



Effect of temperature on the morphology and electro-optical properties of liquid crystal physical gel



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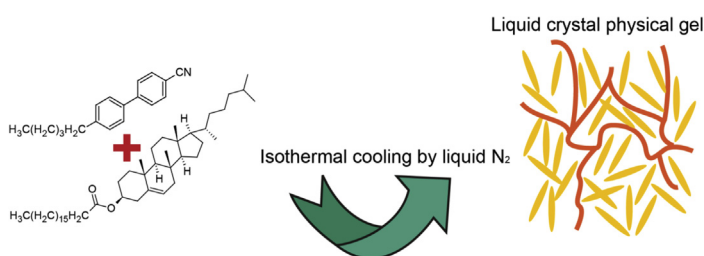
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HIGHLIGHTS

- 5CB nematic liquid crystal was successfully gelled by cholesteryl stearate gelator.
- The morphology of gel network was controlled by different cooling conditions.
- Thinner gel network was formed by the rapid cooling using liquid nitrogen.
- Enhanced electro-optical properties of liquid crystal physical gel were achieved.

GRAPHICAL ABSTRACT



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ABSTRACT

Liquid crystal physical gels were (thermally) prepared with cholesteryl stearate as a gelator in nematic liquid crystal, 4-cyano-4'-pentylbiphenyl. The electro-optical performance of liquid crystal physical gels is almost entirely dependent on the gels' inherent morphology. This study involved an empirical investigation of the relationships among all of the gelation temperature, morphology, and electro-optical properties. Besides continuous cooling at room temperature, isothermal cooling was also performed at both 18 and 0 °C, corresponding to near-solid and solid phases of 4-cyano-4'-pentylbiphenyl respectively. Nevertheless, the liquid crystal physical gel was also isothermally rapidly cooled using liquid nitrogen. Polarizing optical microscopy showed that the gel structure became thinner when isothermal cooling was carried out. These thinner gel aggregates then interconnected to form larger liquid crystal domains. Moreover, it was also revealed that the gel networks were randomized. Electron spin resonance results showed that the liquid crystal director orientation was severely randomized in the presence of gel networks. Conversely, isothermal cooling using liquid nitrogen generated a higher liquid crystal director orientation order. The 6.0 wt% cholesteryl stearate/4-cyano-4'-pentylbiphenyl physical gel that was isothermally cooled using liquid nitrogen showed the lowest response time in a twisted nematic mode optical cell.

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1. Introduction

Liquid crystal (LC) physical gel is a functional anisotropic composite in which the liquid crystal is embedded into the

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interconnected networks of self-assembled low molecular weight gelators. This is in contrast to polymer dispersed liquid crystal [1] and liquid crystal chemical gel [2], in which liquid crystal physical gel is thermally reversible, since the self-assembly process is solely driven by non-covalent interactions such as hydrogen bonding and π - π interaction. Furthermore, this material possesses properties of both liquid crystals and gelators, whilst the stimuli-responsive properties of liquid crystal may be enhanced [3]. Hence, it has received great attention for electro-optical applications such as large area displays, switchable windows and light shutters.

