band from the E7 liquid crystal changes as the crystal is brought into a nematic phase from an isotropic state. Let A_I be the absorbance of the liquid-crystal-specific band in its isotropic state and A_N be the absorbance of the liquid-crystal-specific band in its nematic state. Furthermore, let Φ be the fraction of liquid crystal in the initial homogeneous mixture with the matrix precursor. Correcting for a constant cell thickness, the absorbance of the liquid-crystal-specific band is given simply by

$$A_{\text{CN,uncured}} = \Phi A_I. \quad (1)$$

If α is the fraction of liquid crystal which phase separates during the polymerization process, then the total fraction of the liquid crystal in the droplets is $\alpha\Phi$ and the overall absorbance of the liquid-crystal-specific band is given by

$$A_{\text{CN,cured}} = A_N \alpha \Phi + A_I (1 - \Phi). \quad (2)$$

Rearranging eqs. (1) and (2) to eliminate Φ gives α as

$$\alpha = \left(\frac{A_{\text{CN,uncured}} - A_{\text{CN,cured}}}{A_{\text{CN,uncured}}} \right) \left(\frac{A_I}{A_I - A_N} \right). \quad (3)$$

RESULTS AND DISCUSSIONS

The electro-optical properties of PDLCs are determined primarily by the characteristics of the liquid crystals dispersed within the cured polymer matrix. In practice, the size and position of the liquid

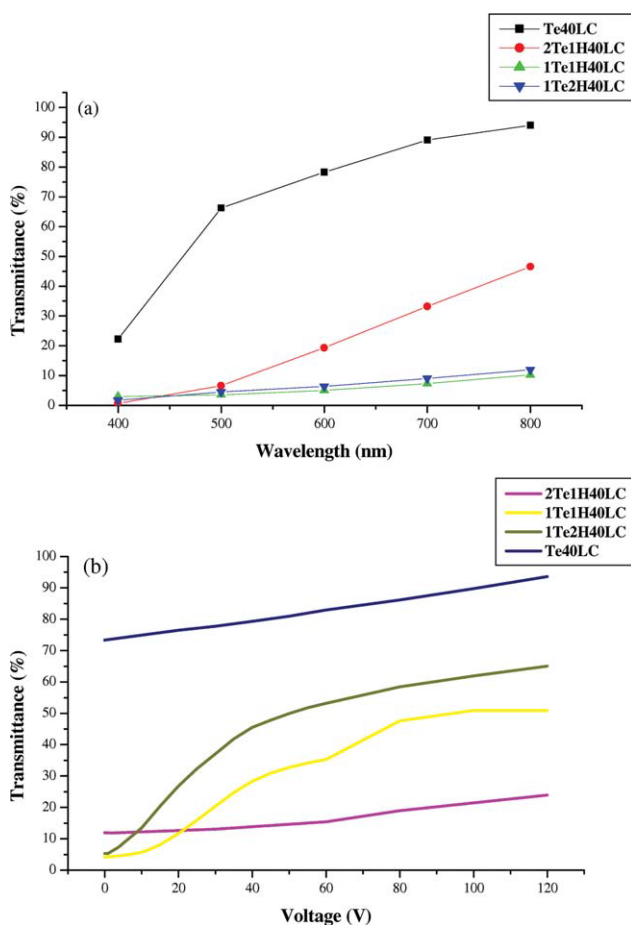


Figure 1 Transmittance properties of PDLC samples with various levels of HEMA addition (a) Variation of off-state transmittance with incident wavelength as function of HEMA mol % (b) Variation of on-state transmittance with voltage as function of HEMA mol %. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

crystals in the current samples depend on the relative contents of the E7 crystals, TeGDA crosslinking agent and HEMA monomer in the original prepolymer mixture. In the preliminary stages of this study, it was found that a liquid crystal addition of approximately 40 wt % yielded the optimal electro-optical properties, i.e. a high contrast ratio and a low driving voltage. Accordingly, all the samples used in the main experimental stage of this study were prepared using a 40 wt % E7 liquid crystal content.

