Photosensitizers and their modified forms are of particular importance in the design of molecular electronic devices.

**FULL PAPER**

Photocontrolled Liquid Transportation in Microtubes by Manipulating Mesogen Orientations in Liquid Crystal Polymers

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Tubular microactuators (TMAs) fabricated by photodeformable liquid crystal polymers (LCPs) pave a significant way for smart microfluidic applications with contactless, spatial, and precise manipulation of liquids. To realize liquid transportation in these TMAs, LCPs should have excellent photodeformation property and suitable mechanical properties. Herein, linear liquid crystal polymers (LLCPs) with different mesogen orientations are used to prepare TMAs to study their liquid transportation behaviors. The mesogen orientation in each LLCP is formed spontaneously and varies with the spacer length, leading to different deformations of LLCP films. It is found that only LLCPs with mesogens oriented out-of-plane realize self-support of the TMAs, whereas the TMAs with mesogens oriented in-plane are so weak that they collapse in the radial direction, indicating the importance of mesogen orientation in fabricating three-dimensional structures. Upon attenuated 470 nm light irradiation, the TMAs deform to an asymmetric conical structure, leading to the motion of liquid slug toward the narrow side. The liquid motion is accelerated in the TMA with longer spacer, showing the control of liquid speed by the mesogen orientation. These photocontrolled TMAs are expected to be applied in biological applications, such as whole blood analysis and flow cytometry, for precise liquid manipulation.

1. Introduction

Tubular microactuators (TMAs) are of particular importance for their potential applications in bionic blood vessels, microfluidic connections, and medical catheters. Compared to silk screens or sponges with liquid collection function, the TMAs can achieve confined liquid storage, directional liquid transportation and liquid recycling, thus showing merits in chemical analysis and biological applications. The current TMAs require external driving setups such as batteries, air pumps, or mechanical pumps to realize liquid control, thereby limiting their applications in pharmaceutical screening or minimally invasive surgery.

To omit these bulky accessories, one approach is to fabricate these TMAs into intelligent systems by using responsive materials that can be controlled by contactless stimuli such as light, electricity, or magnetic field. Particularly, using light to manipulate liquid in the TMAs is a new paradigm for the actuation of microfluidic systems with contactless, spatial, temporal and precise control. Recently, we have fabricated photodeformable TMAs by using liquid crystal polymer (LCP) to accomplish liquid propelling, stirring, fusing and climbing through photinduced asymmetric capillary forces which require neither complex optical setups nor solution environments. The utilization of LCPs presents an opportunity to realize fast and macroscopic deformation, whereas their mechanical properties and deformation behaviors are strongly influenced by the mesogen orientation. Broer and co-workers designed a cilia-like microactuator with LCPs to create mixing and flow, whose deformation is amplified by the splayed orientation. Azimuthal, radial, twisted, and even more complex orientations were developed in the previous work, leading to various deformations such as conical shape, saddle shape, helix shape and accordion shape. Therefore, it is critical to control the mesogen orientation for the design of the TMA. However, how the mesogen orientation affects TMA preparation and liquid transportation behavior remains unclear.

In this work, we synthesized three photodeformable linear liquid crystal polymers (LLCPs) with spontaneous mesogen orientations (Figure 1a). Through the elongation of spacer length, we obtained two types of mesogen orientations (in-plane and out-of-plane), thus managing the bending behaviors of the LLCP films (Figure 1b). The mechanical robustness and photodeformation behaviors of the TMAs were tuned by the mesogen...
orientation for optimizing the fabrication and liquid transportation of the TMAs (Figure 1c). Photocontrolled transportation of liquid slug was realized in the TMAs, which was accelerated by the variation of mesogen orientations (Figure 1d).