The electro-optical properties of liquid crystal physical gels are greatly dependent on the microphase separated structures. For example, Kato and co-workers first reported that nematic liquid crystal gel by oriented fibrous networks of L-isoleucine derivative exhibits a faster response, lower driving voltage and higher contrast ratio in twisted nematic (TN) mode display [4–6]. On the other hand, light scattering type optical materials are commonly prepared by dispersing randomly interconnected gel networks within liquid crystal. The light scattering states are induced by the mismatch of refractive indices between liquid crystal domains and gel networks. One example of this is the combination of 1,3:2,4-bis(3,4-dimethylbenzylidene)sorbitol (DMDBS) and 4-cyano-4'-pentylbiphenyl [7]. Overall, the orientation and morphology of gel networks are controlled by the concentration of the gelator, as well as the combination of the gelator and liquid crystal. For the latter, the order of the sol-gel transition temperature ($T_{\text{sol-gel}}$) and the isotropic-LC transition temperature ($T_{\text{iso-LC}}$) determines the phase-separated structure [8]. In the case $T_{\text{sol-gel}}$ is higher than $T_{\text{iso-LC}}$, randomly dispersed gel networks are formed. In contrast, in the case $T_{\text{iso-LC}}$ is higher than $T_{\text{sol-gel}}$, oriented gel networks are formed by the template effect of the liquid crystal host. Only a few attempts have yet been performed for controlling the phase-separated structure by tailoring the gelation temperature, especially for liquid crystalline physical gel. To the best of the researchers' knowledge, Hiroshi reported the isothermal cooling onto the self-assembly of *N*-bezyloxycarbonyl-L-isoleucylaminooctadecane in a 4-cyano-4'-pentylbiphenyl nematic host [9]. However, isothermal cooling was only performed near the $T_{\text{sol-gel}}$ of the gelator.

Among the purposes of this study is to control the growth and alignment of gel networks by tailoring the gelling temperature. The hypothesis is a lower gelling temperature and a denser molecular packing can suppress the growth of gel to higher-order structure. The isothermal cooling was therefore performed at the near-nematic-solid and solid-state of 4-cyano-4'-pentylbiphenyl. Nevertheless, rapid isothermal cooling was also conducted using liquid nitrogen.

2. Experimental

2.1. Liquid crystal physical gels preparation and characterization

Liquid crystal physical gels were prepared using 4-cyano-4'-pentylbiphenyl (5CB) and cholesteryl stearate (G1). First, suitable amount of G1 was mixed into 5CB followed by heating to 150 °C and kept for 1 h to achieve isotropic state. Solution was cooled to room temperature and kept for several hours to ensure complete gelation. Concentration of gelator was increased gradually until successive gel was obtained. For isothermal cooling, mixture was heated to 150 °C and kept for 1 h. After that, mixture was cooled to fixed temperature (18 and 0 °C) using ice bath for 1 h. Same procedure was followed for liquid nitrogen isothermal cooling, but isotropic sample was dipped into liquid nitrogen for 10 min.

Differential scanning calorimetric (DSC) studies were performed to determine the phase transition temperature of liquid crystal physical gels. Measurements were carried out with PerkinElmer

DSC 8500. Sample was heated to 120 °C and maintained for 3 min followed by cooling to room temperature at a rate of 10 °C min⁻¹.

The orientations and distributions of liquid crystal gel networks were observed using metallurgical optical microscope (AmScope-ME520TA) equipped with polarizers.

Electron spin resonance (ESR) spectra were recorded using JEOL JES-FA 100 ESR spectrometer to investigate the effects of gelator network to the director orientation of liquid crystals [10–12]. Spin probing technique was applied using 3 β -DOXYL-5 α -cholestane free radical due to the diamagnetic properties of liquid crystal physical gels. For sample preparation, spin probe in dichloromethane (10⁻⁴ M) was placed into sample tube followed by the removal of solvent. Mixture of liquid crystal and gelator was placed into the same sample tube. Finally, mixture was heated using hot air gun followed by appropriate cooling.

2.2. Electro-optical measurement

The electro-optical properties were measured in twisted nematic optical cell with cell gap of 6.0 μm . Samples were heated to isotropic phase and injected into cells by capillary action. The samples were cooled down in a few different cooling conditions. Transmission as a function of applied voltage was measured using an experimental setup as shown in Fig. 1 [13]. Optical cell was placed between He-Ne laser and photodiode (Thorlabs, PDA36A-EC) that connected to oscilloscope (Tektronix, TDS 3052C). He-Ne laser (632.8 nm) was used as an incident light source. Frequency of AC electric field was set to 300 Hz. The light intensity of optical cell filled with toluene was set as the maximum light intensity. The threshold voltage (V_{10}) and saturation voltage (V_{90}) were defined as the applied voltage to obtain 10% and 90% of the maximum transmittance of each sample, respectively. The contrast ratio (CR) was calculated as the ratio of maximum transmittance at 0 V to minimum transmittance at 10 V. The rise and decay times were measured as time to reach 90% and drop back to 10% of the maximum transmittance, respectively. All measurements were performed at room temperature.

