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Photoalignment of liquid crystals by a covalently attached self-assembled ultrathin film

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In this paper, the polyanion-containing cinnamoyl group (PACSS-CF₃) was self-assembled with diazo-resin (DR) to form a kind of stable covalent ultrathin film by irradiation with 365 nm UV light. The photoalignment properties of the DR/PACSS-CF₃ covalent film were investigated. The covalent film was found to have anisotropy after irradiation by 297 nm linearly polarised ultraviolet light (LPUVL), and could induce uniform alignment of liquid crystals (LCs). The pretilt angle of the LC was 2.5°. The stability of the film was enhanced by the covalent bonds. The films were thermally stable to 180°C. Polarised UV-Vis spectroscopy was utilised to investigate the photochemical process of the covalent film. It was found that cinnamoyl moieties parallel to the polarisation direction of the LPUVL were consumed by the photoreaction faster than those perpendicular to the polarisation direction. It can be concluded that the selective photoreaction induced the anisotropy of the films. The anisotropic films induced the homogeneous alignment of LC.

Keywords: covalent ultrathin film; photoalignment; liquid crystal; pretilt angle

1. Introduction

There has been much interest in the photoalignment of liquid crystals (LCs), a promising candidate to replace mechanical rubbing technology [1–3]. In conventional photoalignment technology, spin-coated films of cinnamate materials, whose photoaligning mechanism was first described by Schadt *et al.* [4, 5] and differed fundamentally from the azo-photo-alignment process of Ichimura *et al.* [6], have been widely used to fabricate films for investigating alignment properties, which are most suitable for the durable LC photoalignment film because of the irreversible [2+2] photodimerisation of cinnamoyl groups [4, 7, 8]. However, the cinnamate polymers are random in this kind of film, which affects the photoreaction of cinnamate and results in a lower photoreaction ratio. Also, the thermal stability of the photoalignment film is not sufficiently strong. Both factors will limit the application of the photoalignment technique in industry. Our previous investigations showed that ordered cinnamate layer-by-layer (LBL) self-assembled ultrathin films as photoalignment films can readily overcome these obstacles [9, 10]. LBL assembly of oppositely charged polyelectrolytes is a simple and powerful method for the construction of composites that are self-assembled planar ordered on a nanometre scale. Since the photosensitive cinnamate is oriented in the LBL films, the [2+2] photoreaction can proceed easily. The Coulombic interaction between layers also enhances the film's stability, as compared with the

van der Waals force in spin-coated films. However, the force between layers in such films is based on Coulombic interaction, which may be insufficient to stabilise the films in certain cases. The pretilt angle of LCs on previous LBL self-assembled films was under 1°, which is not enough to prevent LC displays from reverse tilt disclinations upon exerting an external electric field.

The purpose of this study was to fabricate a new LC photoalignment film that possesses a large pretilt angle and good stability. In this work, new covalently attached LBL self-assembled ultrathin films were prepared for photoalignment studies. The ultrathin films were fabricated using diazo-resin (DR) as polycation and poly [2-Trifluoromethyl-4-sulfonate-phenyl 4-acryloxycinnamate sodium salt] (PACSS-CF₃) as polyanion (chemical structure as illustrated in Figure 1), in aqueous solution via electrostatic attraction. The ionic bonds between the diazonium ions and sulfonate ions in the film were converted into covalent bonds by irradiated with 365 nm UV light. The prepared covalently attached self-assembled ultrathin film became anisotropic because of the axially selective photoreaction of cinnamate in the film after irradiation with 297 nm linearly polarised ultraviolet light (LPUVL). The product film could align LCs uniformly. The pretilt angle could reach 2.5°. The stability of the film was also enhanced by the linkage conversion of the DR/PACSS-CF₃ layers from ionic to covalent bonds.

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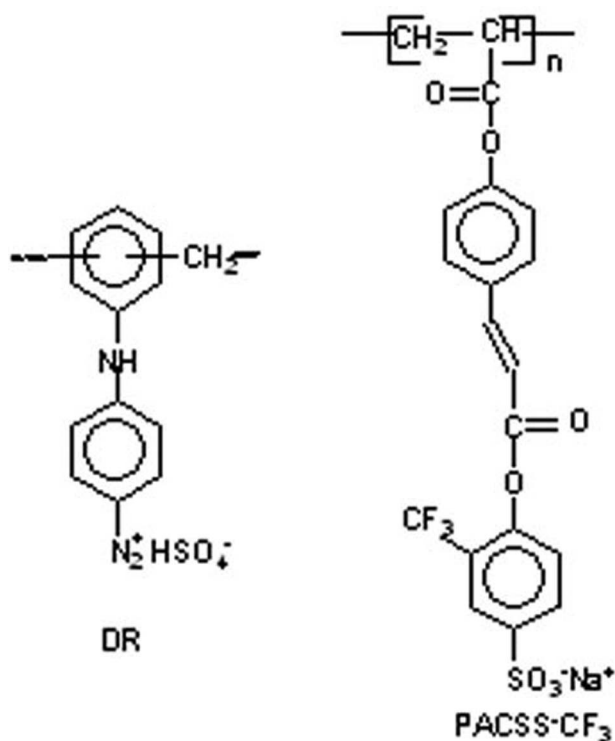


Figure 1. Chemical structures of diazo-resin (DR) and poly [2-trifluoromethyl-4-sulfonate phenyl 4-acryloyloxy cinnamate sodium salt] (PACSS- CF_3).

