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Enlarging the Kerr constant of polymer-stabilised blue phases with a novel chiral monomer

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ABSTRACT

Polymer-stabilised blue phase liquid crystals (PS-BPLC) have attracted many attentions in the area of display and photonic applications because of the superior electro-optical properties. Before application, the problem of high driving voltage should be resolved first. In this report, a new chiral monomer (C5011) has been synthesised and its influence on PS-BPLC was investigated. By adopting this chiral monomer, the Kerr constant of the PS-BPLC could be increased dramatically, which means improved driving capacity can be achieved.
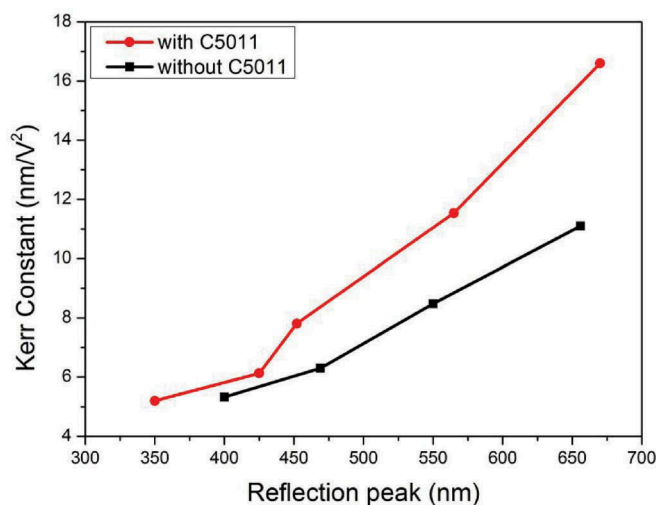
C5011
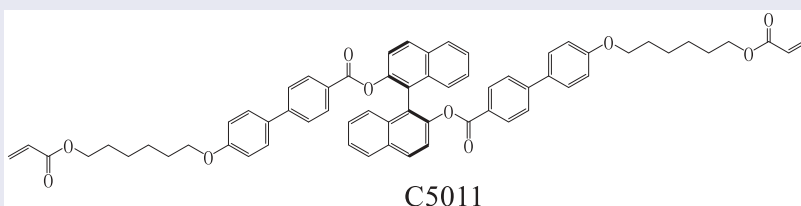
1. Structure of the chiral monomer synthesized.

2. By adopting this chiral monomer, the Kerr constant of the PS-BPLC could be increased

1. Introduction

Blue phase liquid crystal (BPLC) has attracted many attention due to its interesting features such as sub-millisecond response time, no need for colour filters and alignment layer,[1,2] optically isotropic dark state for wide and symmetric viewing angle and cell gap insensitivity if an in-plane-switching (IPS) cell is

employed.[3–5] Since blue phase is a coexistence of double twist cylinders and disclinations, its temperature range is narrow (usually less than a few Kelvin).[6] This limits the applications of blue phases. In 2002, Kikuchi et al. reported a series of PS-BPLCs in which the temperature range was extended to over 60 K including room temperature.[7] Coles et al. [8] presented a thermally stable blue phase from −125 to 125° C by refilling the blue phase templated polymer network with achiral liquid crystal (LC). However, the high driving voltage, residual birefringence and hysteresis still hinder its application. Researchers have made many efforts to improve the properties of blue phase, and detailed progress and new ideas can be seen in recent review papers.[9–14] Among these three problems, high driving voltage is the root cause. If the driving voltage is low enough, then the electrostriction effect causing lattice distortion would be prohibited and hysteresis would be negligible. Therefore, the most fundamental issue for BPLC studies is to lower the operation voltage.[15,16] Except for the method of optimising device structure[17] and increasing the effective electric field,[18,19] enlarging the Kerr constant using improved material is another way to reduce operating voltage.[20–23] To improve Kerr constant, LC hosts of high $\Delta\varepsilon$ (>300) have been developed.[15] However, these hosts usually exhibit a large viscosity which slows down the response time dramatically. Moreover, LC host with high value of $\Delta\varepsilon$ may also meet the low temperature and high frequency operation limits.[23] Sun et al. [24] and Rao et al. [25] found the Kerr constant of the LC cell could be increased by adding reactive diluents. In this paper, a novel monomer with a chiral centre was synthesised and the Kerr constant of PS-BPLC could be increased by applying it.