3. Results and discussion

3.1. Liquid crystal physical gels preparation and characterization

The gel formation was confirmed by the tube inversion method [14]. A physical gel-looked compound was formed after the isotropic mixture of 6.0 wt% G1/5CB preserved for several hours at room temperature. The compound was highly viscous and did not flow upon being inverted upside down as shown in Fig. 2.

The phase transition temperatures of 6.0 wt% G1/5CB were confirmed by DSC thermogram, as depicted in Fig. 3(a). The aim was to find out the sequence between the isotropic-nematic phase transition of 5CB and the sol-gel phase transition of G1. The phase-separated structure of liquid crystal physical gel could be in the

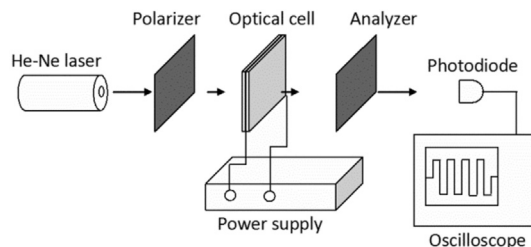


Fig. 1. Setup for electro-optical measurement.

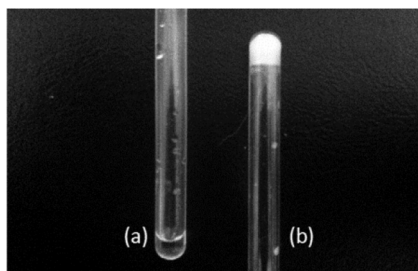


Fig. 2. Photograph of 6.0 wt% cholesteryl stearate/5CB at (a) isotropic and (b) gel state.

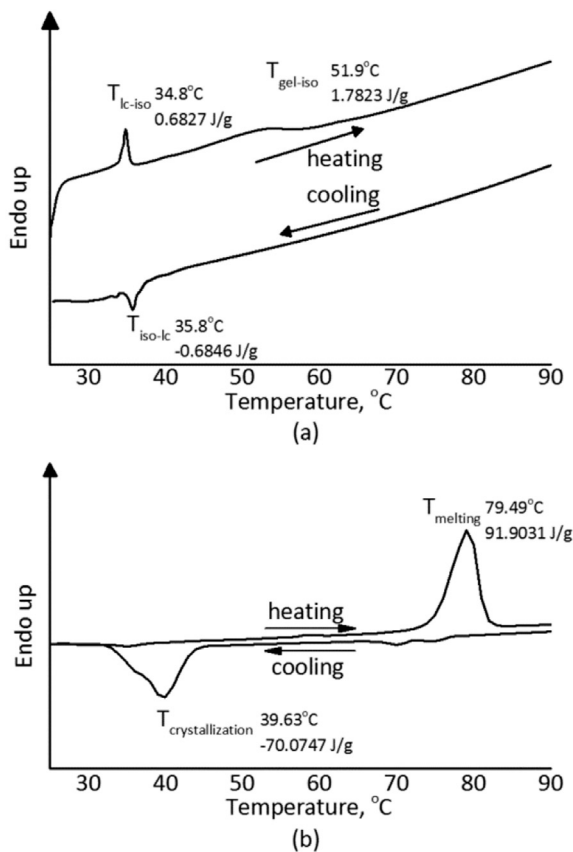


Fig. 3. DSC thermogram of (a) 6.0 wt% G1/5CB and (b) G1.

aligned manner if the isotropic-nematic transition temperature is higher than the sol-gel transition temperature, and vice versa [3].