2. Experimental details

2.1 Synthesis

DR was synthesised from polyformaldehyde and diphenylamine-diazonium salt in concentrated sulphuric acid, $M_n \approx 2000 \text{ g mol}^{-1}$, $\eta_{sp/c} = 0.12 \text{ g dL}^{-1}$ [11]. 2-Hydroxybenzotrifluoride and p-hydroxycinnamic acid were purchased from Aldrich Co. and used without further purification. The polyanion (PACSS- CF_3) was synthesised according to the reaction scheme shown in Figure 2. All reagents were obtained from Shanghai Chemical Inc., and used directly. Dry solvents were freshly distilled under anhydrous conditions.

2.1.1 3-trifluoromethyl-4-hydroxy benzenesulfonic acid sodium salt

In a flask, 2-Hydroxybenzotrifluoride (3.24 g, 0.02 mol) was melted. Then, concentrated sulphuric acid (6 mL, 0.1 mol) was added. The mixture was stirred at 100°C for 36 h. After cooling to room temperature, the reaction mixture was neutralised with Na_2CO_3 . The crude product was filtered off and recrystallised in alcohol. Yield: 30%. FTIR (powder,

KBr, cm^{-1}): 1029 (S=O,v), 1205 (S=O,v), 1345 ($-CF_3$,v), 3584 (O-H,v).

2.1.2 2-Trifluoromethyl-4-sulfonate-phenyl 4-acryloyloxy cinnamate sodium salt (ACSS- CF_3)

4-acryloyloxy cinnamic acid (shown in Figure 2) was synthesised according to the literature method [10]. 4-acryloyloxy cinnamic acid (4.36 g, 0.02 mol) and 5 mL thionyl chloride (freshly distilled) were heated under reflux in 25 mL toluene for 4 h, with the addition of a few drops of DMF as catalyst. The resultant solution was cooled, and needle-like crystals of 4-acryloyloxy cinnamic acid chloride were produced. After filtering, the crude product was recrystallised from dry toluene.

In a 125 mL flask, 3-trifluoromethyl-4-hydroxybenzenesulfonic acid sodium salt (2.64 g, 0.01 mol) and 4-acryloyloxy cinnamic acid chloride (2.6 g, 0.011 mol) were dissolved in 50 mL THF, and cooled to $0-5^\circ\text{C}$. Triethylamine (1.111 g, 0.011 mol) was then added dropwise with stirring at the same temperature. After stirring at room temperature for 24 h, some of the solvent was removed by vacuum distillation. The precipitated was filtered and washed with ice water. The crude product was purified via column chromatography. THF was used as the solvent. Yield: 33%. FTIR (powder, KBr, cm^{-1}): 1058 (S=O, v), 1169 (C-O, v), 1214 (S=O, v), 1403 ($-CF_3$, v), 1633 (C=C, v), 1688 (C=O), 1739 (C-O) cm^{-1} .

2.1.3 Poly [2-Trifluoromethyl-4-sulfonate-phenyl 4-acryloyloxy cinnamate sodium salt] (PACSS- CF_3)

ACSS- CF_3 (1 g) was polymerised in 20 mL THF with 0.02 g AIBN as the initiator at 65°C under nitrogen atmosphere. The polymers were isolated after polymerisation for 12 h by adding the reaction solution to an excess of petroleum ether, purified by reprecipitation from THF into petroleum ether, and dried under vacuum. Yield: 50%. FTIR (powder, KBr, cm^{-1}): 1169 (C-O, v), 1214 (S=O, v), 1407 ($-CF_3$, v), 1633 (C=C, v), 1697 (C=O), 1739 (C-O) cm^{-1} .

2.2 Fabrication of covalently attached films

The films were constructed on quartz wafers for UV-Vis measurement, on CaF_2 slides for IR spectroscopy and on indium tin oxide (ITO) glass for the assembly of LC cells. The quartz slides were treated in fresh piranha solution (v/v = 1:3, 30% H_2O_2 : 98% H_2SO_4) for 1 h, then carefully washed with deionised water and dried. ITO glass slides were cleaned by washing with deionised water. The prepared substrates were

