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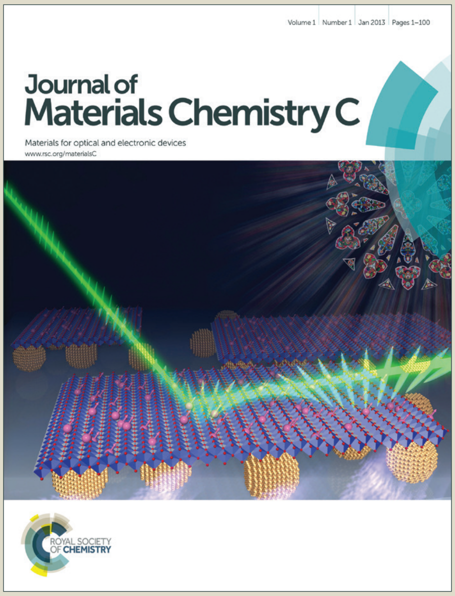

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Photomechanical Response of Polymer-Dispersed Liquid Crystals/Graphene Oxide Nanocomposites

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ABSTRACT: Recyclable, fast and visible-light responsive polymer−dispersed liquid crystal (PDLC)/graphene oxide (GO) nanocomposite films were successfully fabricated by combination of solution casting and mechanical stretching. In the PDLC/GO nanocomposite films, one low−molecular−weight nematic LC (5CB) formed the separated phase and GO−dispersed polyvinyl alcohol (PVA) was designed as the film matrix. Upon irradiation with visible light, PDLC/GO nanocomposite films showed photomechanical response, bending toward the light source along the stretching direction. Here, GO functioned as the light absorbent and nanoscale heat source to thermally-induce phase transition of 5CB from homogeneous alignment to isotropic phase. Thus volume contraction occurred on the surface area of the nanocomposite films due to the photothermal effect of GO, whereas little changes took place in the opposite area, resulting in the visible light-induced photomechanical response in a bimetal-like mode. These PDLC/GO nanocomposite films can be potentially applied in soft actuators and micro-optomechanical systems with visible light as energy source.

1. Introduction

In recent years, photo−driven polymers including hydrogels, shape−memory polymers (SMPs) and liquid−crystalline elastomers (LCEs) have sparked enormous interests due to their promising applications in soft actuators and micro-optomechanical systems.[1−3] Among them, LCEs have been widely studied because they integrate the self−organization performance of LCs and mechanical property of polymers. Especially, photoresponsive LCEs containing photochromic moieties such as azobenzene (AZ) have been successfully applied in fabrication of a variety of soft actuation devices with remote control only by light,[3−6] including the inchworm walkers,[7] motors[8] and artificial arms.[9] However, several bottlenecks are still required to be solved. Firstly, the fabrication process of LCEs including the orientation of LC monomers and the fixation of the mesogenic alignment is complicated and in the harsh condition. Secondly, the mechanical and film−forming properties of LCEs are poor from the viewpoint of practical applications. Majority of soft actuation devices have been fabricated based on bilayer structure that composed of active layer of LCEs and passive layer of general plastics with excellent mechanical and film-forming capability.[9] Thirdly, the stimulating light source of ultraviolet (UV) is less penetrative than the visible or infrared (IR) light, which usually does harm to biosamples.[10] Thus, the development of photoactuators triggered by visible/NIR-light is more convenient for practical applications.[11]

As a result, novel polymer materials with easy ways of fabrication for light−to−mechanical energy transform should be developed particularly exhibiting visible−light responsive properties. For the sake of developing photo-driven polymers with easy fabrication process, good film-forming and mechanical properties and recyclable utilization, we previously fabricated polymer−dispersed liquid crystal (PDLC)−like hybrid films with photoresponsive LC polymers as dopants and photoinert polymers as a host material by coupling with mechanical stretching.[12] Homogeneous alignment of LC polymers was achieved by mechanical treatment and the hybrid films with ultralow mesogenic concentration exhibited similar photomechanical behaviors as LCEs.[3,4,12]. However, the light source triggering the deformation of hybrid films was still UV light because the photomechanical behavior was attributed to the photoresponsive AZ mesogens. Furthermore, the responsive rate was very slow due to high glass transition temperature of the used LC polymer although the photodeformable behavior has been achieved at room temperature.[12]

