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Article

Characteristics Analysis of Plasticized Polyvinyl Chloride Gel-Based Microlens at Different Temperatures

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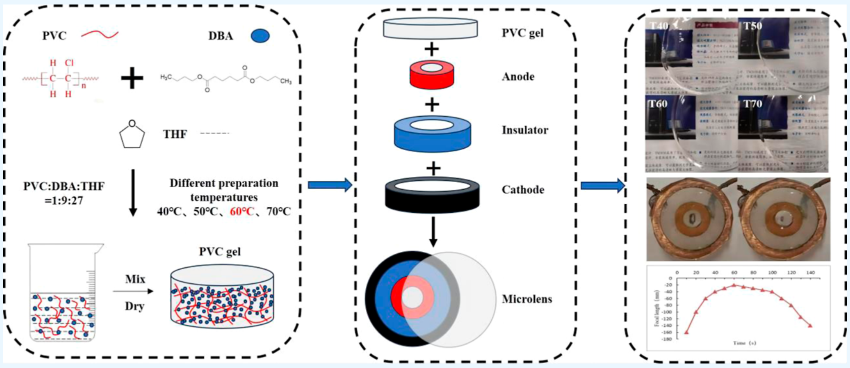

ABSTRACT: Temperature plays a crucial role in the preparation of polyvinyl chloride (PVC) gels for optical applications. Incorrect temperature selection can lead to various issues such as poor surface roughness, inadequate light transmission, and insufficient solution for optical devices. To address this challenge, this study focuses on the preparation of PVC gel samples by combining PVC powder (n = 3000), eco-friendly dibutyl adipate, and tetrahydrofuran at different stirring temperatures ranging from 40 to 70 °C. The PVC gel preparation process is categorized into four groups (*T*40, *T*50, *T*60, and *T*70) based on the mixing temperatures, employing a controlled test method with specific temperature conditions. The prepared PVC gel samples are then subjected to analysis to evaluate various properties including surface morphology, tensile strength, light transmittance, and electrical response time. Among the samples, the PVC gel prepared at 60 °C (referred to as T60) exhibits excellent optical properties, with a transmittance of 91.2% and a tensile strength of 2.07 MPa. These results indicate that 60 °C is an optimal reaction temperature. Notably, the PVC gel microlenses produced at this temperature achieve their maximum focal length (ranging from −8 to −20 mm) within approximately 60 s, and they recover their initial state within around 80 s after the power is switched off. This focal length achievement is twice as fast as reported in previous studies on microlenses. It is observed that the reaction temperature significantly influences the solubility of the resin-based raw materials and the homogeneity of the gel. Consequently, these findings open up possibilities for utilizing PVC gel microlenses in novel commercial optics applications, thanks to their desirable properties.

■ INTRODUCTION

Smart polymer gels undergo shape changes in response to external stimuli such as pH, ambient temperature, external electric fields, and other factors.[1−3] Soft electro-stimulated gels, which are made from electroactive polymers, have demonstrated remarkable mechanical motion when activated with electrical energy. These gels exhibit enhanced mechanical and responsive properties through the application of a low-level electrical field.[2] Specifically, polyvinyl chloride (PVC) gel is an innovative material that mimics the mechanical deformation of human muscles and possesses a unique cross-linked three-dimensional structure formed by strong chemical bonds combined with physical field effects. The affordability of raw materials makes PVC gels a cost-effective option, and they have gained prominence in the field of flexible actuators and lenses due to various advantageous factors.[4−8]

A PVC gel-based actuator was developed by researchers, consisting of a positive metal mesh, a negative copper sheet, and a PVC gel positioned in the center. The actuator exhibited