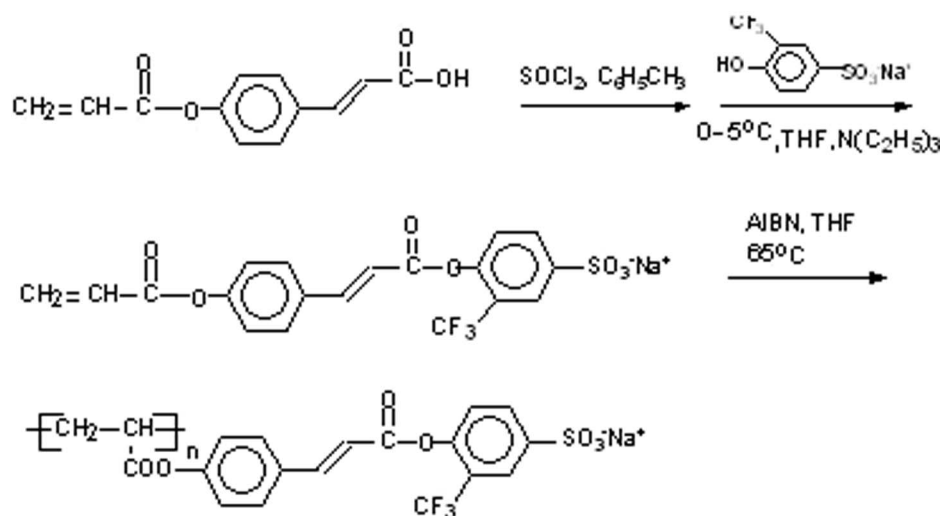


Figure 2. Synthesis of poly [2-trifluoromethyl-4-sulfonate phenyl 4-acryloyloxy cinnamate sodium salt] (PACSS-CF₃).

then alternately immersed in aqueous solution of DR (0.1 mg/mL) and PACSS-CF₃ (0.1 mg/mL) for 10 min each, with deionised water rinsing and nitrogen gas blow-drying between each deposition, according to well-reported procedures [12]. In all cases, DR was the first layer and PACSS-CF₃ was the last layer (outermost layer). All fabrication processes were performed in darkness. The DR solution and films were also kept in the dark before the irradiation. The well-fabricated films were exposed to 365 nm UV light for a given time to ensure the photoreaction proceeded completely, which changed the linkage between the layers of these films from ionic to covalent. The covalently attached film was prepared.

2.3 Fabrication of LC cells

The covalently attached films were obliquely irradiated by 297 nm LPUVL with an incident angle of 30°, schematically depicted in Figure 3. A pair of substrates with photoirradiated covalently attached films was assembled together in the anti-parallel direction of LPUVL irradiation by using 20 µm spacers. Commercial LC material TEB30A (Slichem Co., China, $T_c = 61^\circ\text{C}$) was injected into cell at 71°C. The LC cell was then cooled slowly to room temperature, to remove any flow-induced memory which had been induced by the LC injection process.

2.4 Instrumentation and measurements

The films were irradiated using a 300 W high-pressure Hg-Xe lamp system with glass band-pass filters (297 and 365 nm for UV light) and a Glan-Taylor lens. The exposure dose was measured using

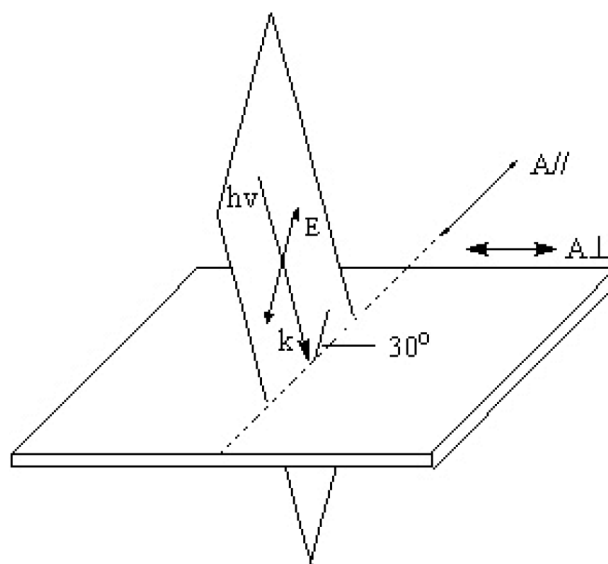


Figure 3. Schematic illustration of oblique photoirradiation with linearly polarised ultraviolet light.

a photometer with a sensor. The intensity of 365 nm UV on the surface of the film was 0.23 mW/cm² (at 365 nm), and the intensity of LPUVL on the surface of the film was 0.58 mW/cm² (at 297 nm). UV-Vis absorption spectra were obtained using Shimadzu UV-3101PC spectrophotometer. Polarised UV-Vis absorption spectra were recorded on the Shimadzu UV-3101PC spectrophotometer equipped with special polarising accessories. IR spectra were carried out on a Bio-Rad FTS3000 spectrophotometer. Polarising microscopy (FOIC-2, China) with a digital camera was also used to evaluate the alignment quality of the LC, and to measure the transmittance when the

polarising directions of two polarisers were crossed. The pretilt angles were measured using the crystal rotation method [13].

