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Phase stabilisation of blue-phase liquid crystals using a polymerisable chiral additive

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ABSTRACT

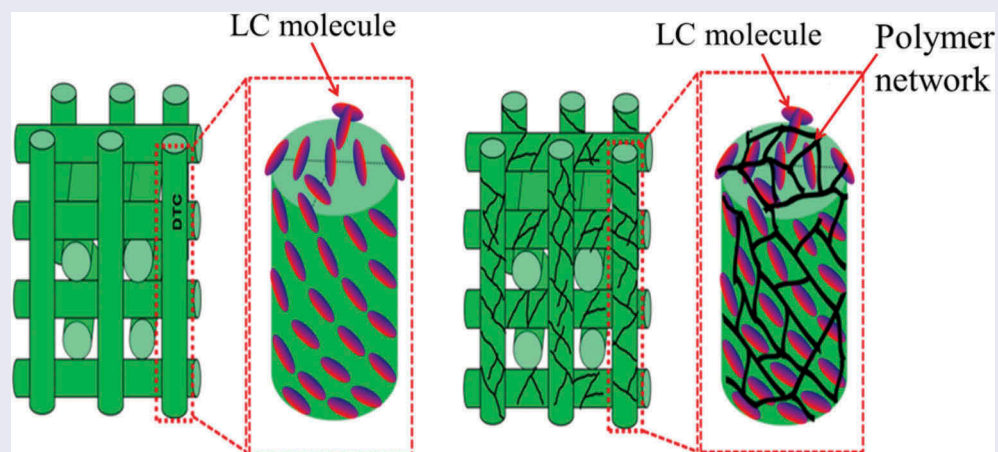
We report a novel polymer stabilisation of blue phase by a chiral agent itself without additional reactive monomers. In this way, the structural difference in phase stabilisation gives rise to novel electro-optic properties such as the hysteresis-free with low-operating voltages. The number of constituents in polymer-stabilised blue-phase mixture is reduced and the polymer network more directly engages the structure of the blue phase, so that the hysteresis is free with less contamination for liquid crystals to be rearranged by electric fields. Also, relatively low operating voltage is achieved because the chiral pitch becomes longer owing to the chiral agent becoming polymerised. This result would open an innovative way from a material perspective on stabilisation of blue-phase liquid crystals.

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reactive chiral monomer;
polymer stabilisation.



1. Introduction

Phase stabilisation is of interest from a perspective of not only industrial breakthrough in liquid crystal display (LCD) but also thermodynamic phase modulation in physical chemistry. In photonic and optical device applications, one of the potential materials for a fast response light modulator is blue-phase liquid crystals as well as for a renovation of LCD panels and flexible display applications. Such blue-phase liquid crystals are advantageous because of a number of features, such as

no need of alignment layer for cell assembly that brings down the device manufacturing cost and simplifies the fabrication process [1], wide viewing angle due to optically isotropic state at a field-off state [2], sub-millisecond response time due to highly confined liquid crystalline elasticity [3] and potential application of a colour filter-free field-sequential full-colour display [4]. From a thermodynamic perspective, the free-energy balance between the confined elasticity and topological defects configures a mysteriously complex nanostructured photonic lattice consisting of double-twist

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cylinders. This sharp energetic balance gives rise to the BP appearing between cholesteric and isotropic phase in a very narrow temperature range by few degrees. In the three-dimensional cubic lattice, the complicated configuration of double-twist affords topological singularity associated with disclination lines. The lattices also form crystallographic space groups thereby classified as BPI, BPII, and BPIII. The BPI and BPII possess body centred cubic structure and simple cubic structure, respectively, while BPIII is an amorphous and known as blue fog [5,6].

By adding photoreactive monomers to a blue-phase liquid crystal mixture and photo-polymerising those, the temperature range of this quasi-stable phase can be extended, called polymer-stabilised blue-phase liquid crystal [7,8]. When applying an electric field to it, electro-optic effect can be expected to have sub-millisecond response time but high operating voltage with significant hysteresis because the polymer network closely interacts with LIQUID CRYSTAL molecules [9,10]. The materials used for conventional polymer-stabilisation method typically consist of host nematic liquid crystal, chiral dopants, reactive monomers, and a photo-initiator. For instance, the chiral dopants should be mixed with sufficient amount to have the short pitch of chirality for double-twist formation, e.g. 25 wt% of CB15 and 10 wt% of ZLI-4725, to host liquid crystal in order to make sure of appearing blue phase [7,10]. Typical concentrations of reactive monomers are 9–15 wt% to a blue-phase liquid crystal mixture; in addition, it is preferred to use combination of mesogenic and non-mesogenic reactive monomers. Here, one may doubt that the additives would be not a guest and treat those as a contamination from pure liquid crystal material perspective, although the role of the additives to pure nematic liquid crystal is quite clear. Based on above facts, again, adding such chiral dopants and monomers are irreplaceable to generate and stabilise blue phases, but, at the same time, are paradoxically unwanted constituents from pure nematic liquid crystal material's view point. These results are one of the most crucial and bad effects in figure of merit for polymer-stabilised blue-phase liquid crystal. Also, the number of additives becomes variable that are not easy to control if too many. Furthermore, the polymer network stabilising the blue phases also paradoxically give rise to high operating voltage. Recent studies on the electro-optic effect of polymer-stabilised blue-phase liquid crystal have focused on low operating voltage and hysteresis free [11–13], long-term stability [14,15] and wide temperature range [16,17]. More recently, enhanced Kerr constant using a chiral reactive monomer was proposed [18–20]. Here, we demonstrate stabilisation of blue

