

Photo-alignment of Low-molecular Mass Nematic Liquid Crystals on Photochemically Bifunctional Chalcone-epoxy Film by Irradiation of a Linearly Polarized UV Light

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Photocrosslinkable chalcone-epoxy compound comprising 1,3-bis-(4-hydroxy-phenyl)-propenone was synthesized for fabricating the photo-alignment layer of liquid crystals. Chalcone group was introduced into the main chain unit of the epoxy oligomer. We observed a photodimerization behavior and an optical anisotropy of this material by irradiation of a linearly polarized UV(LP-UV) light. With a trace amount of cationic photoinitiator (TRS-HFA), polymerization of epoxy groups was also conducted at the similar wavelength range used for photodimerization. Linearly polarized UV irradiation on the chalcone-epoxy films with cationic photoinitiator induced optical anisotropy of the film and the resultant film can be used for alignment layers for low molecular weight nematic liquid crystals.

Keywords : Optical anisotropy, Chalcone-epoxy, Photodimerization, Photopolymerization, Liquid crystal alignment.

Introduction

Conventional rubbing technique in practical application has been widely used for the liquid crystal alignment. But, it has several problems such as dust and static charge due to the mechanical contact of rubbing cloth, causing mechanical damage of active display layer. Moreover, the general method to achieve high performance for liquid crystal device (LCD) applications requires surface anchoring conditions that cannot be induced by a traditional mechanical rubbing method. Therefore, the photo-alignment technique for LC display has received much attention in recent years because of its clean and photo-patternable system.¹⁻⁷ Many kinds of photoreactive polymers that can undergo anisotropic isomerization or photocrosslinking reactions have been reported for the photoalignment of LC.³⁻⁹ Nematic liquid crystal (NLC) can be aligned either homogeneously or homeotropically on the photoreactive polymer film which is usually exposed to a linearly polarized light (LPL).¹⁰⁻¹³

For example, anisotropic [2+2] cycloaddition of polyvinylcinnamate (PVCi) film can induce irreversible low molecular weight-liquid crystal (LMW-LC) alignment.¹⁴

The polymers containing chalcone group in the side chain were also frequently studied for application to LC photo-alignment layer.¹⁵ The induction mechanism of optical anisotropy is identical with that in polyvinylcinnamate.

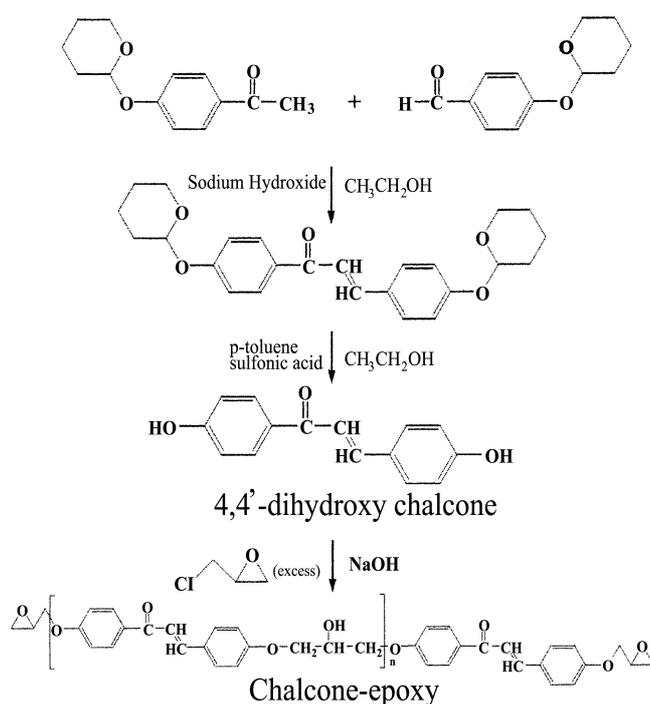
However, most of the photocrosslinkable side chain polymer showed that the optical anisotropy of the thin film increased at the initial stage of UV irradiation and gradually diminished under a continuous irradiation.¹⁶ If the optical anisotropy continue to increase under a continuous UV

irradiation, LC alignment can be observed to quite stable. In order to overcome these problems, we designed and synthesized the photoreactive main chain oligomer bearing the chalcone group in the repeating unit and epoxy groups in the terminal ends.