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responsiveness when a voltage was applied.[9,10] PVC gels have demonstrated excellent direct current (DC) voltage response. Upon applying a DC voltage to the surface of the PVC gel, the polar molecules and plasticizer particles within the gel migrate toward the anode due to their polarity.[4,11] Another study focused on investigating the polarity and electrical stimulation response characteristics of PVC gels. Polarity primarily affects electrical conductivity by influencing the mobility of charge carriers. Electric fields are generated in polar materials like water or certain organic compounds due to the separation of positive and negative charges. These electric fields can exert forces on charged particles, influencing their movement. However, when a voltage was applied to the PVC gel, the negative charges from the cathode migrated through the gel toward the anode, resulting in bending deformation.[12−14] This deformation occurred due to the Maxwell stress effect, where the plasticizer dibutyl adipate (DBA) molecules in the PVC gel became charged under the applied voltage. As a result, the DBA molecules were polarized, causing the PVC chains to open and allowing DBA to pass through the PVC gel network.[9,15] In our recently reported work, we developed a novel soft composite gel by blending functionalized carboxylated cellulose nanocrystals (CCNs) with plasticized PVC. These PVC/CCN gel composites exhibited increased polarity and electrical conductivity, making them suitable as filling materials for soft body actuators with fast response times.[16]

PVC gel is predominantly composed of organic solvents and plasticizers. Through the evaporation of volatile organic solvents from the solution, a soft PVC gel is obtained. The mechanical strength and other properties of the PVC gel are influenced by the quantity of plasticizers used.[17] In a published study, a PVC gel was formulated with a PVC to DBA ratio of 1:4 (by mass). This PVC gel-based actuator has been proposed as a means to assist individuals with paralysis in walking.[18,19] PVC gel represents a softer variation of traditional PVC material. Further research has explored shaping the PVC gel into jagged waveforms using molds. This novel shape increases the contact area with the anode surface, resulting in more pronounced deformation during actuation.[20] A recent development in the production of PVC gel involves employing a solvent-free injection molding method using electroactive PVC resin. This approach enhances the environmental sustainability of the resulting PVC gel product.[21]

However, due to the favorable electrical stimulation response characteristics and high transparency of PVC gel, several studies have utilized an assembly of indium tin oxide (ITO) conductive glass. In this assembly, the PVC gel is positioned as the middle layer, sandwiched between the negative and positive metal rings. The assembled structure is then subjected to stimulation through DC high-voltage action. By shrinking the stimulated gel toward the anode hole, target imaging is achieved. When the DC voltage ranges from 200 to 800 V/mm, the focal length of the microscope varies from 3.8 to 22.3 mm.[22] In another published work, the utilization of ITO glass and its impact on the material's light transmission and absorption were examined. Additionally, a "bridge" microlens structure, comprising a positive, insulating, and negative ring configuration, was proposed. The PVC gel is sandwiched between these electrodes, and a voltage ranging from 0 to 1000 V/mm is applied to the surface. The deformed PVC gel undergoes creep along the surface of the positive circular ring, resulting in a concave lens-like effect. The measured focal length of the microlens ranges from −mm to −21.3 mm.[23,24]

Based on previous research, it has been observed that when PVC gel is fabricated at room temperature, its transparency is negatively affected, thereby impacting the performance of microlenses. In order to address this issue, we hypothesized that the preparation temperature plays a significant role in determining the transparency of PVC gel. Therefore, we carefully selected a temperature range above room temperature yet below the safe usage temperature for tetrahydrofuran (THF). Considering these factors, the present study aimed to prepare PVC gel samples by mixing PVC, DBA, and THF at temperatures ranging from 40 to 70 °C. For the preparation of the PVC gel samples, we opted for a mass ratio of PVC/DBA/THF = 1:9:27. To assess the properties of the PVC gel samples, we conducted experiments using a self-assembled setup specifically designed for microlens evaluation. Moreover, we characterized the mechanical properties, surface characteristics, and transparency of the PVC gel samples.

■ EXPERIMENTAL SECTION

Materials. The details of materials used to prepare PVC gels are given in Table 1.

