



AFRL-AFOSR-JP-TR-2020-0021

Development of New Azobenzene Photo-Alignment Molecules on
Controlling the Tilt Angle of Liquid Crystals

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10/14/2020
Final Report

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REPORT DOCUMENTATION PAGE				<i>Form Approved</i> OMB No. 0704-0188	
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1. REPORT DATE (DD-MM-YYYY) 14-10-2020		2. REPORT TYPE Final		3. DATES COVERED (From - To) 22 Feb 2019 to 21 Jan 2020	
4. TITLE AND SUBTITLE Development of New Azobenzene Photo-Alignment Molecules on Controlling the Tilt Angle of Liquid Crystals				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER FA2386-19-1-4008	
				5c. PROGRAM ELEMENT NUMBER 61102F	
6. AUTHOR(S) Kwang-Un Jeong				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) CHONBUK NATIONAL UNIVERSITY 567 Baekje-daero, Deokjin-gu Chonju, Chonbuk, 54896 KR				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) AOARD UNIT 45002 APO AP 96338-5002				10. SPONSOR/MONITOR'S ACRONYM(S) AFRL/AFOSR IOA	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) AFRL-AFOSR-JP-TR-2020-0021	
12. DISTRIBUTION/AVAILABILITY STATEMENT A DISTRIBUTION UNLIMITED: PB Public Release					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT Cholesteric liquid crystals (CLCs) have been of great interest due to their selective reflection of light (maximum reflection 50%) and can be used as optical fibers, light shutters, smart windows and displays. Previously, dynamic electro-optic responses of polymer stabilized CLCs (PSCLCs) have been reported, including red or blue shifting tuning, bandwidth broadening, and switching response. Compared to the normal angle of incidence, a single PSCLC cell exposed to a large angle of incidence (> 50 degree) exhibits very low transmittance (high optical density) in the reflection band (> 99% reflection) and high out-of-band transmission. However, in order to be used an optical device requiring high optical density, a large prism having an angle of 60 degree or more must be used, which is a big hurdle to overcome. Instead of using a prism, an alternative approach is to tilt the helical axis of CLC across the cell thickness. In this project, light-responsive azobenzene functionalized POSS derivatives are designed and synthesized as new photo-alignment materials. By controlling the orientation of the light-responsive POSS molecules in the LC medium, the tilt angle of the LC and the helical axis of the CLC can be adjusted. The goal of this work is to control the angle of the helical axis of the CLC in the cell by exposure to linearly polarized blue light. The reflection efficiency (optical density) increases as the helical axis of the CLC tilts towards the cell surface. A single PSCLC sample with a tilted CLC helical axis (> 50 degree) will provide excellent optical density and high out-of-band transmission.					
15. SUBJECT TERMS Cholesteric liquid crystals, Azobenzene Photo-alignment molecules, tilt angle					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT SAR	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON KNOPP, JEREMY
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER (Include area code) 315-227-7006

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- BAA Name: FA9550-18-S-0003
- Relevant Section: Organic Materials Chemistry
- Principle Investigator: Kwang-Un Jeong
- Key Researcher involved in the Project: Michael E. McConney
- Period-of-Performance: 1 year
- Total Cost: 65,000 US dollars

1. ABSTRACT

Cholesteric liquid crystals (CLCs) have been of great interest due to their selective reflection of light (maximum reflection $\leq 50\%$) and can be used as optical fibers, light shutters, smart windows and displays. Previously, dynamic electro-optic responses of polymer stabilized CLCs (PSCLCs) have been reported, including red or blue shifting tuning, bandwidth broadening, and switching response. Compared to the normal angle of incidence, a single PSCLC cell exposed to a large angle of incidence (> 50 degree) exhibits very low transmittance (high optical density) in the reflection band ($> 99\%$ reflection) and high out-of-band transmission. However, in order to be used an optical device requiring high optical density, a large prism having an angle of 60 degree or more must be used, which is a big hurdle to overcome. Instead of using a prism, an alternative approach is to tilt the helical axis of CLC across the cell thickness. In this project, light-responsive azobenzene functionalized POSS derivatives are designed and synthesized as new photo-alignment materials. By controlling the orientation of the light-responsive POSS molecules in the LC medium, the tilt angle of the LC and the helical axis of the CLC can be adjusted. The goal of this work is to control the angle of the helical axis of the CLC in the cell by exposure to linearly polarized blue light. The reflection efficiency (optical density) increases as the helical axis of the CLC tilts towards the cell surface. A single PSCLC sample with a tilted CLC helical axis (> 50 degree) will provide excellent optical density and high out-of-band transmission.