Among many kinds of photochemical reaction, photopolymerization under UV light has also been highlighted in practical applications. The photoinitiated cationic ring-opening polymerization of epoxy compounds has been widely investigated.¹⁷⁻¹⁹ Among many promising photosensitive groups, a chalcone group has been well studied and can be used in photocrosslinkable polymers because it affords high sensitivity to UV radiation ($\lambda = 300-360$ nm).²³ Because of poor solubility arising from the rigid-rod nature of chalcone backbone polymers, however, most research works involved only polymers that contain the chalcone unit in the side chain.²⁰⁻²²

In this work, we synthesized a new photoreactive chalcone-epoxy oligomeric compound. Chalcone group was introduced into the main chain unit of the new epoxy compound, which is highly sensitive to the LPL photochemically. This compound can show the photodimerization and photopolymerization behavior with photoinitiator which was described in our previous work.²³ The optical anisotropy of the film induced by irradiation of a linearly polarized UV light is also investigated by using the polarized UV absorption spectroscopy. The film sample that underwent photodimerization and photopolymerization showed that very high anisotropy could be achieved under a continuous UV irradiation. We also investigated the azimuthal orientation of LC molecules and the direction of LC alignment determined by monitoring the dichroism of a dichroic dye (Disperse blue 14) dissolved in nematic liquid crystals.

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Scheme 1. Synthetic procedure for the chalcone-based epoxy oligomer used in this study.

Experimental Section

Synthesis of chalcone-epoxy compound. Scheme 1 shows the chemical structure of the photosensitive material such as chalcone-based epoxy oligomer used in this study. Starting material, 1,3-bis-(4-hydroxy-phenyl)-propenone was synthesized according to the literature.²³ Chalcone-epoxy compound was synthesized by condensation reaction with 4,4'-dihydroxychalcone and epichlorohydrin.

4,4'-Dihydroxychalcone²⁵ (5 g, 0.02 mole) was dissolved in aqueous NaOH (1.76 g) solution (5 wt/vol%) under nitrogen purging. The epichlorohydrin (9.25 g, 0.1 mole) was added into the reaction mixture with stirring. The mother solution was kept at 80 °C for 12 hr. The solution was poured into tetrahydrofuran (THF) and was filtered for removing the solid. The THF solution was concentrated into a half volume of the initial one and the crude product was collected after precipitation into excess hexanes. The yellowish powder was filtered and dried under vacuum thoroughly. Yield, 80%

¹H-NMR (DMSO-d₆) δ (ppm) 2.65, 2.77 (tt, 2H in epoxy ring), 3.35 (t, 1H in epoxy ring), 3.65 (m, 1H in -CH₂-CH(OH)-CH₂-), 3.82-4.25 (m, 4H in -CH₂-CH(OH)-CH₂-), 7.00 (m, 4H in aromatic group), 7.35 (d, 1H in ethylenic -CH=CH-C(=O)-), 7.50 (d, 2H in aromatic group), 7.65 (d, 1H in ethylenic -CH=CH-C(=O)-), 8.05 (d, 2H in aromatic group).

Characterization

UV-Vis absorption spectroscopy: Absorption spectra were obtained using a Hewlett Packard UV-Vis 8453 spectrophotometer (PDA type, $\lambda = 190$ -1100 nm). The epoxy resin with cationic photoinitiator was dissolved in THF and

cast onto quartz plate followed by drying in vacuo at 50 °C for 2 hr. All the epoxy films on quartz plates were irradiated with a 1 kW high-pressure mercury lamp (Nanotek Co.) equipped with bandpass filter ($\lambda = 300$ -360 nm) and a liquid optical cable. The distance between the probe and the sample was set to be 5 cm. The intensity was measured to be 26 mW/cm² by using a 13PEM001 (MELLES GRIOT) detector and power meter.

For the polarized absorption spectroscopic study to investigate the order parameter during UV illumination, we equipped Glan-Taylor polarizer in front of the probe beam in the spectrophotometer. For irradiation of a LPL, we employed a linearly polarized He-Cd laser (Liconix 3675NX, $\lambda = 325$ nm, $I = 210$ mW/cm²).