Herein, we present a general approach to fabricate recyclable, fast responsive and visible−light responsive PDLC/graphene oxide (GO) nanocomposite films composed of GO, one nematic LC (5CB) and polyvinyl alcohol (PVA). In the

PDLC/GO nanocomposite films, GO may function as the photo−absorbent and nanoscale heat source, 5CB as LC dopants and PVA as the photoinert polymer host just providing the good film-formation and mechanical property. The designed structure and composition might endow the PDLC/GO nanocomposite films with unique properties, like easy fabrication, low cost and good photomechanical performance. Besides, fast response could be obtained because of the photothermal effect and high thermal conductivity of GO as well as the low viscosity of low−molecular−weight (LMW) LC (5CB) comparing with LC polymers. Furthermore, the PDLC/GO nanocomposite films were easily fabricated and recyclable since the mixed materials showed different solubility in water and organic solvents.

2. Results and Discussion

2.1 Fabrication of PDLC/GO nanocomposite films

As shown in Figure 1a, the PDLC/GO nanocomposite films were prepared by directly casting aqueous PVA solution containing a certain amount of homodispersed GO and 5CB on a clean glass substrate. The one-pot method to fabricate photoresponsive PDLC/GO nanocomposite films was facile and scalable compared to complicate preparation process of LCEs.[1−3] The chemical structure of 5CB selected as the LC dopant is given in Figure 1b. Here, 5CB was chosen because it is a typical nematic LC with a melting point of 24 °C and the nematic LC−to−isotropic transition temperature (T_{ni}) of 35 °C, exhibiting lower viscosity at room temperature. In this strategy to design the PDLC nanocomposite films, GO acted as the visible light−absorbing materials and nanoscale heat source to thermally-trigger the phase transition of 5CB from ordered alignment LC to disordered isotropic phase, resulting in a large change in volume. On one hand, GO showes good absorption in the visible and near-IR region (Figure S1). On the other hand, GO is more compatible with solvents without any complicate surface modification, comparing with other nanomaterials taking the function of photo-absorbent and nanoscale heat source such as carbon nanotubes and gold nanoparticles, which enables it beneficial to conveniently designing functional nanocomposites. In addition, high thermal conductivity of GO made the heat effectively transfer in the nanocomposite films that improved the responsive speed. Furthermore, GO can be easily detached by dissolving the nanocomposite films in the water and centrifugation. LC and PVA can be separated by the extraction because LC and PVA exhibited different solubility in the water and organic solvents. Thus, the recyclable utilization was achieved in our PDLC/GO nanocomposite films, which is almost unavailable for LCEs with highly chemical cross-linking. [1–4]

2.2 Microstructure of the nanocomposite films

It has been reported that little volume change can be detected when the phase transition occurred from non-aligned LC to isotropic phase.[13] As a result, homogeneous alignment of mesogens in PDLC/GO nanocomposite films is the necessary precondition for their photomechanical behaviors.[4,12] As shown in Figure 2a, one simple method of mechanical stretching was utilized to induce the mesogenic alignment along the stretching direction. By control the amount of LC (5CB), they were uniformly dispersed in the PVA matrix in the form of separated microspheres with a diameter about 20−40 μm, which can be directly obtained with polarizing optical microscope (POM, Figure 2b and Figure S2a) due to the birefringence of LC domains upon microphase separation.

Before the mechanical treatment, 5CB inside the separated LC domains was randomly distributed because no alignment was induced in the fabrication process. No photomechanical response was observed upon irradiation of visible light because of the little change in volume when phase transition occurred from non-aligned LC to isotropic phase.[13] After the mechanical stretching, structural anisotropy of LC domains was clearly observed due to the shape deformation from spheres to ellipsoids (Figure 2c & 2f and Figure S2b). Measuring with POM, the highest transmittance occurred when the angle between the stretching direction and the polarization direction of either polarizer was 45° (Figure 2d), while the lowest appeared when the stretching direction was parallel to one of the polarization directions (Figure 2e).[12b] This periodic change of dark and bright field of view was observed by rotating PDLC/GO nanocomposite films with an interval of 45°, indicating that 5CB was homogenously oriented along the stretching direction. As presented in Figure 2f, SEM image of the cross-sectional morphology of PDLC/GO nanocomposite films further confirmed the shape deformation of LC domains from spheres to ellipsoids upon the mechanical stretching.

