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Application of photosensitive polyimides as alignment layer to optical switching devices of a nematic liquid crystal

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We have explored the change in alignment of a nematic liquid crystal, 4'-pentyl-4-cyano-biphenyl (5CB) with three types of photosensitive polyimide as the alignment layer by photoirradiation at 366 nm. The photosensitive polyimide alignment layer induced a reversible change in alignment of 5CB. It was observed that the 5CB molecules became aligned from homogeneous alignment to homeotropic on photoirradiation with a d.c. electric field as a bias, and reversed to the homogeneous state when photoirradiation was ceased. This result indicates that optical switching could be repeated by on and off switching of the excitation light at 366 nm. The optical switching of the nematic liquid crystal might be mainly due to a photophysical change in the polyimide surface which is affected by the chemical structures of the polyimides at the temperature at which 5CB exhibits a nematic phase. The optical switching of nematic liquid crystals with photosensitive polyimides as the alignment layer is a novel driving method for nematic liquid crystals.

1. Introduction

Photonics, that is the control of light by another light acting as a stimulus, has received increasing attention recently in place of electronics because of advantages in high speed processing of information. In photonics, switching devices play an important role in the control of light and change their own physical properties with the light stimulus. Liquid crystals (LCs) show a large optical anisotropy, and a large change in refractive index can be obtained by changing their alignment. Owing to these excellent properties, which are obviously advantageous for photonic applications, LC materials are expected to be used not only in display devices, but also for various photonic applications. Many studies have been reported on the construction of optical switching devices by the use of LCs [1,2]. So far, we have performed studies on photochemical phase transitions of LC materials containing photochromic molecules, and have reported optical switching by transmission [3–6], reflection [7, 8], and scattering mode [9] analyses. Rapid

optical switching of transmitted probe light through two crossed polarizers could be achieved by means of photosensitive LCs [3, 4].

At present, LCs, usually nematic (NLCs), are used only as active media in display devices because the response of LCs to change in the electric field is slow. In such displays, the static and dynamic properties of NLCs are significantly affected by the alignment layer in the NLC cell. In fact, alignment of NLC molecules depends entirely on the surface properties of the alignment layer [10, 11]. Therefore, if the physical and/or chemical properties of the alignment layer surface can be changed by light as a stimulus, a change in LC alignment can be induced by light, which results in a light induced change in the refractive index of the system [12]. The optical switching of LCs by means of photosensitive alignment layers can also be achieved and is favourable for the fabrication of switching devices with a high speed response. It is a novel driving method for LCs.

Among various polymer materials, polyimides (PIs) are generally used as alignment layers for obtaining homogeneous alignment of NLCs because of their simplicity

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and reliability in handling [13–15]. PIs have become of interest due to their outstanding thermal, chemical, mechanical and electrical properties, and their potential applications. The electrical and mechanical properties of PIs have been extensively explored [16–20], but little research has been performed on their photoresponsive properties with the aim of development of photonic materials.

In this study, we have synthesized two types of aromatic PIs and one type of aliphatic PI to manipulate the alignment of NLC molecules by light as a stimulus and explored the change in alignment of a NLC on photoirradiation with a d.c. electric field as a bias.

2. Experimental

2.1. Materials

3,3',4,4'-Benzophenonetetracarboxylic dianhydride (BPDA), 4,4'-diaminophenylenemethane (DPM), 1,9-diaminononane (DN), 4,4'-bipthalic anhydride (BTDA), and 4,4'-diaminobenzophenone (DBP) were purchased from the Tokyo Chemical Industry Co. Ltd., and used without further purification. Dimethylformamide (DMF), *N*-methyl-2-pyrrolidone (NMP), and γ -butyrolactone were purchased from the Kanto Chemical Co. Inc., and used as solvents without further purification. The NLC, 4'-pentyl-4-cyanobiphenyl (5CB), was supplied by Merck Japan Ltd. 5CB exhibited an absorption maximum at 286 nm (figure 1).