NMR spectroscopy: Proton NMR spectrum was recorded with Varian 200 NMR spectrometer. DMSO-d₆ was used as a solvent for recording NMR spectra. Degree of polymerization, n of the chalcone-epoxy oligomeric compound was determined by using peak integration method.

Photoirradiation: In order to make thin polymer films for LC alignment layer, 0.5 wt% solutions of the chalcone-epoxy compound were prepared with tetrachloroethane, filtered using 0.2 μ m PTFE membrane filter (Acrodisc) and were spin-coated on quartz plate and borosilicate glass.

Light from high pressure Hg lamp (Nanotek Co. 1 kW) was passed through IR filter (59341, Oriel Co.) and broad bandpass filter ($\lambda = 300$ -360 nm) (UG 11, Melles Griot) to isolate the certain wavelength range of the light. A UV linear polarizer (27320 UV linear dichroic polarizer, Oriel Co.) was used for linearly polarized light irradiation.

Determination of LC photoalignment: A LC cell was assembled by sandwiching nematic liquid crystals between

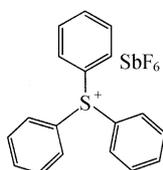
two glass plates spin coated with chalcone-epoxy compound. Photoirradiation of chalcone-epoxy film was performed before LC cell fabrication. The NLC (5CB, Merck Co.) was filled into a cell in its isotropic state by a capillary action. In order to eliminate the capillary effect, the cell was heated to 42 °C above T_{NI} (38 °C) and filled into the cell. The extent of the LC photo-alignment was determined by measuring the transmitted light intensity of a linearly polarized light from He-Ne laser ($I = 1 \text{ mW/cm}^2$) through the LC cell as a function of the cell rotation angle under crossed polarizers. The direction of LC alignment was determined by monitoring polarized absorbance of a dichroic dye (Disperse blue 14) dissolved in 5CB rotating the sample under single polarizer.

Results and Discussion

Synthesis and characterization: Synthetic procedure was simply illustrated in Scheme 1. New photoreactive epoxy oligomer contains unsaturated carbonyl moieties as photosensitive groups in the main chain. 4,4'-Bis(4-tetrahydropyran-2-yloxy) chalcone was prepared by reacting 4-tetrahydropyran-2-yloxy benzaldehyde with 4-tetrahydropyran-2-yloxy acetophenone in ethanol/water mixture in the presence of sodium hydroxide.²⁵ Then using p-toluene-sulfonic acid, we removed the pyran group from the HO-protected chalcone compound to obtain 4,4'-dihydroxy-chalcone. This procedure afforded 4,4'-dihydroxychalcone in a good yield (~70%).

The feeding ratio of 4,4'-dihydroxychalcone and epichlorohydrin was selected to be 1 : 5 in the presence of aqueous NaOH (5 wt/vol%) solution. The degree of polymerization, n was determined to be around 5.0-5.5 by peak integration in NMR spectrum.

UV-Vis absorption spectroscopic study in photocross-linking reaction: The chalcone-epoxy film was fabricated using the mixed solution of chalcone-epoxy compound and cationic photoinitiator that is triarylsulfonium hexafluoroantimonate (TRS-HFA). The structure of the photoinitiator is as follows:



Triarylsulfonium hexafluoroantimonate (TRS-HFA)

The photoirradiation of the epoxy film with cationic photoinitiator (4 wt%) was carried out by using a randomly polarized light from a 1 kW high pressure mercury lamp equipped with a bandpass filter (UG 11, Melles Griot) and IR cut off filters. The light intensity was measured to be 2.0 mW/cm^2 . The UV-Vis absorption spectra of the thin film of the chalcone-epoxy compound are given in Figure 1. The spectra were recorded during UV irradiation simultaneously.

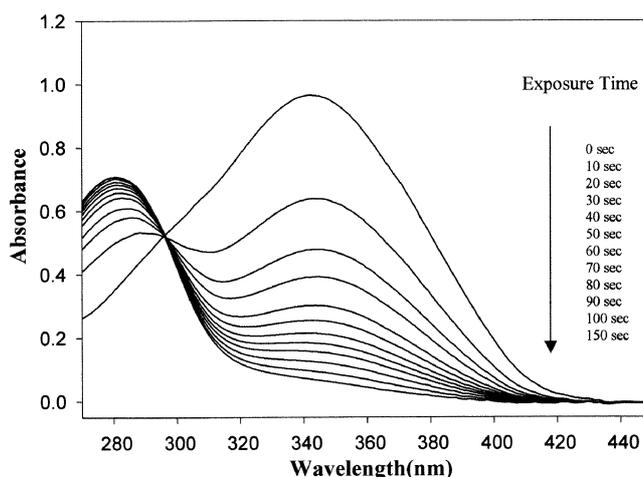


Figure 1. Absorption spectral change of the chalcone-epoxy film irradiated with random polarized UV light at room temperature.