Notably, it was obviously found that LC domains were inclined to locate in the upper part of PDLC/GO nanocomposite films, which is attributed to the fabrication process.[12a] Upon directly casting the mixture aqueous solution of GO, 5CB and PVA on the clean glass substrate to prepare the nanocomposite films, 5CB floated to the upper part of the mixture solution in the process during slow evaporation process of the solvent due to its lower density of 5CB compared with water (Figure S3). Thus, the bilayer structure of PDLC/GO nanocomposite films preferably formed based on this method; taking benefits to their light−responsive behaviors since most of photoresponsive behaviors of LCE films have been attributed to the so−called bimetal mechanism.[3]

2.3 Photoresponsive property of the nanocomposite films

The photoresponsive behaviors of stretched PDLC/GO nanocomposite films were studied using a visible light beam at 450 nm with an intensity of 54 mW/cm^2. As shown in Figure 3, the films bent toward the light source along the stretching direction upon photoirradiation, just like other LCE materials containing homogeneously aligned mesogens.[3,4] The in−situ measurement of the light−responsive process (Video S1) indicated that PDLC/GO nanocomposite films showed more faster responsive rate than our previously fabricated PDLC-like films stimulated with UV light.[12] This photoinduced bending behavior was completed within 4 s and longer photoirradiation did not cause anymore apparent changes to nanocomposite films. Turning off the light, the photoinduced deformation remained without detectable relaxation, indicating that bent nanocomposite films were obtained in a stationary state. For comparison, photomechanical behaviors of three mechanically−stretched sample films were fabricated and they are pure PVA, PVA/GO nanocomposite and PVA/5CB PDLC, respectively. It was discovered that all these films showed no response upon irradiation of the same visible light even with a higher intensity.

To further elucidate the mechanism of photomechanical behaviors of the PDLC/GO nanocomposite films, thermally−responsive characteristics of the three sample films stretched to the elongation rate of 100% were systematically studied (Figure S4), respectively. In these films, neither the pure PVA nor the PVA/GO nanocomposite films showed response when the temperature was higher than Tni (35 °C) of 5CB, even higher than the glass-transition temperature (Tg) (80 °C) of PVA. However, the PVA/5CB PDLC films apparently exhibited bending deformation behavior when the temperature was just higher than T_{ni} (35 °C), which was the same as that occurred in PDLC/GO nanocomposite films upon light irradiation. These obviously indicate that the thermal response of PVA/5CB PDLC films was not simply caused by the thermal expansion of PVA, but actually attributed to the LC−to−isotropic phase transition. When the temperature was higher than the Tni of LC, the LC exhibited remarkable contraction because the order degree of LC decreased due to the LC−to−isotropic phase transition which has been reported by many groups[1,3,6]. Thus, the contraction of pre-aligned 5CB in the upper layer of PDLC films due to the LC−to−isotropic phase transition was responsible for the mechanical behavior of PVA/5CB PDLC films. Therefore, it is reasonable to infer that light-responsive property of the present PDLC/GO nanocomposite film was related with some kind of thermal effect.

2.4 Mechanism for the photoresponsive property of nanocomposite films

In the PDLC/GO nanocomposite films, GO was designed as the only photo−absorbent and nanoscale heat source since both the LCs and the PVA are inert to visible light. When the visible light was switched on, GO can effectively absorb the visible light and transferred the light into thermal energy, thereby severing as nanoscale heater instantly increased the temperature of PDLC/GO nanocomposite films. As shown in Figure 4a, the temperature of PDLC/GO nanocomposite films increased from 25 °C to 48 °C within 4 s upon photoirradiation and fast decreased to 25 °C within 4 s when the light was switched off. The rapid response of the temperature to the visible light was attributed to highly thermal conductivity of PDLC/GO nanocomposite films by the incorporation of GO.[14] Reference to bimetal models for photomechanics of LCEs, possible mechanism of the light-responsive behaviors of the present PDLC/GO nanocomposite films can be proposed as Figure 4b.

