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Journal Name

ARTICLE

Stretchable Light Scattering Display Based on Super Strong Liquid Crystalline Physical Gels with Special Loofah-like 3D gel networks

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A unique stretchable liquid crystal light scattering display is reported in this paper with clear images shown at a maximum strain of 145% of the original length, which is based on super strong liquid crystalline physical gels with special loofah-like 3D gel networks.

Introduction

Liquid crystal molecules are a kind of fantastic materials that have special stimuli-response properties to lights, heat, magnet, electric, solvent and et al., and are used everywhere in our lives especially as display materials. Nowadays, flexible and soft display materials are no longer news for us,¹ and even stretchable displays² are reported to enable expandable and foldable screens for smart phones, wearable or fashionable electronic clothing, rollable or collapsible wallpaper-like lamps and biocompatible light sources for in-vivo or epidermal medical devices^{3,4}. The most popular stretchable displays research nowadays are basically focused on light-emitting displays (LED) with the obvious advantages of wide viewing angle, high brightness, and simpler constructions mainly attributed to the avoid of using polarizing films which is necessary in the internal structures of liquid crystal displays (LCDs). However, comparing to LEDs, LCDs are quite stable to oxygen and humidity, sensitive to all kinds of stimuli and the liquid crystal molecules in which have simpler structures that are easier to access. But considering the special nature of liquid crystals that is a kind of matter with its molecules may be oriented in a crystal-like way but can flow like a liquid, stretchable ability sounds like a mission impossible for LCDs. The very first problem needs to be solved is that a soft solid is needed instead of a liquid. Although changing liquid crystals into polymers or elastomers by polymerization or crosslinking is a traditional method to get solid state photomechanical optical devices⁵, gelators seems to be a better choice attributing to its ability that quite a few addition through a much simpler way could change a large amount of liquids (solvents) into gels, which is a typical kind of soft material. If liquid crystals are

used as solvents, we could get liquid crystalline physical gels that are a novel class of stimuli-responsive soft materials with applications in electro-optical switchable devices,^{6, 7} LC semiconductors with high mobility,^{8, 9} fast-response switching in twisted nematic cells,¹⁰⁻¹² stabilization of the orientation of ferroelectric LCs,¹³ et al. Besides the multiple stimuli-responsive properties including electric response, thermal response and photo response, LC physical gels have easy processing ability due to its sol-gel transition behavior. One can pour sol into any shape of containers and after the gelation process, a certain shape of gel could be obtained, and such "pour" process could take good advantage of many processing methods including pouring, spin-coating, roller coating, injection molding, and even 3D-printing¹⁴. But to the best of our knowledge, nearly all the LC physical gels reported are not strong enough to be utilized with their mechanical properties, not to mention the comprehensive application of both stimuli-response and mechanical properties.

We reported a novel kind of POSS core gelators with strong gelation abilities that can self-assembly into a special loofah-like three dimensional (3D) gel networks in normal organic solvents,¹⁵ which is supported by a sectional type hexagonal columnar assembly mode rather than physical entanglement of the length-limited fibers normally reported in the literatures. This unique structure can macroscopically manifest itself as a highly efficient gelator, and has a strong ability to resist external stress and exhibits a distinct viscous flow state. Such kinds of gelators with strong self-assembly properties were introduced into LC host with azobenzene dopant in this paper and a species of super strong LC physical gels with quite high $T_{sol-gel}$ (sol-gel transition temperature of gels) and strain resistance could be obtained with quite low amount of gelator addition. Furthermore, the photo-responsive properties coming from the azobenzene dopant provide the result LC physical gels photo-responsive properties in addition of thermo-responsibility of the LC host. As a result, the contrast ratio between the different responsive states of the LC physical gels is large enough to show clear images so that the indispensable polarizers in traditional LCDs are totally deprecated. Although a similar kind of

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light scattering display without polarizes has already been researched years ago^{16,17}, the mechanism and multiple applications of which are also based on the contrast of scattering/transparent state of the light scattering display, getting rid of polarizes makes the display system so simple that provides us an outstanding opportunity to make full use of the extremely strong properties of the LC physical gels to obtain a stretchable LC light scattering display which could remain clear images even being stretched to 145% of the original length, which has never been reported before.