Table 1. Test Material Parameter Information

experimental materials	CAS number	properties	remarks
PVC resin powder	9002-86-2	melting point: 170−195 °C	please refer Figure 1a
polymerization degree		boiling point: 0.100 °C	
(n = 3000)		density: 1.4 g/mL at 25 °C (lit.)	
		refractive index: 1.45	
		storage conditions: 2−8 °C	
		form: powder	
DBA plasticizer	105-99-7	melting point: −32.4 °C	please refer Figure 1b
		boiling point: 305 °C	
		density: 0.962	
		flashpoint: >110	
		form: liquid	
THF organic solvent	109-99-9	boiling point: 66 °C	please refer Figure 1c
		water solubility: soluble	
		density: 0.89 g/cm^3	
		flashpoint: −14 °C	
		form: liquid	

Preparation of Gel Samples. PVC resin powder, plasticizer DBA, and THF organic solvent were used to prepare the PVC gel samples. The PVC gel samples were prepared from raw materials by the solvent casting method with a mass ratio of PVC/DBA/THF = 1:9:27.[23] To investigate the effect of reaction temperature on PVC gel, four PVC gel samples were prepared at four different temperatures. For stirring, the water bath heating device was warmed to 40, 50, 60, and 70 °C and the mixing speeds were set to 1000 samples rad/min. After mixing, the gel mixture was poured into a 100 mm glass Petri dish and dried at room temperature for 3 days to remove the solvent. Finally, the desired PVC gel was obtained, and the thickness of the final prepared gel was set at 1 mm. The obtained PVC gel samples at temperatures (40−70 °C were named T40, T50, T60, and T70, respectively. Figure 2 depicts the above preparation

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Figure 1. Pictures of the materials used for the preparation of the PVC gels: (a) PVC powder; (b) DBA plasticizers; and (c) THF solvent. Photograph courtesy of “Li Xudong”. Copyright 2023.

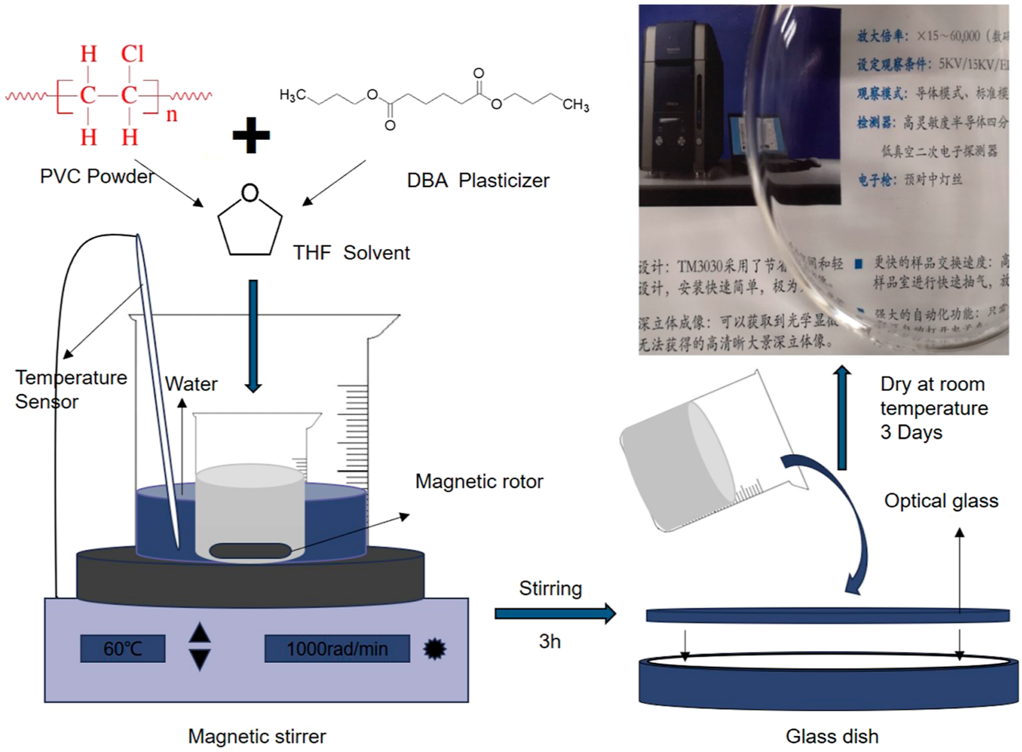

Figure 2. PVC gel samples’ preparation process. Photograph courtesy of “Li Xudong”. Copyright 2023.

process while Figure 3 depicts the schematic diagram of the optical flat glass assembly.