Figure 1(a) illustrates the variation of the off-state transmittance of the current samples with changes in the wavelength of the incident light. It can be seen that the transmittance of the Te40LC sample (with no HEMA content) has a very high transmittance at all values of the incident wavelength. However, the addition of 33.77 mol % HEMA results in a significant reduction in the transmittance. Furthermore, it is observed that the transmittance continues to reduce as the molar ratio of HEMA to TeGDA is

increased. In other words, the results show that for a constant liquid crystal content (40 wt %), the molar ratio of the single functional monomer (HEMA) to the double functional crosslinking agent (TeGDA) has a significant effect upon the light-scattering properties of the liquid crystals and therefore provides a convenient means of tuning the transmittance of the PDLC film in accordance with the desired application.

Figure 1(b) illustrates the variation of the on-state transmittance of the current PDLC samples as a function of the magnitude of the applied electric field. Note that the results are obtained using a constant illumination wavelength of 550 nm. The electro-optical properties of the four samples are summarized in Table I. As shown, the transmittance of the sample with no HEMA monomer (i.e., Te40LC) increases from 73.37 to 93.59% as the voltage is increased from 0 to 120 V. Thus, the contrast ratio is just 1.28 : 1 (i.e., 93.59/73.37). Furthermore, V_{sat} and V_{th} are found to be 110 and 13 V, respectively. When 33.77 mol % of HEMA monomer is added to the prepolymer mixture (i.e., sample 2Te1H40LC), the transmittance is found to increase from 11.94 to 23.91% as the applied voltage is increased from 0 to 120 V, corresponding to a contrast ratio of 2.01 : 1. When the prepolymer mixture is prepared using a 1 : 1 mole ratio of TeGDA and HEMA, respectively, (i.e., sample 1Te1H40LC), the contrast ratio is improved significantly to a value of 12.75 : 1. Finally, when the molar ratio is increased to 1 : 2 (i.e., sample 1Te2H40LC), the transmittance increases from 5.03 to 65.3%, corresponding to a contrast ratio of 13 : 1. Furthermore, compared to the 1Te1H40LC sample, it is observed that the saturation voltage of the 1Te2H40LC sample increases slightly (i.e., from 78 to 85 V), but the threshold voltage reduces significantly (i.e., from 16 to 10 V). The addition of 66.49 mol % HEMA increases the contrast ratio from a value of 1.28 : 1 in the sample prepared using TeGDA and E7 liquid crystals only to a value of 13 : 1, whilst simultaneously reducing the threshold voltage from 13 to 10 V, the saturation voltage reducing from 110 to 85 V. Overall, the results presented in Table I reveal that the 1Te2H40LC sample possesses the best electro-optical properties of the current samples, namely the highest contrast ratio and the lowest driving voltage.

Table I shows that the refractive indices of the current PDLC samples vary in the range 1.5054–1.5098, corresponding to a variation of just 0.044. Thus, even though the relative contents of the TeGDA and HEMA monomer in the PDLC prepolymer mixture have a significant effect upon the electro-optical properties of the PDLC film, they have no more than a marginal effect on the refractive properties of the sample.

