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Effect of Curing Temperature on the Properties of Electrically Controlled Dimming Film with Wide Working Temperature Range

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Abstract: In this paper, a polymer dispersed liquid crystal (PDLC) film with good electro-optical properties and wide working temperature range was prepared by the UV-polymerization induced phase separation (PIPS) method by optimizing the curing temperature using a LC with wide temperature range. The investigation found that when the polymerization temperature was at the clearing point of the prepolymer/LC mixture, the film had better electro-optical properties and a high contrast ratio of 51 at 90 °C, and the rise time and decay time were respectively as fast as 241.5 ms and 1750 ms at −20 °C. This study provides further methodological guidance for the curing process of PDLC film, and promotes its application in outdoor smart windows.

Keywords: electro-optical properties; curing temperature; PDLC; working temperature

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1. Introduction

Polymer dispersed liquid crystal (PDLC) film is a kind of functional LC composite material, which combines the excellent mechanical and thermal capabilities of polymer with the external field stimuli-responsive properties of LC [1–4]. Moreover, the adhibition of PDLC film involves various fields, such as smart window, projection, LC grating, holographic film, thermal devices, nonlinear optics, and flexible display due to it having high sensitivity, high resolution, and relatively fast response time, and it can be cheaply made into films of various shapes and thicknesses [5–7].

At present, polymerization-induced phase separation (PIPS) is a common method to prepare PDLC films, as an appropriate polymerization method selected by the reaction characteristics of the prepolymer, including free radical polymerization, polycondensation reaction, photothermal-initiated polymerization, etc. Then, the LC is embedded in the polymer as submicron or micron-scale droplets to realize the preparation of PDLC film. It is a truism that the properties of PDLC films depend on the content of LCs, the size and shape of LC domains (polymer microscopic morphology) and the properties such as elasticity, viscosity, and dielectric constant of raw materials [8,9]. When the raw material and ratio are determined, the micro morphology will be the important influence on the electro-optical (E-O) properties of the PDLC film, which are largely determined by the rate of polymerization and the viscosity of the polymer monomer/LC mixture. Therefore, researchers have undertaken a host of investigations on the process conditions of PDLC film preparation, such as polymerization temperature, ultraviolet (UV)-light intensity, and polymerization time.

F Z Elouali found that the enhancement of curing light intensity accelerated the phase separation of PDLC film and sharply reduced the diameter of LC domain, and the formation of PDLC film microstructure and size gradient could be controlled by effectively regulating the curing light intensity [10]. Li systematically studied the influence of sample proportion and polymerization conditions on the micromorphology of PDLC films, and the conclusion showed that the size of LC domain in PDLC films accreted with the fall in the proportion of prepolymer and the weakening of UV radiation intensity. However, with the heating of polymerization temperature, the size of LC domain first increased and then decreased [11]. In the thermoset PDLC film system, H. Hakemi found that the polymerization rate attenuated at the appropriate curing temperature (close to the coexistence curve), resulting in suitable morphology and better electro-optical properties [12].

It is worth noting that there are few studies on the influence of curing temperature on the properties of PDLC films prepared by UV-initiated polymerization. However, as a key factor affecting the properties of PDLC films, it is necessary to explore the influencing mechanism of curing temperature. Additionally, for the practical application of PDLC dimming film, its working temperature is also an aspect worthy of attention. Even though the PDLC film has excellent electro-optical properties at room temperature, the birefringence (Δn) of the LC drops sharply at high temperatures, especially close to the clearing point of the LC [13,14], which will lead to the improvement of the off-state transmittance of the PDLC film [15]. Moreover, the variation of the LC viscosity at colder temperatures can significantly worsen the driving voltage and response speed of PDLC films [16–18]. These situations seriously damage the practical application of PDLC film, so the study of the working temperature of PDLC film must be concerned with optimizing the electro-optical properties of the PDLC film.

Therefore, one of the primary purposes of this work is to explore the influence mechanism of curing temperature on the properties of PDLC films prepared by UV initiating polymerization, which provides experimental support for the optimization of PDLC film curing process. Secondly, the photoelectric properties of PDLC films at different temperatures are investigated to determine the possibility of its outdoor application.