The main experimental equipment in this experiment is a magnetic heating stirrer, vacuum drying oven, and freeze dryer. The experimental process requires using the water bath heating method and the preparation of raw materials in a beaker. The stirring operation is completed on the magnetic stirrer. Finally, the stirred and mixed gel solution is dried in the vacuum drying oven. When the samples are tested for electrical stimulation response, the drive model is also used with a high-voltage DC power supply. Customized experimental equipment, such as planar optical glass, evaporative crystallization dishes, etc., are also used in the research work.

Figure 3. Evaporating crystallization dish (left) and optical flat glass (right). Photograph courtesy of “Li Xudong”. Copyright 2023.

Testing and Characterizations. The PVC gel samples used in this study were cut into various shapes for testing. The PVC gel samples used for the tensile strength test were cut as rectangles of 10 × 60 × 1 mm and tested by using a universal electronic material testing machine (Universal electronic material testing machine 9002-86-2 , Dongguan Ruiyu Chemical Co.) at room-temperature conditioning. The speed of the tensile machine was set to 20 mm/s and the standard was ASTM D638. Scanning electron microscopy (SEM, Phenom Pro) was used to analyze the surface morphology of pure PVC gels. For SEM testing, the samples were first freeze-dried while the surface of

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ACS Omega 2023, 8, 28924−28931

the freeze-dried samples was metallized and the surface morphology was observed under vacuum conditions at room temperature. The PVC gel samples used for the light transmission tests were set up as 5 × 30 × 10 mm rectangles and tested at room temperature using a visible spectrophotometer (F98 Shanghai, Prism Technology Co). During the experiments, the gel samples were required to be placed in the sample chamber of the photometer, and a light source with a wavelength of 200−1200 nm was applied to the samples to test the loss (transparency) in each wavelength band after the light source passed through the samples.

The PVC gel samples used for the microlens electrode assembly were arranged in the shape of a $\varphi 10 \times 1$ mm disk. The electrode assembly is cylindrical in shape and consists of a concentric and equal-height positive metal inner ring with a plastic insulating barrier and a negative metal outer ring. A DC voltage of 1000*V* is applied after PVC the gel is laminated to the electrode surface. A laser displacement meter is used to observe and record PVC gel deformation and response time.

RESULTS AND DISCUSSION

Effect of Reaction Temperature on the Properties of Gel Materials. *Influence of Reaction Temperature on PVC Gel Transmittance.* As shown in Figure 4, the PVC gel films of each group were initially compared in their normal state. The PVC gel of T60 clearly has the highest transparency.

Figure 4. Comparison of gel transparency effects of PVC gel samples. Photograph courtesy of "Li Xudong". Copyright 2023.

Several factors influence the transparency of optical devices, including light transmission, absorption, and light scattering by material impurities. The uneven surface of the material affects the transparency, which affects the scattering effect of the material. As a result, a visible spectrophotometer was used to measure the transmittance and absorbance of the gel samples, and the results are shown in Figures 5 and 6. The results showed that in the visible wavelength range (300−1100 nm), the transmittance of the samples increased and then decreased as the reaction temperature increased. The highest transmittance of the samples in the T60 group was 92.8%, nearly comparable to the market optical flat glass of the same thickness. These findings