phases using a chiral monomer that plays a role of both the chiral dopant and the reactive monomer [21], thereby reducing the chirality after polymer stabilisation and lowering down the twist power to unwind when applying voltage. In this novel stabilisation method, the blue-phase mixture only consists of nematic liquid crystal and chiral monomer; therefore, the mixture is expected to become relatively pure and to entail novel electro-optic properties due to innovative architecture in structural confinement.

2. Theory

The influence of external electric field in blue phases is numerically demonstrated as [22,23],

$$f_{bp} = f_{dist} + f_{thermo} + f_{elec} \quad (1)$$

where f_{dist} , f_{thermo} , and f_{elec} denote distorted elastic free energy, thermodynamic free energy, and electric field energy, respectively. Among the terms, the last term is only with negative sign and contributes against the other terms.

When electric field is applied to the blue phase, the double-twist structure unwinds and the constituent liquid crystal molecules gradually orient in the field direction, thus generate field-induced birefringence, and restore back to self-organisation on field removal. The electro-optical phenomenon of isotropic materials, called Kerr effect, can be described by quadratic formula [24], $\Delta n_{ind} = \lambda K E^2$, where the induced birefringence (Δn_{ind}) is proportional to wavelength (λ), Kerr constant (K) and an external field (E). This model has been extended for higher accuracy that the Δn_{ind} saturates up to maximum induced birefringence (Δn_s) by saturation field (E_s). This is expressed as the extended Kerr effect model [25], $\Delta n_{ind} = \Delta n_s \left(1 - \exp \left[- \left(\frac{E}{E_s} \right)^2 \right] \right)$. The Δn_{ind} reaches to Δn_s as E becomes larger than E_s . The E_s and Δn_s determine the electro-optic behaviour of BP. High Kerr constant is crucial for low operating voltage, so that it is worth describing the variables that influence the Kerr constant,

$$K = \Delta n \Delta \epsilon \epsilon_0 P^2 / k \lambda (2\pi)^2, \quad (2)$$

where Δn , ϵ_0 , P and k are dielectric anisotropy, vacuum permittivity, chiral pitch length and elastic constant of liquid crystal [26]. The chiral pitch can be estimated by simple formula,

$$P = \frac{1}{\sum_{i=1}^n c_i \cdot HTP_i}, \quad (3)$$

where c and HTP denote concentration of chiral agent and helical twisting power, respectively.

On the other hand, the selective Bragg reflection is another characteristic nature of the blue phase. When light passes through this medium, Bragg reflection/diffraction occurs upon Bravais lattice planes depending on how the lattice site standing and competitive to the wavelength. Typically, such colourful appearance shows micrometre-sized platelet domains with distinct reflected/diffracted colour. The Bragg reflection can be expressed as [27–29], $\lambda = \frac{2\bar{n}a\cos\theta}{\sqrt{x^2+y^2+z^2}}$, where a is the lattice constant, θ is the angle of incidence, \bar{n} is the average refractive index of nematic liquid crystal, and x, y, z are miller indices. The lattice constant is equal to one pitch length in the BPI while half the pitch length in the BPII. As the formula shows, the reflected wavelength range is function of both the lattice constant, that is, the length of chiral pitch and the lattice planes with respect to the light propagation direction.

One of the greatest advantages of blue phase is fast response upon on and off of an applied electric field, and the decaying time can be determined as [8],

$$\tau_{off} \approx \frac{\gamma_1 P^2}{k(2\pi)^2}, \quad (4)$$

where γ_1 is the rotational viscosity. The low viscosity of the liquid crystal mixture is crucially related to the response time, and the chiral pitch also quadratically influences it. We note that the P should be importantly optimised from the perspectives between the operating voltage and the response time.