2. Characterisation

The structures of the final products and intermediates were confirmed by NMR. ^{1}H-NMR with tetramethylsilane as internal standard was recorded on a Bruker AV500 (500 MHz) instrument. The phase transition temperatures of the final product were measured by DSC (TA DSC-Q20) in nitrogen atmosphere at heating and cooling rates of 5°C/min. In this paper, the reported phase transition temperatures were peak values of the transition on DSC curves.

3. Synthesis

The target molecular 5 is a derivative of R-(+)-1,1′-bi-2-naphthol. Synthetic route is illustrated in Scheme 1. 6-Chlorohexyl acetate was used instead of 6-chlorohexan-1-ol to avoid byproducts. The biphenyl structure was designed to enlarge the helical twist power of the chiral monomer.

3.1 Materials

4′-Hydroxy-[1,1′-biphenyl]-4-carboxylate and 6-chlorohexyl acetate were purchased from Wuxi Yangshi Chemical Co. Ltd. and used as received. DMA, DMAP and DCC were purchased from Zhejiang Bulk
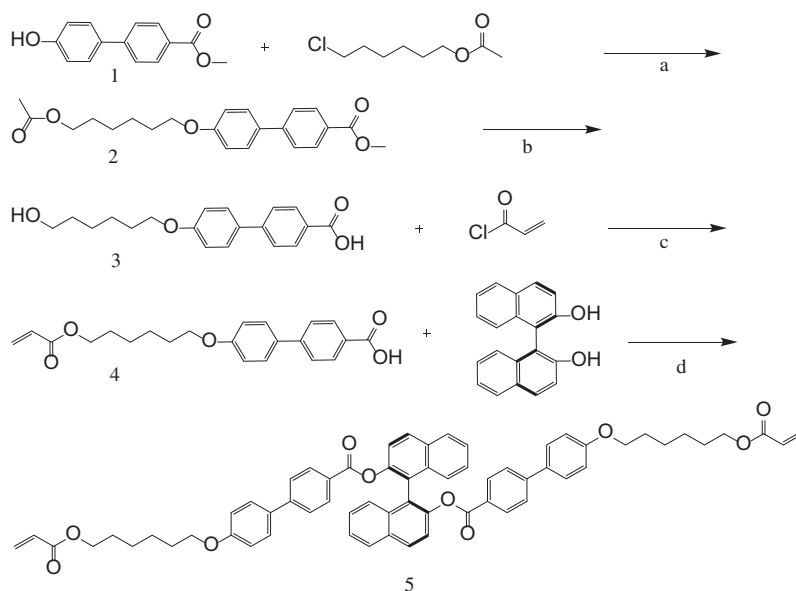
Scheme 1. General synthetic procedure of chiral monomer C5011. (a) K_2CO_3, KI, DMF, reflux for 4 h. (b) NaOH, H_2O, EtOH, reflux for 8 h, HCl. (c) DMA, DCM, rt, 20 h. (d) DMAP, DCC, DCM, rt, 24 h.

Chemical Co. Ltd. R-(+)-1, 1′-bi-2-naphthol was purchased from Lian YunGang Chiral Chemical Co. Ltd.

3.2 Synthesis of methyl 4'-((6-acetoxyhexyl)oxy)-[1, 1'-biphenyl]-4-carboxylate (2)

10 g (0.044 mol) of methyl 4′-hydroxy-[1,1′-biphenyl]-4-carboxylate and 9.97 g (0.056 mol) of 6-chlorohexyl acetate were dissolved in 200 ml of DMF, then 13.8 g (0.1 mol) K_2CO_3 and 8.3 g (0.05 mol) KI were added to the solution. The mixture was refluxed for 4 h, then cooled to room temperature and filtered to remove the insoluble salts, a yellow solution was achieved. The solution was then dumped into 1000 ml of water, stirred for 1 h and filtered to obtain the white particles (14.6 g).