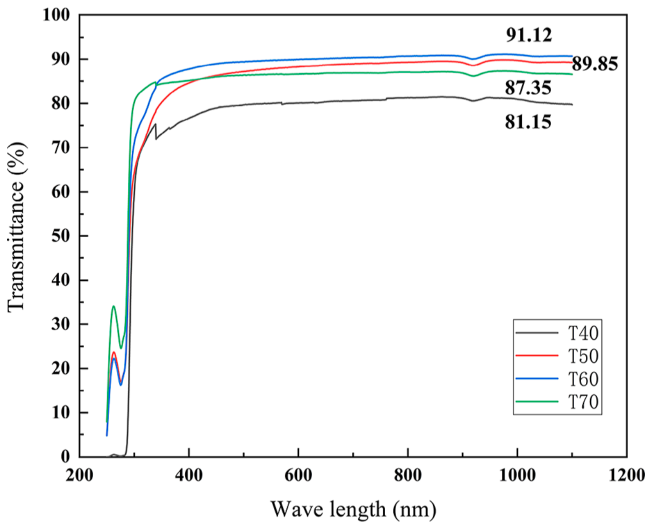

Figure 5. Gel light transmittance test.

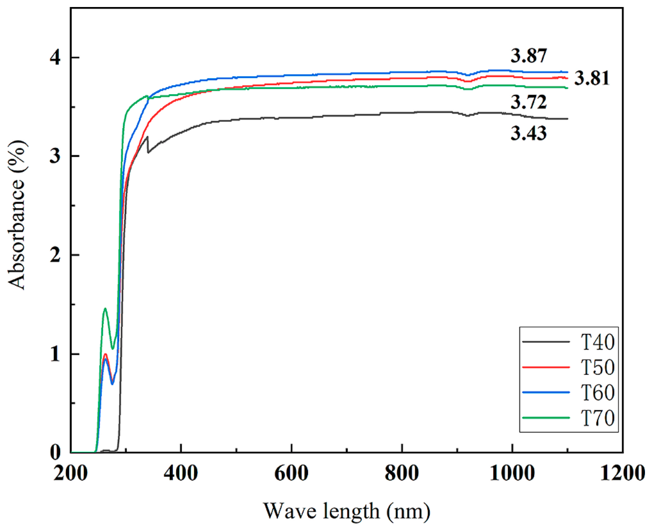

Figure 6. PVC gels' absorbance test.

are attributed to the reaction temperature exceeding the solvent's evaporation temperature. After testing, the measured value of the material's absorbance still showed an increasing trend with temperature in the visible wavelength range, which is consistent with the theory of the material's optical properties and proves the accuracy of the test results. This result also confirms that 60 °C is an ideal reaction temperature.

Surface Morphology. SEM was used to characterize the surface morphology of PVC gels at various temperatures. In the first step, the PVC gel samples were frozen at −20 °C for 12 h before being dried in a vacuum freeze-drying oven for 24 h. Gold spraying was used to examine the surface morphology of the dried samples, and the results are shown in Figure 7.

Some degree of agglomerations was observed on the surface of the samples T40 and T50 of PVC gel due to the slightly less dispersion of particles. However, the surface morphology of sample T60 is more uniform and has fewer PVC agglomerates than the other three PVC gel samples. The surface properties of sample T70 were poor because the boiling point of THF was 66 °C, resulting in solvent evaporation and insufficient dissolution of the PVC particles. The SEM images confirmed that PVC gel prepared at 60 °C is a relatively good reaction temperature