The UV absorption changes at λ_{max} in the epoxy compound caused by photoreaction were investigated using thin films on quartz plate. Chalcone unit undergoes a crosslinking reaction by [2+2] cycloaddition between a UV excited chalcone group and an unexcited chalcone group (ground state) on another. The maximum absorbance at $\lambda = 341 \text{ nm}$ due to the π -electronic conjugation decreased rapidly. Although the photoisomerization is possible to be observed, the photodimerization was predominantly performed under a prolonged UV irradiation. The photopolymerization through epoxy group under cationic initiation and photocrosslinking behavior were well described in our previous results.²³

Polarized UV-Vis absorption spectroscopy under irradiation of a linearly polarized light: The films of chalcone-epoxy oligomeric compound with cationic photoinitiator showed anisotropic spectra after exposure to LPL from He-Cd laser. In Figure 2A, the absorption spectra were illustrated using the sample with photoinitiator. We can observe the change in UV absorption spectrum and dichroism. A_{\parallel} denotes the parallel absorbance and A_{\perp} perpendicular to electric field of the incident LP light. Exposure time dependence of the parallel and perpendicular absorbance at 341 nm was investigated during irradiation of the linearly polarized UV light from He-Cd laser. For this experiment, we prepared the sample films coated on borosilicate slide glass. The sample contained 4 wt% photoinitiator (TRS-HFA) of chalcone-epoxy compound, which dissolved in propylene carbonate (50 wt%). The decrease in the absorption at 341 nm was evident, which is attributed to the formation of cyclobutane rings through [2+2] cycloaddition of the carbon-carbon double bonds in chalcone moieties. (See Figure 2B)²³

The parallel absorbance at 341 nm was observed to be smaller than the perpendicular absorbance in the whole range of the irradiation time period. From the difference of UV absorbance, we could analyze the optical anisotropy of the thin film in terms of the difference of the absorbance and the order parameter.

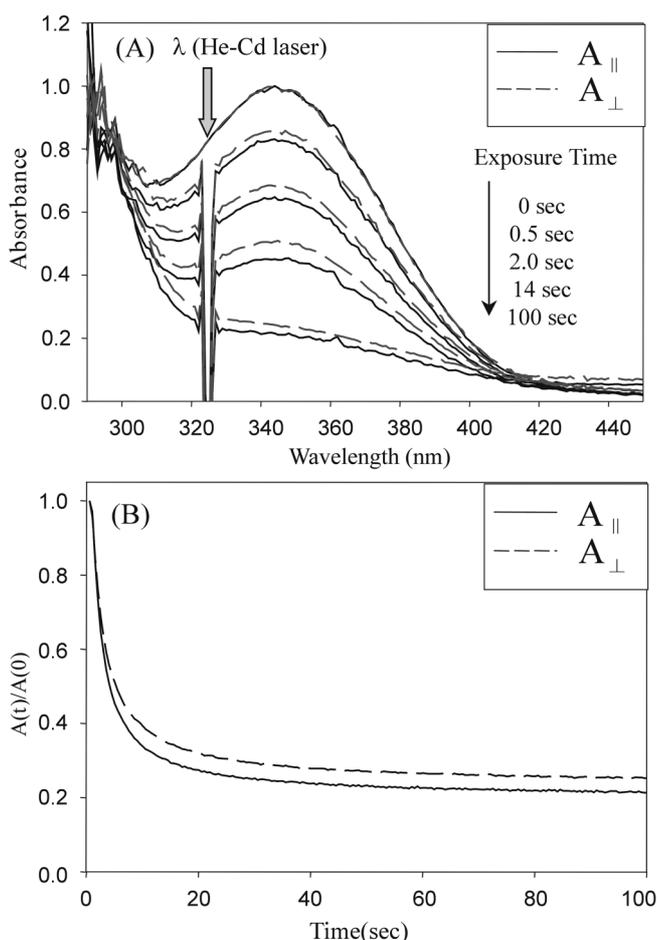


Figure 2. (A) Polarized absorption spectra of the chalcone-epoxy film with photoinitiator exposed to a linearly polarized UV light with the change of the irradiation time. (B) Change of polarized absorbance of chalcone-epoxy film with photoinitiator at 341 nm as a function of UV exposure time.