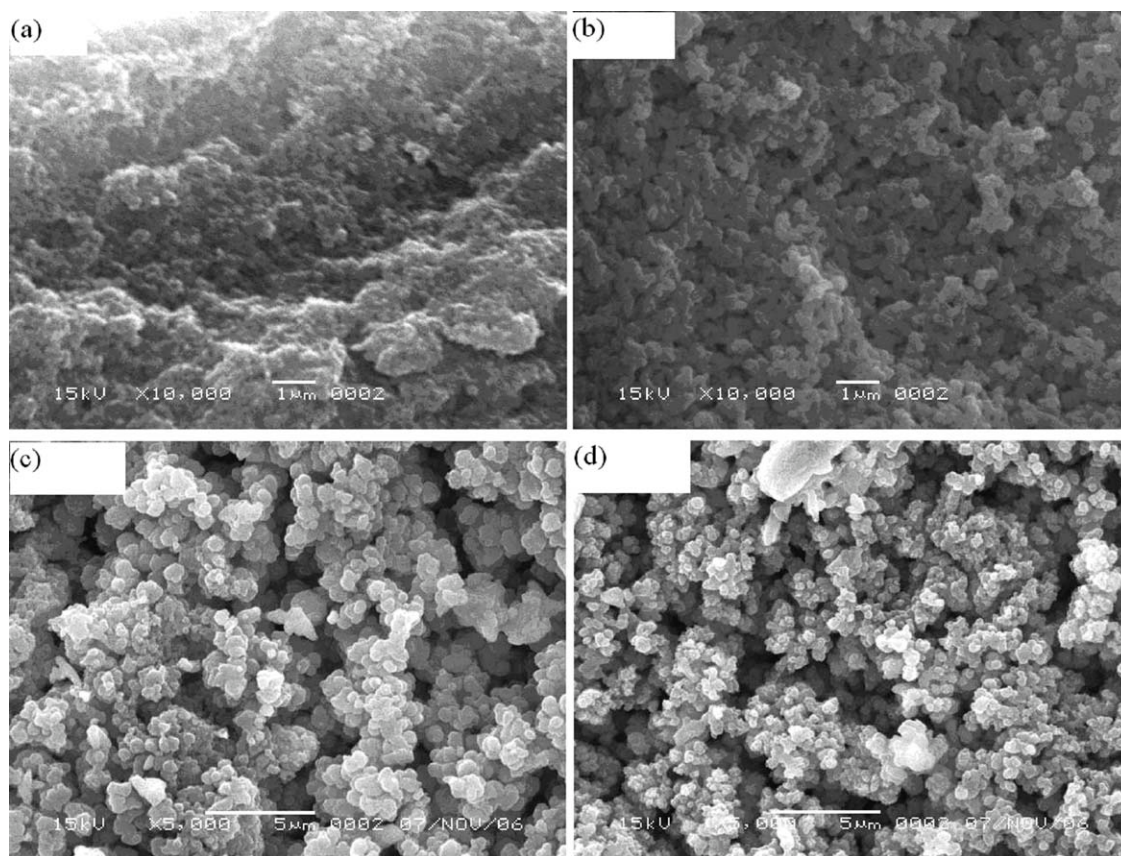


Figure 2 SEM micrographs of PDLC samples (a) Te40LC, (b) 2Te1H40LC, (c) 1Te1H40LC, and (d) 1Te2H40LC.

An understanding of the phase separation process is important because it determines the morphology of the films.¹⁷ Figure 2 presents SEM images of the microstructures of the four PDLC samples. Figure 2(a) shows that the microstructure of the Te40LC sample with no HEMA content has a coral reef-like appearance with many micro holes in the matrix and very small agglomerations of liquid crystals. During the exposure stage of the PIPS procedure, the liquid crystal agglomeration process competes with that of polymer matrix polymerization,¹⁸ and the liquid crystals form into separate agglomeration domains while the monomers link together to form a polymer reticular structure. In the Te40LC sample, a rapid polymerization of the crosslinking monomers occurs due to the absence of HEMA, and thus the liquid crystals in the prepolymer mixture are

rapidly surrounded by polymer matrix, which suppresses their agglomeration during the phase separation process. As a result, the Te40LC PDLC sample has a poor light-scattering property in the V_{off} condition. By contrast, the addition of the single functional HEMA monomer to PDLCs reduces the crosslinking density of the polymer matrix due to its strong terminal group and therefore produces a better phase separation effect, which in turn improves the electro-optical properties of the sample.