With regards to the cooling cycle, the DSC thermogram of 6.0 wt% G1/5CB showed a sharp exothermic peak at 35.8°C . The enthalpy change was measured as -0.6846 Jg^{-1} . The corresponding endothermic peak was found at 34.8°C (0.6827 Jg^{-1}) in the heating cycle. This value is compatible with several previous studies of the isotropic-nematic transition temperature of 5CB [15–17]. Hence, it is reasonable to assign this peak as T_{iso-LC} of 5CB [16]. $T_{sol-gel}$ was not found in the cooling cycle of 6.0 wt% G1/5CB. Comparing with the DSC thermogram of G1 as depicted by Fig. 3(b), the crystallization transition temperature should appear at 39.63°C . The absence of the $T_{sol-gel}$ peak indicated that the gelation of 5CB was too slow to be detected in a scan rate of $10^\circ\text{C min}^{-1}$. Therefore, it can be deduced that the gelation occurred in the nematic phase of 5CB, hence the gel network was expected to form under the template effect of the liquid crystal [18].

Fig. 4 shows the polarizing optical microscope (POM) micrographs of 6.0 wt% G1/5CB prepared by various cooling conditions. In the case of continuous cooling, the gel aggregates were well distributed in the liquid crystal as shown in Fig. 4(a). In addition, gel aggregates extended towards a single direction, as indicated by the red arrow. It was clear that gel networks grew gradually along the director of liquid crystal on a micron scale. When observed under $100\times$ magnification, gel aggregates were in an elongated shape of about $6 \mu\text{m}$ width and $30 \mu\text{m}$ length. Liquid crystal domains consisted of a wide range of sizes in micron units. For isothermally cooling at 18°C , which is near to the nematic-solid transition temperature of 5CB, a homogeneous texture was obtained as depicted in Fig. 4(b). Uniaxial alignment of gel networks was not observed. Besides, the gel aggregates were significantly thinner. Interconnected gel aggregates encapsulated liquid crystal in smaller domains. The POM micrograph of 6.0 wt% G1/5CB prepared by isothermal cooling at 0°C (Fig. 4(c)) was quite similar to that of prepared by isothermal cooling at 18°C . The gel fibrils were significantly thinner compared to that of prepared by continuous cooling. Besides, the fibrils were mostly arbitrarily interconnected. This was explained by the closely packed-like-liquid crystal molecules in the solid state of 5CB that suppressed the growth of aggregates to a higher order structure. A more rapid isothermal cooling was performed using liquid nitrogen. In this case, the resulted gel aggregates were even smaller, forming larger liquid crystal domains compared to others as shown in Fig. 4(d). On average, the gel aggregates were about $2 \mu\text{m}$ in size. The optical micrographs in Fig. 4 conclusively show that the gelation temperature greatly affected the physical structure of the aggregates. For isothermal cooling, the growth of gel aggregates was efficiently suppressed. More importantly, the template effect of liquid crystals was neglected as well. Thus, random or oriented gel networks can be produced by tailoring the gelation condition.

Fig. 5 shows the ESR spectra of 5CB single component and 6.0 wt% G1/5CB physical gels prepared at various cooling conditions. For bulk 5CB, the ESR spectra consisted of three sharp hyperfine tensors with hyperfine spacing of 0.87 mT from centre as shown in Fig. 5(a). This value represents the aligned orientation of 5CB molecules. This is due to the parallel alignment of the positive anisotropic 5CB molecules to the direction of the magnetic field [19–21]. In contrary, ESR spectra of 6.0 wt% G1/5CB were partially averaged out