3.3 Synthesis of 4'-((6-hydroxyhexyl)oxy)-[1,1'-biphenyl]-4-carboxylic acid(3)

14.6 g of methyl 4′-((6-acetoxyhexyl)oxy)-[1,1′-biphenyl]-4-carboxylate (2) was added to a three-necked bottle followed by 50 ml water, 50 ml EtOH and 5.6 g (0.14 mol) NaOH. The mixture was refluxed for 8 h, and then cooled to room temperature. 20 ml concentrated HCl was added to the solution and a white precipitation appeared. The white precipitation was filtered and purified by recrystallisation with EtOH. Yield 86%, 11.9 g. ^{1}H-NMR (CDCl$_3$, 500 MHz) δ (ppm): 12.92(s, 1H), 8.03 ~ 7.96(m, 2H), 7.78 ~ 7.71(m, 2H), 7.70 ~ 7.64(m, 2H), 7.07 ~ 6.99(m, 2H), 4.35 (s, 1H), 4.00(t, J = 6 Hz, 2H), 3.53(t, J = 6 Hz, 2H), 1.79 ~ 1.65(m, 2H), 1.50 ~ 1.30(m, 6H); IR (KBr, cm^{-1}): 2938, 2862, 2554, 1682, 1603, 1528, 1498, 1474, 1434, 1290, 1251, 1199, 1126, 1077, 1063, 998, 918, 835, 773, 715, 695, 547, 492.

3.4 Synthesis of 4'-((6-(acryloyloxy)hexyl)oxy)-[1,1'-biphenyl]-4-carboxylic acid (4)

11.9 g of 3 and 7.24 g (0.06 mol) N,N-dimethylaniline were added to 100 ml DCM, the mixture was stirred for 1 h, and then 4.16 g (0.046 mol) acryloyl chloride was dropped and stirred for 20 h. After that 2 ml of concentrated HCl was added to the solution, and stirred for 0.5 h, and then the mixture was extracted and washed by 300 ml of water for three times. The organic phase was dried with MgSO$_4$ and evaporated, and 14.1 g white solid was obtained. Yield 97%. ^{1}H-NMR (CDCl$_3$, 500 MHz) δ (ppm): 12.91(s, 1H), 8.04 ~ 7.94 (m, 2H), 7.79 ~ 7.71(m, 2H), 7.70 ~ 7.62(m, 2H), 7.10 ~ 6.95(m, 2H), 6.37 ~ 6.28(m, 1H), 6.22 ~ 6.13 (m, 1H), 5.96 ~ 5.89(m, 1H), 4.12(t, J = 5 Hz, 2H), 1.80 ~ 1.69(m, 2H), 1.68 ~ 1.60(m, 2H), 1.50 ~ 1.34(m, 4H); IR (KBr, cm^{-1}): 2945, 2869, 2545, 1720, 1679, 1602, 1561, 1529, 1498, 1411, 1299, 1248, 1194, 1609, 1039, 999, 897, 828, 776, 718, 551.

3.5 Synthesis of R-[1,1'-binaphthalene]-2,2'-diyl bis(4'-((6-(acryloyloxy)hexy-l) -oxy)-[1,1'-biphenyl]-4-carboxylate) (5)