3. Results and discussion

3.1 Covalently attached self-assembled ultrathin film formation

The prepared films were clear, transparent and uniform, with good optical quality as seen by the naked eye. The LBL deposition of polyelectrolyte DR/PACSS-CF₃ pair-layers was investigated by UV-Vis absorption spectroscopic measurement. Figure 4 shows the UV-Vis absorption spectra of DR/PACSS-CF₃ film on quartz slide with increasing

number of bilayers. The absorption spectra displayed two broad bands centred at 380 nm and 290 nm. The band at 380 nm was due to the diazonium group of the diazoresin, and the band at 290 nm belonged to the π - π^* absorption of the double bonds conjugated with the phenyl group of the cinnamoyl group. The adsorption occurred essentially uniformly. The absorbance of the two peaks increased linearly with the number of DR/PACSS-CF₃ pair-layers. The linear increase of the two peaks with the number of bilayers clearly indicated the regular growth of the DR/PACSS-CF₃ layers, the same amount being deposited on each cycle. The π - π^* absorption of the double bonds conjugated with the phenyl group of the cinnamoyl group in the film was red-shifted

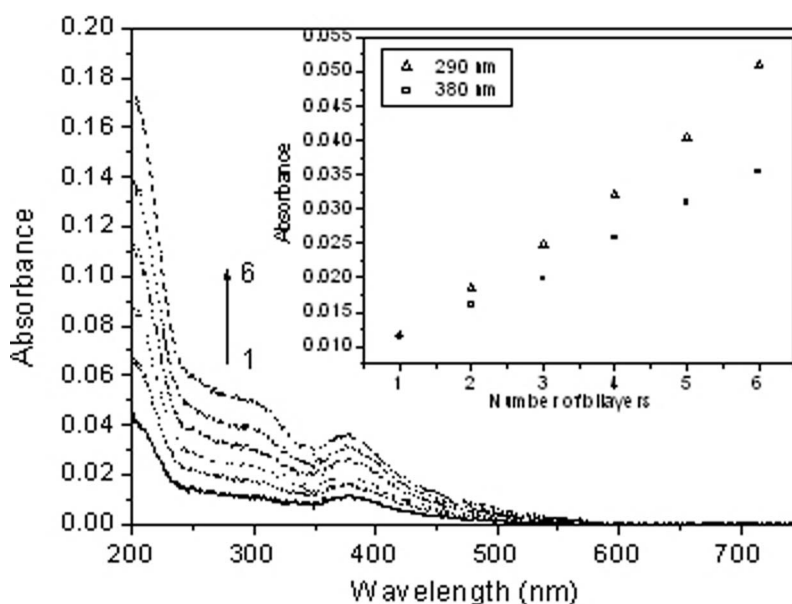


Figure 4. UV-Vis absorption spectra of DR/PACSS-CF₃ films on quartz wafer as a function of the number of bilayers. The inset diagram shows the increase of absorbance at 290 nm and 380 nm with the number of bilayers.

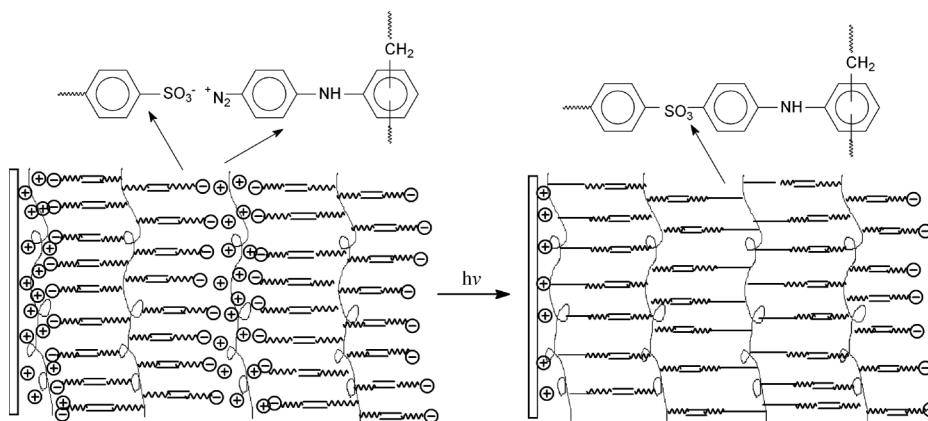


Figure 5. Self-assembly DR/PACSS-CF₃ multilayer films: schematic illustration and conversion of the interlayer linkage from ionic to covalent under UV irradiation.