Experimental

Materials

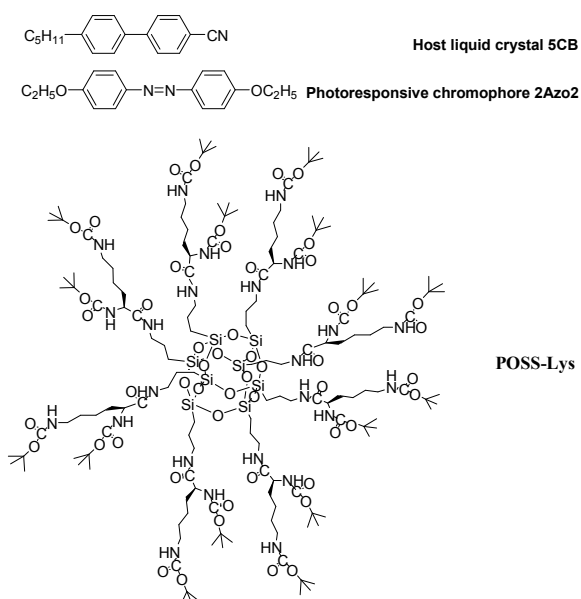
All the materials required in this reaction were commercial available. Octa(3-aminopropyl) silsesquioxanes hydrochloride (Octa Ammonium POSS-HCl) was purchased from hybrid plastic (Hattiesburg, MS). 1-hydroxy benzotriazole (HOBt), N-(3-Dimethylaminopropyl)-N'-ethylcarbodiimide hydrochloride (EDCI), N α ,N ϵ -dit-BOC-L-lysine [(di-t-BOC)-L-lys-OH], N-Methylmorpholine (NMM) were supplied by Aladdin. Co., Ltd (Shanghai, China) and 4,4'-Diethoxyazobenzene (97%, 2Azo2) were supplied by Alfa Aesar (China) Chemical Co., Ltd and used as received. The nematic liquid crystals 5CB (as shown in Scheme 1, $T_{IN} = 35.0$ °C) were purchased from Shijiazhuang Kerun Display Material Co., Ltd in China. All the solvents used in the synthesis were analytical pure and used without further purification. Silica column chromatography was carried out using silica gel (200-300 mesh) provided by Qingdao Haiyang Chemical. Co., Ltd (Qingdao, China). Thin layer chromatography was performed on commercially available glass backed silica plates. A thermal crosslinkable urethane liquid rubber compound provided by Zhixin Science and Technology Co., Ltd (Shanghai, China), (ClearFlex95, mixed at a weight ratio of 2:3 parts A:B) are used as elastic substrate.

Characterization

The structure of the product was determined by NMR (Bruker Avance III, 500 MHz), ESI-TOF MS (Agilent 6210) and MALDI-TOF MS (Bruker Autoflex III TOF/TOF) in linear mode with α -cyano-4-hydroxycinnamic acid as a matrix.

Tube inversion method is operated in the following procedure. The gelator sample is mixed with host mixtures in a sealed test tube and the mixture was heated to certain temperature until the solid was completely dissolved. Then the solution is spontaneously cooled to room temperature in air, and finally the test tube was inverted to observe whether the solution inside could still flow. Gelation was considered to occur when a homogeneous "gel-like" material was obtained which exhibited no gravitational flow. The gel-sol transition temperature (T_{gel}) was measured with the "tube inversion" method, in an oil bath which was heated slowly.