2. INTRODUCTION

Control of liquid crystal (LC) alignment has attracted considerable interest in the fundamental scientific aspects of partially ordered soft materials as well as in the engineering application of LC electro-optical devices. LC alignment strongly depends on the surface environments and LC properties. For conventional LC alignment layer, mechanical rubbing on the polymer-coated surface has been the most widely used. However, the polymer-based rubbing alignment method has some limitations, such as long processing times, low yield, and high cost due to the direct contact process. Recently, photo-alignment methods have become technologically important and attractive for controlling LC orientation because of their contact-free and photo-patternable characteristics.

Inspired by natural creatures, cholesteric liquid crystals (CLCs) have been extensively investigated due to their periodic nanostructure. CLCs exhibit selective reflection due to the one-dimensional (1D) band gap formed by the helicoid structure. At the normal incidence, the position of the reflection band is defined as $\lambda = np$, where n is the average reflective index of the LCs and p is the helical pitch. CLCs

reflect circular polarized light matching the handedness of helicoidal superstructure, while transmitting circularly polarized light with opposite handedness. According to this polarization selectivity rule, CLCs reflect maximum 50% for unpolarized incident light. The narrow reflection band gap and handedness-sensitive reflectance of CLCs limit a wide range of applications in areas, such as reflective colored displays, smart laser protections, and etc. Recently, the orientation of the helical axis of the CLCs controlled by different external stimuli is a fascinating issue. Dynamic control of the helical axis of the CLCs in 2D and 3D has been achieved and opens up several potential applications.

In this context, this project is to develop achiral and chiral photo-alignment molecules that can control the tilt angle of the LC and the helical axis of the CLC to overcome the limitations of conventional CLC. A series of photo-alignment materials based on azobenzene functionalized polyhedral oligomeric silsesquioxane (POSS) derivatives was synthesized *via* mono-functionalization. Azobenzene molecules (AZ) can be useful for precisely controlling the tilt angle of helical axis. Under linearly polarized UV or blue-green light, the tilt angle of the LC molecule can be determined by *trans*-to-*cis* isomerization or *trans*-*cis*-*trans* reorientation of azobenzene. The structure-property study between the photo-alignment layer and the LC orientation will offer a new solution to overcome the limitations of the CLC system.

3. EXPERIMENTAL SECTION

3.1. Materials

Aminopropylisobutyl POSS (Hybrid Plastics), 6-Bromohexanoyl Chloride (97%, Sigma), triethylamine (TEA, 99%, Sigma), 4-nitrobenzoyl chloride (98%, TCI), (*S*)-1-(naphthalen-2-yl)ethanol (99%, Sigma), phenol (99% Sigma), 4-aminobenzonitrile (98%, TCI), 4-butylaniline (97%, TCI), tetrahydrofuran (THF, 99.9%, Samcheon Chem.), chloroform (CHCl₃, 99.9%, Samcheon Chem.), hexane (99.9%, Samcheon Chem.), ethyl acetate (EA, 99.9%, Samcheon Chem.), ethanol (99.9%, Samcheon Chem.), dichloromethane (DCM, 99.9%, Samcheon Chem.), acetone (99.9%, Samcheon Chem.), 2-butanone (99.9%, Samcheon Chem.), Isopropyl alcohol (IPA, 99.9%, Samcheon Chem.), hydrazine monohydrate (98%, Sigma), Raney-nickel catalyst (Al/Ni, Ni ≥90%, Al ≥6%, Sigma), hydrochloric acid (HCl, 37%, Sigma), sodium hydroxide (NaOH, 93%, Showa), potassium carbonate (K₂CO₃, 99%, Showa), potassium iodide (KI, 99% Showa), anhydrous sodium sulphate (Na₂SO₄, 99.5%, Showa), sodium nitrite (NaNO₂, 97%, Showa), and nematic liquid crystal (E7, Merck Co.) were used as received.