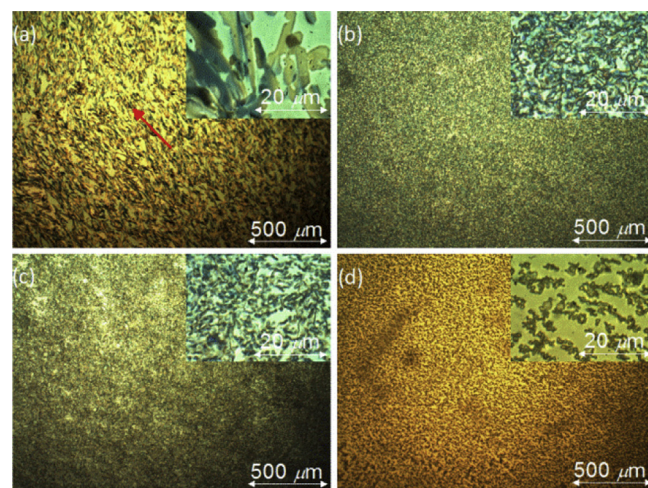


Fig. 4. POM micrographs of 6.0 wt% G1/5CB prepared by (a) continuous cooling at room temperature, (b) isothermal cooling at 18°C , (c) isothermal cooling at 0°C and (d) rapid cooling using liquid nitrogen. Insets are the respective micrographs of $100\times$ magnification.

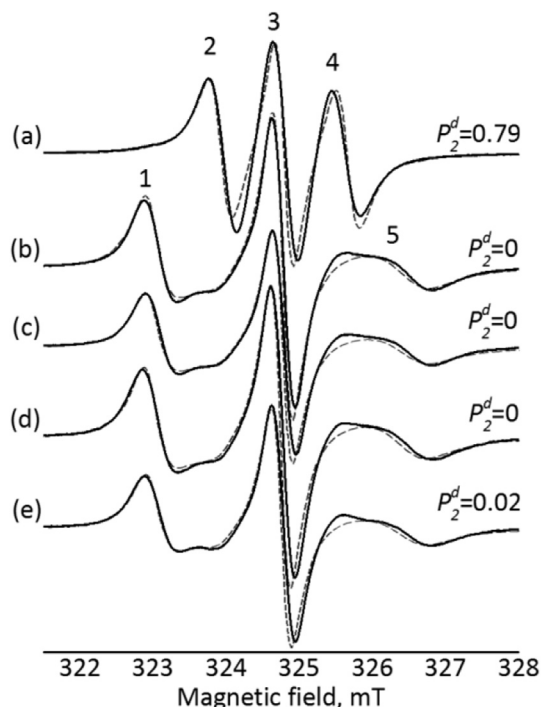


Fig. 5. Experimental (solid line) and simulated (dash line) ESR spectra of (a) 5CB, 6.0 wt% G1/5CB prepared with (b) continuous cooling, (c) isothermal cooling at 18 °C, (d) isothermal cooling at 0 °C and (e) rapid cooling using liquid nitrogen.

into a powder pattern (Fig. 5(b–e)). This is because of the longer diffusion time of the spin probe in different liquid crystal director orientations exceeding the time scale of ESR spectroscopy (10^{-8} s). This indicated that the interaction of liquid crystal molecules with the gel fibrils surface was larger than the aligning power of the static magnetic field in the ESR (0.3 T). The intensity of the hyperfine tensor 2 and 4 decreased substantially, indicating that liquid crystal director orientation of 5CB in aligned forms was destroyed. Simultaneously, peaks 1 and 5 appeared, although the intensity of peak 5 was too low to be distinguished. Hyperfine spacing was recorded to be 1.79 mT. All of these observations indicated that the existence of 6.0 wt% gel networks in 5CB massively randomized the director orientation of 5CB. Fig. 5(e) shows the ESR spectrum of 6.0 wt% G1/5CB prepared by rapid cooling using liquid nitrogen. The lineshape was slightly different, especially in the range of 323–324 mT. To be specific, the intensity of peak 2 was higher. This indicated that 6.0 wt% G1/5CB prepared by rapid cooling using liquid nitrogen consisted of a slightly higher portion of aligned liquid crystal director.