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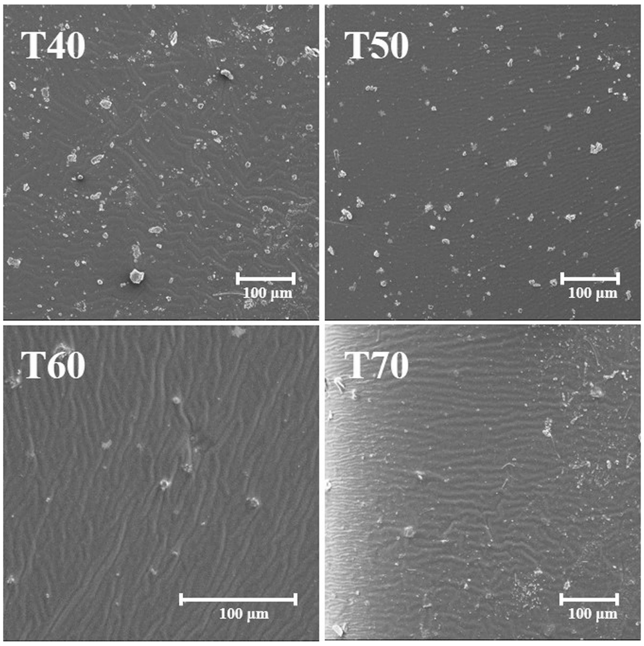
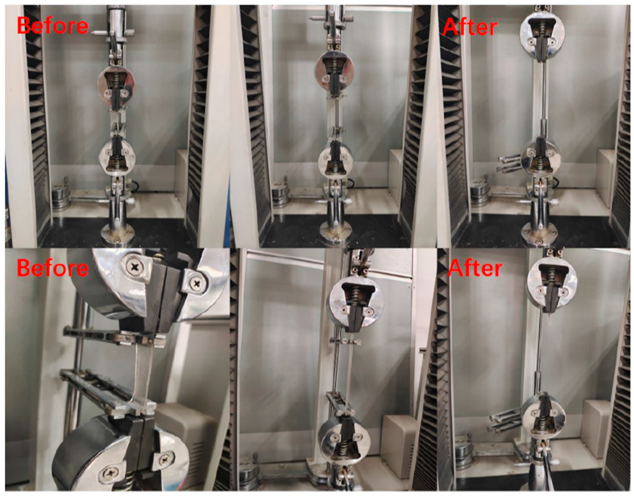


Figure 7. SEM surface test of the gels.

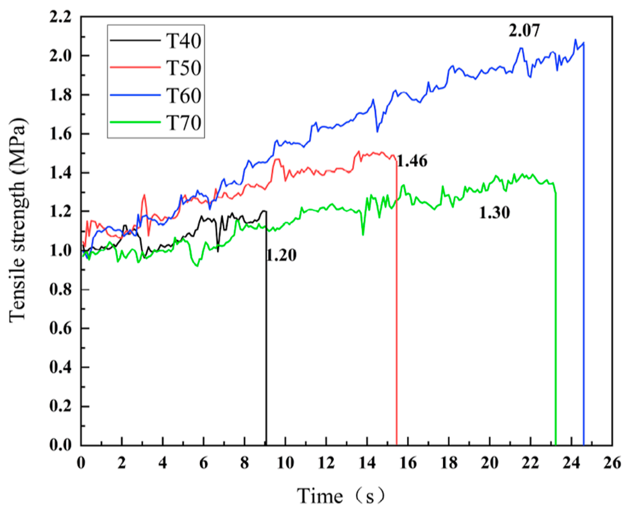
point. Therefore, it is also demonstrated that better surface morphology significantly impacted the PVC gel's light transmission.

Mechanical Properties. The mechanical properties of noncrystalline elastic materials are primarily determined by the degree of cross-linking of the material's molecular chains, i.e. the degree of dispersion of the PVC resin powder. However, it affects the material's mechanical properties, such as tensile strength and yield strength. The tensile strength of PVC gels T40, T50, T60, and T70 obtained are 1.2, 1.4, 1.3, and 2 MPa, as depicted in Figure 8. The tensile strength of T40 and T50 is low because the material's surface properties indicated that the degree of PVC resin is less dispersed in the system. Meanwhile, the tensile strength of the T60 sample of PVC gel demonstrated the best performance, with a maximum tensile strength of 2.06 MPa. The trend of the tensile strength of each gel group with temperature change is similar to that of the light transmission rate, demonstrating that the reaction temperature and raw material solubility are necessary conditions affecting material properties.