2. Results and Discussion

2.1. Preparation and Mesogen Orientation Characterization of LLCP TMAs

The LLCPs abbreviated as PC-AB6 without spacer and PCnAB6 with different lengths of spacers (n is the number of carbons in the spacers, n = 3, 7) were synthesized by the ring-opening metathesis polymerization (ROMP) (Scheme S1, Supporting Information). Thanks to this living polymerization method, the molecular weights of LLCPs (Table S1, Supporting Information) are at least one order of magnitude larger than those of the general LCPs, which enhanced the mechanical robustness of these LLCPs. Typical schlieren texture for all the LLCPs was identified at room temperature by polarized optical microscopy (POM) (Figure S2a, Supporting Information). The differential scanning calorimetry (DSC) curves of the LLCPs showed an increase in the clearing temperature (Tc) from 47.6 to 87.0 °C with the elongation of the spacer, suggesting the increase in the liquid crystal phase stability. The endothermic peaks at 35.0 and 42.3 °C are identified as the crystallization point of the spacers in PC3AB6 and PC7AB6, respectively (Figure S2b, Supporting Information).

LLCP TMAs were fabricated by solution processing. Solutions of LLCPs in CH2Cl2 (~2 wt%) were filled into a glass capillary. After evaporation of CH2Cl2 at 50 °C, the inner surfaces of glass capillaries were uniformly coated with the LLCPs. The coated capillaries were annealed at 50 °C for 30 min, and the glass substrates were then etched by hydrofluoric acid. To understand the phase structures of these TMAs, they were cut along the length direction and flattened out to be annealed films. The LLCP films form large-scale spontaneous orientation, which is beneficial for the construction of actuators with complex structures to enhance their deformation property. As investigated by 2-dimensional X-ray diffraction (2D-XRD) in Figure 2a–c, the splitted diffractions in both the low- and high-angle regions demonstrated spontaneous formation of uniform mesogen orientation. The diffraction arcs in the low-angle region appeared on the meridian in the PC-AB6 film while on the equator line in PCnAB6 films, implying the formation of the smectic phase with different normal directions of the smectic layer in PC-AB6 (parallel to the film plane) and PCnAB6 (perpendicular to the film plane). In the high-angle
region, two diffraction arcs on the equator in the PC-AB6 film demonstrated the smectic A phase with in-plane orientation of the mesogens (Figure 1b and Figure 2d), whereas the diffractions splitted into four folds in PCnAB6 films that illuminated the smectic C phase with out-of-plane orientation. The tilting angles in the smectic C phase were $58^\circ$ and $63^\circ$ for PC3AB6 and PC7AB6, respectively (Figure 1b and Figure 2e,f). The details of the phase behaviors of LLCPs are discussed in the Supporting Information (Figure S3 and S4, Supporting Information).

2.2. Manipulating Bending Behavior via Controlling Mesogen Orientations

The bending behaviors of annealed LLCP films (10 mm $\times$ 1.57 mm $\times$ 20 $\mu$m) with spontaneous orientation, which were cut along the length direction of TMAs and flattened out, were manipulated by UV/visible light irradiation. These films were fixed by two glass slides to maintain the vertical state to eliminate the influence of gravity. Two different types of bending behavior could be observed with the same irradiation of UV light (365 nm, 80 mW cm$^{-2}$): bending toward the light source (PC-AB6, within 6 s) and bending away from the light source (PC3AB6 and PC7AB6, within 2.5 and 2 s, respectively). Meanwhile, all the bent films could recover their initial states after illumination with visible light (530 nm, 140 mW cm$^{-2}$) with recovery time of 20, 12 and 6 s for PC-AB6, PC3AB6, and PC7AB6, respectively (Figure 3 and Movie S1, S2, S3, Supporting Information).

The distinguishing bending behaviors of LLCP films verified that the mesogen orientations in these films were different. In the PC-AB6 film, as the mesogens were oriented in-plane, an anisotropic contraction induced by the trans–cis photoisomerization of the azobenzene moieties was generated on the surface region of the film along the mesogen orientation direction upon UV light irradiation, resulting in the bending toward the light source.[42] In contrast, the trans–cis photoisomerization caused the expansion on the film surfaces of PCnAB6 as the mesogens are oriented out-of-plane, which leads to the bending behaviors in the opposite direction (Figure 1b).[43] The bending angle $\gamma$ shown in Figure 3 represented the maximum bending of the films after 365 nm exposure. The PC-AB6 film exhibited the largest bending due to the considerable anisotropic contraction. In the PCnAB6 films, the surface expansion enlarged gradually with the increase in the tilting angle, giving rise to larger bending in PC7AB6 than in PC3AB6. The angular speed defined as the distance (in degree) covered per second increased monotonically with the spacer length. A maximum speed of $32.5^\circ$ s$^{-1}$ was achieved for PC7AB6, whereas the speed of only $20^\circ$ s$^{-1}$ and $15.3^\circ$ s$^{-1}$ was reached for PC3AB6 and PC-AB6, respectively. The acceleration of bending could be explained by the increase in the spacer length, which reduced the restriction of the azobenzene moieties caused by polymer backbones.