3. Experiments

The prepared blue-phase mixtures used in this study consist of two different chiral agents, SRM17 and SRM3 as the molecular structures shown in Figure 1. SRM17 is a chiral dopant while SRM3 plays both roles of chiral dopant and a reactive monomer. The blue-phase liquid crystal mixtures are identically composed of a nematic liquid crystal mixture MLC-2053, which exhibits large dielectric anisotropy and high birefringence ($\Delta\epsilon = 42.6$, $\Delta n = 0.235$ and $T_{NI} = 86^\circ\text{C}$). The experimental procedures are schematically represented in Figure 2. As shown in Table 1, a sample BP-pure contains 5 wt% of SRM17 with high helical twisting power ($HTP \sim 166 \mu\text{m}^{-1}$) aimed no polymer stabilisation. For polymer-stabilised blue-phase liquid crystal, both 5 wt% of SRM17 and various concentrations (1, 3 and 5 wt%) of SRM3 ($HTP \sim 11 \mu\text{m}^{-1}$) are mixed with the nematic liquid crystal, thereby naming samples as

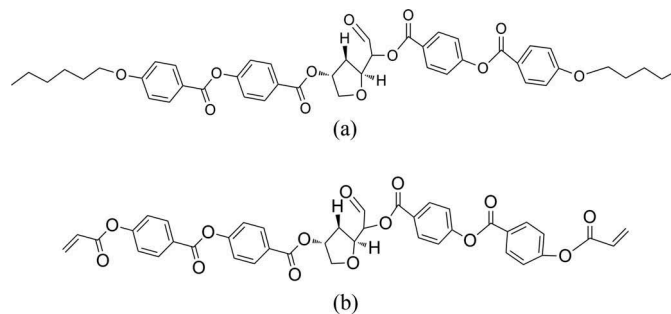


Figure 1. Molecular structures of (a) chiral dopant (SRM17) and (b) photo-reactive chiral monomer (SRM3).

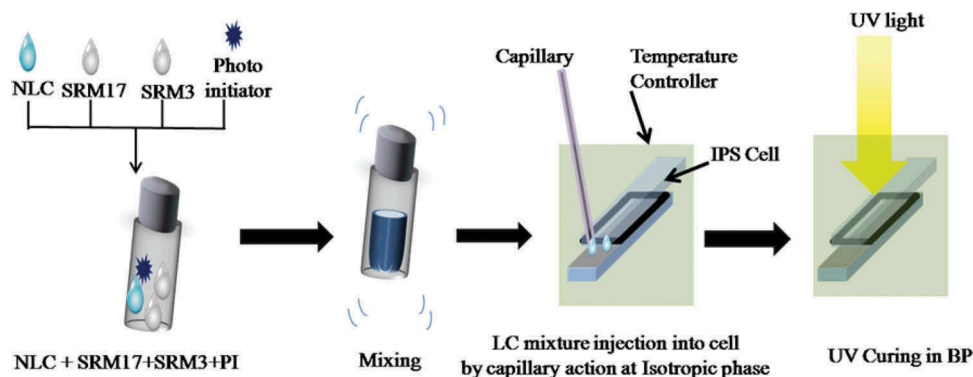


Figure 2. (Colour online) Schematic representation of sample processing for polymer-stabilised samples. No photo initiator and SRM3 added to the sample BP pure (no UV curing).

Table 1. The constituents and concentrations used for blue phase and polymer-stabilised blue phase.

Sample	Liquid Crystal, MLC-2053 [wt%]	Chiral dopant, SRM17 [wt%]	Photoreactive chiral monomer, SRM3 [wt%]
BP-pure	95	5	–
PS-BP1	94.05	4.95	1
PS-BP3	92.23	4.85	3
PS-BP5	90.30	4.75	5

PS-BP1, 3 and 5, respectively. The concentration of SRM3 varies to characterise the blue phases with respect to the photonic effect up on the chirality, stability of the polymer stabilisation, and electro-optic property of polymer-stabilised blue-phase liquid crystal.

Cells are prepared as blue-phase mixtures are sandwiched by two glass substrates with inter-digitated indium-tin-oxide (ITO) electrodes on one of the substrates. The width and spacing between electrodes are 4 μm and 4 μm , respectively. The cell gap is kept uniformly by 10- μm diameter-sized silica ball spacers.