2. Experimental

2.1. Materials

The nematic LC HTW111740-200 (Δn = 0.251, n_e = 1.757, $T_{Cr\text{-}N} < -40$ °C, $T_{N\text{-}I}$ = 125 °C, Jiangsu Hecheng Display Technology Co., Ltd. Nanjing, China). The monomers, isobornyl acrylate (IBOA) and acrylic acid hexyl ester (HA), and the crosslinking agent 1,4-butanediol diacrylate (BDDA) were acquired from Beijing InnoChem Science & Technology Co., Ltd., Beijing China. The UV photoinitiator Irgacure 651 was provided by Shanghai Macklin Biochemical Co., Ltd., Shanghai China. The chemicals used in this paper were used directly without further purification, and their structural formulas are depicted in Figure 1.

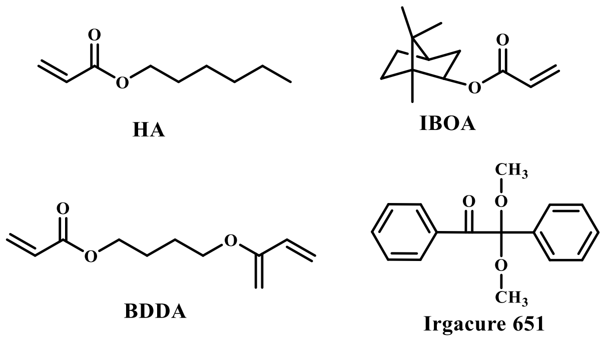

Figure 1. The chemical structural formula of the material used.

2.2. Sample Preparation

According to the formula in Table 1, the acrylates and LC were mixed in a centrifuge tube, and the Irgacure 651 was added to the total mixture at 2 wt.%. The mixture was stirred evenly with a vortex mixer (Kylin-Bell VORTEX-5, Kylin-bell Lab Instruments Co., Ltd., Haimen, China) and was transfused into a cell with a spacing of 20 µm by capillary action. Eventually the filled cells were placed on a cold and hot stage (Linkam LTS 420, Linkam Scientific Instruments Ltd., Tadworth, Surrey, UK) and the PDLC films were prepared by UV irradiation (365 nm, 14 mW·cm^{-2}) for 5 min at a certain temperature.

Table 1. The formulas and polymerization conditions of all samples.

	Formula		**Polymerization Conditions**	
Sample	**Composition**	**Weight Ratio (wt. %)**	**Curing Temperature (°C)**	**Curing Light Intensity (mW/cm^2)**
A	HA/IBOA/BDDA/LC	18/6/6/70	50	14
B	HA/IBOA/BDDA/LC	18/6/6/70	40	14
C	HA/IBOA/BDDA/LC	18/6/6/70	30	14
D	HA/IBOA/BDDA/LC	18/6/6/70	20	14
E	HA/IBOA/BDDA/LC	18/6/6/70	10	14
F	HA/IBOA/HDDA/LC	18/6/6/70	0	14
a	HA/IBOA/BDDA/LC	18/6/6/70	12	14
b	HA/IBOA/BDDA/LC	18/6/6/70	14	14
c	HA/IBOA/BDDA/LC	18/6/6/70	16	14
d	HA/IBOA/BDDA/LC	18/6/6/70	18	14
e	HA/IBOA/BDDA/LC	18/6/6/70	20	14
f	HA/IBOA/HDDA/LC	18/6/6/70	22	14
g	HA/IBOA/HDDA/LC	18/6/6/70	24	14

2.3. Characterizations

To examine the surface morphologies of the PDLC polymer, a Hitachi S-4800 scanning electron microscope (Hitachi Science System Ltd., Tokyo, Japan) was utilized. To completely eliminate the LC phase, the PDLC films were immersed in a 7:3 volume ratio of cyclohexane and acetone for 10 days.