2.3. Liquid Transportation in LLCP TMAs

The 3D-structured TMAs show different mechanical properties along the length direction and radial direction attributed to out-of-plane and in-plane mesogen orientations in PCnAB6 and PC-AB6, respectively. As a result, the TMAs fabricated by PCnAB6 were self-supported, but the PC-AB6 TMA collapsed in the radial direction of the microtube (Figure 4a). That is because the Young’s modulus of the LLCP film parallel to the mesogen orientation direction is about twice as large as that of the perpendicular one (Figure S5 and Table S2, Supporting Information). The out-of-plane oriented mesogens from the PCnAB6 TMA provided enough modulus along the radial direction, whereas
the in-plane oriented mesogens in PC-AB6 TMA wall failed to support its 3D structure for liquid transportation.

Liquid transportation was performed in the PCnAB6 TMA with an inner diameter of 500 μm (Figure 4b). Instead of UV irradiation as performed in the deformation analysis, 470 nm light was applied to avoid aging of the PCnAB6 TMAs. Upon irradiation of 470 nm light whose intensity was attenuated from left to right, an isopropanol slug with asymmetric concave surfaces was self-propelled toward the right; when the direction of attenuation was reversed, the moving direction of the slug changed to the left (Figure 4c). The liquid transportation performance of PC3AB6 was the same as that of PC7AB6 TMAs (Movie S4, Supporting Information).

The liquid transportation ability of the TMA is attributed to asymmetric photodeformation in response to attenuated 470 nm light. It has been reported that azobenzene mesogens can be realigned along the propagation direction of actinic linearly unpolarized blue light after repetition of trans–cis–trans isomerization cycles, which is known as the Weigert effect.[44] Here, the effect was testified by irradiation of parallel 470 nm light to the PC7AB6 films, wherein the mesogens were aligned along the light propagation direction and changed from the smectic C phase to the smectic A phase, as shown in Figure S6, Supporting Information. Therefore, when the TMA was exposed to unpolarized 470 nm light, whose actinic direction is perpendicular to the length direction of the TMA, the azobenzene mesogens were reoriented along the propagation direction of the light. The change in the tilting angles θ of azobenzene mesogens in the different exposed areas was different because the smectic layers of the LLCP were arranged coaxially in the TMA wall (Figure 5a). To facilitate understanding of the photo-induced reorientation, the irradiation of parallel light on the TMA was transferred to the irradiation of diffused light on the flattened film. The incident angles of the actinic light on the different areas of the plane ranged from 0° to 90°, leading to an expansion along the width direction and a contraction along the thickness direction of the film plane. This expansion and contraction resulted in an elongation of the perimeter of the TMA and a decrease in the thickness of the TMA wall, which together caused an increase in the cross-sectional area of the TMA. The upper surface of the TMA wall was displaced toward the light source upon exposure to 470 nm light and returned to its initial position immediately when the light was switched off. The obtained freestanding TMA possessed the ability to generate fast deformation-recovery conversion upon light on and off for many cycles (Figure 5b). Moreover, the cross-sectional area enlarged with the increase in light intensity. Therefore, the TMA deformed to an asymmetric conic geometry, which generated adjustable capillary force to propel the liquid slug in the direction of light attenuation (Figure 1d).[14] We also investigated the

Figure 3. Photodeformation behavior of a) PC-AB6, b) PC3AB6, and c) PC7AB6 annealed films. The bending and recovery are induced by 365 nm UV light (80 mW cm⁻²) and 530 nm green light (140 mW cm⁻²). The maximum bending angles of PC-AB6, PC3AB6, and PC7AB6 reach 92°, 50°, and 65°, respectively. The size of the films is 10 mm × 2 mm × 20 μm, and the scale bar is 2.5 mm.
bending behaviors of the annealed LLCP films upon irradiation of 470 nm blue light. The incident light was nearly parallel instead of diffused in the tube case and penetrated the whole film, leading to only small fluctuation but not obvious bending (Figure S7, Supporting Information).