FESEM measurements were taken on a Hitachi S-4700 field emission scanning electron microscope (FESEM, Hitachi, Japan) for the morphological analysis. The samples were prepared as follow: the gel formed in a glass vial by a heating-cooling process. And the pre-prepared gels was put into an extractor with n-hexane as solvent and extracted for 24 h. After that, the samples were taken out and severally allowed to dry under vacuum to a constant weight. Then



Scheme 1 The structure of the host liquid crystal (5CB), photoresponsive chromophore (2Azo2) and POSS based dendrimer gelator (POSS-Lys)

the resulting xero-gel was respectively coated with a thin layer of gold before investigation.

Rheological measurements were carried out on freshly prepared gels by using a controlled-stress rheometer (MCR302, Anton Paar, Austria). These gels were obtained by a heating-cooling process. Parallel-plate geometry of 25 mm diameter and 1 mm gap was employed throughout the dynamic oscillatory work. The tests were performed as follows: the sample was submitted to this parallel-plate very quickly to minimize solvent evaporation, and then increased the amplitude of oscillation up to 100% apparent strain shear (kept a frequency of 1 rad s⁻¹) at 25 °C.

The transparency switched for the LC gels with UV irradiation was measured by using Transmittance Tester (WGT-S, Changfang Optical instrument co., LTD, Shanghai, China). The test sample of the LC gel display device was obtained by injecting the sol into a 7 μ m thick LC cell at 150 °C and then cooled to room temperature. The initial transparency was tested. Then the samples were exposed to 35.4 mW/cm² UV irradiation of 365 nm wavelength for every 30 seconds and tested the transparency immediately until no clear changes could be observed.

Synthesis and Sample Preparation

Synthesis of Gelators

The chemical structures of the commercial available LC host 5CB, azobenzene dopant 2Azo2 and POSS-Lys are shown in Scheme 1.

The synthesis of POSS-Lys was reported in the literature [13]. The structure of the gelator is shown in Scheme 1. ESI-TOF: The calculated $[M+3Na]/3$ of POSS-Lys was 1191.96 and the tested result was 1192.3; calculated $[M+2Na]/2$ was 1776.44 and the tested result was 1776.9. MALDI-TOF (m/z , $[M+Na]^+$): 3528.87 (calculated for C₁₅₂H₂₈₈N₂₄O₅₂Si₈Na), 3528.80 (measured). PDI 1.02 (GPC, polystyrene). ¹H NMR (CDCl₃, 500 MHz, ppm), δ 7.58

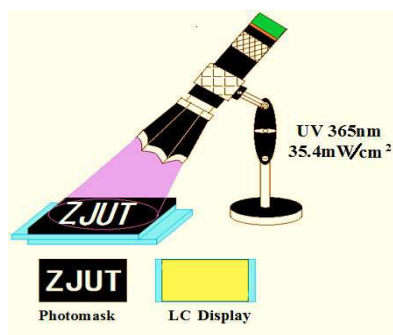


Figure 1 Schematic illustration of the writing process of the stretchable LC light scattering display

(8H, br, CONH), 5.88 (8H, br, NHBoc), 4.85 (8H, br, NHBoc), 4.22 (8H, br, COCH(R)NH), 3.30 (16H, br, CH₂NHCOCH(R)), 3.08 (16H, br, CH₂NHBoc), 1.71-1.35 (208H, m, CH₂, CH₃), 0.63 (16H, m, SiCH₂CH₂(R)).

Preparation of Substrate

The elastic substrate was obtained through introducing a precursor solution consisting of ClearFlex95 into the small gap between two pre-treated glass slides and then cured at 110 °C for 10 min before peeling off as a free-standing film. The glass slides were cleaned by acetone and isopropanol before being applied a hydrophobic layer. The thickness of the elastic substrate was determined by the fixed spacer between the glass slides.

Preparation of LC physical gels

The LC physical sol was prepared by dissolving certain amount of gelators and 2Azo2 in 5CB at 150 °C in stoppered tubes on a hot stage under constant agitation.

The liquid crystal light scattering display was prepared by drop-casting the homogeneous sol onto the elastic substrate and then cooled to room temperature.