Since the bilayer structure spontaneously formed during the fabrication process of PDLC/GO nanocomposite films, upper layer of nanocomposite films was rich in LC domains. Obviously, the lower layer of the nanocomposite films was short of the LC domains. Upon irradiation with visible light, the temperature rapidly increased to the temperature over Tni of 5CB. As a result, the volume contraction occurred along the mechanical stretching direction in the upper layer of nanocomposite films caused by the LC−to−isotropic phase transition of 5CB,[12] while the lower layer showed few changes because of lacking of LC domains. Thus, PDLC/GO nanocomposite films bent toward the light source along the stretching direction, as that of previously reported in LCEs.[15] In these films, the light−responsive behavior was based on the photothermal effect of GO leading to the phase transition of 5CB, unlike photoisomerization and photoinduced phase transition of AZ mesogens in LCEs.[16,17] Moreover, their structures are similar to photoresponsive bilayer nanocomposite films composed of active layer and passive layer reported recently by other groups.[18] Fortunately, unique bilayer structure spontaneously formed during the one-pot fabrication process of the PDLC nanocomposite films, which is potential for mass production from the viewpoint of applications.

Further to confirm our conjecture for the mechanism for the photomechanical response of nanocomposite films, we placed PDLC/GO nanocomposite films with lower layer lacking of LC domains towards the light source (see in Figure S5). Upon irradiation, the nanocomposite films bent away from the light source, which fully demonstrates that photoresponsive property of nanocomposite films was caused by the bilayer structure of nanocomposite films and phase transition of 5CB.

As mentioned above, the driving force for light−responsive behaviors of PDLC/GO nanocomposite films originated from the LC−to−isotropic phase transition of 5CB thermally−triggered by the photothermal effect of GO. To further understand the mechanism of the light−responsive property, effect of the 5CB concentration and mechanical stretching on the photomechanical behaviors of the PDLC/GO nanocomposite films were systematically investigated. Here, a parameter of bending degree (F) was introduced to quantitatively evaluate the macroscopic deformation, seen in Figure 5a. It is expressed as follows:

$$F = \frac{L_0 - L}{L_0} \tag{1}$$

where L_0 and L are linear length of PDLC/GO nanocomposite films before and after irradiation, respectively.

As shown in Figure 5b, the elongation rate exhibited great influence on the light−responsive behaviors of PDLC/GO nanocomposite films. When the elongation rate was lower than 120%, the bending degree almost linearly increased with increasing the elongation rate. While the elongation rate was higher than 120%, the F exhibited few changes. At a lower elongation rate, mesogens in the separated LC domains was not well aligned along the direction of the mechanical stretching, the LC domains showed no obvious volume change upon the LC−to−isotropic phase transition, resulting in small deformation of the nanocomposite films. On the contrary, good alignment of 5CB in the deformed LC domains achieved at a higher elongation rate showed a large volume change can cause the larger deformation. When the elongation rate was higher than 120%, almost all the 5CB mesogens in LC domains were aligned in one direction. Further stretching the nanocomposite film could not enhance the LC alignment, thereby little effected on the visible−light responsive behaviors.

Meanwhile, the 5CB concentration also showed great effect on the light−responsive behaviors of PDLC/GO nanocomposite films. The bending degree increased with increasing LC concentration at the same elongation rate. Obviously, at the same elongation rate, the volume change determining the deformation of nanocomposite films was almost proportional to the concentration of 5CB. Thus, larger deformation was achieved at a higher 5CB concentration. Above all things, the relationship between light−responsive behaviors of nanocomposite films and the LC concentration and mechanical stretching further confirmed that photomechanical response should be attributed to the LC-to-isotropic phase transition of 5CB in the separated mesogenic domains. Moreover, the light-responsive behaviors can be well controlled by adjusting the elongation rate and LC concentration.

