ISSN: 2414-1895 DOI: 10.6919/ICJE.202505_11(5).0003

Glycerol-Crosslinked Chitosan/Polyvinyl Alcohol/Polyaniline Conductive Hydrogel for Wearable Strain Sensors with High Sensitivity and Environmental Durability

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Abstract

In this chapter, a PANI/CS/PVA/GL composite conductive hydrogel with excellent mechanical properties, electrical conductivity, and environmental stability was successfully prepared by incorporating conductive polyaniline (PANI) into the chitosan (CS)/polyvinyl alcohol (PVA) hydrogel system, along with the plasticizing and anti-freezing effects of glycerol (GL). Fourier transform infrared spectroscopy (FTIR) analyses confirmed the hydrogen bonding interactions among PANI, CS, and PVA, as well as the successful introduction of PANI. The experimental results demonstrated that the incorporation of PANI not only enhanced the mechanical properties of the hydrogel but also endowed the composite hydrogel with outstanding electrical conductivity. Additionally, the inclusion of glycerol imparted excellent freeze resistance and dehydration resistance to the hydrogel, enabling it to maintain stable mechanical and electrical performance even under extreme environmental conditions.

Keywords

Natural Polysaccharide Hydrogel; Conductive Hydrogel; Flexible Sensor.

1. Introduction

Conductive hydrogels are typically fabricated by incorporating conductive materials, such as conductive polymers, inorganic fillers, salts, or ionic liquids, into hydrogel matrices[1-3]. As a result, they combine the flexibility of hydrogels with the conductivity of conductive materials, making them highly attractive for applications in flexible sensors, energy storage devices, and other fields. Conductive polymers like polypyrrole (PPy) and polyaniline (PANI) are particularly favored due to their non-toxicity, excellent stability, and biocompatibility[4]. However, when conductive hydrogels are used in wearable sensing devices, they come into direct contact with human skin. This necessitates that the hydrogel matrix be composed of non-toxic and biocompatible materials[5].

Chitosan, a natural polysaccharide derived from the shells of marine arthropods such as shrimp and crabs, is known for its non-toxicity and excellent biocompatibility[6,7]. It exhibits antimicrobial activity, inhibiting the growth and reproduction of certain fungi, bacteria, and viruses, and is biodegradable within a specific timeframe. Chitosan hydrogels also possess thermoreversible properties. To leverage this, an irreversible polyaniline network can be introduced into chitosan hydrogels to create conductive composite hydrogels with shape memory capabilities. Hydrogels prepared through the repeated freeze-thaw method using polyvinyl alcohol (PVA) are purely physically crosslinked, containing no initiators or crosslinking agents, thereby minimizing harm to cells and tissues[8]. However, due to their high water content, hydrogels are prone to dehydration and freezing at low temperatures, leading to the loss of flexibility, conductivity, and other essential

ISSN: 2414-1895 DOI: 10.6919/ICJE.202505_11(5).0003

properties[9]. This significantly limits their application scope and lifespan. Therefore, enhancing the freeze-resistance, dehydration resistance, and overall durability of conductive hydrogels is crucial[10].

Flexible wearable strain sensors demonstrate significant application potential in health monitoring, motion tracking, and human-machine interaction due to their excellent flexibility, high sensitivity, biocompatibility, and multifunctional integration capabilities[11]. However, they still face numerous challenges in terms of environmental stability, signal interference, long-term durability, energy supply, and data security. For instance, variations in environmental temperature and humidity may affect their performance, body movements can introduce noise, prolonged use may lead to material fatigue, reliance on external power sources, and concerns over the privacy of physiological data. Addressing these issues requires breakthroughs in material innovation, structural optimization, algorithm enhancement, and interdisciplinary collaboration to improve their reliability and promote widespread adoption in practical applications[12].

In this chapter, a PANI-CS/PVA/GL composite conductive hydrogel was prepared by replacing H2O with a binary solvent system of H_2O and glycerol (GL), and incorporating sulfonated conductive polyaniline (PANI) into the CS/PVA network. The mechanical properties, dehydration resistance, and freeze resistance of the composite hydrogels were optimized based on the influence of different components. Furthermore, the compressive properties, dehydration and freeze resistance, electrical conductivity, strain sensing performance, and shape memory functionality of the composite hydrogels were systematically investigated. The PANI-CS/PVA/GL composite hydrogel demonstrates significant potential for applications in flexible sensors.