The displayed image on the device was obtained by the use of a "ZJUT" photomask, as shown in Figure 1: the thin cell was covered with the "ZJUT" photomask and exposed to 35.4 mW/cm² UV irradiation of 365 nm wavelength for 3 min at room temperature, and erased with visible lights (455 nm, 26.5 mW/cm²) for 5 s at room temperature.

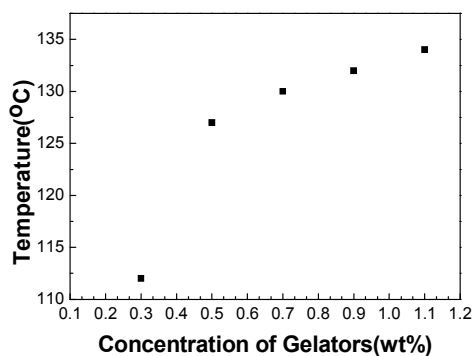


Figure 2 Phase diagram of LC gels

Results and discussion

Gelation behaviors

The POSS-Lys gelator has a very strong gelation ability that could self-assemble into a special loofah-like gel network in common organic solvents including MMA, benzene, chlorobenzene and ethyl acetate.¹⁵ The POSS-Lys gelator could also uniformly dissolved in the LC host of 5CB with 2Azo2 (5 wt%) mixtures at temperatures higher than $T_{sol-gel}$. The relationship between gelator concentration and $T_{sol-gel}$ is shown in Figure 2. Most of the LC physical gels reported in the literatures¹⁸ have a much lower $T_{sol-gel}$ than the isotropic phase transition temperature (T_{iso-LC}), indicating only weak interactions existing in the gel networks and gel could no longer exist at the isotropic phase of the LC host. But because of the special gelation ability of POSS-Lys gelators, the resultant LC physical gel has a so much higher $T_{sol-gel}$ that it could keep the stable gel status at the temperature much higher than T_{iso-LC} . The host LC mixtures have a nematic-isotropic phase change temperature (T_{n-i}) of 42 °C. From Figure 2 we could find out that even at the lowest gelator concentration of 0.3wt%, the $T_{sol-gel}$ is still as high as 112 °C, which is 70 °C higher than the T_{iso-LC} of the host LC mixtures, indicating a super strong LC physical gels was obtained through the little amount addition of such special POSS core gelators.

As mentioned above, the POSS-Lys gelator reported by our group¹⁵ has super strong self-assembly ability attributed to the special loofah-like gel networks in normal organic solvents which is supported by a sectional type hexagonal columnar assembly mode rather than physical entanglement of the length-limited fibers. The organic solvents we have tried including MMA, benzene, chlorobenzene and ethyl acetate, and the typical loofah-like xerogel network morphology obtained in MMA is shown in Figure 3a. When the host was changing into liquid crystals, that is 5CB doped with 2Azo2 (5 wt%) in this case, the morphology of xerogel aggregations of POSS-Lys formed in the mixture of 2Azo2/5CB was shown in Figure 3b, which was obtained from drying the gels acquired by throughout removing the LC host molecules with hexane. The process could be understood as the balance of the gelator-gelator and gelator-solvent interactions. Considering the interactions between LC molecules are stronger than the normal solvents, gelators in LC host forms more loose aggregates, shown as in Figure 3b with a much larger scale bar. Additionally, it is worth mentioning that removing the LC host using hexane leads to the collapse of those xerogels at such low gelator content of 0.5 wt%, that's the reason why the morphology in Figure 3b is not as well-shaped as in

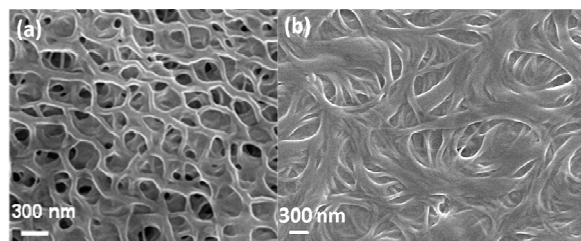


Figure 3 SEM images of the loofah-like xerogel network morphology obtained in (a)MMA (0.5 wt%); (b)LC host (0.5 wt%) which has been removed by rinsing with hexane.