2.2. Synthesis of poly(amic acid)s (PAAs)

2.2.1. PAA (BTDA/DBP)

The PI precursor, poly(amic acid) PAA (BTDA/DBP), was prepared by the reaction between BTDA (0.69 g, 2.4 mmol) and DBP (0.50 g, 2.4 mmol) in DMF (22.7 g) at ambient temperature for 6 h. First, DBP was dissolved in DMF, then an equimolar amount of BTDA was added incrementally to the solution under a nitrogen

atmosphere. After the reaction was completed, the crude PAA was precipitated from the DMF solution into methanol, filtered off, and dried at ambient temperature for 24 h. The PAA was obtained as a yellow solid (1.09 g, 91% yield). IR (KBr, cm^{-1}) 1776, 1718, 1656, 1602, 1510, 1363, 1275, 737.

2.2.2. PAA (BTDA/DPM)

This PAA (94% yield) was similarly prepared from BTDA and DPM. IR (KBr, cm^{-1}) 1775, 1721, 1618, 1512, 1371, 1222, 738.

2.2.3. PAA (BPDA/DN)

An aliphatic PAA (59% yield) was obtained at a higher temperature (140°C) than the aromatic PAAs because of the low reactivity of DN towards BPDA. IR (KBr, cm^{-1}) 1772, 1714, 1437, 1394, 1294, 724.

2.3. Preparation of PI alignment layer

The PAAs were first dissolved in a 2:1 (by weight) mixture of γ -butyrolactone and NMP at a concentration of 3 wt %. Glass substrates coated with indium tin oxide (ITO), with dimensions $25 \times 20 \text{ mm}^2$, were washed with neutral detergent, distilled water, and 2-propanol in an ultrasonic bath and then dried at 60°C. The PAA was spin-coated onto the ITO glass substrate. The film obtained was subsequently heated at 100°C for 1 h to evaporate the solvent and then thermally imidized by heat treatment at 250°C for 2 h. The PI film thickness was $48 \pm 6 \text{ nm}$ which was estimated by SEM (Hitachi, S4200). The chemical structures of the PIs used as alignment layers in this study are shown in figure 2.

An LC cell was assembled using a pair of the substrate plates prepared above; these had been rubbed to obtain uniaxial alignment with their rubbing directions anti-parallel (figure 3). The gap of the LC cell was controlled by using 5 μm thick Mylar spacer. 5CB was introduced

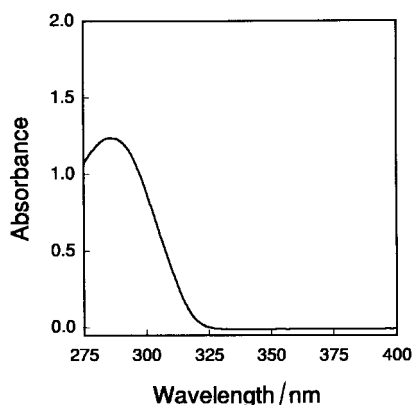


Figure 1. UV-vis absorption spectrum of 5CB in dimethyl sulfoxide at a concentration of $5.37 \times 10^{-5} \text{ mol l}^{-1}$.

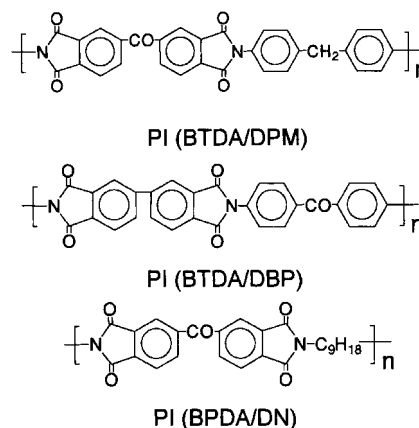


Figure 2. Chemical structures of PI (BTDA/DBP), PI (BTDA/DPM), and PI (BPDA/DN) used in this study.

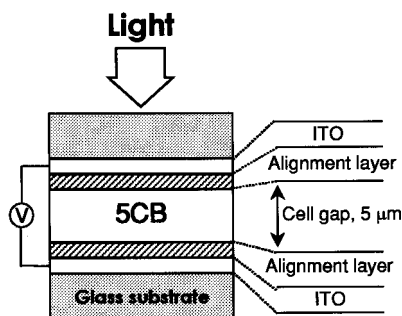


Figure 3. A cross section of the NLC cell used for evaluation of optical switching behaviour.

into the LC cell in the isotropic phase by capillary action. 5CB exhibits phase transitions from crystal to N at 296.6 K and from N to isotropic at 308.3 K.