In order to determine the temperature range and optic/photonic properties, the prepared blue-phase mixtures are injected into the cells in few degrees above clearing temperature (T_c). After injecting the mixtures, cells are loaded onto a thermally insulated hot-stage connected to a temperature controller (INSTECH, HSC402-STC20U), which is equipped on a polarising optical microscope (POM) (Nikon, ECLIPSE E600) with a CCD camera (Nikon, DXM 1200). The temperature is lowered down slowly from isotropic phase and closely observed under crossed polarisers. Once blue phase appears, the platelets become visible under significantly high intensity of the source lamp, compared to nematic or chiral nematic phases. The reflection spectra of the samples are measured by a UV-VIS spectrometer (Ocean Optics, USB2000+) to

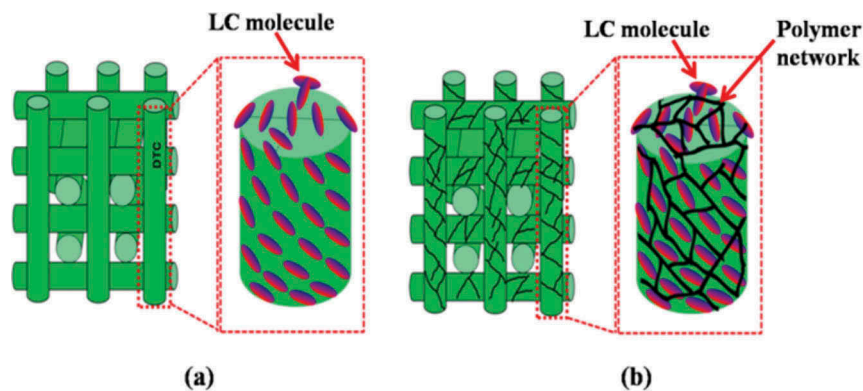
determine the Bragg reflection peak of blue phases at various chirality conditions.

Polymerisation takes place at desired blue phases by UV irradiance (10 mW/cm^2 at $\lambda = 365 \text{ nm}$) for 5 min, and then POM images, temperature range and reflection spectra are observed again for comparison before and after the polymer stabilisation. Finally, electro-optic hysteresis and operating voltage of polymer-stabilised blue-phase liquid crystal measure by voltage-dependent transmittance curves. The cells are placed between crossed polarisers in the way that the long-side electrode direction is 45° to the polarisers, and transmittance is detected by a photodetector using He-Ne light source ($\lambda = 633 \text{ nm}$) while applying voltage by a function generator (Tektronix, DPO2024B). After the measurement, the cell submerged into Hexane for 48 hours to wash out the liquid crystals, and then observed by field emission scanning electron microscopy (FE-SEM).

4. Results and discussion

The main difference of proposed stabilisation method using photoreactive chiral monomer SRM3 from conventional polymer stabilisation is expected as schematically illustrated in Figure 3. In the conventional method, polymer network is mostly formed in the space of disclination, whereas the photo reactive chiral monomer is polymerised where the polymer network would be randomly distributed and formed in space between the liquid crystal molecules.

From a perspective between elasticity and interfacial anchoring of polymer network, the proposed method would be much more efficient to the polymer network interacting with liquid crystal molecules than the conventional polymer-stabilisation method. In the proposed way, we can build assumptions on the effect after polymerisation: (1) the chiral pitch length should

**Figure 3.** (Colour online) Schematic representation of (a) structure of blue phase and (b) polymer networks tightly formed between liquid crystal molecules hence holding a double-twist cylinder.

become longer; (2) the T_c of mixture would be changed because the amount of photo reactive chiral monomer is reduced to become polymerised; (3) as in the Equation (2), the longer chiral pitch length P , the higher Kerr constant K , hence less operating voltage (4) the electro-optic hysteresis would become significantly reduced because the polymer network closely interacts with liquid crystal molecules.

The first and second assumptions can be proven by following series of POM image of the prepared samples in Table 1. The POM images of sample BP pure shows the blue phase range from 85.6°C to 81.7°C, the range is 3.9°C as shown in Figure 4. In Figures 5(a,c,e), POM images of blue phases are observed for samples PS-BP1, PS-BP3 and PS-BP5 with various concentrations of SRM3 corresponding to the colour of textures. The blue phases appear at 84.7°C, 84.7°C and 79.1°C and turn into chiral nematic phases at 80.8°C, 80.9°C and 77.1°C, that is, the ranges of 3.9°C, 3.8°C and 2.0°C for samples PS-BP1, PS-BP3 and PS-BP5, respectively (Table 2). In order to verify the role of SRM3, UV curing has been done while the temperature of samples is kept at 83.8°C, 81.7°C and 78.8°C for PS-BP1, PS-BP3 and PS-BP5, respectively. After UV curing, the temperature ranges were observed as 5.3°C, 6.1°C and 7.4°C for PS-BP1, PS-BP3 and PS-BP5, respectively, as shown in Figures 5(b d f) and Table 2. The overall colour of POM images shows that the chiral pitch length becomes longer after the photo-polymerisation because the lattice size becoming bigger means the

selective Bragg reflection peak shifts to longer wavelength region. Figures 5(a,b) represent sample PS-BP1 before and after the photo-polymerisation. Broadening of blue-phase range can be easily noticed by comparing these two images. Similar results are observed for samples PS-BP3 and PS-BP5 as shown in Figures 5(c,d) and Figures 5(e,f), respectively. The T_c is lowered down after the photo-polymerisation as the chiral agent becomes polymerised and separated from nematic liquid crystals.