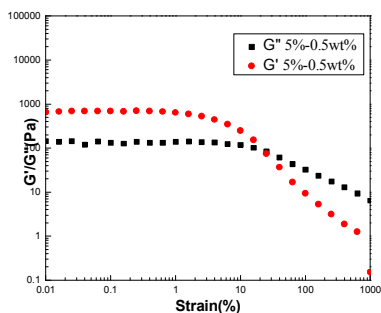


Figure 4 Dynamic rheological measurement of storage modulus G' and loss modulus G'' versus strain at angular frequency of 6.28 rad/s of the LC gel (0.5wt%).

Figure 3a. Although the morphologies from Figure 3a and 3b seems not exactly the same, the existed intertwined self-assembled nanobricks to form 3D networks in these LC xerogels also tell the truth that POSS-Lys self-assembly into a similar loofah-like gel network in LC host.¹⁹⁻²¹ And such special self-assembly process could also provide a super strong LC physical gel with quite high sol-gel transition temperature.

Dynamic rheological characterization

To further research the mechanical properties of the LC physical gels, dynamic rheological measurement was performed in the strain sweep test with a frequency of 6.28 rad/s at 25°C to characterize the mechanical properties of the above LC gels, and the measured storage modulus (G') and loss modulus (G'') are shown in Figure 4. From Figure 4 we could find out that when strain < 1%, the G' and G'' of the LC gel keep nearly unchanged which means the gel is at the linear viscoelastic region that the elastic and viscous of which are constant. When the strain increases, G' decreases quickly while G'' is invariant until intersecting with G' and then also decreases quickly. The intersection point of G' and G'' is the point of gel-sol transition, before which $G' > G''$, which indicates that the gel system formed a stable continuous elastic network. After the intersection point, $G' < G''$ and the gel network is no longer continuous so that the mixture is in viscous state. The intersection point is as large as 25% strain here in the LC gel, which is nearly 50 times larger with a at least 40 times lower gelator content than that of microparticle-LC composite gels.²²

Photo-controllable ability

So how is the photo-controllable ability of such LC physical gels supported by the special loofah-like 3D gel networks with strong mechanical properties? Considering 5% photosensitizer 2Azo2 is added into the host mixtures which have trans-cis photochromic behaviors, the transparency could also be controlled by the wavelength of the incident light. That is saying that using the trans-cis transition to destroy the alignment of 5CB will result in the isotropic phase of the host LC mixtures to obtain a transparent state, which is because when the azo dyes dispersed within the host liquid crystals itself are dis-oriented by illumination with UV light, dis-alignment can be induced in the host liquid crystal, the mechanism

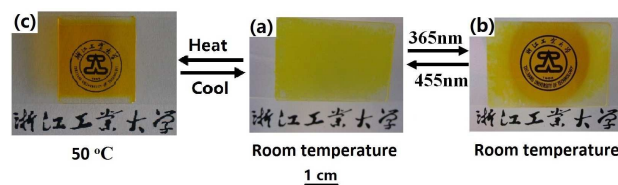


Figure 5(a) The sample in the initial opaque state or after irradiation at 455nm at room temperature; (b) The sample after irradiation at 365nm at room temperature; (c) The sample after being heated at 50°C.

of which is shown in Figure S1 in ESI†, as reported in the literature^{23,24}. The opaque state (with the trans perform of 2Azo2) and the transparent state (with the cis perform of 2Azo2) of the LC gels is so obvious that such kind of LC gels could be used as a new kind of LC displays without polarizing films. We all know that in normal LC displays, polarizing film is indispensable because liquid crystal molecules could only adjust the birefringence to light but could not acquire dark field by themselves, and two orthogonal polarizing films are necessary for produce a dark field for the LC cell between them, as shown in Figure S2 in ESI†. And the existence of polarizing films is the reason why liquid crystal displays have a relatively narrow visual angle. Getting rid of the polarizing films makes the assemble of LCD devices more easier and give the possibility of produce stretchable displays based on liquid crystals.