Generally, fascinating reversible photomechanical behaviors of LCEs are often attributed to the phase transition of mesogens and excellent alignment of mesogens fixed by the crosslinked network.[1−4,12] The present nanocomposite films showed visible-light responsive property due to the LC−to−isotropic phase transition of 5CB. Without crosslinked network, the alignment of 5CB cannot automatically recover to their initial state induced by the mechanical stretching. However, randomly oriented 5CB can be easily realigned by restretching deformed nanocomposite films due to lower viscosity and low phase-transition temperature, endowing re-stretched nanocomposite films with light-responsive property again.[12] As a result, visible-light responsive behaviors can be achieved reversibly in the present nanocomposite films just by repeating mechanical stretching.

3. Conclusion

In summary, recyclable, fast and visible−light responsive PDLC/GO nanocomposite films based on GO, 5CB and PVA were fabricated by combination of solution casting and mechanical stretching. Bilayer structure spontaneously formed during the fabrication process of PDLC/GO nanocomposite films because of the lower density of 5CB compared with water. The light-responsive behaviors were attributed to the unique bilayer structure and LC−to−isotropic phase transition of 5CB induced by photothermal GO. The fast response was due to the low viscosity of LMW LC and high thermal conductivity of GO. Accordingly, most of general polymer materials can be utilized to fabricate visible-light responsive PDLC/GO nanocomposite films. These nanocomposite films can be potentially used in soft actuators and micro-optomechanical systems with visible light as the energy source.

4. Experimental Section

Typical fabrication process of PDLC/GO nanocomposite films was as follows. The graphene oxide (GO) solution was obtained through ultrasonic treatment of graphite oxide (30.3 mg) in 54 ml deionized water for two hours. Then it was mixed with 3.0 g PVA and 30.3 mg 5CB. After stirring the mixture solution for 48 hours at 95 °C, it was directly casting on clean glass slide and dry for one day at room temperature. Finally, PDLC/GO nanocomposite films were achieved by peeling off the glass slide. The dimensions of all prepared PDLC/GO nanocomposite films were around 70mm×20mm×0.8mm. In all nanocomposite films, PVA was fixed at 3.0 g and GO (30.3 mg) was used, about 1wt% of PVA. Moreover, four different 5CB concentrations 1, 3, 5 and 8wt% of PVA were selected for the purpose of investigating the effect of 5CB concentration on the light−responsive behaviors of PDLC/GO nanocomposite films.

For investigation of visible−light responsvie behaviors, the PDLC/GO nanocomposite films with the same dimension of 20mm×5mm×0.8mm were mechanically stretched to different elongation rates by a tensile test machine at the glass transition temperature (Tg) of PVA. Moreover, all the stretched PDLC/GO nanocomposite films were hold for 24 hours at room temperature to ensure the complete relaxation of residual stress.

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Figure Captions

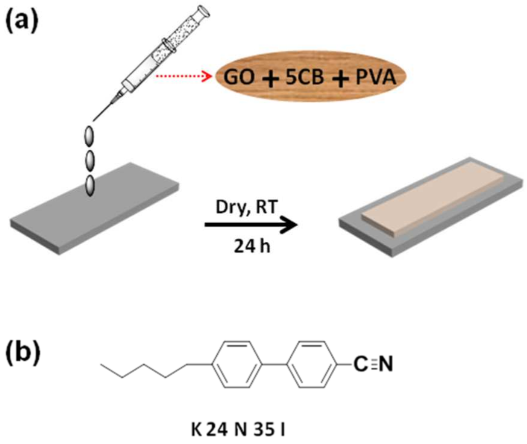
Figure 1. Materials used for the fabrication of the PDLC/GO nanocomposite films. (a) Schematic illustration of the fabrication process. (b) Chemical structure and thermal property of 5CB. K, crystal; N, nematic LC; I, isotropic phase.

Figure 2. Structural characterization of the PDLC/GO nanocomposite films. (a) Schematic illustration of the mechanical stretching process. The mechanical stretch behavior is conducted at 90°C. (b) & (c) are optical microscopic images of nanocomposite films with 3wt% 5CB and 1wt% GO before and after stretching with an elongation rate of 100%. (d) & (e) are POM images of mechanically stretched films with stretching direction parallel to one of the polarization direction and angle between the stretch direction and polarization direction of either polarizer was 45°. The scale bar is 40 μm. A, analyzer; P, polarizer. (f) is the SEM image of cross-section of the nanocomposite film with 3wt% 5CB and 1wt% GO.