2. Material and Methods

2.1 Sample Preparation

Dissolve 3.0 g of chitosan (CS) in 97 mL of an aqueous acetic acid solution with a mass fraction of 1 wt%, and stir uniformly until the CS is completely dissolved. Dissolve 10.0 g of polyvinyl alcohol (PVA) in 90 mL of water, and heat the mixture at 90 °C with continuous stirring for 2 hours until the PVA powder is fully dissolved. The prepared 3 wt% CS solution and 10 wt% PVA solution were mixed in specific ratios (1:3, 1:5, 1:7, 1:10) and stirred thoroughly until homogeneous. The mixture was then poured into a dedicated gel mold and left to stand at room temperature for 6 hours to eliminate bubbles in the gel precursor solution. Subsequently, the solution was subjected to three cycles of freezing at -18 °C and thawing to obtain the CS/PVA hydrogel.

Based on the component ratio of the aforementioned CS/PVA hydrogel (CS:PVA = 1:7), different amounts of PANI were dissolved in 3 mL of water and ultrasonicated for 30 minutes to achieve uniform dispersion (the PANI amounts were 10 mg, 20 mg, and 30 mg, corresponding to 1-PANI, 2-PANI, and 3-PANI, respectively). Then, 5 mL of CS solution (100 mg) and 7 mL of PVA solution (700 mg) were thoroughly mixed. The prepared PANI aqueous solution was poured into the mixture and stirred uniformly for 2 hours. The mixture was transferred to a dedicated mold and left to stand for 6 hours to remove bubbles. Subsequently, the hydrogel was subjected to three cycles of freezing and thawing to obtain the PANI-CS/PVA hydrogel. Finally, the hydrogel was immersed in a glycerol (GL)-water mixed solution (volume ratio of Gel:H_2O = 1:3) for 12 hours to produce the PANI-CS/PVA/GL composite conductive hydrogel.

2.2 Characterization

The structure of 3-PANI/CS/PVA hydrogels was observed by field emission scanning electron microscopy (SEM) (JEOL FEG-XL30S). FT-IR spectra were recorded on a PerkinElmer Spectrum100 using KBr particles. The TH2830 digital bridge test records the rate of change in relative resistance. The mechanical properties of the hydrogel were tested by an electronic universal testing machine controlled by the Pine Shield LDW-2 electronic testing machine. The water retention and dry-holding properties of the hydrogel are calculated by recording the weight for different time periods in a thermostatic drying oven: the calculation formula is:

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$$Sr(\%) = \frac{Wt}{W0} * 100\% \quad (1)$$

3. Results and Discussion

3.1 Preparation of PANI-CS/PVA/GL Composite Conductive Hydrogel

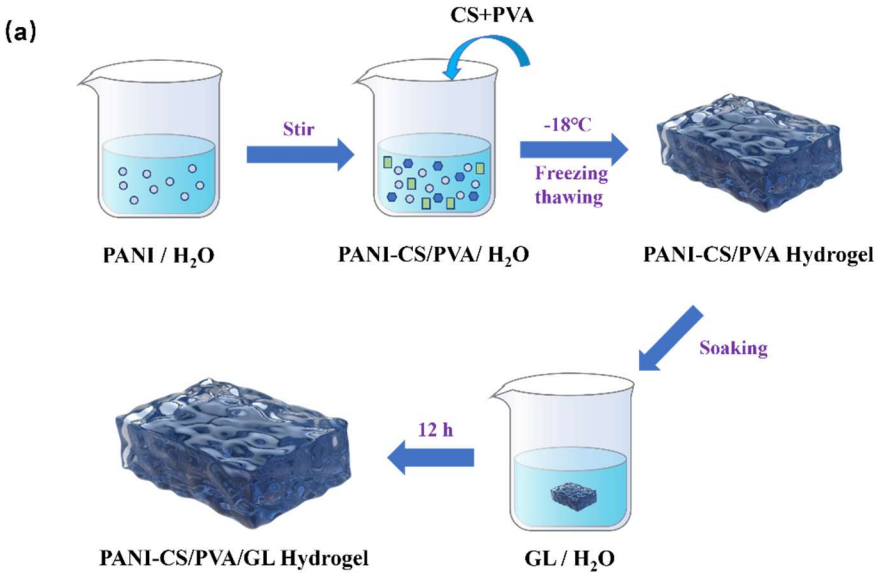

Figure 1. Preparation of PANI-CS/PVA/GL Composite Conductive Hydrogel.