Although the observation of PS-BP1, PS-BP3 and PS-BP5 was carried out at the each photo-polymerisation temperature, the POM images showed no selective Bragg reflection colours in the range 83.8°C–79.5°C for PS-BP1, 81.7°C–78.5°C for PS-BP3, and 78.8°C–76.5°C for PS-BP5. It would be a reasonable expectation that the polymer network stabilises double-twist cylinder rather than the lattice structure because the photo reactive chiral monomer is polymerised while staying still in the double-twist cylinder. Therefore, it can be possible that double-twist cylinder is, at the photo-polymerisation temperature, stabilised regardless of the lattice structure, hence being amorphous structure as BPIII and appears optically isotropic without photonic effect. Also, it needs to be noticed that the reason of relatively less-extended temperature range than that with conventional polymer-stabilisation method is the low monomer concentration without using monomers which can stabilise the disclination lines.

In order to quantitatively verify the reflection band of blue phases regarding the effect of photo-polymerisation, the reflection spectroscopy has been performed as shown in Figure 6. In case of the sample BP pure, the reflection band slightly shifts to longer wavelength range with decreasing temperature. For the case of samples PS-BP1, PS-BP3 and PS-BP5, as the concentration of the photoreactive chiral monomer increases,

Table 2. The measured blue phase ranges at various concentration of photo reactive chiral monomer before and after polymerisation.

Sample	ΔT_{BP} before UV exposure [°C]	Extended ΔT_{BP} [°C]
BP-pure	3.9	-
PS-BP1	3.9	5.3
PS-BP3	3.8	6.1
PS-BP5	2.0	7.4

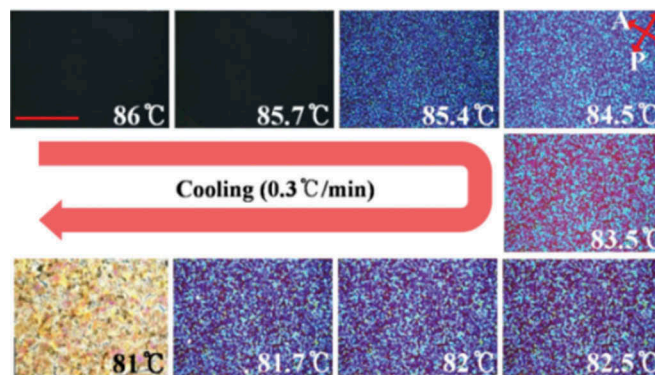


Figure 4. (Colour online) The POM textures of sample BP pure, which consists of 5 wt% of SRM17 in nematic liquid crystals (cooling rate of 0.3°C/min). The scale bar equals to 50 μm .