Figure 3. Visible−light responsive behavior of the stretched PDLC/GO nanocomposite films. (a) Scheme illustration of photomechanical behaviors. (b) & (c) are photographs of the nanocomposite films before and after irradiation of visible light.

Figure 4. Photothermal effect of PDLC/GO nanocomposite films upon irradiation of visible light. (a) Temperature of PDLC/GO nanocomposite films as a function of on and off cycles of visible light (54 mw/cm^2). (b) Scheme illustration of possible mechanism of light-responsive behaviors.

Figure 5. Influence factors on the light-responsive behaviors of PDLC/GO nanocomposite films. (a) Definition of bending degree of PDLC/GO nanocomposite films. (b) Relationship between the 5CB concentration and mechanical stretching and the light-responsive behaviors.



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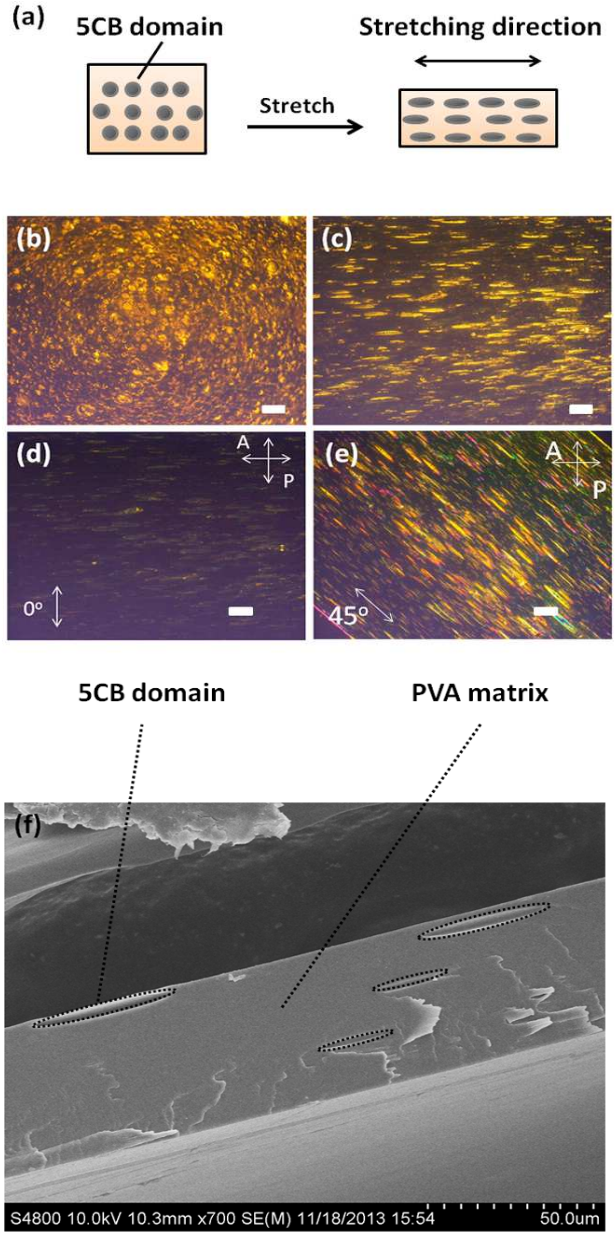
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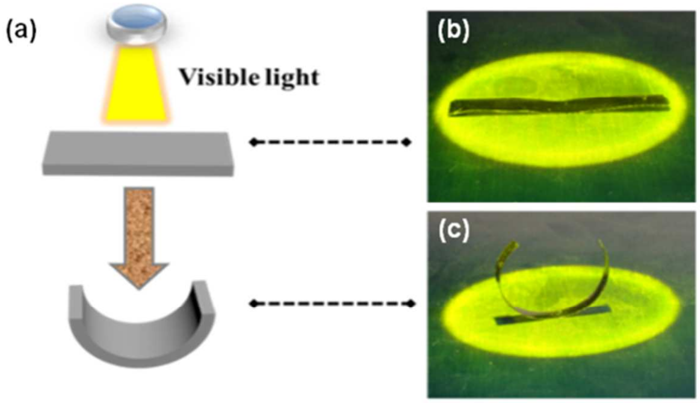