2.4. Characterization

Thermal properties of the PIs were evaluated with a differential scanning calorimeter (DSC, Seiko I&E SSC-5200 and DSC 220C) at a heating and a cooling rate of $10^{\circ}\text{C min}^{-1}$. Fluorescence and UV-visible absorption spectra of the PIs were measured at ambient temperature in air using a fluorimeter (Hitachi F-4010) and a spectrophotometer (Jasco V-550). Change in the chemical analysis of the PI surface components from before to after photoirradiation was measured by electrospectroscopy (ESCA, VG Microtech MT 500/L). IR spectra of the PI films were recorded on a Jasco FTIR-610 infrared spectrometer at a resolution of 4 cm^{-1} . The PI films were prepared as KBr disks for IR measurements under the same condition as for the PAAs.

2.5. A measurement system for optical switching of NLCs

Figure 4 shows the measurement system for the optical switching behaviour of NLCs. The NLC cell was placed between two crossed polarizers and temperature-

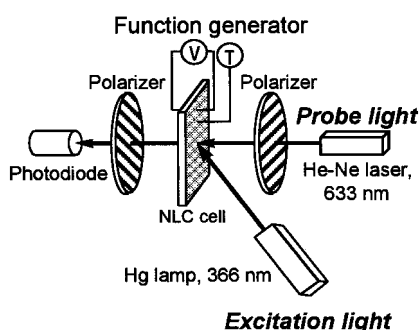


Figure 4. Schematic diagram for electro-optical response measurement of NLC (5CB) with the photosensitive PIs as an alignment layer.

controlled, and a d.c. bias voltage was applied across the cell. A He-Ne laser (NEC, GLC5370, 5 mW) was used as a probe light source, and the transmittance at 633 nm was measured with a photodiode and automatically recorded with a personal computer. We defined the 100% transmittance as the transmittance of the probe light through parallel polarizers, with the LC cell containing the NLC in an isotropic state between them. Photoirradiation was carried out using a high pressure mercury lamp giving light at 366 nm which was obtained by a combination of glass filters (Toshiba, UV-D36A + UV-35 + IRA-25). The intensity of the light was measured with a power meter (TQ8219).

3. Results and discussion

3.1. Electro-optical switching of the NLC

We explored the change in alignment of 5CB with PI (BTDA/DBP) as an alignment layer using light as a stimulus. Figure 5 shows the transmittance–voltage (T – V) profile of 5CB with PI (BTDA/DBP) as the alignment layer. The measurement was performed at 30°C . The 5CB molecules in the cell without an electric field are aligned parallel to the glass substrate (homogeneous alignment) and thus they exhibit a maximum value of transmittance. It was observed that with increasing applied d.c. voltage across the cell, the transmittance remained unchanged until $\sim 4\text{ V}$ and then it decreased steeply and eventually reached $\sim 0\%$. Change in the transmittance was induced by the external d.c. electric field. This is well known as an electro-optic effect of a NLC, which results from change in the alignment of the NLC from homogeneous to homeotropic, and its behaviour is affected by alignment layer thickness and cell gap. A threshold d.c. voltage for the change in the alignment of 5CB was about 6 V. We also measured the T – V profile under photoirradiation at 366 nm (light

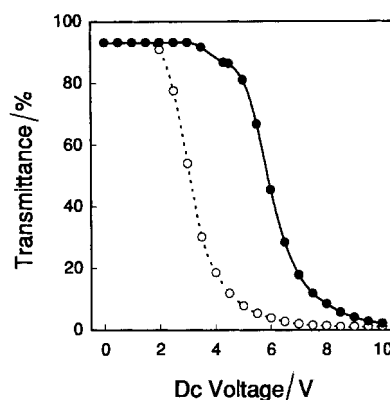


Figure 5. The electro-optical response of 5CB with PI (BTDA/DBP) as an alignment layer at 30°C : ● before photoirradiation; ○ during photoirradiation at 0.6 mW cm^{-2} . The pretilt angle of the cell was 4.7° .

intensity 0.6 mW cm^{-2}). Very interestingly, the T - V profile under photoirradiation shifted to a low d.c. voltage and the threshold voltage decreased to 3 V as shown in figure 5.