3.2. Characterization

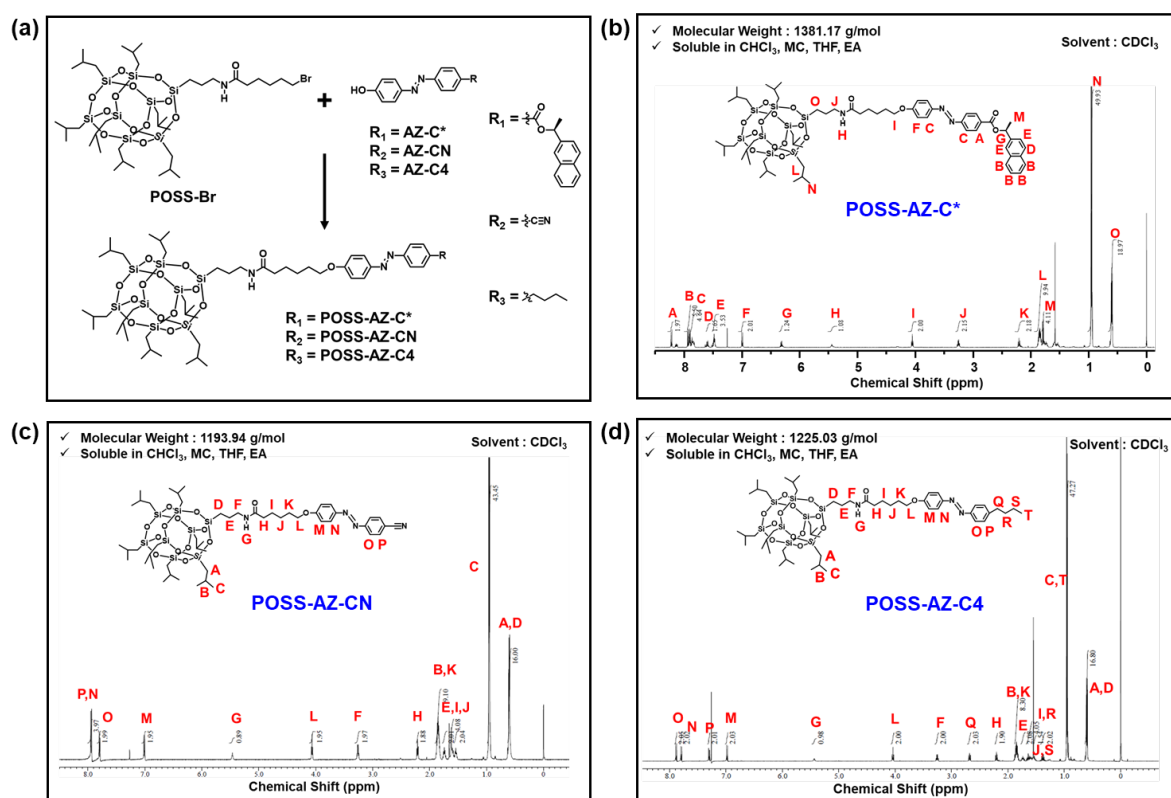
The chemical structure and purity of the POSS-AZ derivatives were confirmed by proton (¹H) nuclear magnetic resonance (NMR, JEOL JNM-EX400). Chemical shifts were quoted in part per million (ppm) with a reference of tetramethylsilane (TMS). Thermal transition behavior was studied using differential scanning calorimetry (DSC, Perkin Elmer PYRIS Diamond). For DSC experiments, the sample weights were controlled to be about 3.0 mg and the pan weights were kept constant with a precision of ± 0.001 mg. Temperature and heat flow scales were calibrated using standard materials at different cooling and heating rates. Heating experiments preceded the cooling experiments to eliminate previous thermal histories, and the same cooling and heating rates were used. The optical texture was observed with a polarized optical microscopy (POM, Nikon ECLIPSE

LV100POL) coupled with a heating stage (Mettler Toledo FP90). One-dimensional (1D) wide angle X-ray diffraction (WAXD) experiments were conducted in the reflection mode of a Rigaku 12 kW rotating anode X-ray (CuK α radiation) generator coupled with a diffractometer. Diffraction peak positions and widths were calibrated with silicon crystals in the high 2θ -angle region ($>15^\circ$) and silver behenate in the low 2θ -angle region. To monitor structural evolutions as temperature changes, a hot stage calibrated within $\pm 1^\circ\text{C}$ error was coupled to the diffractometer. Samples were scanned over a 2θ -angle range of 1.5° to 35° at a scanning rate of $2^\circ\text{C}/\text{min}$.

Figure 1. Synthetic procedures (a) and ^1H NMR spectra of (b) POSS-AZ-C*, (c) POSS-AZ-CN and (d) POSS-AZ-C4.

3.3 Synthesis of POSS-Br

Aminopropylisobutyl POSS (3.43 mmol) and 6-Bromohexanoyl Chloride (6.86 mmol) were dissolved in 30 mL of THF. To this solution, TEA (27.44 mmol) was slowly added dropwise to the



solution and stirred at room temperature for 24 h. After the reaction, the undissolved compound was filtered off and the filtrate was extracted 3 times with CHCl₃ and water. After drying the organic layer with Na₂SO₄, the crude product was purified by column chromatography filled with silica gel using hexane:EA = 3:1 as an eluent. The resulting product was purified by recrystallization from ethanol to afford POSS-Br as a white solid (yield: 68%).