LC gel display device

The LC gel display device is obtained by injecting the sol into a 7μm thick LC cell at 150°C and then cooled to room temperature. Initially, a homogeneous opaque LC gel is obtained as shown in Figure 5a. After being irradiated by 365nm UV lights (35.4 mW/cm²) for about 3 min at room temperature, the LC gel changes to transparent state as shown in Figure 5b. Such transition could be controlled through the wavelength of the incident light. And a similar opaque-transparent state transition could also be obtained through changing the ambient temperature of the sample from room temperature to higher than 42°C (T_{n-i}), as shown in Figure 5c. The contrast between these two statuses could be directly used to get a clear image without using polarizing films. And the quantitative data regarding to the value of the transparency switched for the LC gels with UV irradiation was shown in Figure 6, indicating a significant difference of the transparency of the transparent and opaque state which could provide clear image information. Furthermore, the UV-vis cyclability test of the LC gel display device verifies that the transparency of both transparent and opaque state shows no obvious change even after 8 cycles, as shown in Figure S3 in ESI†. When the LC gel was exposed to UV light through a “ZJUT” pattern photomask, a light-induced isomerization in the irradiation site took place, resulting in apparent image information shown clearly on the LC cell, as shown in Figure 7. Although the picture shown in Figure 7 was taken at room temperature, the clear image could keep stable until the T_{n-i} of the host LC mixtures (42°C). It was named light scattering displays based on the imaging mechanism, which is shown in Figure S2 in ESI† as reported in the literature^{18,23,24}.

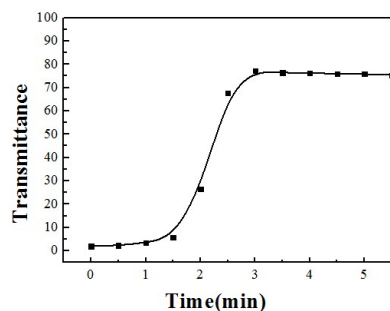


Figure 6 Transparency of LC gels with different UV irradiation time



Figure 7 Liquid crystal physical gel for light scattering display at room temperature

Stretchable light scattering displays

Successively getting rid of the polarizing film makes the fabrication of light scattering displays so much easier that we further try to make full use of the characteristics of gels, including soft and good processing abilities, and super strong mechanical properties, to obtain stretchable light scattering displays. As a traditional display material, LC molecules could easily fulfil all the requirements for displays, not to mention getting rid of polarizing films solved the narrow view angle problem. By the mean time, the sol-gel property provides a really easy method for preparing LC displays, and the addition of photosensitizer in this paper provide a non-contact writing and erasing way to display the image. So we try to drop-cast the LC gel onto a stretchable substrate through sol-gel process and using different wavelength of incident lights to long-distance control the image without a contact with the LC display films. Images on the soft stretchable substrate could be achieved as clear as on glass substrate by using the same writing process. After that, the obtained stretchable display is fixed on a homemade tensile stage and stretched as uniformly as we could by manual rotating the control nuts (as shown in video 1 in supporting information). The screenshot of the initial state of the display is shown in Figure 8 (a) with an effective length of 3.5cm. And the images keep clear during the stretch process and can be stretched as long as 5.1cm as shown in Figure 8(b). So the LC display can be stretched up to 145% of its original length with the pictorial information clearly shown on it.

Conclusions

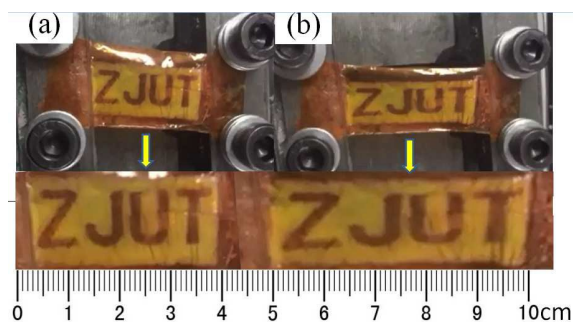


Figure 8 The screenshot of (a) the initial state of the display with an effective length of 3.5cm;(b) the LC display is stretched up to 145% of its original length with an effective length of 5.1cm.