3.68 g (0.01 mol) of compound 4, 0.06 g (0.0005 mol) 4-dimethylaminopyridine and 2.48 g (0.012 mol) DCC were added in sequence to 20 ml DCM, and then a solution of 30 ml DCM and 1.20 g (0.0042 mol) of R-(+)-1,1′-bi-2-naphthol was dropped and stirred for 24 h. The solution was filtered to remove the insoluble salts, then washed by HCl (5%) and aqueous Na$_2$CO$_3$ (5%) three times, respectively. The organic phase was dried with MgSO$_4$ and concentrated. The residue was purified by silica-gel column chromatography, and 2.65 g white solid was obtained. Yield 64%. ^{1}H-NMR (CDCl$_3$, 500 MHz) δ (ppm): 7.99 (d, J = 5 Hz, 2H), 7.95 (d, J = 5 Hz, 2H), 7.69(d, J = 10 Hz, 4H), 7.59(d, J = 10 Hz, 2H), 7.50 ~ 7.38(m, 12H), 7.37 ~ 7.30(m, 2H), 6.92(d, J = 10 Hz, 4H), 6.44 ~ 6.35(m, 2H), 6.17 ~ 6.07(m, 2H), 5.85 ~ 5.77(m, 2H), 4.21 ~ 4.14(m, 4H), 4.02 ~ 3.94(m, 4H), 1.88 ~ 1.77(m, 4H), 1.76 ~ 1.66 (m, 4H), 1.57 ~ 1.41(m, 8H); IR (KBr, cm^{-1}): 2938, 2864, 1731, 1604, 1525, 1497, 1472, 1407, 1268, 1251, 1183, 1064, 1014, 998, 873, 827, 808, 765, 720, 695.

4. Experimental section

Two series of experiments were carried out to investigate the influence of C5011 on PS-BPLC. The samples of series 1 have the same components, but were curved under different intensities of UV light. Series 2 was conducted with different components, but curved under the same intensity of UV light. The BPLC host consists of a major positive nematic LC (The nematic LC host has following properties: Δn = 0.2 at λ = 633 nm, $\Delta \varepsilon$ = 96 at 1 kHz and 21°C, and clearing temperature Tc = 96°C), the chiral dopant R5011 (HCCH) was added to induce blue phase, two kinds of ultraviolet (UV)-curable monomers were prepared: trimethylolpropane triacrylate and (TMPTA, Sigma Aldrich, St. Louis, MO, USA), RM257 (Sigma Aldrich).

The first series has four samples, with the same component (LC host, 85.54%; C5011, 2.73%; R5011, 2.73%; RM257, 5.4%; TMPTA, 3.6%), all of them were curved at 68°C (in the blue phase). Samples 1–3 were held exposing to the UV light for 30 min with different intensities (30, 8 and 6 mW/cm^2) and sample

Table 1. Composition of N*-LC mixtures of series 2.

Sample	Host	R5011	C5011	RM257	C12A
1	83.5%	4.0%	2.5%	6.0%	4.0%
2	84.0%	3.5%	2.5%	6.0%	4.0%
3	84.5%	3.0%	2.5%	6.0%	4.0%
4	85.0%	2.5%	2.5%	6.0%	4.0%
5	85.5%	2.0%	2.5%	6.0%	4.0%
6	86.0%	4.0%	–	6.0%	4.0%
7	86.5%	3.5%	–	6.0%	4.0%
8	87.0%	3.0%	–	6.0%	4.0%
9	87.5%	2.5%	–	6.0%	4.0%

4 was exposed to UV light with an intensity of 1 mW/cm^2 for 60 min.

In order to achieve blue phase with selected reflection-band, different proportions of chiral dopant R5011 were used in five samples of series 2. The doping amount was between 2% wt. and 4% wt. The constituent fractions of the mixtures were shown in Table 1. After these components were homogeneously mixed, the mixture was injected in an IPS cell at the isotropic temperature and then cooled down at 0.5°C/min.

In order to stabilise the BPLC, the cell was exposed to a UV light (intensity = 4 mW/cm^2, wavelength = 365 nm) for 15 min at 65°C. The electrode width (w) and distance (l) between interdigitated electrodes were 8 μm and 12 μm, respectively, and the cell gap was 10 μm. BPLC cells placed between crossed polariser and the textures were observed using POM. In the IPS cell, the electric field-induced birefringence was in the direction lateral to the in-plane field and the striped electrode was oriented at 45° to the transmission axis of the polariser to maximise the transmittance.
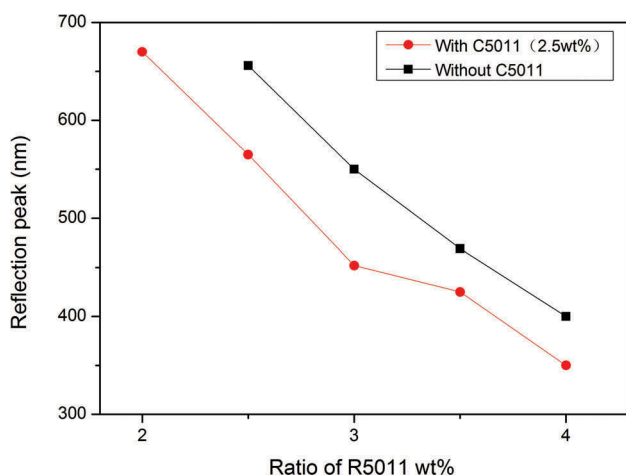
5. Results and discussion