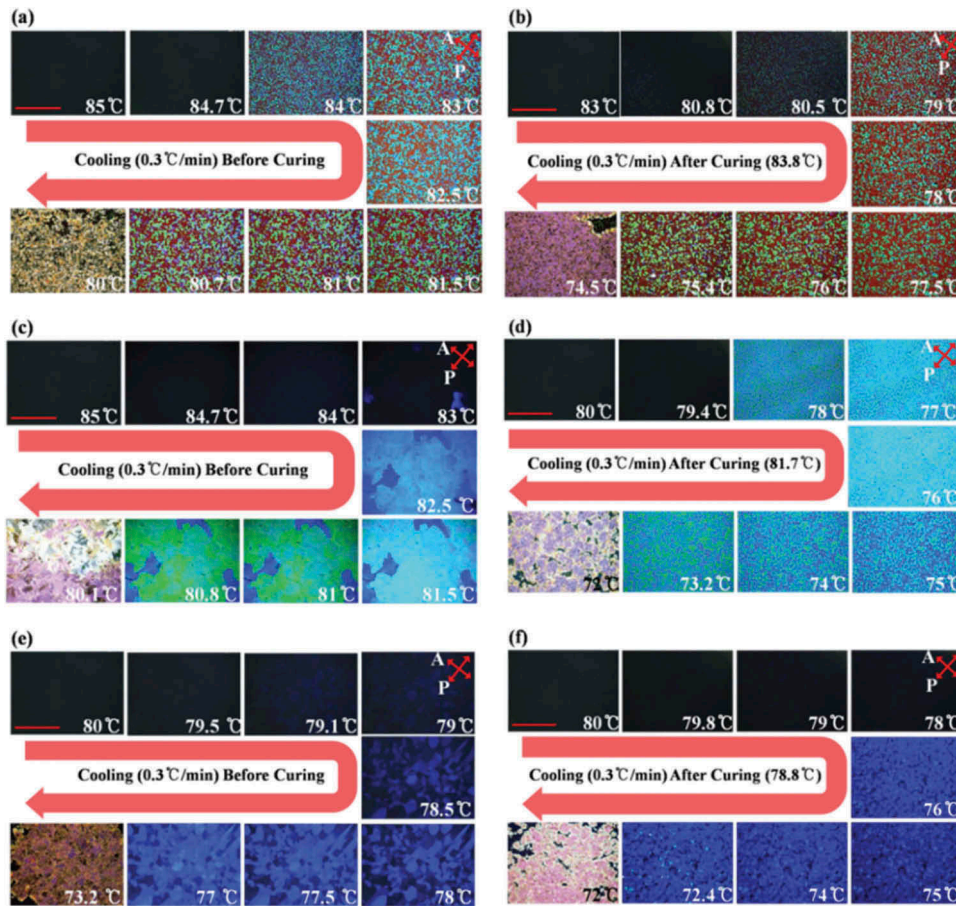


Figure 5. (Colour online) The POM textures of samples PS-BP1, PS-BP3 and PS-BP5, which consist of 1 wt%, 3 wt% and 5 wt% of SRM3 in nematic liquid crystals (a, c, e) before and (b, d, f) after UV curing. The scale bars are 50 μm .

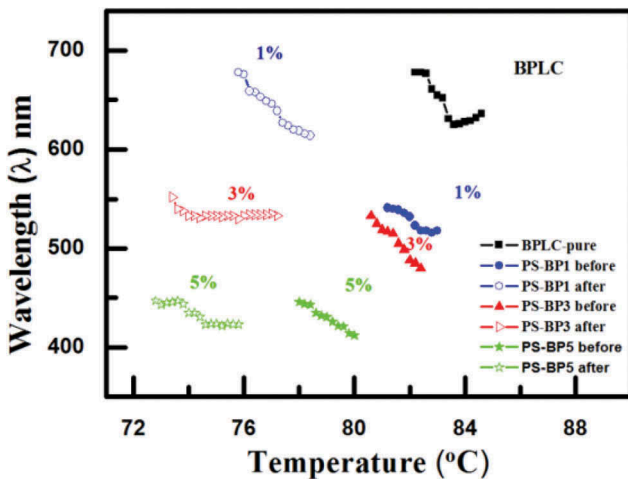


Figure 6. (Colour online) The wavelength of Bragg reflection peaks as a function of temperature and SRM3 concentration (solid symbols) before and (hallow symbols) after UV curing.

c increases, the reflection band shifts to shorter wavelength range. As POM images show, the reflection band after the photo-polymerisation shifts to longer wavelength range because of reducing chiral agent in

the liquid crystals. It should be noticed that the degree of the Bragg reflection band shift gets lower as c increases as shown in Figure 6. (PS-BP1: from 518–541 to 614–678 nm; PS-BP3: from 480–533 to 533–552 nm; PS-BP5: from 412–446 to 423–447 nm). This is because the P , which decides the Bragg reflection band, is inversely proportional to c in Equation (3). So, when c becomes high, the same amount of the chiral reactive monomer withdrawn for polymerisation contributes to small change in P .

The third and fourth assumptions can be demonstrated by voltage-dependent transmittance measurement with ascending and descending voltages as shown in Figure 7. In this measurement, threshold voltage (V_{th}), operating voltage (V_{op}) and hysteresis are determined as voltages at 10%, 90% of transmittances, and the voltage difference between ascending and descending at half of transmittance to the voltage at maximum transmittance, respectively, which are summarised in Table 3.

Remarkably, the V_{th} , V_{op} and hysteresis are reduced at higher concentration of the photo reactive chiral monomer, thus revealing enhanced electro-optic