3.4 Synthesis of (S)-1-(naphthalen-2-yl)ethyl 4-nitrobenzoate

To a stirred mixture of 4-nitrobenzoyl chloride (4.35 mmol), and (S)-1-(naphthalen-2-yl)ethanol (2.90 mmol) in 30 mL of DCM at 0°C was slowly added TEA (5.81 mmol). After the reaction mixture was stirred at room temperature for 12 h, the solution was washed with water three times.

The organic layer was dried over Na₂SO₄, filtered and evaporated under reduced pressure to obtain a white solid. This compound was recrystallized from ethanol (yield: 81%).

3.5 Synthesis of (*S*)-1-(naphthalen-2-yl)ethyl 4-aminobenzoate

Hydrazine monohydrate (3.11 mmol) was added drop-wise to a solution of (*S*)-1-(naphthalen-2-yl)ethyl 4-nitrobenzoate (1.56 mmol) in EtOH (40 mL). After the solution was heated to 45 °C, the activated Raney-nickel was added in portions until no further reaction was observed. The resulting mixture was filtered off, and the ethanol was removed under reduced pressure. The crude was dissolved in CHCl₃ and washed with water and brine, and then dried with Na₂SO₄. After distilling the solvent, the product was obtained as brownish solid which was used without further purification (yield: 93%).

3.6 Synthesis of AZ-C*, AZ-CN and AZ-C4

A 2.5 M NaNO₂ aqueous solution (1.00 mL) was added slowly dropwise at a temperature below 5 °C to a heterogeneous mixture of (*S*)-1-(naphthalen-2-yl)ethyl 4-aminobenzoate (2.47 mmol) in 5 M HCl aqueous solution (1.48 mL). The mixture was held below 5 °C and added carefully to a solution of phenol (2.47 mmol) in 2 M NaOH aqueous solution (2.47 mL). The product was precipitated by the addition of small portion of water. By the recrystallization of the product in hexanes, the purified yellow crystal was collected (yield: 72%).

For AZ-CN, similar procedure was adopted using 4-aminobenzonitrile (2.6 mmol) as starting material. After the reaction, the purified yellow crystal was collected (yield: 85 %).

For AZ-C4, similar procedure was adopted using 4-butylaniline (2.1 mmol) as starting material. After the reaction, the purified yellow crystal was collected (yield: 80 %).

3.7 Synthesis of POSS-AZ-C*, POSS-AZ-CN and POSS-AZ-C4

A solution of POSS-Br (0.48 mmol), AZ-C* (0.95 mmol), K₂CO₃ (2.38 mmol) and catalytic amount of KI in 20 mL of 2-butanone was refluxed at 80 °C for 48 h. After reaction, solvent was removed in vacuum and the remaining residue was dissolved in CHCl₃ and washed with distilled water three times. The organic layer was dried over Na₂SO₄. It was purified by column chromatography with silica gel using CHCl₃:Acetone = 15:1 as an eluent. The resulting product was recrystallized with hexane and the orange powder was obtained. (yield: 61%)

For POSS-AZ-CN, similar procedure was adopted using AZ-CN (0.56 mmol) as starting material. After the reaction, the purified yellow crystal was collected (yield: 41 %).

For POSS-AZ-C4, similar procedure was adopted using AZ-C4 (0.78 mmol) as starting material. After the reaction, the purified yellow crystal was collected (yield: 60 %).

3.8 Fabrication of LC test sandwich cell

LC test cells were fabricated by sandwiching two ITO-coated glasses. The ITO-coated glasses were carefully washed with distilled water, IPA, and acetone several times and then dried in a vacuum oven at 80 °C for 3 h. A 10 μm cell gap was maintained by using cell gap tape. POSS-AZ derivatives and nematic LC (E7) were mixed with DCM. After solvent evaporation and vacuum drying, the homogeneous mixture was injected into the test cell by capillary forces.

4. RESULTS AND DISCUSSION

4.1. Azobenzene-Functionalized POSS Derivatives

To control the tilt angle of the liquid crystal (LC) or the helical axis of the cholesteric LC across the cell thickness, three azobenzene-functionalized POSS-derivatives (POSS-AZs = POSS-AZ-CN, POSS-AZ-C4, and POSS-AZ-C*) were newly designed and synthesized. POSS-CN, POSS-C4, and POSS-C* consist of hydrophobic isobutyl-based POSS and photo-responsive azobenzene groups with the cyanobiphenyl, hydrocarbon chain, and (*R*)-configured naphthyl units, respectively (Figure 2). POSS-AZs in the nematic mixture can migrate onto the polyimide alignment layer and form a monolayer where POSS attaches to the PI alignment layer and azobenzene stretches toward liquid crystals. The functions introduced at the end of azobenzene are useful for precise control the LC orientation. The chemical structures and purities of POSS-CN, POSS-C4, and POSS-C* R were confirmed by ^1H NMR spectra.