Interestingly, we found that the transportation speed of the isopropanol slug confined in the PC7AB6 TMA was 32.0 mm min\(^{-1}\), which was 17.6% faster than that in the PC3AB6 TMA (27.2 mm min\(^{-1}\)), resulting from the variation in the tilting angle \(\theta\) (Figure 5c). As mentioned earlier, the increase in the cross-sectional area of the TMA is relative to the change in \(\theta\). Compared to PC3AB6, PC7AB6 owned larger \(\theta\) which induced stronger expansion along the width direction as well as weaker contraction along the thickness direction upon 470 nm light irradiation, leading to larger cross-sectional area of the TMA and higher liquid transportation speed. Thus, the faster transportation speed of the isopropanol slug of 40.4 mm min\(^{-1}\) was found in PC11AB6 TMA with \(\theta\) of 65°, and it is expected that the transportation speed will reach the maximum if the mesogens are oriented along the radial direction coaxially in the TMA with \(\theta\) of 90°.

3. Conclusion

In conclusion, we prepared photodeformable TMAs using LLCPs with different spacer lengths (the carbon number in the spacer is 0, 3 and 7, respectively). Two types of mesogen orientations, in-plane and out-of-plane, were obtained in the LLCP TMAs, leading to distinguishing reversible bending behaviors of the annealed films obtained by cutting the TMAs along the length direction and flattening out. The PC-AB6 film bent toward the UV source and reversed by 530 nm light, whereas the PC\(_7\)AB6 film bent toward the opposite direction. The Young’s modulus of LLCP TMAs parallel to the mesogen orientation direction was about twice as large as that for the perpendicular one. Therefore, PC-AB6 TMA collapsed in the radial direction, whereas PC\(_{11}\)AB6 TMAs were well self-supported, which demonstrated that out-of-plane orientations in the LCPs were essential to afford enough mechanical robustness in fabricating 3D tubular structures. The transportation speed of isopropanol in PC3AB6 and PC7AB6 TMAs reached 27.2 and 32.0 mm min\(^{-1}\) upon irradiation of 470 nm light, respectively, reflecting the acceleration of liquid transportation with the increase of \(\theta\) in
the smectic layer. The transportation speed is speculated to reach the maximum if the mesogens are oriented along the radial direction coaxially in the TMA with $\theta$ of 90°/C14. With controllable liquid transportation manipulations, these TMAs are expected to have potentials in many biological analyses such as whole-blood analysis and flow cytometry.

4. Experimental Section

Synthesis of C-AB6 and CnAB6: The synthetic routes of PC-AB6 and PCnAB6 are shown in Scheme S1. For the synthesis of C-AB6, 5 mmol 4-((4-(hexyloxy)phenyl)diazenyl)phenol (molecule (1) in Scheme S1), 5 mmol 4-(cyclooct-4-en-1-yloxy)-4-oxobutanoic acid (molecule (3) in Scheme 1) and 5.5 mmol 4-dimethylaminopyridine (DMAP) were added to a 500 mL flask and dissolved in 200 mL CH$_2$Cl$_2$. Under the protection of Ar, 10 mmol dicyclohexylcarbodiimide (DCC) in 50 mL CH$_2$Cl$_2$ was added to the mixture and stirred at 0°C for 24 h. After the reaction, the precipitate was filtered out, and the monomer product was obtained by column chromatography with silica gel using tetrahydrofuran:chloroform = 1:8 (yield: 84%).