The largest difference in the T - V profile before and during irradiation occurred at 4.5 V. Therefore, we irradiated the cell at 366 nm while 4.5 V (d.c.) was applied across the cell as a bias voltage. As shown in figure 6, the transmittance of the He-Ne laser beam at 633 nm decreased on photoirradiation and recovered to the initial value when photoirradiation ceased. This behaviour is due to change in the alignment of the 5CB molecules from the homogeneous state to the homeotropic state. This optical switching could be repeated by on and off switching of the excitation light at 366 nm. These results indicate that the alignment of 5CB can be reversibly changed by light when PI (BTDA/DBP) with a photosensitive moiety is used as the alignment layer; this is entirely due to physical and/or chemical changes of the PI (BTDA/DBP) by the light, because 5CB absorbs no light at 366 nm as shown in figure 1.

In order to explore the effect of temperature on the optical switching behaviour of the cell with the PI (BTDA/DBP) alignment layer, we investigated response times of the cell by irradiation of the N phase of 5CB as a function of temperature with a d.c. electric field as a bias. We defined the response time as the time necessary to reduce the transmittance to 10% of the maximum value, a change which results from change in the alignment of the 5CB by the light. As shown in figure 7, the response time of the cell with the d.c. bias voltage of 4.5 V decreased abruptly at $\sim 29^\circ\text{C}$ and remained nearly constant at higher temperatures. This result may be interpreted in terms of the viscosity of 5CB in the N phase since this decreases to a significant extent with temperature.

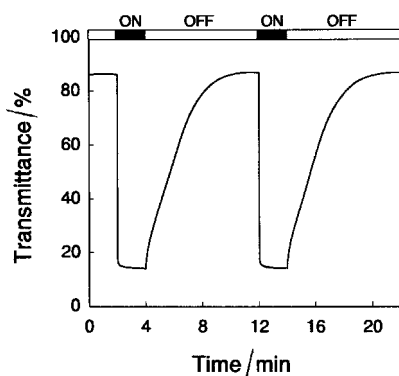


Figure 6. Optical switching of 5CB with PI (BTDA/DBP) as an alignment layer. The switching behaviour was examined at 30°C and photoirradiation was performed at 366 nm (light intensity, 0.6 mW cm^{-2}) with a d.c. bias voltage of 4.5 V.

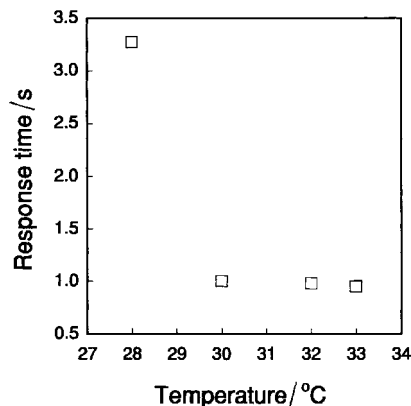


Figure 7. The effect of temperature on optical switching behaviour of the cell with PI (BTDA/DBP) as an alignment layer. Photoirradiation was performed at 366 nm (light intensity, 0.6 mW cm^{-2}) with a d.c. bias voltage of 4.5 V.

We next explored the optical switching of 5CB with PI (BTDA/DPM) as an alignment layer. We measured the T - V profile of the cell before and during photoirradiation as previously stated, and the results are shown in figure 8. The T - V profile during irradiation shifted to a low d.c. voltage and the threshold voltage decreased to 5 V. Optical switching of 5CB was also achieved by means of the PI (BTDA/DPM) alignment layer and its behaviour was similar to that of PI (BTDA/DBP). In this case, however, the slope of the T - V profile was not so steep as that shown in figure 5 and the threshold voltage for the change in the alignment was higher than for PI (BTDA/DBP) having a benzophenone moiety. We applied 7 V (d.c.) as a bias, which produced the largest difference in the T - V profile before and during irradiation, and examined the optical switching behaviour of the NLC cell. As shown in figure 9, the transmittance could also be reversibly changed by on and off switching

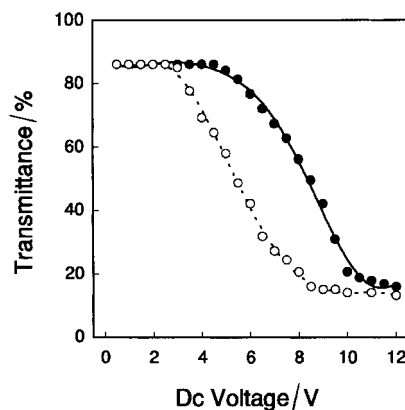


Figure 8. The electro-optical response of 5CB with PI (BTDA/DPM) as an alignment layer at 30°C : ● before photoirradiation; ○ during photoirradiation at 0.6 mW cm^{-2} . The pretilt angle of the cell was 4.3° .