Investigation of the Response Time of PVC Gel Electrical Stimulation. Each sample of PVC gel was tested for practical application before being applied to the gel microlens of variable focal length and attached to the electrode surface. The PVC gel-based microlens setup was fabricated from PVC gel and electrodes' assembly. The inner hole structure of the microlens was enlarged from 1 to 4 mm for easier observation. The model was made up of positive and negative metal rings made of brass, with an insulating rubber layer in the middle. The positive metal ring's inner hole was set to 4 mm, the edge size to 8 mm, and the thickness to 1 mm. With a concentric circle structure, the internal hole size of the negative metal ring was set to 14 mm, the edge size to 18 mm, and the thickness to 1 mm. Wires were connected to the circular electrodes in turn, and then to the positive and negative terminals of the DC power supply. The assembled structure was kept directly under the laser displacement meter, the marker sat beneath the drive assembly, and the laser displacement meter was controlled by the height and position of the iron frame table. Response time data were collected after controlling the microlens conditions by applying a constant DC voltage (1000V) to observe gel deformation and the computer terminal connected to the laser displacement meter. Figure 8 depicts the test procedure schematically. After accessing the DC voltage, the laser displacement meter's laser test point was placed in the center of the gel, and the change in the radius of curvature of the gel was recorded. However, the imaging focal length was converted using the concave lens's focal length calculation formula, and the focal length conversion formula 9 in ref 25 is as follows



(a)



(b)

Figure 8. PVC gels' tensile performance test: (a) pictorial view of UTM during testing and (b) tensile strength of PVC gels. Photograph courtesy of "Li Xudong". Copyright 2023.

$$f = \frac{1}{(n_1 - 1) \times \left(\frac{1}{r_1} - \frac{1}{r_2}\right)}1 \quad (1)$$

where f is the focal length in mm. r_1 is the radius of curvature of the upper surface of the PVC gel, in mm, r_2 is the radius of curvature of the lower surface of the PVC gel, in mm, and n_1 is the refractive index of the PVC gel.

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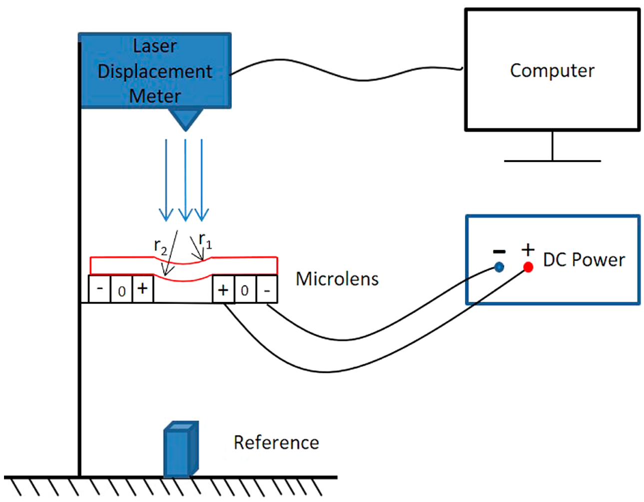

Figure 9. Schematic diagram of the focal length test process.

Figure 10 shows the recorded deformation effect of the PVC gel microlens. Using the T60 PVC gel sample as an example, the reference beneath the gel gradually changed after the energization began. It stopped changing around 60 s, when the power was disconnected, and the imaging change was observed and recorded. The deformation of the PVC gel's upper and lower surfaces after stimulation was measured. The specific focal length, on the other hand, was calculated using eq 1. When the focal length of the microlens reached its maximum, for example, the upper surface of the PVC gel was depressed by 0.4 mm and the lower surface was depressed by 0.3 mm. The deformation was then converted into curvature radii R1 and R2 to calculate the focal length at this point. Furthermore, the test data were converted and organized to produce a graph of the PVC gel's focal length change versus time, as shown in Figure 11.

Figure 11 shows that the response time for the T60 PVC gel to reach maximum focus after power on is 60 s, and the recovery time after power off is 80 s, reducing the response time data by half compared to the previous studies.