Negative dichroism and the difference of the absorbance between the parallel and perpendicular spectrum was again observed in these traces. In Figure 2B, normalized absorbance change in both parallel and perpendicular direction to the pump polarization direction was shown as a function of the exposure time. The difference of the absorbances was sustained even after long time exposure in the case of the sample with cationic photoinitiator.

We also could calculate the order parameters using the parallel and perpendicular absorption spectra. In Figure 3, exposure time dependence of the order parameter reflects the inducing optical anisotropy. The order parameter was calculated using the following equation (1).

$$\text{Order Parameter (O.P.)} = [A_{\parallel} - A_{\perp}] / [2A_{\parallel} + A_{\perp}] \quad (1)$$

The value from the sample with photoinitiator decreased quite fast at the initial stage of irradiation. Then, the following reaction was retarded slightly.

Assuming that the order parameter is mainly attributed to dimerized photoproducts and the polymerized epoxy groups

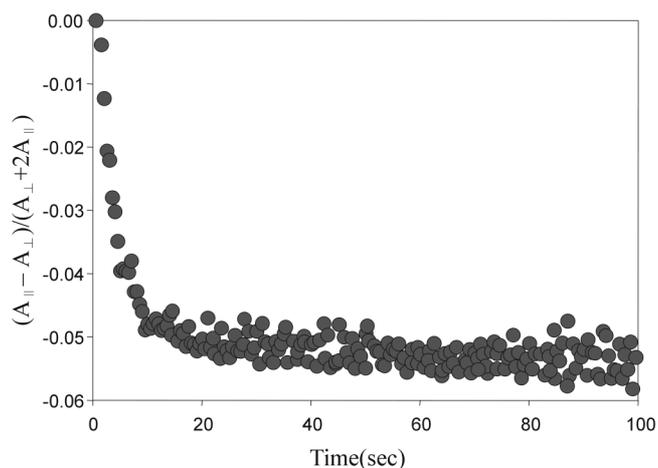


Figure 3. Exposure time dependence of the order parameter of the chalcone-epoxy film with cationic photoinitiator.

do not show anisotropic behavior due to 3-D network structure, the photopolymerization retards the photocrosslinking reaction for optical anisotropy. Most of the photocrosslinkable side chain polymer bearing the cinnamates or chalcone group showed that the molecular orderness is reduced as the power of the UV light goes higher.^{16,24} Although the irradiation continued longer subsequently, the order parameter of the chalcone-epoxy sample with photoinitiator decreased continuously to show high anisotropy.

The cationic photopolymerization used herein retards the photodimerization due to the geometrical hindrance from the polymerization through the end groups of the epoxide.²⁷ While the cycloaddition reaction was retarded, the optical anisotropy from the linearly organized molecular structure can be highly stabilized under our strategy.

Photocontrol of LC alignment: There are many reports that dealt with the LC alignment using the photoreactive side chain polymer containing cinnamate and chalcone group.^{3-9,14} The orientational direction of LC molecules caused by linearly polarized light irradiation of polymers with photoreactive side chains including azobenzenes, cinnamates, and benzylidenephthalimides is perpendicular to the electric field vector of actinic polarized light.²⁵ Thin films of chalcone-epoxy compound on a substrate plate were exposed to polarized UV light ($\lambda = 300-360$ nm) followed by the fabrication of LC cell using the above two plates coated with chalcone-epoxy compound.