When 33.77 mol % of HEMA is added to the PDLC mixture, the microstructure retains a coral reef appearance, but the size of the liquid crystal agglomerations increases [Fig. 2(b)]. Figure 2(c) shows that when the level of HEMA addition is increased to 50 mol %, the reticular microstructure observed in the Te40LC and 2Te1H40LC samples is

TABLE II
Domain Mean Diameter Distributions for PDLCs

Samples	N/Nt (%)										
	0.05–0.1 (μm)	0.1–0.2 (μm)	0.2–0.3 (μm)	0.3–0.4 (μm)	0.4–0.5 (μm)	0.5–0.6 (μm)	0.6–0.7 (μm)	0.7–0.8 (μm)	0.8–0.9 (μm)	0.9–1.0 (μm)	>1.0 (μm)
2Te1H40LC	40.28	39.93	11.46	5.56	1.04	1.04	0.69	0.00	0.00	0.00	0.00
1Te1H40LC	0.00	40.19	19.63	10.28	9.35	5.61	1.87	0.93	0.93	0.00	11.21
1Te2H40LC	0.00	39.90	19.21	11.33	6.40	4.43	4.43	3.94	1.48	0.49	8.37

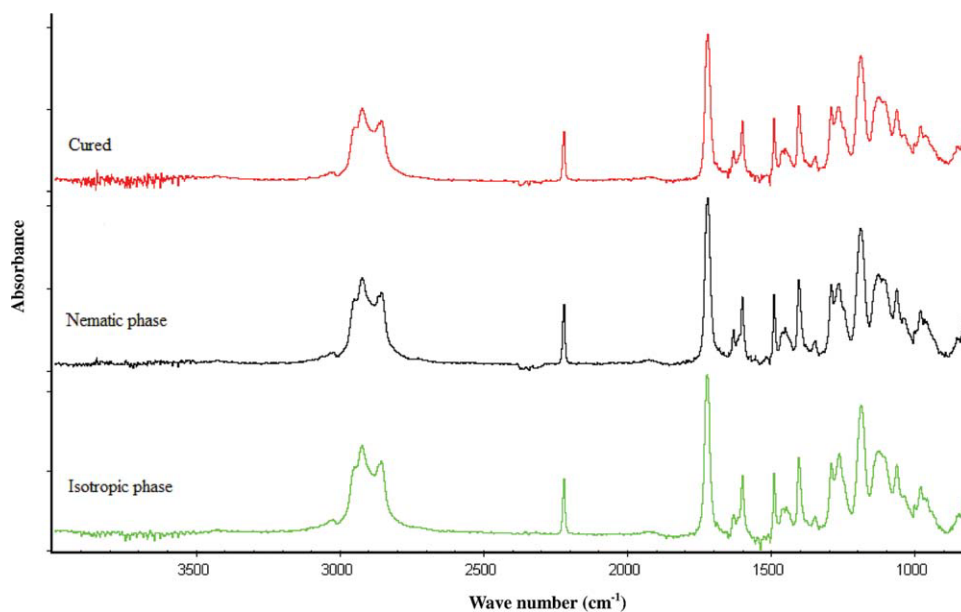


Figure 3 FTIR spectra of 2Te1H40LC PDLC sample. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

replaced by a structure comprising a large number of small spherical formations). Finally, when 66.49 mol % of HEMA is added to the prepolymer mixture, the microstructure retains the same spherical features as that in the 1Te1H40LC sample, but has a larger liquid crystal domain. Table II summarizes the distribution of the diameters of the holes in the microstructures of the three samples containing HEMA monomer. From inspection, it is found that in the sample with 33.77 mol % HEMA (i.e. 2Te1H40LC), 97.23% of the holes have an average diameter of less than 0.4 μm . However, with HEMA additions of 50 mol % and 66.49 mol %, respectively, the percentage of the holes with an average diameter of less than 0.4 μm reduces to around 70%. As a result, TeGDA/HEMA molar ratios of 1 : 1 and 1 : 2 yield an improved light scattering effect and a higher contrast ratio. As shown in Table I, the smaller liquid crystal domain in the 2Te1H40LC sample results in a significantly higher saturation voltage ($V_{\text{sat}} = 100$ V) than that in either the 1Te1H40LC sample or the 1Te2H40LC sample, i.e. $V_{\text{sat}} = 75$ V and 85 V, respectively. Note that the LC was removed before microscopy, so the dark areas in the micrographs, revealing the absence of mate-

rial, are representative of the original LC domains. The degree of PIPS and the growth of LC domains by diffusion and coalescence are strongly hindered by the higher rate of polymerization, leading to the decrease in droplet size of LC domains dispersed in matrix. The small LC droplets contribute to high threshold voltage.