We have shown that a high-performance, stretchable LC light scattering display can be fabricated using a relatively simple, solvent free, sol-gel based process. Such LC light scattering display is fabricated by adding quite little amount (≤ 0.5 wt%) POSS core gelators into LC host doped with 5% photosensitizers and drop-casting the result LC sol onto stretchable substrate. The non-contact writing process was done by using 365nm wavelength incident beam through a mask, and as a result, clear image could be shown on the LC display because of the high contrast between the exposure and unexposure area. In this way, no polarizing films are needed and that makes the fabrication process even simpler. The resulting LC display exhibits stretchability at room temperature and can clearly show pictorial information at strains as large as 145%. Considering the fabrication process is quite simple, can be achieved through many kinds of traditional process technologies including drop-casting, spin-coating and high temperature flame plating, and the size of the LC display can be easily obtained as large as you want (larger sample with about 10 times area of the sample in Figure 8 was shown in Figure S4 in ESI†, which could also be stretched up to 145% of its original length with the pictorial information clearly shown on it), we believe that fully stretchable LC light scattering displays will have widespread applications in the future.

Acknowledgements

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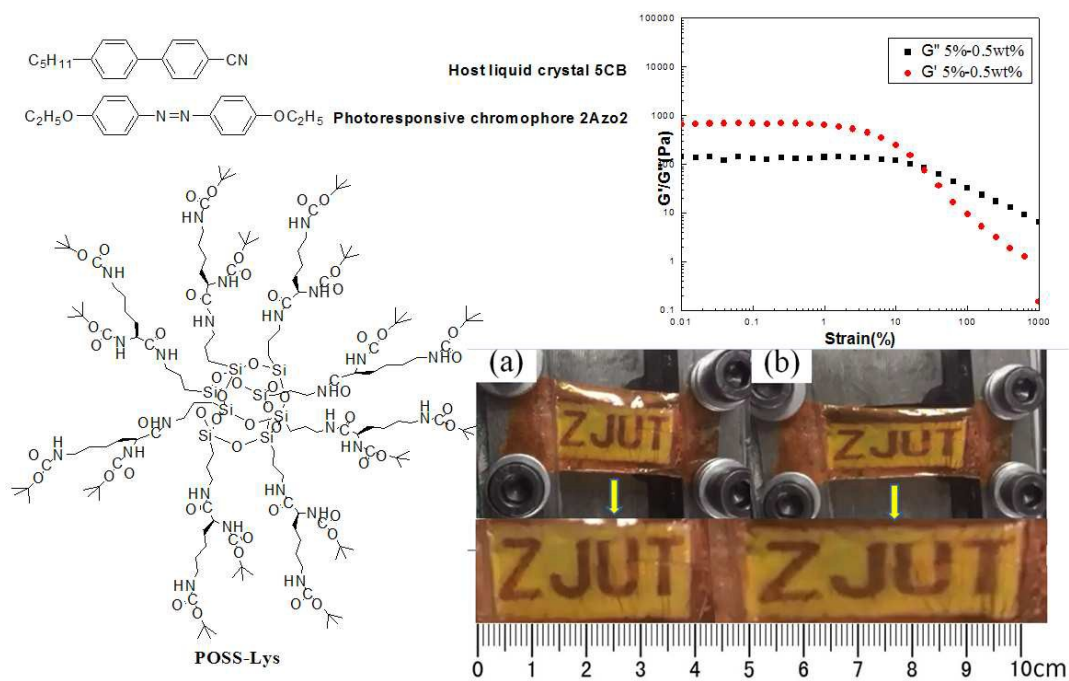
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