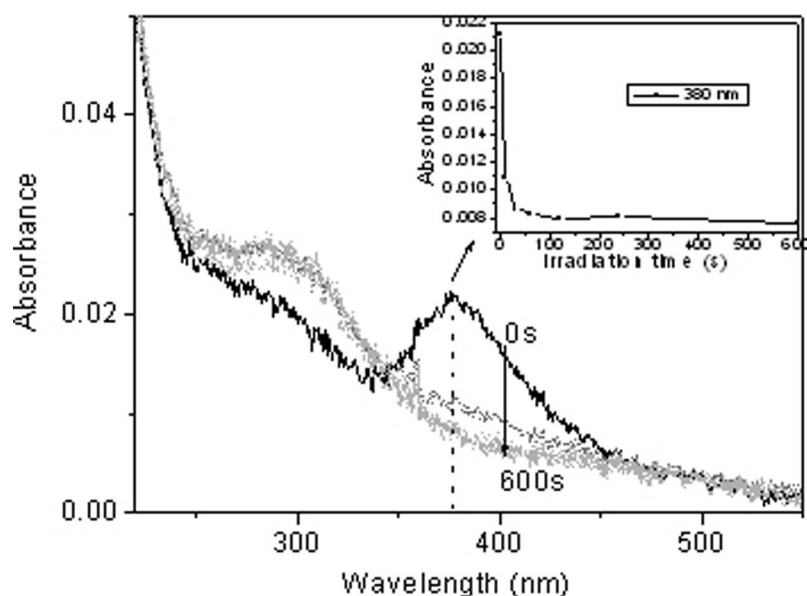


Figure 6. UV-Vis absorption spectra of three-bilayer DR/PACSS- CF_3 film under 365 nm UV irradiation for different time. Irradiation time (s) (from top to bottom): 0, 10, 30, 60, 120, 240 and 600. Irradiation intensity (at 365 nm): 0.23 mW cm^{-2} . The inset plot shows the decrease of absorbance at 380 nm with the irradiation time.

7 nm compared with the 1 mg/ml PACSS- CF_3 solution ($\lambda_{\text{max}} = 283 \text{ nm}$). The red-shift behaviour of the DR/PACSS- CF_3 film showed the formation J-aggregates in each layer where the orientation of the cinnamoyl groups could be described as end-to-end [14]. In contrast to the solution, the moieties were not independent from each other in the films.

The polycation DR and cinnamate polyanion were linked by electrostatic interaction between the cationic diazonium group ($-\text{N}_2^+$) and the

anionic $-\text{SO}_3^-$ to form a LBL ultrathin film. The DR was a photosensitive polymer and decomposed readily by UV irradiation [15, 16]. The ionic bond between $-\text{N}_2^+$ and O^- could be easily transformed into a covalent bond under UV light (shown in Figure 5). This improved the stability of the film. Figure 6 shows the changes by means of UV-Vis spectra of the films after 365 nm UV irradiation. After 365 nm UV irradiation, the absorbance at 380 nm decreased rapidly, which indicated diazonium group

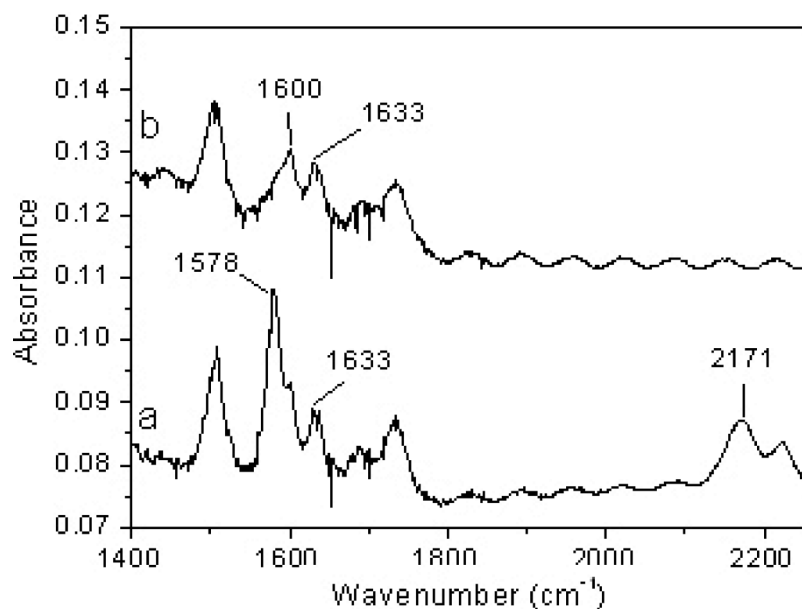


Figure 7. IR spectroscopy of a 100 bilayers DR/PACSS- CF_3 film on a CaF_2 substrate (a) before and (b) after UV irradiation. Irradiation intensity (at 365 nm): 0.23 mW cm^{-2} . Irradiation time: 1 min.