Photoinduced azimuthal LC alignment was investigated by measuring transmitted intensity of a polarized He-Ne laser beam as a probe light passed through a LC cell as a function of the rotational angle of the cell. Irradiation with linearly polarized UV light resulted in the control of azimuthal orientation of LC for chalcone-epoxy compound as revealed by the regular appearance of periodicity in transmitted light intensity at 90° difference. As shown in Figure 4, the azimuthal orientation of LC layers was generated under exposure does of polarized UV light, 1.5 J/cm^2 . Minimum exposure dose of about 0.5 J/cm^2 is required for the generation of homogeneous alignment by chalcone-epoxy

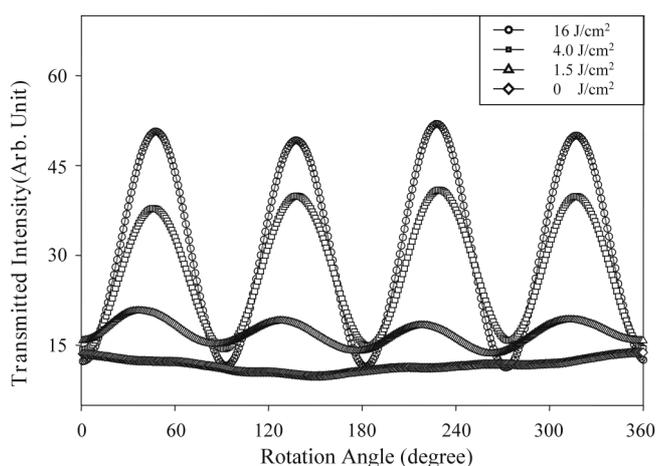


Figure 4. Angular dependence of transmitted light intensity of a linearly polarized He-Ne laser beam passed through the LC cell with the change of the intensity of the LP-UV light.

film. As the exposure energy increased, the homogeneous LC alignment was more clearly observed, as is usual. (See Figure 4).

Direction of LC alignment: The direction of LC alignment was determined by measuring the dichroic property of a dichroic dye (Disperse blue 14, 0.2 wt%) dissolved in 5CB. Thin films of chalcone-epoxy compound were exposed to linearly polarized UV light and used for cell assembly. We irradiated the thin film under 16 J/cm^2 of the UV light in order to maximize the induced optical anisotropy. On this alignment layer, the direction of LC alignment was observed to be parallel to the electric field vector of the LP-UV light.

Figure 5 illustrated the circular diagram of absorbance of the dichroic dye (Disperse blue 14) dissolved in 5CB (0.2 wt%) in an LC cell fabricated by using the chalcone-epoxy film with photoinitiator. The arrow indicates the electric field vector of the LP-UV light. In Figure 6, exposure energy

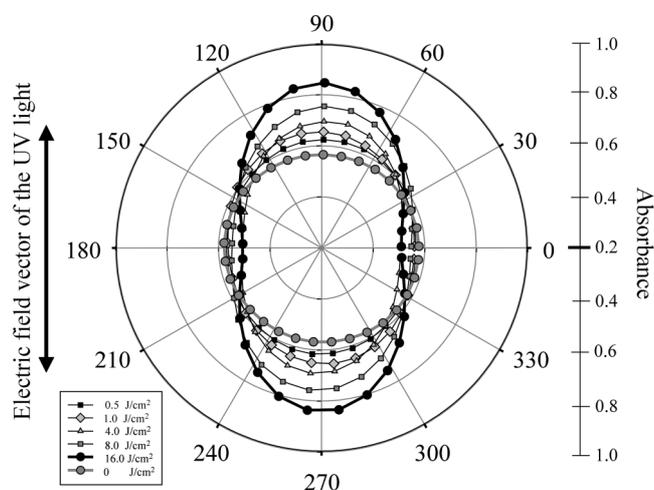


Figure 5. Circular diagrams of absorbance of the 0.2 wt% dichroic dye (Disperse blue 14) dissolved in 5CB with the change of the intensity of the LP-UV light. The electric field vector of the LP-UV light is shown by the arrow.