A LC device parameter tester (LCT-5016C, Changchun Liancheng Instrument Co., Ltd., Changchun, China) was used to determine the E-O performances of PDLC films. The incident light was emitted by a HeNe laser with a wavelength of 560.0 nm. The external square wave electric field's frequency and maximum operating voltage evaluated in this article are 1000 Hz and 100 V, respectively, and to standardize the sample transmittance, the air transmittance is set to 100%.

A temperature controller was added to the sample rack of the LC device parameter test instrument to evaluate the E-O performances of PDLC films at different working temperatures. Between −20 °C and 90 °C, the films were tested for electro-optical performance every 10 °C.

A polarizing microscope (ZEISS Axio Scope.A1, Carl Zeiss AG, Oberkochen, Baden-Württemberg, Germany) was used to characterize the phase separation degree of the mixture of LC/prepolymer at different temperatures. Herein, the cold and hot stage (Linkam LTS 420, Linkam Scientific Instruments Ltd., Tadworth, Surrey, UK) was used in conjunction with polarizing microscope to control the temperature of the test sample.

The phase transition point of the mixture of LC/prepolymer was characterized by a differential scanning calorimeter (Perkin-Elmer DSC 8000, Perkin Elmer, Inc. Waltham, MA, USA) in a nitrogen atmosphere, and the scanning speed was 10.0 °C/min.

3. Results and Discussion

3.1. Analysis of Phase Transition Point of the Mixtures of LC/Prepolymer

The phase transition temperature of the mixture of LC/prepolymer was investigated by polarizing microscopy and DSC. First of all, the mixture was poured into the LC cell without alignment layers and taken a polarized photo after holding at a certain temperature for 5 min, as shown in Figure 2. The mixture exhibits a LC state at 10 °C and below, and a black field above 20 °C, indicating that it has reached the isotropy. The accurate clearing-point of the mixture was characterized by DSC. In Figure 3, there is an endothermic peak at 17.5 °C when the temperature is raised, which is the clearing-point of the mixture of LC/prepolymer.

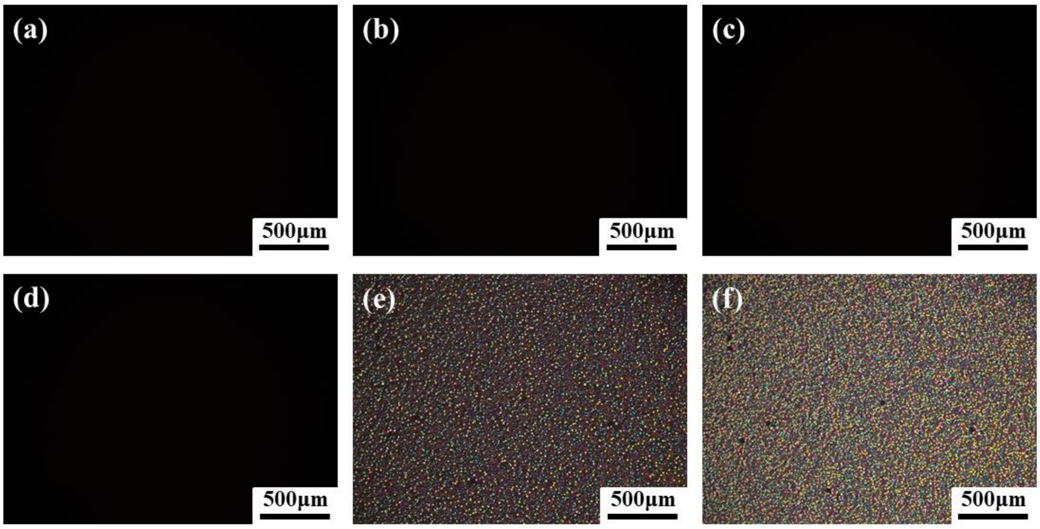

Figure 2. Temperature-dependent polarized images of LC/acrylate mixtures: (**a**) (50 °C), (**b**) (40 °C), (**c**) 30 °C), (**d**) (20 °C), (**e**) (10 °C), (**f**) (0 °C).