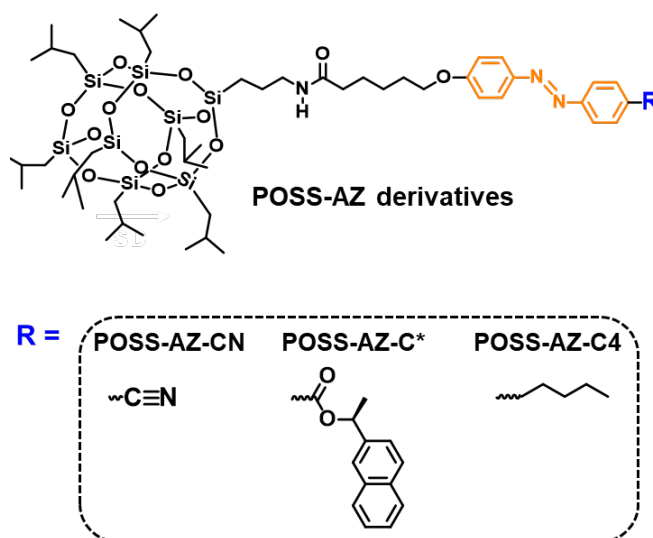


Figure 2. Chemical structures of azobenzene-functionalized POSS derivatives.

4.2. Phase Behaviors and Ordered Structure

The phase behaviors of POSS-AZs in the bulk state were studied by differential scanning calorimetry (DSC), polarized optical microscopy (POM), and wide-angle X-ray diffraction (WAXD). DSC experiment was conducted at cooling and heating rates of 2.5 - 20 °C/min to determine the thermal transition temperatures and quantitative thermodynamic property. As shown in Figure 3a and 3b, POSS-AZ-CN and POSS-AZ-C4 have a single endothermic peak at 152.4 °C and 163.8 °C and an exothermic peak at 120 °C and 110 °C during heating and subsequent cooling process at 10 °C/min, respectively. POSS-AZ-C* shows the LC mesophase appeared after crystal melting above 128.1 °C and becomes isotropic at 131.7 °C. Meanwhile, a single peak during cooling is detected. As shown in Figure 3a and 3d, the large dendritic domains of POSS-AZ-CN are formed where the spherulites of POSS-AZ-C4 are observed under cross-polarized light at -10 °C/min (Figure 3b and 3e). The weak birefringence of POSS-AZ-C* is exhibited at 125 °C, which is often detected in a smectic LC phase. Decreasing the temperature further to room temperature results in solidification with birefringent defects.

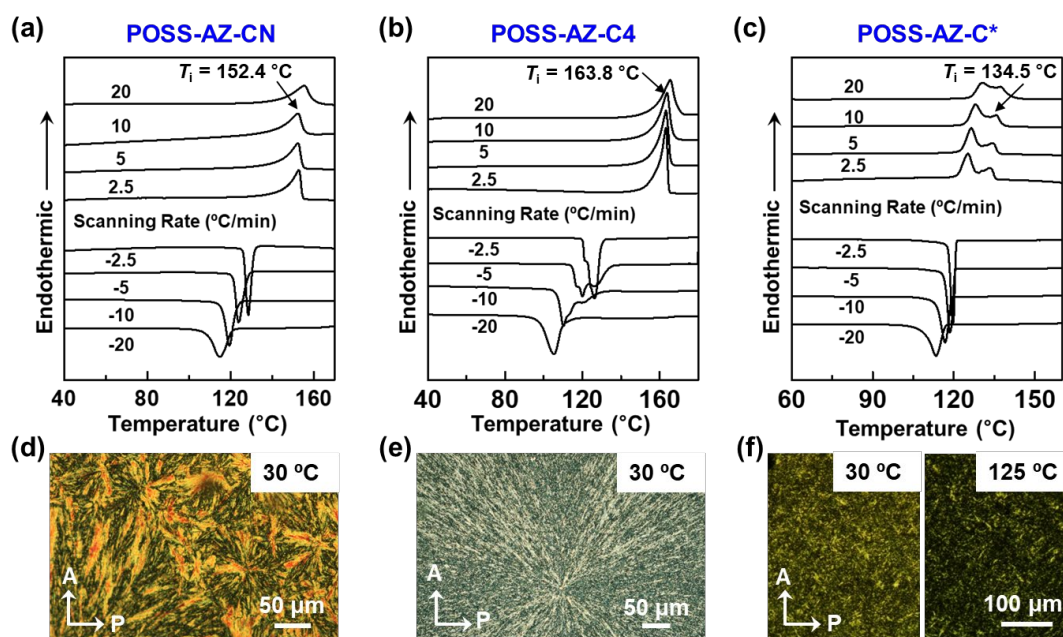


Figure 3. DSC thermogram and POM images of (a,d) POSS-AZ-CN, (b,e) POSS-AZ-C4, and (c,f) POSS-AZ-C*.