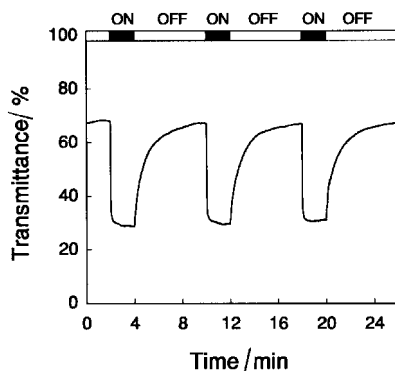


Figure 9. Optical switching of 5CB with PI (BTDA/DPM) as an alignment layer. The switching behaviour was examined at 30°C and photoirradiation was performed at 366 nm (light intensity, 0.6 mW cm^{-2}) with a d.c. bias voltage of 7 V.

of the excitation light at 366 nm. The change in the transmittance, however, was smaller than that of the NLC cell with PI (BTDA/DBP).

We next investigated the T - V profile of 5CB before and during irradiation with an aliphatic PI (BPDA/DN) alignment layer. In this case, the change in the T - V profile of the cell before and during photoirradiation was so small that we could obtain only a very small change in the transmittance as shown in figure 10.

It is worth mentioning here that when we used poly(vinyl alcohol), which has no absorption at 366 nm, as an alignment layer instead of the PIs shown in figure 2, no shift in the threshold voltage in the T - V profile was observed before and during photoirradiation at 366 nm. Furthermore, we observed no optical switching behaviour with the poly(vinyl alcohol) alignment layer. This control experiment clearly indicates that the PIs as alignment layers are responsible for the observed optical switching phenomena.

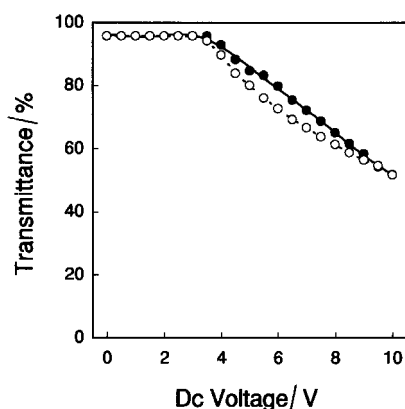


Figure 10. The electro-optical response of 5CB with PI (BPDA/DN) as an alignment layer at 30°C: ● before photoirradiation; ○ during photoirradiation at 0.6 mW cm^{-2} .

3.2. Analysis of PI films exposed to UV light

In order to discuss the mechanism of the photoinduced reversible change in alignment of the NLC molecules, the change in the PI (BTDA/DPM) surface components before and after irradiation at 366 nm was measured with ESCA. As shown in the table, the PI surface exposed to light (light intensity, 0.6 mW cm^{-2}) remained unchanged until $\sim 1 \text{ h}$ and then gradually changed to a more polar state as the light intensity increased. The nitrogen atom hardly changed within the range of the light energy examined. This result indicates that a photochemical reaction occurred on the PI surface brought about by high energy levels of irradiation [21–24]. In fact, Wang *et al.* reported that a chemical change of PI was caused by linearly polarized light of a long wavelength at 366 nm [25, 26]. However, the energy necessary to induce the optical switching of 5CB in this study was small ($\sim 0.6 \text{ mJ cm}^{-2}$) and it was not absorbed at the surface of the PI under the present experimental conditions which are unlikely to have caused any appreciable photochemical change to the surface of the PI when optical switching was induced by light. The change in alignment might not involve a photochemical process, but might occur mainly by photophysical change of the PI surface. To confirm this, we investigated the changes of the surfaces of the other PIs after irradiation. PI (BTDA/DBP), which exhibited the largest absorption at 366 nm among the PIs used, also showed a similar behaviour to that of PI (BTDA/DPM), as shown in the table, where the surface components are hardly seen to change on photoirradiation at 0.6 mW cm^{-2} for 1 h. The aliphatic PI (BPDA/DN) exhibited little change in the surface within the range of the light energy examined (at $\sim 5 \text{ mW cm}^{-2}$ for 4 h).