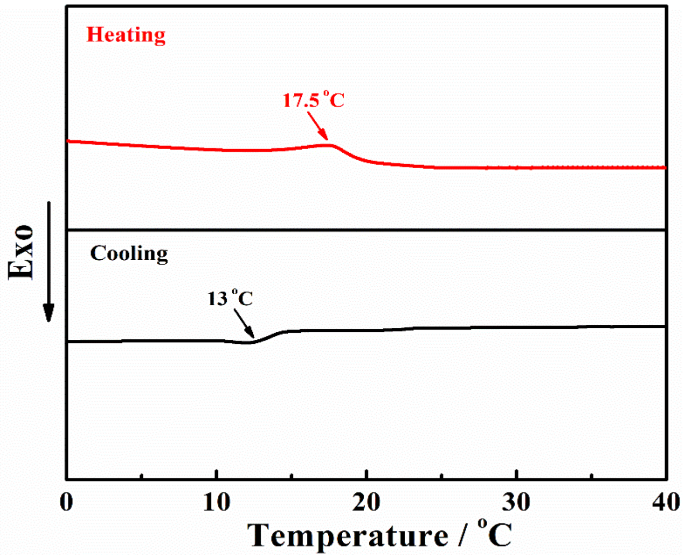

Figure 3. DSC spectra of liquid crystal/acrylate mixture system.

3.2. Micromorphology of PDLC Films at Various Curing Temperatures

The polymer matrix morphologies of the PDLC films prepared at various curing temperatures are depicted in Figure 4. The size of the polymer mesh diminishes noticeably as the temperature rises when the curing temperature is above the clearing point of the LC/acrylate mixtures. When PDLC film is polymerized at 50 °C, it is evident that the polymer cavities have very small dimensions and are quite dense. In addition, when the curing temperature is lower than the clearing point of the LC/acrylate mixtures, the morphologies with coexistence of sparse large mesh size and dense small mesh are formed (Figure 4e,f) due to the precipitation of some LC droplets. The large aggregation of LC droplets in the LC/prepolymer system can also be demonstrated in Figure 2e,f showing the polarized images.

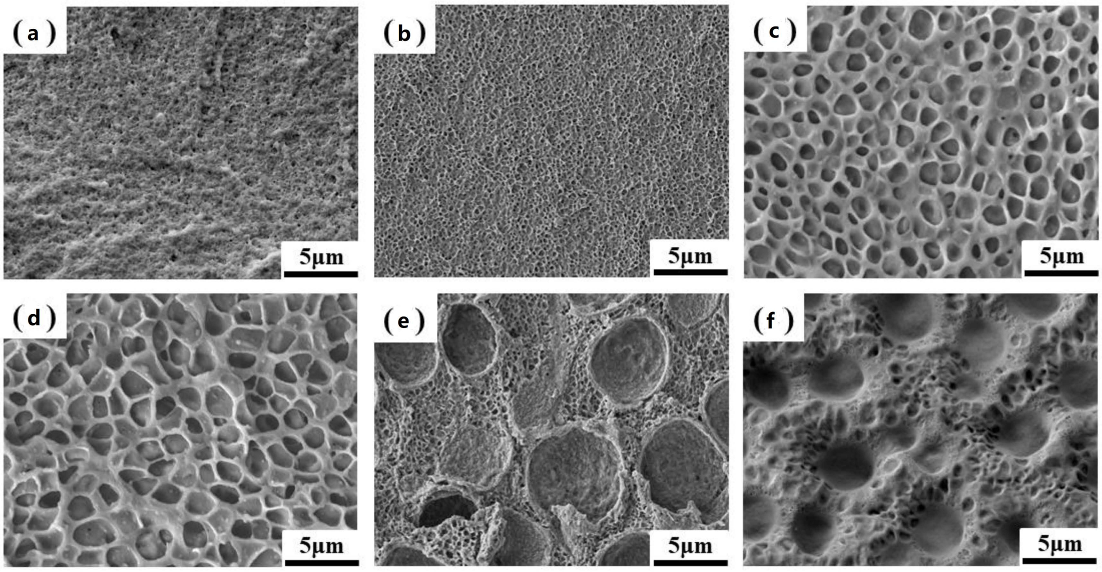

Figure 4. The microscopic morphology of the PDLC polymer matrix at various curing temperatures: (**a**) (50 °C), (**b**) (40 °C), (**c**) (30 °C), (**d**) (20 °C), (**e**) (10 °C), (**f**) (0 °C).