To gain more detailed insights about the liquid crystal director distribution of 6.0 wt% G1/5CB prepared at various conditions, ESR spectra were reproduced using a simulation derived from the torque balance equation developed by Luckhurst [22,23]. The liquid crystal director distribution is described in Equation (1):

$$f(\beta) = \lambda^2 / (\lambda^2 - (\lambda^2 - 1)\cos^2 \beta)^{3/2} \quad (1)$$

where λ is the single parameter controlling the liquid crystal director distribution. When λ is united, the director is randomly distributed in three dimensions. On the other hand, the director is aligned parallel to the magnetic field as λ increases. The second rank order parameter of the liquid crystal director can be obtained by λ in the form of (2).

$$\bar{P}_2^d = (\lambda^2/2) \left((2\lambda^2 + 1) / (\lambda^4 - \lambda^2) - 3 \arctan(\lambda^2 - 1)^{3/2} \right) \quad (2)$$

The 6.0 wt% G1/5CB prepared by isothermal cooling using liquid nitrogen had a higher second rank order parameter of 0.02, whilst others were perfectly randomized. This result is acceptable, since the POM micrograph showed that the gel structure prepared by isothermal cooling using liquid nitrogen had larger liquid crystal domains formed by thinner gel networks. It was expected that the liquid crystals at the interface near the gel aggregates' surface were immobilized. However, there was a small portion of liquid crystal molecules at the centre of the void, which was less affected by the surface anchoring force of the gel networks, hence, was free to be aligned by the magnetic field.

3.2. Electro-optical measurement

Fig. 6 shows the dependence of the light transmittance on the applied voltage for composites in the TN optical cell. The operating voltage range was between 0 and 100 V for 6.0 wt% G1/5CB prepared by continuous cooling. Such a higher operating voltage range was caused by the thick aggregates in the composite, which hindered the.

transmittance of the electric field throughout the system. A resultant higher power consumption is not practical for actual

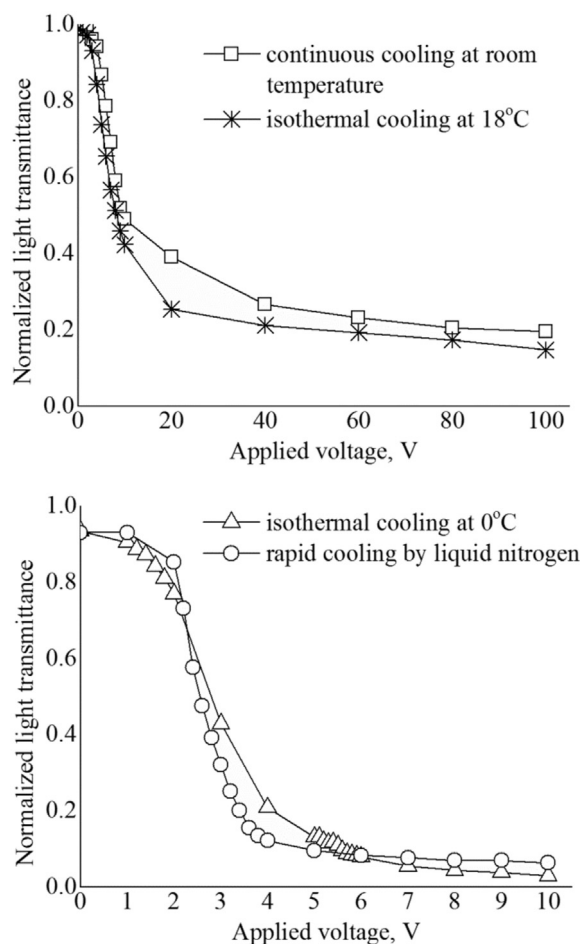


Fig. 6. Relationship between the light transmittance and the applied voltage of 6.0 wt% G1/5CB prepared at different cooling conditions.