Figure 11 shows the UV-vis and fluorescence spectra of the PIs. Aromatic PIs exhibited distinct fluorescence

Table. ESCA atomic compositions of PI (BTDA/DPM) and PI (BTDA/DBP) films after photoirradiation at various light intensities.

Film	Atomic concentration/%		
	C	O	N
PI (BTDA/DPM)			
unexposed	80.9	13.9	5.2
exposed to light at 366 nm			
at 0.6 mW cm^{-2} for 1 h	80.8	13.8	5.4
at 0.6 mW cm^{-2} for 10 h	78.1	17.6	4.3
at 5 mW cm^{-2} for 4 h	74.5	20.3	5.2
PI (BTDA/DBP)			
unexposed	77.9	17.4	4.7
exposed to light at 366 nm			
at 0.6 mW cm^{-2} for 1 h	77.8	17.4	4.8

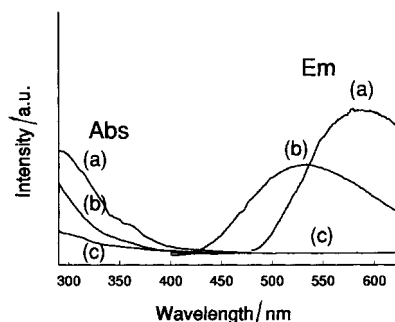


Figure 11. The UV-vis absorption and fluorescence spectra of PIs: (a) PI (BTDA/DBP); (b) PI (BTDA/DPM); (c) PI (BPDA/DN). Abs and Em denote absorption and emission spectra, respectively; excitation wavelength, $\lambda_{ex} = 366$ nm.

due to the formation of charge transfer (CT) complex resulting from interaction between electron-donor (diamine) moieties and electron-acceptor (dianhydride) moieties [27–30]. An imide ring of the PIs is known to have a low-lying π^* orbital that acts as a good electron acceptor in the CT process [31]. PI (BTDA/DBP) has contiguous π donor and acceptor moieties; therefore, a possibility for the CT process exists upon excitation at 366 nm. In contrast, in PI (BPDA/DN) a saturated methylene spacer replaces the phenyl ring, and therefore no π donor orbitals are available to take part in the CT process. In PI (BTDA/DPM), a methylene spacer separates the donor and the acceptor, and this may partially inhibit the CT process. Figure 11 shows fluorescence spectra of three PIs obtained by excitation at 366 nm, which was the same excitation wavelength as that for optical switching. At 366 nm, however, the absorbance of each PI was different: 0.20 for PI (BTDA/DBP), 0.07 for PI (BTDA/DPM) and 0.04 for PI (BPDA/DN). We next measured the fluorescence spectra of the PIs at excitation wavelengths where the three PIs showed the same value of the absorbance (0.20): 366 nm for PI (BTDA/DBP), 327 nm for PI (BTDA/DPM) and 285 nm for PI (BPDA/DN). It was found that the fluorescence spectra observed were similar to those shown in figure 11.

Since Dine-Hart and Wright originally proposed that CT may occur in PIs [27], many researchers have reported that upon excitation the CT process occurred due to electron transfers from the donor to the acceptor [27–32]. Furthermore, they used the CT theory to explain a number of properties of PIs such as fluorescence [33–35], photoconductivity [36–38], colour and ordering [39]. Such changes in the properties of the PIs induced by the CT processes might affect the optical switching behaviour of the NLC cell, but the mechanism of the optical switching of 5CB is unclear at present. Further work is necessary to understand this phenomenon fully.

4. Conclusions

We found a novel driving method for a NLC using PIs having photosensitive moieties as an alignment layer by irradiation at 366 nm. The photosensitive PI alignment layer induced the optical switching of 5CB. The alignment of the NLC was altered from a homogeneous state to a homeotropic state on photoirradiation with a d.c. electric field as a bias. The optical switching could be repeated by on and off switching of the light and its behaviour was dependent on the structure of the PIs and temperature. Based on the following results, this photoinduced reversible change in alignment might not be due to photochemical change, but mainly to photo-physical change in the PI surface. First, the light energy used in this study caused no appreciable chemical change of the PI surface on irradiation. Second, the alignment of 5CB was reversibly changed by the light. This new method, the change in alignment of a NLC brought about by light acting on photosensitive PIs as the alignment layer, might be applied to optical switching devices of NLCs.

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