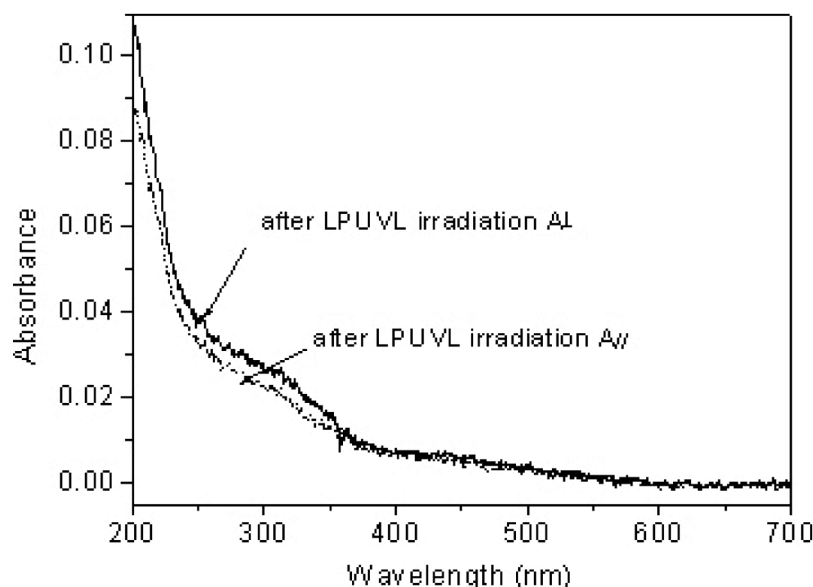


Figure 8. Polarised UV-Vis absorption spectra of three-bilayer covalent film of DR/PACSS-CF₃ irradiated with 297 nm linearly polarised ultraviolet light for 10 min. Irradiation intensity (at 297 nm): 0.58 mW cm⁻².

decomposition. The absorbance at 300 nm increased simultaneously, and an isosbestic point at 340 nm appeared in accordance with the results from previous investigations with DR as a component in planar films [16]. The decomposition was complete within 1 min. In order to further confirm this reaction, films of 100 bilayers of DR/PACSS-CF₃ were constructed on CaF₂ slides, and IR spectra were recorded before and after irradiation. As shown in Figure 7, it was found

that the peak at 2171 cm⁻¹ (the asymmetric stretching vibration of the diazonium group) disappeared completely after 365 nm UV irradiation for 1 min, which implies that the diazonium group of DR/PACSS-CF₃ film decomposes under UV irradiation. At the same time, the absorbance at 1580 cm⁻¹ corresponding to the vibration of the benzene ring conjugated with an unsaturated group in DR/PACSS-CF₃ film shifted to 1600 cm⁻¹, which was the normal absorption of

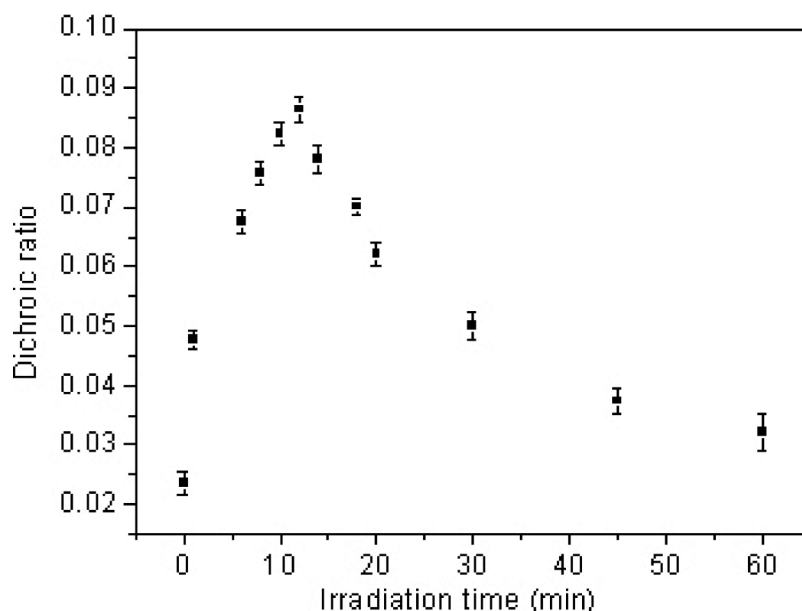


Figure 9. Change in dichroic ratio at 290 nm of a six-bilayer covalent DR/PACSS-CF₃ film irradiated with linearly polarised ultraviolet light as a function of the irradiation time.

the benzene ring. The band at 1633 cm^{-1} due to the vinylenic C=C of the cinnamoyl group stretching vibration has no obvious change. Thus the ionic bond between diazonium and sulfonate group converted to a covalent bond by the first irradiation, whereas the photosensitive double bond in the PACSS-CF₃ did not change. Covalently attached self-assembly films containing photosensitive cinnamoyl group have been fabricated.