However, the overall difference in the degree of focal length variation was different when compared to the results of previous studies. It leads to the conclusion that the effect of the energized

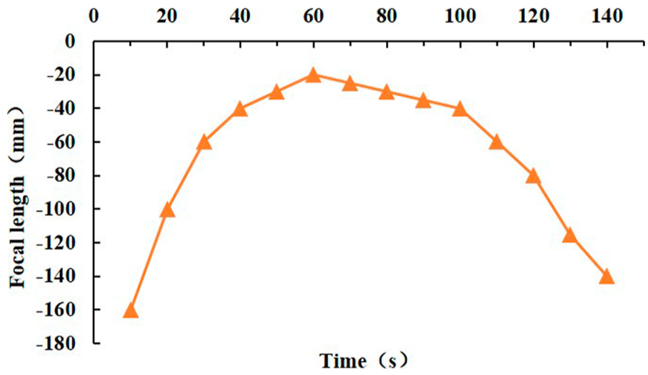

Figure 11. Relationship between response time and focal length.

deformation of the material is the same with the same raw material. As a result, the stirring temperature has less of an impact on the gel's driving development. Furthermore, the factors influencing the response time of the gel remain in the polarity and conductivity of the material itself, which provide new ideas for the study's future work.

■ CONCLUSIONS

In this study, PVC gel samples were prepared at various temperatures 40−70 °C and successfully tested. The T60 group PVC gel had the highest light transmittance compared to the other PVC gel samples, reaching 92.8%, comparable to an optically flat glass of the same thickness. The SEM images observed some degree of agglomerations on the surface of the T40 and T50 PVC gel samples. However, the SEM image of T60 showed more uniformity due to the homogeneous mixing of PVC, DBA, and THF. Furthermore, the T60 PVC gel's tensile stress test results demonstrated excellent tensile properties, with a fracture tensile limit of 2.06 MPa and the longest tensile resistance time. These results exposed the good interactions between PVC and DBA.

Moreover, the electro-response data were recorded using a laser displacement meter: the response time and focal length of self-fabricated PVC gel-based microlens under an applied voltage of 1000 V. The PVC gel microlens at this applied voltage reached their maximum focal length −8 to −20 mm,

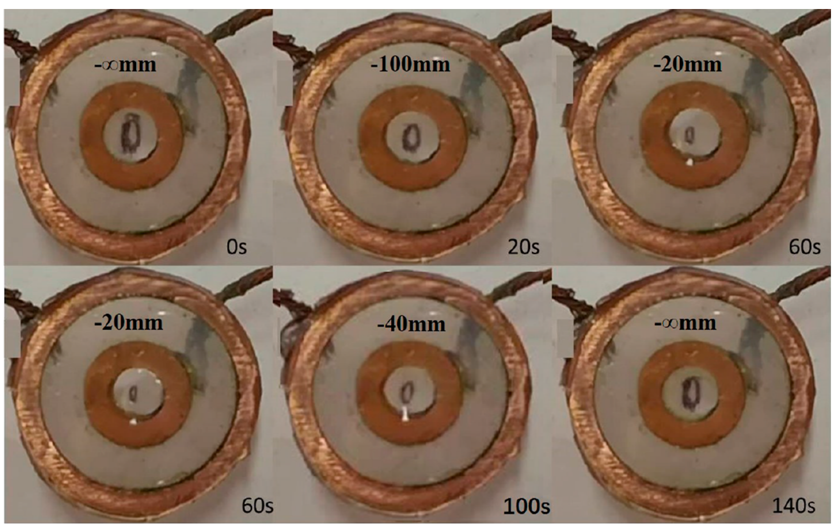

Figure 10. Imaging changes of energized microlens. Photograph courtesy of "Li Xudong". Copyright 2023.

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ACS Omega 2023, 8, 28924−28931

responded in 60 s, and recovered in about 80 s after the applied voltage was shut down. The results revealed that the focal length of microlens change and response time of the gels under an applied voltage was nearly doubled compared to previous studies. This paper concludes that the polarity of the raw material is an important factor influencing the response time of the PVC gel material to electrical stimulation. For future direction, it is proposed that investigations must be carried out to understand better the mechanisms behind improving the material's mechanical, thermal, and electrical properties. Advanced techniques and thermal analysis could provide valuable insights into the structure and properties of the material. Additionally, further research could focus on developing more sustainable and environmentally friendly methods for synthesizing and processing PVC gel. The proposed PVC gel microlenses could be used in various devices such as new electrically tunable optical microlenses, flexible electronic screens, wearable transparent devices, and flexible skin applications.

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Notes

The authors declare no competing financial interest.

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