To obtain structural information, powder patterns of 1D WAXD were obtained. Figure 4a and 4b show many reflection peaks of POSS-AZ-CN and POSS-AZ-C4 owing to the formation of highly ordered crystal phases. Particularly, in the small angle region, since diffractions at 30 °C are resolved in three peaks in a ratio of 1:2:3, the formation of the layer structure is clear. In the high angle region, multiple diffractions are observed indicating crystallization within the layer. POSS-AZ-C* also shows the integer multiple peaks in the small angle region at 30 °C while single peak was observed at $2\theta = 19.3^\circ$. After crystal melting, the layer structure is maintained. At the isotropic temperature of POSS-AZs, the peak at $2\theta = 8.0^\circ$ ($d = 1.09$ nm) is observed identically, where the diffraction halo corresponds to the quasi long-range correlation of the POSS group. Investigation on their self-assembly behaviors reveals that POSS-AZs are strongly formed into a lamellar structure driven mainly by POSS-POSS interactions in bulk state. Among them, POSS-AZ-C* containing a chiral component has regularly lateral interactions between the azobenzene mesogens.

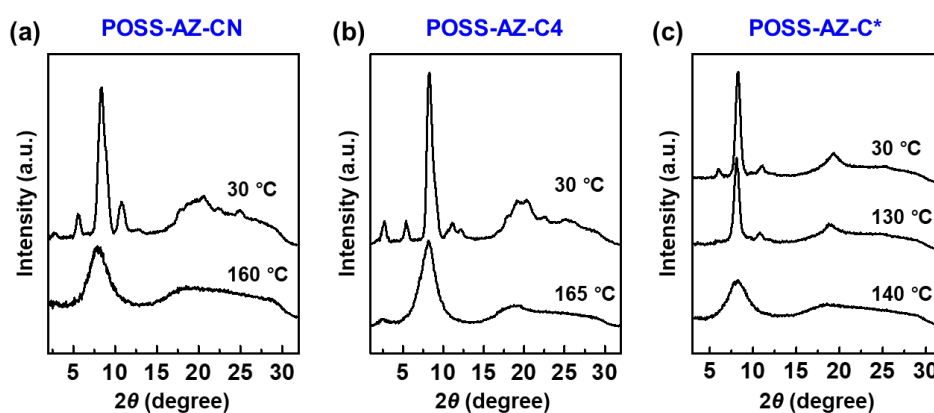


Figure 4. Temperature-dependent 1D WAXD patterns of (a) POSS-AZ-CN, (b) POSS-AZ-C4, and (c) POSS-AZ-C*.

4.3. Construction of Automatic VA Layer by Directly Doping POSS-AZ Derivatives into NLC Medium

To confirm that the POSS derivatives construct automatic vertical alignment (VA) of NLC medium, a series of POSS-AZ derivatives/NLC mixtures was prepared with 0.1/99.9 wt%. For nanoparticle induced VA materials, the VA state is affected by the chemical affinity between the nanoparticles and the NLC medium. Therefore, two types of NLC (5CB and E7) were selected for NLC medium. The homogeneously mixed LC mixtures with different contents of POSS derivatives were injected into the LC test cell fabricated by sandwiching two ITO-coated glass substrates (cell gap: 10 μm).

The macroscopic photographs of NLC (5CB, E7)/POSS-AZ-derivatives taken at room temperature with a common digital camera under crossed polarizers are shown in Figure 5a-f. The dark state images under the cross-polarizers can be observed in both macroscopic (Figure 5a-c) and microscopic POM images (Figure 5a'-c'). The dark state images can be observed in several situations when the sample is truly or accidentally isotropic (isotropic effect), the optical axis of the sample is parallel to one of the polarizers (zero amplitude effect), and the optical axis of the sample is parallel to the propagation direction of light (zero birefringence effect). In this case (Figure 5a-c), the dark images are observed by the birefringence effect. The formation of a Maltese cross in the conoscopic POM images is direct evidence of the VA of NLC (Inset images of Figure 5a'-c'). In the test cell used as E7 host NLC (Figure 5d-f), Schlieren texture are observed instead of dark states. These results indicate that the chemical affinity and solubility between the host NLC and the dopants are important variables for automatic VA formation.