5.1 Optical properties

Figure 1(a) shows the image of blue phase before curve of the series 1. Figure 1(b)–(d) depicts samples 1–3 of series 1 after polymerisation at 23°C. Sample 4 was not further tested because of strong scatting. The reflection peaks of samples 1–3 after polymerisation are 485, 500 and 518 nm, respectively. It is obvious that the reflection peak increases as the decrease of the UV light intensity. As shown in Figure 2, the reflection peak of samples with C5011 is shorter than that of without. Like samples 1 and 6, both contain the same ratio of R5011 (4%wt), while the reflection peak of the sample 1 was 350 nm, shorter than that of sample 6 (425 nm).

The reflection wavelength of BPLC is determined by lattice parameter (a). Lattice parameter (a) is related to the pitch length of the double twist cylinder of blue phase, for example, a is equal to pitch length in the case of BPI.[26] The reflection spectroscopy shows that by adding chiral monomer (C5011) a blue phase of shorter pitch length could be obtained. During polymerisation, the produced polymers segregate from the low molecular weight LCs and a phase separation occurs at submicrometre level.[27] The chiral monomer would be confined in the polymer by segregation and lost chirality.[24] However, the resulted reflection peak wavelength of samples containing C5011 was shorter than these with the same fraction of non-polymerisable chiral dopant. It is conceivable that inducing C5011 to PS-BPLC system can decrease the pitch length of blue phase. This phenomenon may result
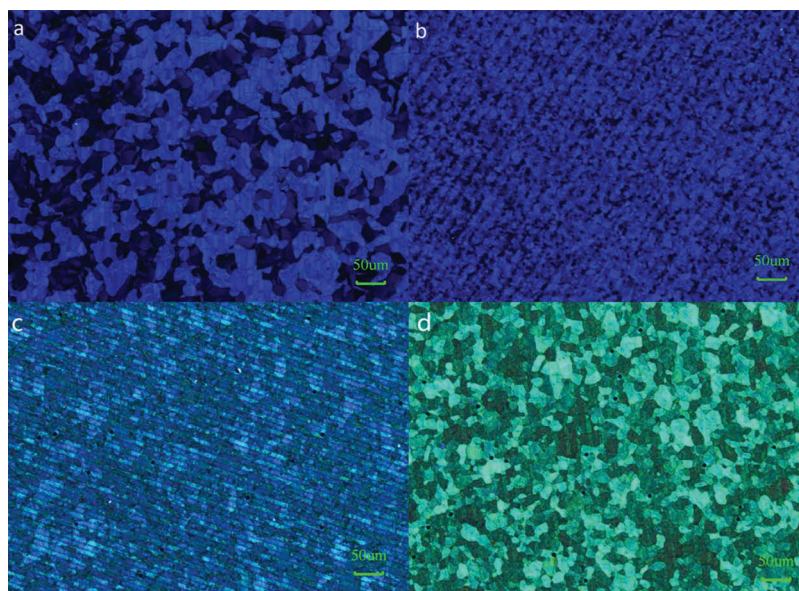
Figure 1. The photos of PS-BPLC textures: (a) series 1 before curve; (b) series 1 exposed to UV light of 30 mW/cm^2 for 30 min; (c) series 1 exposed to UV light of 8 mW/cm^2 for 30 min; (d) series 1 exposed to UV light of 6 mW/cm^2 for 30 min.
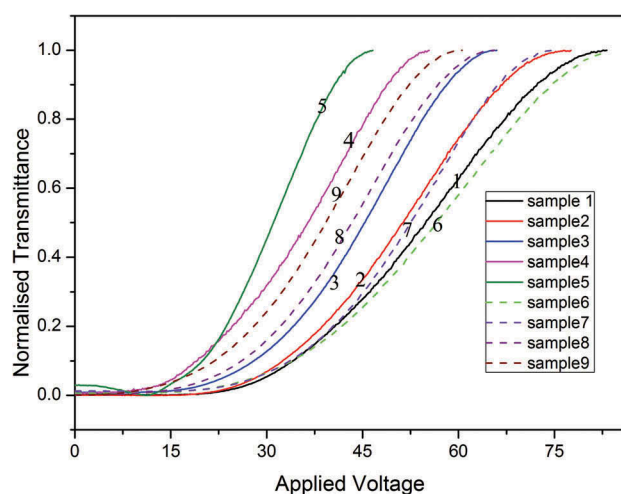
Figure 2. (color online) Reflection peak of samples with different ratio of R5011 (series 2).