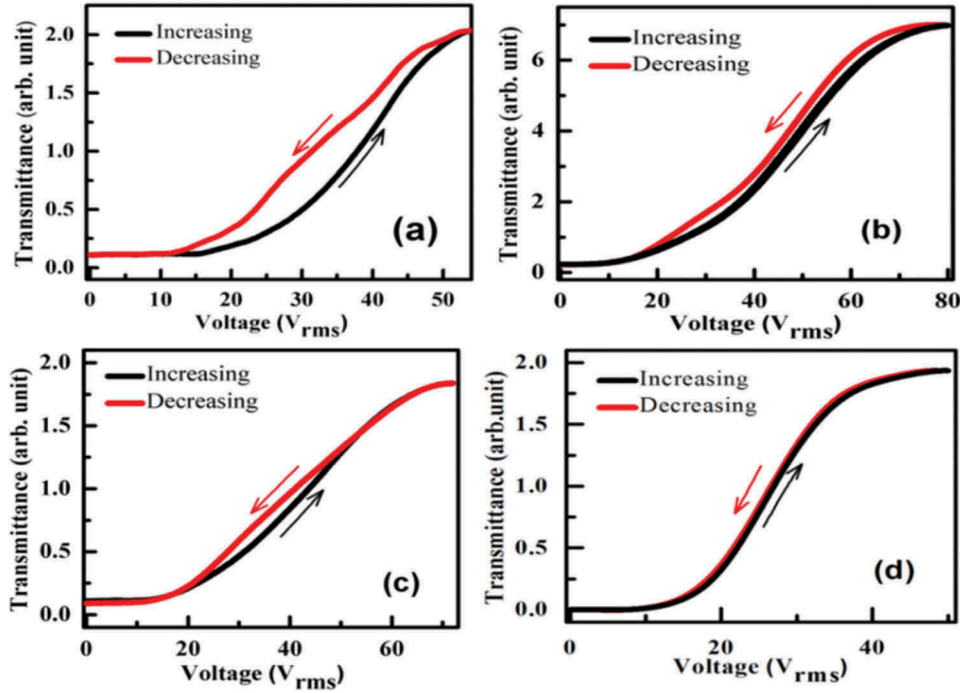


Figure 7. (Colour online) Measured voltage-dependent transmittance curves of (a) BP pure, (b) PS-BP1, (c) PS-BP3 and (d) PS-BP5. The upward and downward arrows indicate ramping up and down of the applied voltage, respectively.

Table 3. Measured electro-optical properties of BP and PSBP using the chiral reactive monomer.

Samples	V_{th} (E_{th}) [V_{rms}] ($[V_{rms}/\mu m]$)	V_{op} (E_{op}) [V_{rms}] ($[V_{rms}/\mu m]$)	Hysteresis, $\Delta V/V_{T100}$ [%]
BP-pure	24.9 (6.22)	48.5 (12.12)	11.32
PS-BP1	21.4 (5.35)	70 (17.50)	3.18
PS-BP3	20 (5.00)	60 (15.00)	4.37
PS-BP5	18 (4.50)	45 (11.25)	1.05

performance even though it is counter-intuitive to the conventional polymer-stabilisation method. The reason would be correlated to the novel architecture of the polymer network; that is, again, the polymer network is formed in the liquid crystalline medium, i.e. formed in the double-twist cylinder, rather occupying isotropic disclination lines. This structural difference gives rise to different response under applied electric fields. Considering the sample BP pure without polymer network under the applied voltage as described in Equation (1), the elastic free energy of liquid crystal medium (twist would be dominated due to high chirality) is significantly influenced against the external electric field. On the other hand, after polymerisation of photoreactive chiral monomer as for the samples PS-BP1, PS-BP3 and PS-BP5, another term of interfacial anchoring energy between liquid crystals and polymer network f_{poly} needs to be added to Equation (1). In the conventional polymer-stabilised regime, there is no

change in f_{dist} regarding the f_{thermo} and f_{poly} ; however, in the proposed regime, f_{dist} becomes significantly reduced due to the decrease in chirality in the liquid crystal medium. Therefore, the f_{elec} would be relatively easy to dominate so that the V_{th} and V_{op} can become lowered than that of the conventional polymer-stabilisation method, which is also consistent with proportionality between Kerr constant K and chiral pitch length P as in Equation (2). And as the degree of chirality in f_{dist} transferring to f_{poly} gets higher, the V_{th} and V_{op} further become lowered as shown in Table 3. Furthermore, because the polymer network strongly and closely interacts with liquid crystal molecules, the hysteresis becomes free as shown in Figure 7 (d). At this point, one may come up with the response time that can be slower by the enlarged P as stated in Equation (4). Here, we address that the dependent variable on the response time can include not only the P but also the correlation length (ξ) of the polymer network [30,31]:

$$\tau_{off} \approx \frac{\gamma_1(\xi)^2}{k(2\pi)^2}, \quad (5)$$

This correlation length means the space in the polymer morphology whether it is continuous or discontinuous, so one can verify how the confined liquid crystal molecules closely interact with the surface of

polymer, such like the cell gap effect in general liquid crystal cells. However, owing to the polymer network generated inside the DTCs, the correlation length of the polymer network becomes shorter than cases that polymer network is generated only within the disclinations comparable to the P . In our method, therefore, the degree of the response time increased by the longer P can be compensated by the shorter ξ of the polymer network. Our measurement result of the decaying time is approximately 1.3 ms. It seems reasonable to exhibit fast response time even with high rotational viscosity of the liquid crystal material used in our experiment, although it is not sub-millisecond response.