The above variation trend of polymer mesh with curing temperature can be explained by the competitive mechanism of LC molecular diffusion and monomer polymerization. The diffusion coefficient of LC molecules at various temperatures can be stated using Fick's second law of diffusion [19,20],

$$D = D_0 \exp(Q/RT) \tag{1}$$

where D_0 is the diffusion coefficient constant, Q is the activation energy, R is the gas constant and T is the absolute temperature. It can be seen from Equation (1) that the diffusion coefficient of LC molecules has an exponential relationship with temperature. When the temperature is below the clear point of LC/acrylate mixtures or even lower, the viscosity of LC can be greatly reduced by raising the curing temperature, which will then encourage the formation of LC domains through coalescence and diffusion, increasing the size of the polymer meshes.

What's more, the polymerization rate of monomers also has an exponential relationship with temperature, as shown in Equation (2) [11,21].

$$\frac{1}{\tau} \propto e^{\frac{-E_{act}}{kT}} \tag{2}$$

where τ is the curing time constant, E_{act} is the activation energy of the reaction, k is the Boltzmann constant, and T is the absolute temperature. The effect of temperature on LC viscosity is weakened when the curing temperature is higher than the isotropic temperature of the LC/acrylate mixture. This means that the speed at which LC molecules diffuse is relatively slow compared to the polymerization rate of monomers at a higher curing temperature, which seriously hinders the phase separation of UV polymerization and the diffusion and aggregation of liquid crystal domain.

3.3. The E-O Properties of PDLC Films at Various Curing Temperatures

The PDLC films' E-O properties under different curing temperatures were characterized by using a LC comprehensive parameter instrument. The following definitions provide the PDLC film's E-O parameters. T_{off} and T_{on} stand for the PDLC film's off-state transmittance and maximum on-state transmittance, respectively, and the ratio of T_{on} to T_{off} is the film contrast ratio (*CR*). The voltages applied when the PDLC film transmittance reaches 10% and 90% of the T_{on} are known as the threshold voltage (V_{th}) and saturation voltage (V_{sat}). The time when the film transmittance rises from 10% to 90% of T_{on} is defined as the rise time (t_R), and after removing the electric field, the time when the film switches from 90% to 10% of T_{on} is the decay time (t_D).

Figure 5a shows the relationship curves between the transmittance of the A-E PDLC film and the applied voltage, it can be seen that sample D (cured at 20 °C) has excellent electro-optical properties. The V_{sat} of samples A-F showed a trend of decreasing and then increasing, as shown in Figure 5b. Generally speaking, the large size of the polymer mesh could reduce the specific surface area of the LC/polymer interface and weaken the anchoring effect, so the lower external electric field could orient the LC molecules [22–24]. However, the polymer cavities of samples E and F have heterogeneous sizes, among which the micro-sized polymer mesh accounts for the majority, which enhances the anchoring effect on the LC molecules, thus increasing the driving voltage. The polymer mesh size of the PDLC film is uniform and large when cured at 20 °C, so the sample D exhibits a lower V_{sat}.