Figure 3. (color online) Comparison of normalized VT curves of various PS-BPLC samples, prepared from sample 1 to sample 9 of series 2.

from the fact that the polymer network of PS-BPLC can act as cast that retaining the 3D structure of blue phase, just like Jau [28] depicts, the polymer network not only stabilise the defects but also the double twist cylinders. Since the excretion of the chiral monomer from the nematic media and the fixation of the blue phase structure take place at the same time, part of the twist power from the chiral monomer was restored by the resulted polymer network. The higher value of UV light intensity leads to a faster polymerisation speed and more restored twist power. By inducing the chiral monomer to the PS-BPLC system, the pitch length of blue phase could be decreased.

5.2 The electrical properties of series 2

5.2.1 Driving properties
Figure 3 depicts the measured VT curves for the PS-BPLC cells. The on state voltage (V_{on} means the applied voltage when the normalise transmittance is 1) of samples 1–5 is 83.2, 77.0, 67.4, 55.4 and 46.8 V, respectively, while that of samples 6–9 is 85.6, 75.0, 65.6 and 56.6 V, respectively. The driving voltage increases with the increasing amount of R5011, whether the samples contain C5011 or not. A lower driving voltage can be obtained by the use of chiral monomer C5011. This trend is similar to the work of Jau [28], the driving voltage was declined effectively by refilling the porous free-standing cast retaining 3D structure of blue phase with achiral mesogen (method of washout and refill). In the method of washout and refill, the helical structure of blue phase was just supported by the polymer anchoring force. However, in the system containing chiral monomer, the helical structure was supported by both the polymer

anchoring force and twist power of non-polymerisable chiral dopants. Therefore, the driving voltage was abated not only by the LC host but also by the chiral dopant.

A decrease in required applied voltage corresponds to an increased Kerr constant.[29] Figure 4 shows plots of Kerr constant of samples 1–9 after polymerisation (calculated from the applied voltage proposed by Yan [29]). The Kerr constant of both series increased with the reflection peak whether the samples contained C5011 or not. Usually the Kerr constant increases with the reflectance wavelength, which corresponds to the pitch length of blue phase. Increase in Kerr constant as decreasing concentration of R5011 in series 2 is
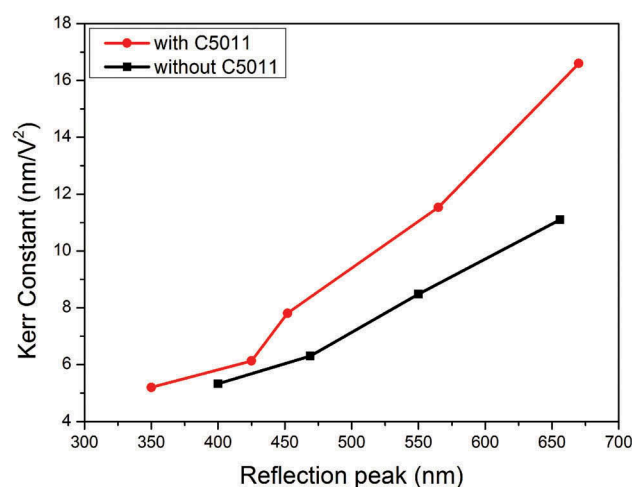
Figure 4. (color online) The variation of Kerr constants with reflection peak wavelengths of the samples with and without C5011.