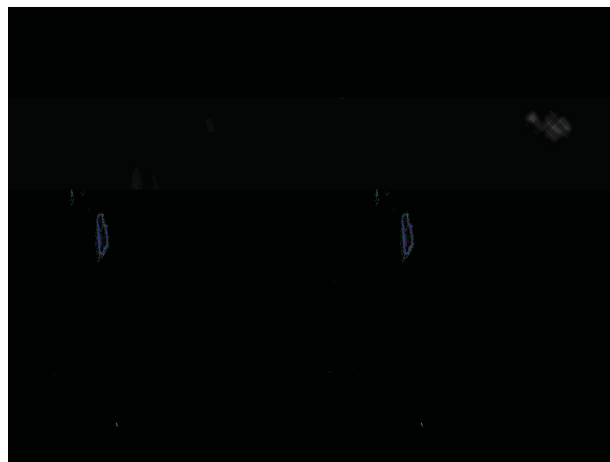
3.2 Anisotropic photoreaction in covalently attached self-assembled ultrathin film

Covalent self-assembled films with one cinnamoyl moiety per chemical repeat unit in the side chains in the polyanion PACSS-CF₃ were then exposed to 297 nm LPUVL with an incident angle of 30° (as shown in Figure 3) to induce anisotropy. The photoreaction procedure in the film was examined by UV-Vis spectra. Figure 8 shows the linearly polarised UV-Vis absorption spectra of a three-bilayer DR/PACSS-CF₃ film irradiated with 297 nm LPUVL for 10 min. The polarised absorbance A_{\perp} ($A_{//}$) was measured with a probing UV-Vis light linearly polarised perpendicular (parallel) to the polarisation direction of the LPUVL. As shown in Figure 8, a difference in the absorbance was observed between A_{\perp} and $A_{//}$ after the irradiation. The result indicates that the cinnamoyl groups with vinylenic units positioned parallel to the polarised direction of the LPUVL ($A_{//}$) were consumed more rapidly by photoreaction than those positioned perpendicular to the polarised direction of LPUVL (A_{\perp}) during LPUVL irradiation. As a result of the direction-selective photoreaction, the vinylenic C=C bonds of unreacted cinnamoyl groups aligned perpendicular to the polarisation direction of the LPUVL were left in a greater numbers relative to those aligned parallel to the polarisation direction. Therefore, the structural anisotropy of the multilayer film was generated from the selective photoreaction of the cinnamoyl group in the film. The LPUVL-induced anisotropic film would give rise to anisotropic interactions with the LC molecules, which might possibly generate the LC homogeneous alignment.

Photoinduced optical anisotropy of the covalently attached film was investigated by measuring absorbance at 290 nm in parallel with ($A_{//}$) and perpendicular to (A_{\perp}) the electric vector of LPUVL. The optical anisotropy was expressed here by the UV dichroic ratio $[= (A_{\perp} - A_{//}) / (A_{\perp} + A_{//})]$. As seen in Figure 9, all the measured dichroic ratios were positive over the irradiation time of ≤ 60 min. With increasing irradiation time, the dichroic ratio increased rapidly to a maximum at 10 min and then began to decrease. This could be reasonably interpreted as a combination

of two kinds of LPUVL-induced photochemical reaction: axial-selective photodimerisation and non-angular selective photodecomposition [9]. The former process proceeded as a result of photodimerisation of cinnamates when the transition moment of the chromophore was in parallel with the polarised direction of LPUVL. This resulted in the axial-selective consumption cinnamoyl group, to bring about optical anisotropy. This process led to the observed initial increase of DR. However, a competing mechanism would cause the dichroic ratio to decrease with irradiation time: the photodecomposition of cyclobutene derivatives. In the first scenario, the rate of photocyclisation was greater than photodecomposition, so the DR increased. However, with increasing irradiation time, the level of remaining cinnamoyl groups in the film fell, and the quantity of cyclobutene derivatives

(a)



(b)

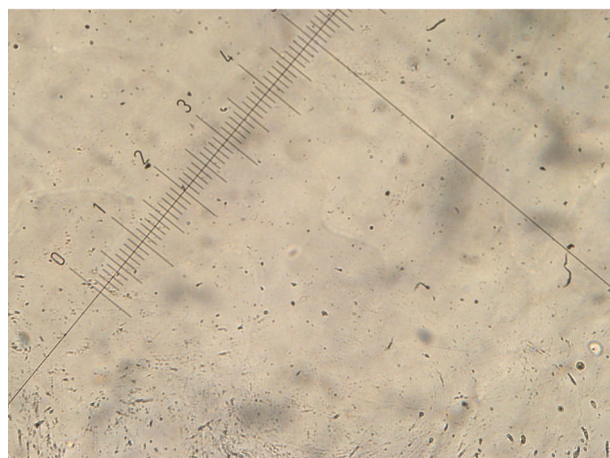


Figure 10. Polarising microscopy texture for a homogeneously aligned nematic liquid crystal cell prepared by the six-bilayer covalent DR/PACSS-CF₃ photoalignment film. The irradiation time was 10 min. (a) dark state, (b) bright state.

increased. While the rate of photodecomposition exceeded the rate of photocyclisation, the dichroic ratio decreased; thus, further irradiation would lead to the gradual reduction of dichroic ratio. Therefore an irradiation time of 10 min was selected for the photoalignment film fabrication.