Table 1
Electro-optical properties of 6.0 wt% G1/5CB prepared at different cooling conditions.

Liquid crystal physical gel	V_{10} (V)	V_{90} (V)	Contrast	T_{on}^a (ms)	T_{off}^a (ms)
Continuous cooling	6.0	47	5.1	—	—
Isothermal cooling at 18 °C	4.5	38	6.7	—	—
Isothermal cooling at 0 °C	1.6	5.2	30.9	23.26	64.14
Isothermal cooling using liquid nitrogen	2.2	3.8	14.5	10.66	18.37

^a Only for samples that achieved 90% light transmittance at 10 V.

operation. As can be seen, isothermal cooling gradually improved the electro-optical performance of 5CB. For isothermal cooling at 18 °C, light transmittance at a respective applied voltage was lower. For isothermal cooling at an even lower temperature, the operation voltage was shortened to 0–10 V.

Several electro-optical parameters are recorded in Table 1. The threshold and operating voltages were low when the isothermal cooling was performed. It is undeniable that more liquid crystal molecules should be anchored on the surface of the thinner fibrils, hence the threshold and saturation voltages were expected to increase. However, the thinner fibrils also improved the penetration of the electric field. As shown in the polarizing optical micrographs, the distribution of the gel networks was more homogeneous throughout the sample. Besides, since more liquid crystal molecules have been anchored on the surface of thinner fibrils, it was deduced that the thinner fibrils could have weakened the interaction between liquid crystal molecules and the glass substrate as well [5]. As a result, the liquid crystal molecules were more easily reorientated by the electric field. Several studies also showed that liquid crystal physical gels with large aggregates always show low electro-optical performances [24].

For the liquid crystal physical gel prepared by rapid cooling using liquid nitrogen, the threshold voltage increased adversely. This was explained by the existence of bigger liquid crystal domains as shown in Fig. 4(d). The domain size ranged from several tens to hundreds micrometer, which was significantly larger than that of the liquid crystal physical gel prepared at isothermal cooling. As calculated using MM2, the size of the energy-minimized 5CB molecule is around 2 nm which is close to the suggested references [25]. In this situation, it was expected that only the molecules in the vicinity close to the surface of fibrils were affected. Whilst, the molecules at the centre of void did not interact with the gel fibrils. This was double proved by our results for the ESR study. The second rank order parameter of liquid crystal physical gel prepared by rapid cooling using liquid nitrogen was slightly higher than others. This showed that a minor portion of liquid crystal molecules was less randomized. As a result, the threshold voltage was slightly higher.

However, thinner fibrils and larger liquid crystal domains actually improved the response time significantly. Compared to 6.0 wt% G1/5CB prepared by isothermal cooling at 0 °C, on-stage and off-stage response time have been shortened about 54% and 72%, respectively. The TN optical cells instantly returned to transparent state when the electric field was removed. This is because the liquid crystal molecules were able to reorient back to original position and the light was twisted through crossed polarizers.

As summary, the thinner gel fibrils improved the penetration of the electric field as well as reduced the interaction between the liquid crystal molecules and the glass substrate. However, the best electro-optical performance is only achieved by optimizing the thickness of fibril and the size of liquid crystal domains.

4. Conclusions

It was revealed that the morphology of the liquid crystal

physical gel was sensitively dependent on the gelation temperatures. Isothermal cooling at a lower temperature depressed the formation of aggregates of a higher order; this discovery is first reported in this work. Well-distributed thinner gel networks in liquid crystal were favourable for electro-optical performance in twisted nematic mode display. Thinner gel networks had embedded liquid crystals in larger domains. Liquid crystal physical gel possessed a higher second rank order parameter. As a result, a faster response time, and a lower operation voltage, were successfully achieved.

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