For the synthesis of CnAB6, 5 mmol 4-((4-(hexyloxy)phenyl)diazenyl)phenol, 5 mmol 3-bromo-1-propanol or 7-bromo-1-heptanol, 10 mmol Cs$_2$CO$_3$ and 0.2 mmol tetrabutylammonium bromide (TBAB) were added to a 500 mL flask and dissolved in 200 mL N,N-dimethylformamide (DMF). The mixture was heated to 110°C and stirred for 10 h. After the reaction, the mixture was poured into 1 L water, and the precipitate was separated by vacuum filtration. The precipitate was washed with water to gain pure 3-(4-((4-(hexyloxy)phenyl)diazenyl)phenoxy)propan-1-ol or 7-(4-((4-(hexyloxy)phenyl)diazenyl)phenoxy)heptan-1-ol (molecule (2) in Scheme 1, yield: ≈90%). Then, the monomers were synthesized using molecule (2) and molecule (1) through the similar procedure for the synthesis of PC-AB6.

Polymerization of the Monomers: The polymers were synthesized by the ROMP method. Solutions of second-generation Grubbs catalyst (2.5 μmol) and the monomer (500.0 μmol) in anhydrous CH$_2$Cl$_2$ (10 mL) were vigorously stirred for 3 h at room temperature in Ar atmosphere. After the polymerization, the concentrated reaction mixture was precipitated with methanol and washed with petroleum ether, and the obtained polymer was dried in vacuum oven (yield: ≈95%).

Characterization Methods: All reagents were commercially obtained and used without further purification. $^1$H NMR and $^{13}$C NMR spectra of the monomers and the LLCPs were recorded on a Bruker DMX500 NMR spectrometer using tetramethylsilane as the internal standard and CDCl$_3$ as the solvent. The thermodynamic properties of the polymers were determined by differential scanning calorimetry (DSC, TA, Q2000) at a heating and cooling rate of 10°C min$^{-1}$. The textures of the LLCP films were evaluated with a polarized optical microscope (POM, Leika, DM2500p) equipped with a Mettler hot stage (Model FP-90 and FP-82). 2D-XRD experiments on the LLCP films were conducted on a Bruker D8 Discover diffractometer with a 2D detector of general area detector diffraction system (GADDS) in

Figure 5. a) Schematics illustrating the reorientation of mesogens in the cross-sectional area of the TMA and the flattened film before and after irradiation by unpolarized 470 nm light. The intensity of the blue light source is 80 mW cm$^{-2}$ in these experiments. b) Plot showing the displacement of the upper surface of the exposed wall of the PC7AB6 TMA by alternately switching on and off 470 nm light. c) Transportation speed of an isopropanol slug in the PC3AB6, PC7AB6, and PC11AB6 TMAs. The length of the slug is 100 μm.
the transmission mode. The X-ray sources (Cu $K_{\alpha}$, $\lambda = 0.154$ nm) were provided by 3 kW ceramic tubes, and the peak positions were calibrated with silicon powder ($2\theta > 15^\circ$) and silver benenate ($2\theta < 10^\circ$). The background scattering was recorded and subtracted from the sample patterns. The tensile stress–strain measurements of the LLCP films were performed using an Instron Universal Testing Machine (Model 5943) at a deformation rate of 60 mm min$^{-1}$ in air. The toughness, a parameter that characterizes the work required to fracture the sample per unit volume, was calculated from the area below the tensile stress–strain curve until fracture. Photographs and videos of the bending behaviors as well as the light-induced liquid motion were taken by a super-resolution digital microscope (Keyence, VHX-100). Visible light at 470 nm was obtained from an light-emitting diode (LED) irradiator (CCS, HLV-24GR-3W). Attenuated 470 nm light was produced by placing a rectangular, continuously variable and metallic neutral density filter (Thorlabs, NDL-25C-4) in front of the 470 nm LED irradiator.

Preparation of sheared LLCP films: The LLCP bulks were heated to clearing temperature on a glass substrate and sheared along one direction quickly by a scraper to form thin films. Then, the samples were moved away from the heater and cooled to liquid-crystal phase rapidly for the sake of maintaining the mesogen orientation induced by shearing. Then, the sheared films were peeled off from the substrate, and the inhomogeneous edges were cut.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

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