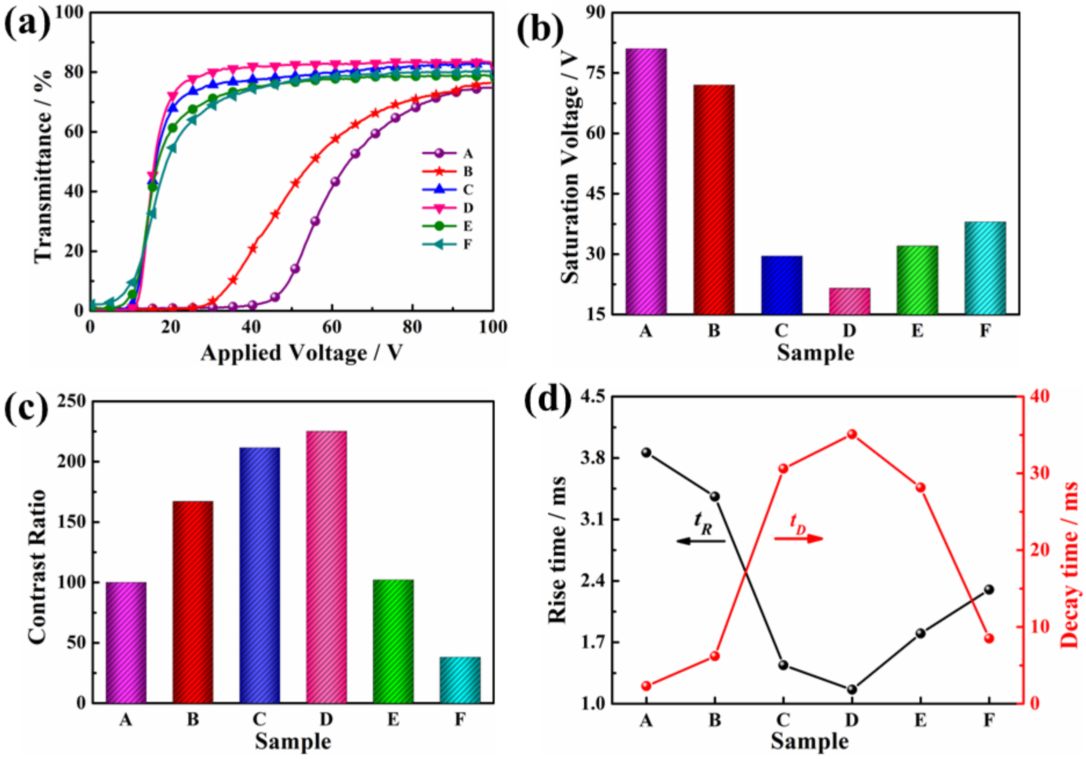

Figure 5. (**a**) The curve of A-F samples transmittance with voltage, (**b**) V_{sat}, (**c**) *CR*, (**d**) t_{R} and t_{D} of A-E sample.

In Figure 5c, the *CR* of the samples A–F shows a trend of increasing and then decreasing. It is well known that small size of the polymer meshes can greatly enhance the light scattering of PDLC films. For a certain system with constant LC content, the size of LC droplet with the best light scattering in the visible light region is generally around a few microns, that is, the LC droplets with sub-micron size (<1 μm) could not effectively scatter visible light [6,25]. Therefore, samples A and B have an average polymer mesh size of

0.6 μm and 0.8 μm, respectively, which exhibit a higher T_{off}. Additionally, the small LC domains will also cause the film to increase light scattering at the on-state, so that samples A and B have lower *CR*. The average sizes of the polymer mesh of samples C and D are 1.8 μm and 2.5 μm, which have better light scattering intensity at the off-state and show a large *CR* value. As we all know, the rising process is a process for the LC molecules to overcome the anchoring effect, so the PDLC film with small polymer mesh has a longer t_R. What's more, the smaller polymer meshes also cause higher distortions in the rising process of the liquid crystal molecules [24,26]. After the power is turned off, the time for the LC molecules return to the initial state becomes shorter, so the t_R and t_D of samples A-F show the variation tendencies of the curve in Figure 5d.

In order to further determine the optimal curing temperature, a batch of PDLC film samples were prepared every 2 °C within the range of 12–24 °C, and their electro-optical curves, V_{sat} and *CR* were analyzed, as depicted in Figure 6, when the curing temperature is 18 °C, the sample has better electro-optical performance. Therefore, with the UV-initiated polymerization of PDLC film, the optimal curing temperature is at the clearing point of the mixtures of LC/prepolymer.