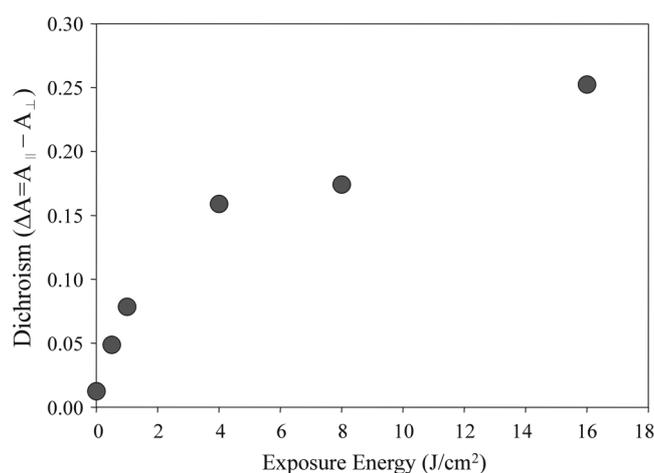


Figure 6. Exposure energy dependence of the dichroism of the LC cell.

dependence of the dichroism ($\Delta A = A_{||} - A_{\perp}$) was shown. From low energy (0.5 J/cm^2) to high energy (16 J/cm^2) of the UV light, the alignment was not changed to sustain the parallel LC direction to the electric field vector of the excitation light. This result is quite different from that in the alignment layer of side chain polymer as we described previously. The chalcone unit in the main chain of this compound showed unique behavior after photocrosslink to align LC molecules. As far as the photoalignment mechanism is concerned, Reznikov *et al.* proposed that parallel alignment results from photoproducts of cinnamates attached to polymer chain while perpendicular alignment is determined by E/Z photoisomerization of cinnamates in the side chain of the polymer.²⁵ The polymer containing the chalcone in the side chain could also show the very similar behaviors.

Obi *et al.* also suggested that the induction of optical anisotropy by photochemical reactions consists of two processes.²⁶ They include the angular-selective photochemical transformation of molecules with a transition dipole moment in parallel with the electric field vector of excitation light and the reorientation of the photoisomerizable groups, leading to the preferential displacement of molecules oriented perpendicularly to the electric field vector of the excitation light. It can be performed in the side chain polymer containing the chalcone group as a side chain unit. The chalcone moiety is rigid rod-shaped when it was tethered to the polymer backbone as a side chain pendent group. However, this is not the case of for chalcone-epoxy film which gives rise to a parallel LC photoalignment. It seems that molecular reorientation of chalcone group induced by linearly polarized light cannot take place. The possibility of the photoreorientation of chalcone unit in this compound is consequently excluded since chalcone group is tethered as a main chain repeating unit so that the molecular motion should be much more restricted. Particularly, the chain end of epoxy group was opened by cationic initiator to be polymerized to form a highly dense structure. The chalcone group was restricted to be E/Z photoisomerized because the two ends were tethered to the next repeating unit and the

chain ends were sewed after cationic photopolymerization. It follows that the LC photoalignment is determined by optical anisotropy generated by angular-selective photochemistry that results in the consumption of the chalcone group and the formation of dimerized photoproducts. Resulting from the photodimerized products, the strong van der Waals interaction between the cyclized photoproduct and liquid crystal molecules results in parallel alignment of liquid crystal molecules.

Although clear evidence has not been available yet by polarized infrared spectroscopic study precisely, it can be assumed that the parallel orientation of LC molecules is governed by the anisotropically photodimerized products of chalcone moieties. It is also assumed that the existence of chalcone unit as a repeating unit in the main chain oligomer plays an important role in determining the mode of photoalignment, although further studies are necessary to measure the dichroic ratio of the LP-UV exposed film of chalcone epoxy compound. Using the LC cell used in this study, we measured the pre-tilt angle with the two different exposure energies of UV irradiation. Under 4 and 16 J/cm², we could obtain 3.071° and 0.927° respectively. As the exposure energy increased, the pre-tilt angle was found to be lower.

Conclusion

We synthesized photo-crosslinkable chalcone-epoxy compound comprising 1,3-bis-(4-hydroxy-phenyl)-propenone. Cyclobutane structures were formed under UV irradiation in epoxy compound between the main chains. We clarified anisotropic LP-UV photoreaction of chalcone-epoxy compound using the polarized absorption spectroscopy. The dichroism of the photoreacted chalcone group was negative and very large order parameter was observed when using as-coated films bearing a photoinitiator. Homogeneous alignment by irradiation of chalcone-epoxy film with linearly polarized UV light was induced when the irradiation was carried out before cell assembly. The orientational direction of LC alignment was parallel to the electric field vector of the excitation light. It was determined by cyclized photoproducts from angular-selective photodimerization and photopolymerized products through epoxy groups in the chain ends.

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