Figure 1 illustrates the preparation flowchart of the PANI-CS/PVA/GL composite conductive hydrogel. As depicted, a certain amount of conductive polyaniline (PANI) is uniformly dispersed in H2O through ultrasonication and stirring. Pre-prepared CS and PVA solutions are then added to the PANI/H2O solution to form a gel precursor solution, PANI-CS/PVA/H2O. After degassing, the aforementioned gel precursor solution is transferred into a specific mold and subjected to three freeze-thaw cycles at -18°C to obtain the PANI-CS/PVA hydrogel. Subsequently, the prepared PANI-CS/PVA hydrogel is immersed in a binary solvent mixture of glycerol (GL) and water for 12 hours to yield the PANI-CS/PVA/GL hydrogel. Here, the volume ratio of glycerol to water in the binary solvent mixture is 1:3.

3.2 Structural and Compositional Analysis of PANI-CS/PVA Hydrogel

To verify whether the aforementioned gel has been successfully synthesized into a hydrogel, Fourier Transform Infrared Spectroscopy (FTIR) was employed to analyze the composition of the composite conductive hydrogel.

As shown in Figure 2a, in the infrared spectrum of conductive polyaniline (PANI), the absorption peak at 699 cm^{-1} is typically attributed to the out-of-plane bending vibration of the C-H bond in the benzene ring. This vibrational mode is characteristic of hydrogen atoms on the benzene ring, specifically representing the out-of-plane bending motion of adjacent hydrogen atoms on the ring. When PANI is introduced into the hydrogel system, this characteristic peak is also observed in the infrared spectrum of the 3-PANI/CS/PVA hydrogel, thereby confirming the successful incorporation of PANI into the hydrogel system.

Simultaneously, the peaks in the range of 3200-3400 cm^{-1} correspond to the stretching vibrations of the N-H bonds in polyaniline (PANI) and chitosan (CS). Upon introduction into the hydrogel system, these peaks may overlap or shift with the O-H stretching vibration peaks in the hydrogel. As illustrated in Figure 2a, the peak gradually shifts to higher wavenumbers with the addition of CS and PANI, reflecting the progressively enhanced hydrogen bonding interactions between chitosan, PANI, and the PVA-based hydrogel. This demonstrates that both conductive polyaniline (PANI) and chitosan (CS) have successfully participated in the formation of the composite hydrogel.

ISSN: 2414-1895 DOI: 10.6919/ICJE.202505_11(5).0003

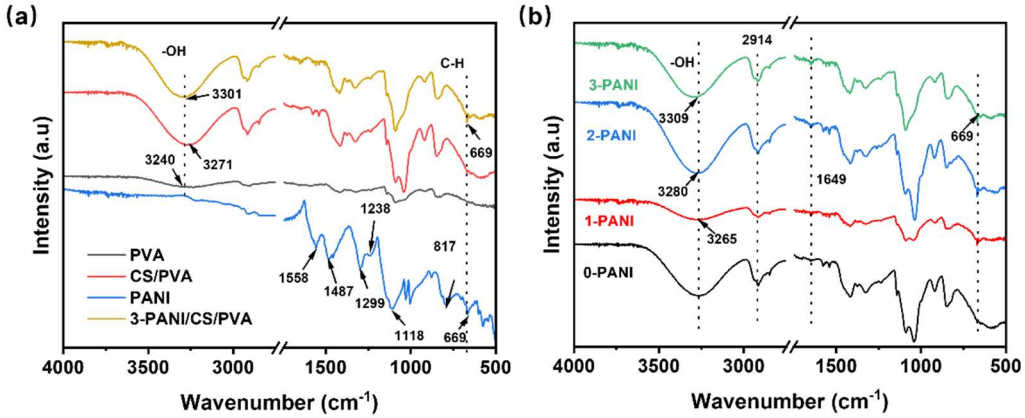

Figure 2. FTIR spectra of different hydrogels. (a) FTIR spectra of PANI, pure PVA, CS/PVA, and 3-PANI/CS/PVA hydrogels. (b) FTIR spectra of PANI/CS/PVA hydrogels with different PANI contents.

The mechanical impact of varying PANI content on the CS/PVA hydrogel is depicted in Figure 2b. As the PANI content increases from 0 to 30 mg to form the 3-PANI/CS/PVA hydrogel, the characteristic hydroxyl (-OH) peak shifts from 3233 cm^{-1} for 0-PANI to 3309 cm^{-1} for 3-PANI, further indicating the strengthening of hydrogen bonding interactions during the hydrogel formation process.