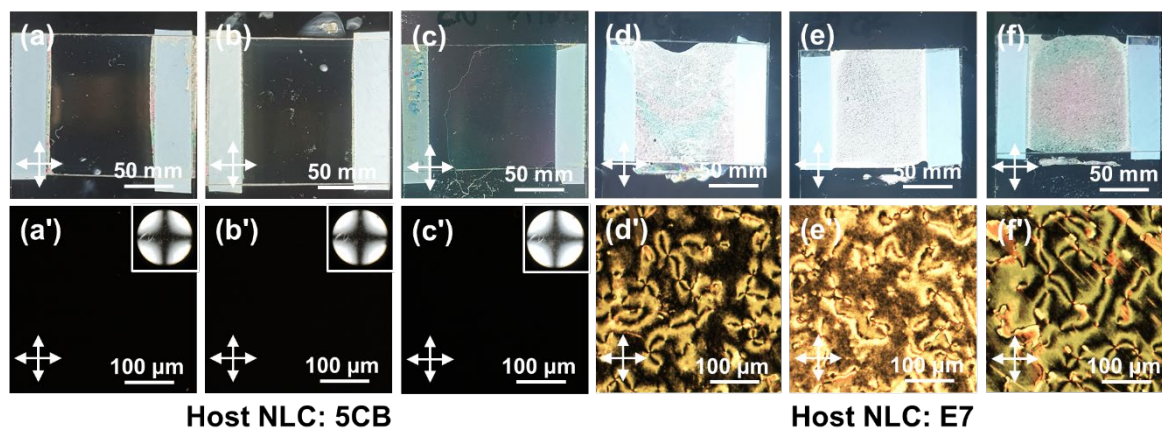


Figure 5. Macroscopic photographs of mixtures of NLC (5CB, E7)/ (a, d) POSS-AZ-C4, (b, e) POSS-AZ-C* and (c, f) POSS-AZ-CN filled LC test cells with 0.1 wt%. The bottom POM images and their inset images are corresponding orthoscopic and conoscopic POM images, respectively.

4.4 Transformation of Alignment of NLC Medium from Vertical to Planar by Inducing Isomerization of POSS-AZ Derivatives

The *trans-cis* isomerization of the POSS-AZ-derivatives upon exposure of UV and vis light was measured by the UV-Vis spectra (Figure 6). Note that the absorption spectra of azobenzene are typically characterized by low energy conversion of the 350 - 360 nm *trans*-isomer (π - π^* transition) and the 430 - 440 nm *cis*-isomer (n - π^* transition). To confirm the photochemical behaviour of POSS-

AZ derivatives, the POSS-AZ derivatives were dissolved in chloroform (1×10^{-3} mg/mL). In case of POSS-AZ-C4, any energy transitions in the UV absorbance are detected. This means that the photoisomerization of POSS-AZ-C4 does not occur. On the other hand, POSS-AZ-CN and POSS-AZ-C* show energy conversions upon irradiation with 365 nm UV light, and reversion is detected under visible light irradiation. Therefore, it is recognized that POSS-AZ-CN and POSS-AZ-C* are more suitable for photoalignment materials than POSS-AZ-C4.

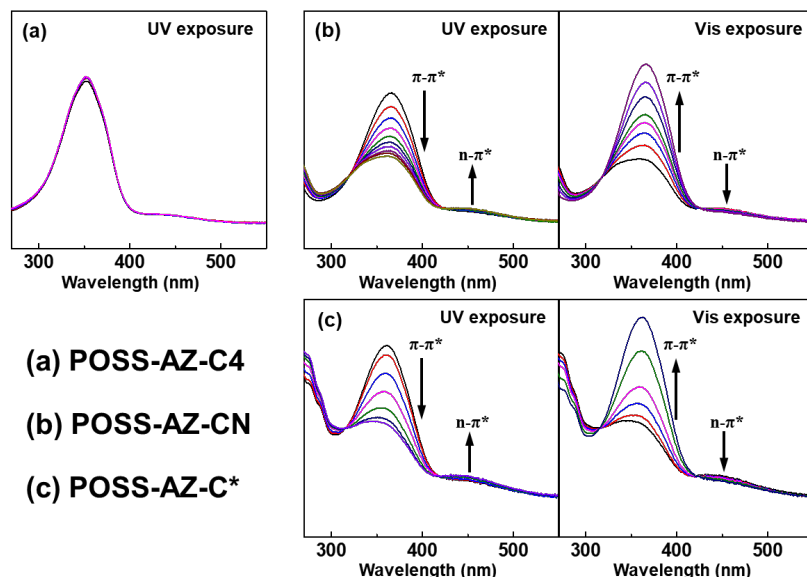


Figure 6. The UV-Vis absorption spectra showing the *trans-cis* isomerization under 365 nm UV light irradiation of (a) POSS-AZ-C4, (b) POSS-AZ-CN, and (c) POSS-AZ-C*, respectively and their *cis-to-trans* conversions under visible light irradiation.