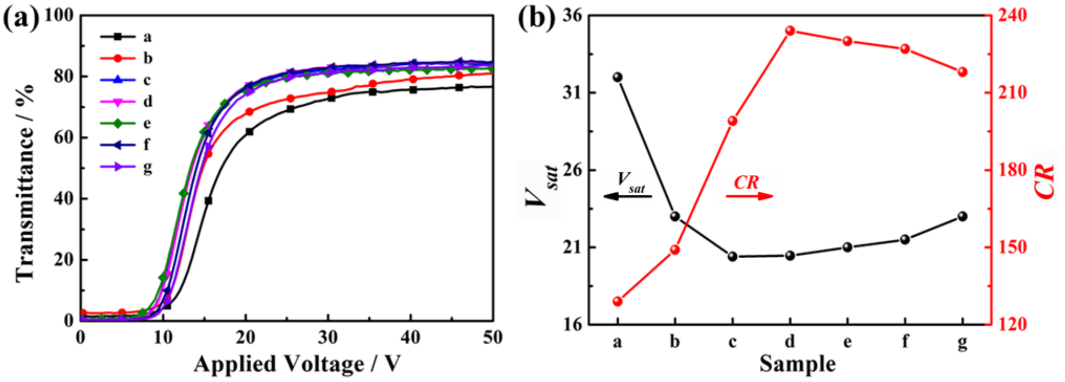

Figure 6. (**a**) The curve of samples a–g transmittance with voltage, (**b**) V_{sat} and *CR* of a–g sample.

3.4. Analysis of Working Temperature of PDLC Film

Since the above-mentioned exploration found that the PDLC film had better electro-optical properties when the curing temperature was 18 °C, this article had investigated the working temperature of sample d. In the range of −20–90 °C, the electro-optical properties of the film were measured every 10 °C. The temperature-dependent relationship between the transmittance of sample d and applied voltage is shown in Figure 7. The relationship between the transmittance of sample d and the applied voltage at different temperatures is shown in Figure 7. Obviously, the E-O curve of the sample moves to the right with the cooling of temperature, that is, the driving voltage of the sample becomes higher. Similarly, it can be seen intuitively that the V_{sat} of sample d drops sharply at a high temperature in Figure 8a. This is due to the rise in the temperature of the LC viscosity resulting in its reduction. Then, under the action of small external electric field, LC molecules can be driven to orientation [19].

It is well known that the birefringence of the LC becomes smaller with the heating of temperature. Then, the light scattering between the LC/polymer interface becomes frailer at the off-state, so the PDLC film exhibits decrease of *CR* values at a high temperature, as shown in Figure 8b, and the *CR* of sample still remains above 51 at 90 °C. In Figure 8c,d, the response time of the PDLC film between −20 °C and 90 °C ascends significantly with the temperature reducing, especially below 10 °C. However, the sample d still shows fast t_R and t_D at −20 °C, which respectively are 241.5 ms and 1750 ms. This variation phenomenon is caused by the reduction of LC viscosity and the enhancement of activity during the heating process.

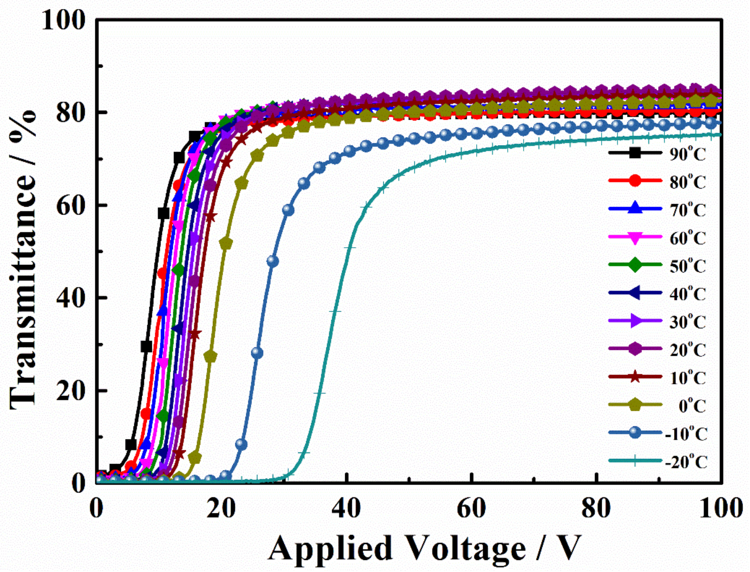

Figure 7. The temperature-dependent relationship between PDLC film transmittance and applied voltage.