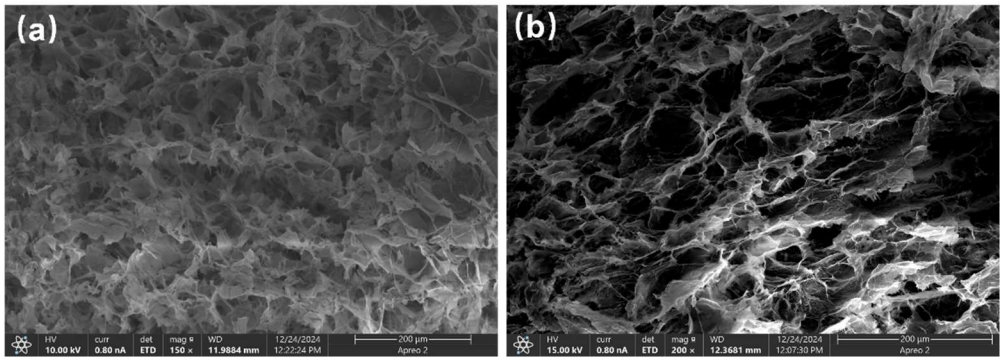

Figure 3. The SEM images of the hydrogel. (b) SEM image of pure PVA hydrogel (c) SEM Image of 3-PANI/CS/PVA Hydrogel.

In this chapter, conductive polyaniline (PANI) is introduced into the chitosan (CS)/polyvinyl alcohol (PVA) hydrogel, and the hydrogel is transformed into an anti-freezing and dehydration-resistant hydrogel by soaking in glycerol (GL). This process involves complex structural changes and intermolecular interactions.

The SEM images in Figure 3 reveal the three-dimensional (3D) porous structure of the freeze-dried PVA and 3-PANI/CS/PVA hydrogels, with pore sizes ranging from fifty to two hundred nanometers. The high-resolution localized magnification image more clearly reflects the regular porous structure. Figure 3a shows the SEM image of the pure PVA hydrogel. As shown in Figure 3b, Furthermore, after the introduction of CS and PANI, the SEM images exhibit a more regular three-dimensional porous structure, indicating an enhancement in mechanical properties compared to the pure PVA hydrogel.

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3.3 Mechanical Properties of the Composite Hydrogel

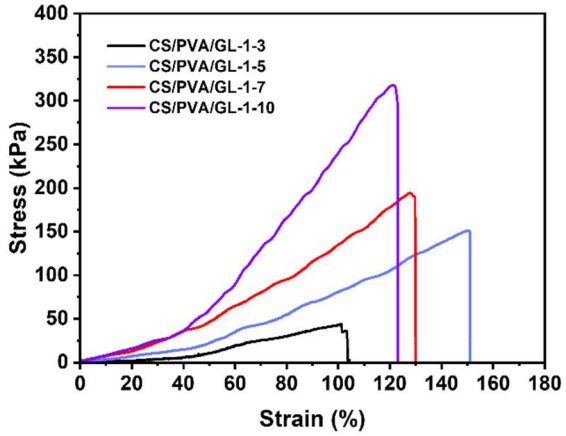

Figure 4. Tensile stress-strain curves of CS/PVA/GL composite hydrogels with different ratios.