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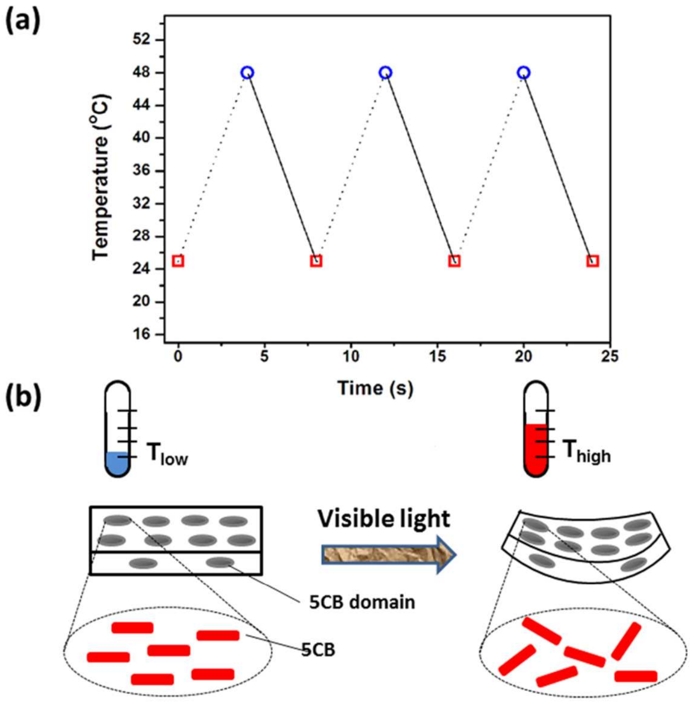
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Figure 4.

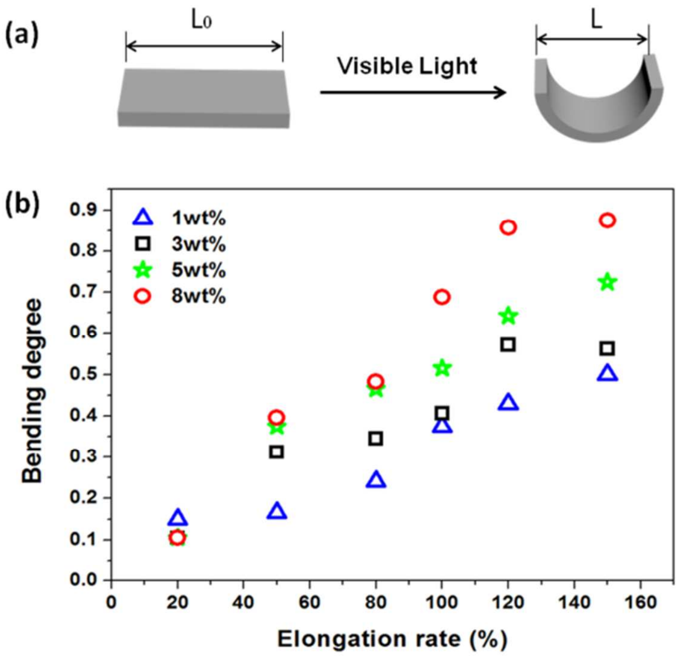

Figure 5.

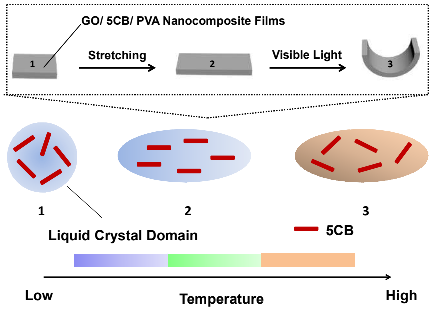

Table of Content

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