3.3 LC alignment properties studies

The alignment of LCs on the covalently attached film was studied using crossed polarised optical microscopy. A uniform homogeneous alignment of LC molecules could be obtained in the anti-parallel cell modified by the irradiated covalent film. Figure 10 showed the micrographs of an anti-parallel LC cell with six-bilayer DR/PACSS-CF₃ covalent film irradiated for 10 min by LPUVL. Figure 10(a) shows the dark state when one of the crossed polarisers was parallel to the optical axis of the sample; Figure 10(b) shows the bright state when the two crossed polarisers were kept at 45° to the optical axis of the sample. Both contained few defects and disclinations, indicating that excellent homogenous alignment was obtained.

The pretilt angle of the LCs in the cells was measured by the crystal rotation method. The pretilt angle varied in the range 1.0–2.5°, depending on the irradiation angle of LPUVL. The pretilt angle was larger than that of cinnamate LBL ultrathin films [9]. It has been reported that the high pretilt angle generated in 4-pentyl-4'-cyanobiphenyl (5CB) on rubbed polyimide surfaces containing trifluoro-methyl moieties was due

to the fluorine atoms in the surface of alignment layer [17]. Some studies also have been carried out on the relationship between the surface energy of the alignment layer and the pretilt angle generated in the LC [18]. LC alignment layers containing fluorine units showed low surface energy, and this was related to the LC alignment properties on the LC alignment layers. The polyanion PACSS-CF₃ containing trifluoro-methyl moieties was related to the pretilt angle generated in the LC. As shown in Figure 11, the pretilt angle initially increased with the increasing irradiation angle. When the angle of incident LPUVL exceeded 30°, the alignment of LCs became poor. The LPUVL irradiation angle of 30° was selected for the photoalignment film fabrication.

The stability of the LC cell was also examined against heat treatment. The cell was injected at 71°C and heated to 75°C for 10 min, and then cooled to room temperature naturally. The LC in the cell maintained good alignment. The cells were then sequentially heated at a temperature 5°C higher than the preceding heating step, and cooled as before. We discovered that even when the temperature reached 180°C the cells could maintain good homogeneous alignment as before. These results indicated that the covalent films maintained their LC alignment properties up to at least 180°C, which was approximately 80°C higher than the thermal stability of conventional LBL photoalignment films. Also, the LC cell was left for 1 month under ambient conditions, and transmittance was monitored. A slight decrease in intensity was observed. These results clearly showed that the stabilities of the covalent photoalignment films were

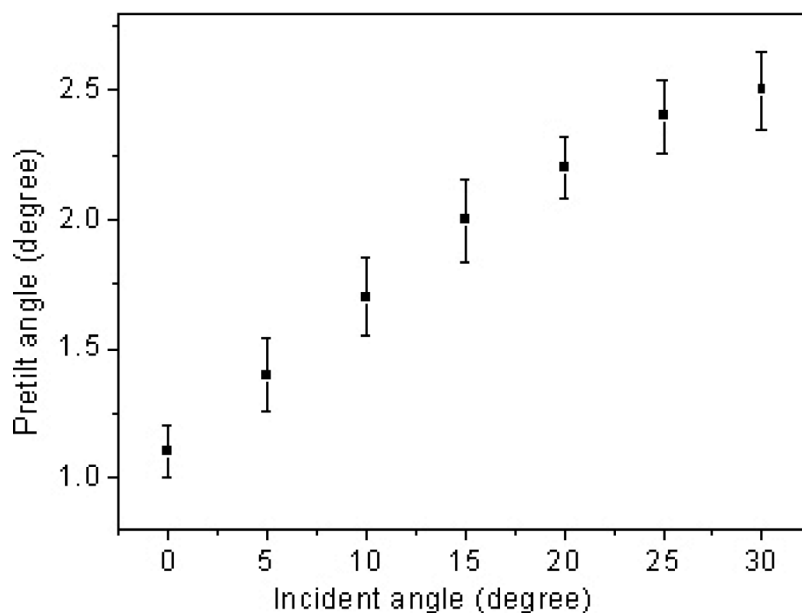


Figure 11. Pretilt angle variations of liquid crystals (LCs) measured in LC cells with different linearly polarised ultraviolet light irradiation angle.

enhanced. This higher stability was ascribed to the linkage conversion of the DR/PACSS-CF₃ layers from ionic bond to covalent bond.

4. Conclusion

In this investigation, new covalently attached multilayer ultrathin films were prepared for LC photoalignment studies. The ultrathin films were fabricated using DR as polycation and PACSS-CF₃ as polyanion by a self-assembly technique. The self-assembled films were irradiated with 365 nm UV light, which changed the linkage between the layers of the films from ionic to covalent. The covalent self-assembled films were irradiated with 297 nm LPUVL to form anisotropic surfaces through the axially selective photoreaction of cinnamates in the films. The films show excellent LC alignment performance. The pretilt angle of LC was 2.5°. The thermal stabilities of the films were enhanced, reaching 180°C. This is sufficient for commercial requirements. Thus, the incorporation of the cinnamoyl moiety using the new covalent LBL film is a promising technique for LC photoalignment studies.

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