To confirm the reorientation of the vertically aligned LC to the planar orientation, UV light with a wavelength of 365 nm is exposed to the cell (Figure 7a). However, no change of NLC alignment with vertical alignment is observed even though the LC samples are exposed by UV light for 150 min. This result indicates that the UV absorption of azobenzene for isomerization is affected by angle of incidence between the direction of light propagation and the optical axis of azobenzene. Therefore, linearly polarized UV light is exposed to the sample at an angle of incidence of 20° in Figure 8a. For the samples containing POSS-AZ-CN, the vertically oriented NLC medium remains unchanged and maintains vertical alignment. However, the samples containing POSS-AZ-C* show reversible reorientation.. As shown Figure 8b, the homogenous LC texture is observed after 30 min exposure of 365 nm UV light at an angle of incidence of 20° . The birefringence of the LC test cell changes as UV light exposed time increases. The vertically oriented LC medium is reoriented to the random orientation regardless of the direction of the exposed UV light. Future studies should be included, such as polymerization of a small amount of monomer, surface modification of the alignment layer, and rubbing process to preferentially aligned the LC molecules in the same direction.

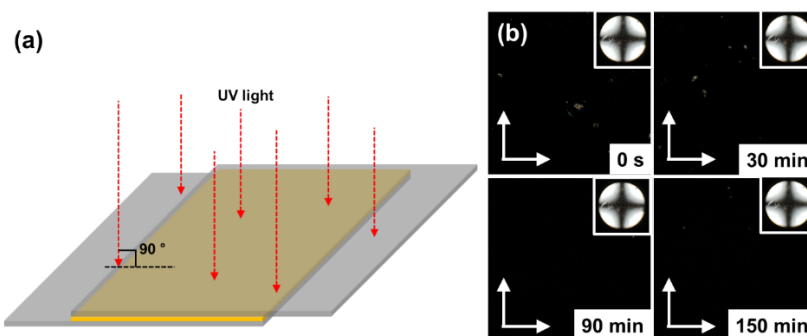


Figure 7. (a) Schematic illustration of UV light irradiation of POSS-AZ-CN/NLC (0.1/99.9 wt%) filled LC test cell. (b) Orthoscopic POM images of the LC test cell according to UV light irradiation time.

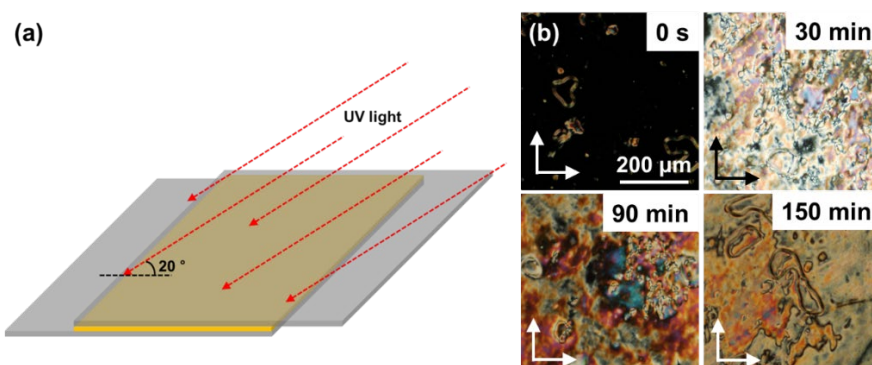


Figure 8. (a) Schematic illustration of UV light irradiation of POSS-AZ-C*/NLC (0.1/99.9 wt%) filled LC test cell. (b) Orthoscopic POM images of the LC test cell according to UV light irradiation time.

6. CONCLUSION

To control the tilt angle of the liquid crystal (LC) or the helical axis of the cholesteric LC across the cell thickness, three azobenzene-functionalized POSS-derivatives (POSS-AZs = POSS-AZ-CN, POSS-AZ-C4, and POSS-AZ-C*) were newly designed and synthesized. The vertical alignment of LC induced by POSS-derivatives was examined in 5CB and E7. When exposed to UV light, trans-to-cis isomerization occurs for POSS-AZ-C* samples, but not for POSS-AZ-C4 and -CN samples. The azobenzene molecule of POSS-AZ-C* is effective in changing the LC orientation from vertical to planar by the conformational change of azobenzene group. This result indicates that the change in the LC orientation induced by the conformational change of azobenzene group is affected by some factors, such as the chemical compatibility between the end functional group and the LC medium. The information obtained from this project can be utilized to understand the mechanism how to control the tilt angle of the LC on a controlled alignment layer.