In order to directly observe the polymer network, the FE-SEM is carried out as shown in Figures 8(a–c) after the liquid crystals are washed out. Although it is not directly proved that the scale of polymer network shows polymer stabilisation of double-twist cylinders, we compare the polymer network to an amorphous blue phase (BPIII) [32] as shown in Figure 8(d). We found that the mesh scale of the polymer networks is similar, and both of the network structures are amorphous. These imply that, if the polymer networks in the samples PS-BP5 are polymerised only in the disclinations, the structure would be periodic and more aligned as the disclination lines are described periodically [5,7,32], which is unlikely in Figure 8. Therefore, we can interpret the polymer network in Figures 8(a–c) is polymerised randomly inside the double-twist cylinders and directly stabilise it, and it is tightly entangled in PS-BP5 using the reactive chiral monomer with relatively small amount of the polymer additive (5% in PS-BP5 vs. 22% in PS-BPIII in Ref [32]). Again, in our method, a question may arise that the sample PS-BP5 shows the amorphous structure of the polymer network in spite of the polymer stabilisation at BPI, and this structural observation would not be distinguished from PS-BPIII. To answer this question, one can think about the relation between the P and ξ . The P of PS-BPIII in Ref [32] and the sample PS-BP5 is estimated to be ~ 180 nm and ~ 260 nm, respectively.

Despite of the different P , the polymer-mesh-scale in PS-BP5 and PS-BPIII is similar as shown in FE-SEM images in Figure 8.

5. Conclusion

We demonstrate a novel method to stabilise blue phases using a polymerisable chiral additive. The blue-phase liquid crystal mixture contains only single additive, so that the purity of liquid crystals was enhanced. Furthermore, the photoreactive chiral monomer stabilises on double-twist structure rather than occupying isotropic disclination line. In this manner, liquid crystal molecules highly interact with polymer network while keeping the operating voltage low and hysteresis free, which are the main novelty of the proposed method.

Although the extended temperature range of blue phase should be further widened for real device application, this would be the matter of optimisation on the amount of chiral monomer concentration and helical twisting power, which should be further studied. The achieved low operating voltage is $45 V_{\text{rms}}$ ($11.25 V_{\text{rms}}/\mu\text{m}$) and hysteresis free (1.05%), which are competitive records among related literatures [20,33,34], while phase extension achieved by 3.7 times wide range with only 5 wt% of the additive.

The observation of the polymer morphology in the sample PS-BP5 is unusual; it is the amorphous structure, not the cubic/periodic structure of BPI. So, we may find out the reason that the amorphous structure generated in BPI in our sample comes from the polymer network generated in the DTCs. Also, with the amorphous structure of the polymer network, it cannot be distinguished from any cases whether the network is generated in DTCs of BPI or that in BPIII because a random structure cannot be distinguished from another random structure. Therefore, we could conclude two facts: (1) both PS-BP5 and BPIII show amorphous polymer network structures; (2) the correlation length of both would be similar although the estimated chiral pitch length is different. If both cases have

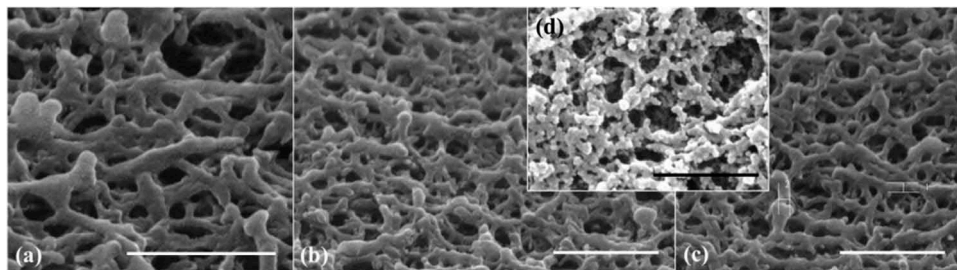


Figure 8. The FE-SEM images of the polymer network in (a) PS-BP1, (b) PS-BP3, (c) PS-BP5 and (d) polymer-stabilised amorphous blue phase, reproduced from Ref [32]. The scale bars are 500 nm.

polymer network generated in disclinations only, then the correlation length should be very different. However, in our result of FE-SEM images, the correlation length of both is similar, so we are convinced that the sample PS-BP5 has the polymer network within DTCs.

The proposed approach opens a breakthrough for the trade-off relation between the phase stabilisation and the high operating voltage in polymer-stabilised blue-phase liquid crystals. We believe the proposed polymer-stabilisation method will contribute to new way in phase stabilisation with highly enhanced electro-optic property.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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