Figure 3 presents an FTIR analysis of the absorbance characteristics of the 2Te1H40LC sample in three conditions, namely the cured condition, the nematic phase condition (25°C) and the isotropic phase condition (65°C). The phase separation parameters of PDLCs using FTIR through baseline and properties of liquid crystals to absorbance of the nitrile stretching ($-\text{C}\equiv\text{N}$ $\nu = 2227$ cm^{-1}), to show the changes when exposure of both Nematic phase at 25°C and Isotropic phase at 65°C. Applying the liquid crystal fraction formulation given in eq. (3) to the FTIR results obtained for each of the four PDLC samples as is shown in Table III, it is found that the extent of the phase separation during the polymerization process increases with an increasing HEMA monomer ratio. Specifically, in the Te40LC sample with no HEMA monomer, the phase separation fraction is found to be just 0.2187, while in the sample with

TABLE III
The Phase Separation Parameter of PDLCs

Sample	A_N	A_I	$A_{\text{CN-cured}}$	Phase separation parameter (α)
Te40LC	0.371	0.5425	0.202	0.2187
2Te1H40LC	0.06	0.18	0.06	0.2500
1Te1H40LC	0.858	1.265	0.439	0.4115
1Te2H40LC	0.079	0.3475	0.092	0.4376

33.77 mol % HEMA addition, the phase separation fraction increases slightly to a value of 0.2500. However, HEMA additions of 50 and 66.49 mol % increase the phase separation fraction significantly to 0.4115 and 0.4376, respectively. Therefore, the phase separation degree between liquid crystals and polymer matrix gets higher. Thus, it is apparent that higher levels of HEMA addition improve the degree of phase separation between the liquid crystals and the polymer matrix, respectively, and therefore enlarge the size of the liquid crystal domain (see the SEM images in Fig. 2) and improve the electro-optical properties of the sample (Table I). The higher of the phase separation parameter is, the better electro-optic properties become.

CONCLUSIONS

This study has examined the effect of HEMA addition on the microstructure and electro-optical properties of PDLC composite films fabricated using E7 liquid crystals and TeGDA crosslinking agent. Overall, the results have shown that whilst the level of HEMA addition has very little effect upon the refractive index of the PDLC films, it has a notable impact on the microstructure and electro-optical properties of the various samples. Specifically, the addition of single functional HEMA monomer (with strong polar terminal group) improves the contrast ratio of the PDLC film and reduces the driving voltage. For a HEMA addition of 33.77 mol %, the improvement in the PDLC properties relative to those of a sample fabricated using TeGDA and E7 liquid crystals only is very small as only a limited phase separation effect occurs and hence the size of the liquid crystal domain is relatively small. However, the addition of 50–67 mol % HEMA monomer yields an effective improvement in the phase separation effect and increases the size of the liquid crystal domain. As a result, a significant improvement in the electro-optical properties of the PDLC film is

obtained. For example, the addition of 66.49 mol % HEMA increases the contrast ratio from a value of 1.28 : 1 in the sample prepared using TeGDA and E7 liquid crystals only to a value of 13 : 1, whilst simultaneously reducing the driving voltage from 13 V to 10 V. Overall, the results therefore show that a PDLC comprising 40 wt % E7 liquid crystals, 33.51 mol % TeGDA and 66.49 mol % HEMA is an ideal candidate for commercial intelligent photoelectric applications.

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