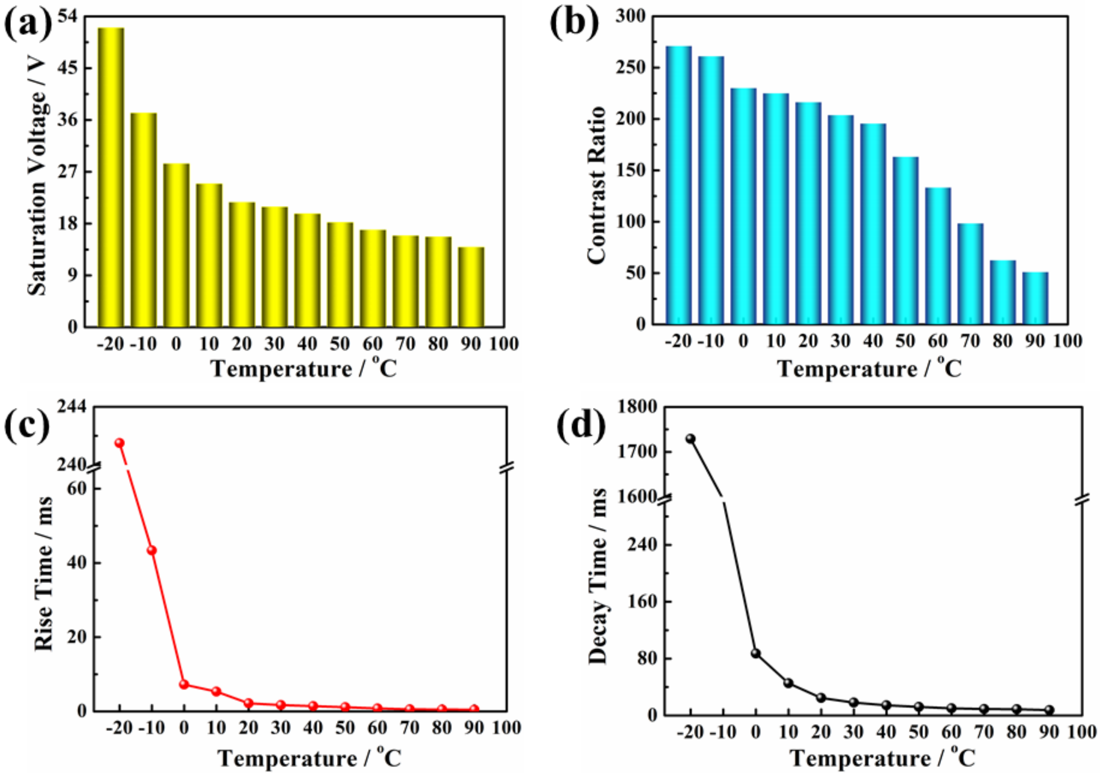

Figure 8. (**a**) The V_{sat}, (**b**) CR, (**c**) t_R and (**d**) t_D of PDLC film at various temperature.

4. Conclusions

In summary, this study mainly examined how the curing temperature affected the electro-optical characteristics of PDLC films created by UV-initiated polymerization, and secondarily explored the variation rules of E-O properties of PDLC films at different temperatures. When the polymerization temperature was at the clearing point of the LC/prepolymer mixture, the film had better electro-optical properties and a better *CR* value of 51 at of 90 °C, and the rise time and decay time were as fast as 241.5 ms and 1750 ms at −20 °C. Thus, it is demonstrated that the PDLC film has excellent performance in the temperature range of −20–90 °C. This work provides some experimental support for optimizing the curing process and outdoor application of PDLC films.

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