related to the increase of pitch length. This trend is agreed with Equation (1) proposed by Gerber [30]:

$$K \approx \Delta n \Delta \varepsilon \frac{p^2}{\lambda k (2\pi)^2},\qquad(1)$$

where λ is the probe beam wavelength, Δn is the intrinsic birefringence, $\Delta \varepsilon$ is the dielectric anisotropy, P is the pitch length and k is the effective elastic constant of this system. As shown in Figure 4, the Kerr constant of samples containing C5011 is larger than that without when the reflection wavelength is the same. It has been discussed in the former part of this paper that the lattice parameter is one helical pitch for BPI. Thus the change of effective elastic constant (k) may be the main reason for the increase of Kerr constant. Since the magnitude of the effective elastic constant is governed by both the polymer anchoring force and the twist power of chiral dopant,[28] a decreasing fraction of chiral dopant will lead to a drop of effective elastic constant. When the mesogenic molecules are tilted by applying a voltage, the reducing of twist power is helpful for the electric field to unwind the helical structure. Inducing C5011 to the PSBP system can reduce the fraction of chiral dopant relatively. This phenomenon can cause a drop in the effective elastic constant in comparison with the original PSBP. An enlarged Kerr constant was observed consequently.

5.2.2 Hysteresis

We have also measured hysteresis curves of the cells. Hysteresis is defined as the difference in voltage at half transmittance between the forward and backward directions. As Figure 5 depicts, samples
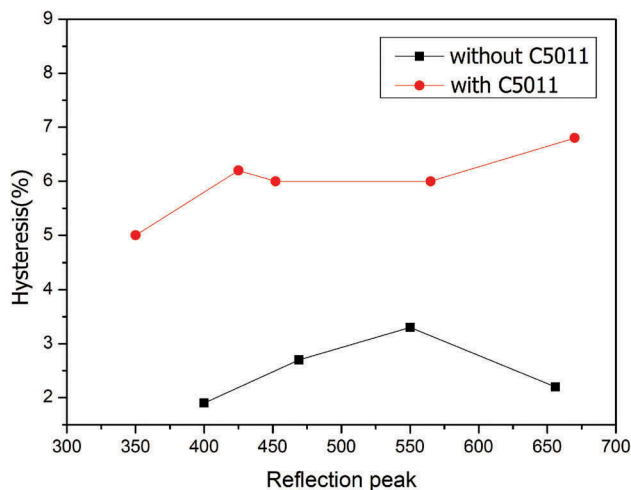

Figure 5. (color online) Comparison of hysteresis without C5011 and stabilized by C5011 (2.5% wt).

containing C5011 show more hysteresis than samples without C5011. This phenomenon is agreed with the findings proposed by Lan et al. [31] that hysteresis increases as chiral concentration decreases. Hysteresis of blue phase is related to relaxation ability. Relaxation means BPLC recovers itself from lattice electrostriction to original BCC structure. Effective elastic constant (k) is the primary factor that governs the relaxation ability.[29] As we mentioned above, the effective elastic constant (k) could be decreased by inducing C5011 to the PS-BPLC system. The decreased effective elastic constant might be major reason for the increase of hysteresis.

6. Conclusion

In summary, a novel chiral monomer was synthesised and applied in PS-BPLC. By doping such a chiral monomer C5011, these BPLC systems need an actual less amount of chiral dopant. It can reduce the 'twist' power from chiral dopant and increase the Kerr constant resulting in decreased driving voltage of PS-BPLC system. This study will provide a method to improve driving capacity of PS-BPLC, and further detail investigation of its application on BPLC should be carried out.

Disclosure statement

No potential conflict of interest was reported by the authors.

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