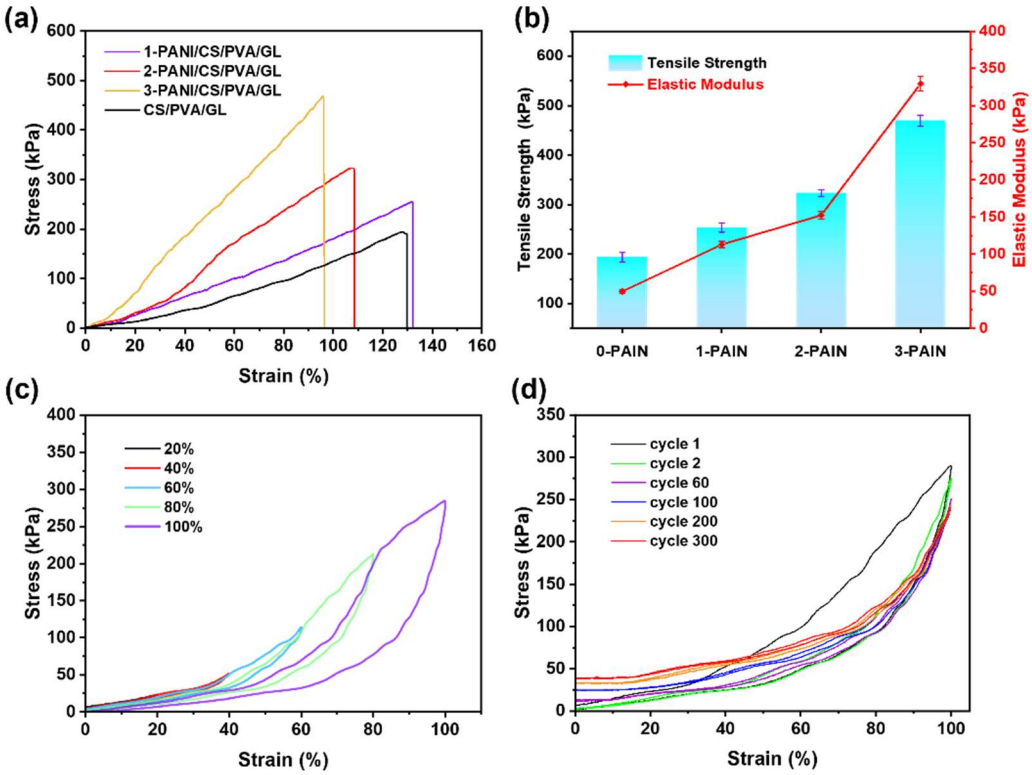

Figure 5. Characterization of tensile properties of composite conductive hydrogels with different PANI content. (a-b) The tensile stress-strain curve, tensile strength and elastic modulus of PANI/CS/PVA/GL composite conductive hydrogels with different PANI content. (c) The tensile cyclic loading curve of 2-PANI/CS/PVA/GL composite conductive hydrogel. (d) The curves of 2-PANI/CS/PVA/GL composite conductive hydrogels with different cycle numbers under the condition of 100% tension.

To determine the optimal mechanical properties of the hydrogel, the tensile stress-strain curves of CS/PVA/GL hydrogels with different ratios are shown in Figure 4. When the mass ratio of CS to PVA is 1:3, the maximum fracture strength of the hydrogel is 43.26 kPa, and the maximum elongation is only 101.28%. As the CS:PVA ratio increases to 1:5, the maximum tensile strength of the hydrogel

ISSN: 2414-1895 DOI: 10.6919/ICJE.202505_11(5).0003

reaches 150.26 kPa, and the maximum tensile elongation is 152.43%. The increase in PVA content enhances the tensile properties of the hydrogel. Comparing the stress-strain curves of CS/PVA hydrogels with ratios of 1:5, 1:7, and 1:10, the tensile strength of the hydrogel gradually increases with the increase in PVA content, while the flexibility gradually decreases, and the maximum tensile elongation becomes shorter. In this chapter, the CS/PVA hydrogel with a ratio of 1:7, which exhibits excellent balance between rigidity and flexibility, is selected as the base gel material for subsequent incorporation of conductive polyaniline.

To investigate the mechanical performance impact of introducing conductive polyaniline (PANI) into the CS/PVA/GL-based hydrogel, a series of tensile performance tests were conducted on composite conductive hydrogels with different PANI contents (Figure 5). As shown in Figure 5a, the tensile stress-strain curves of the composite conductive hydrogels with varying PANI contents reveal that the CS/PVA/GL hydrogel without PANI has a maximum tensile strength of 193.92 kPa and a maximum elongation at break of 130.21%. With the introduction of 10 mg of PANI into the CS/PVA/GL hydrogel, the 1-PANI/CS/PVA/GL composite conductive hydrogel exhibits an increased maximum tensile strength of 253.26 kPa, a year-on-year growth of 0.34, and a slight increase in maximum elongation. Comparing the three composite conductive hydrogels with different PANI contents (10 mg, 20 mg, and 30 mg), as the PANI content increases from 10 mg to 20 mg, the 2-PANI/CS/PVA/GL composite conductive hydrogel achieves a maximum tensile strength of 322.87 kPa, while the maximum elongation at break decreases to 108.18%. Simultaneously, the 3-PANI/CS/PVA/GL composite conductive hydrogel with 30 mg of PANI shows a maximum tensile strength of 468.57 kPa and an elongation at break of 96.02%. The tensile strength and elastic modulus of the PANI/CS/PVA/GL composite conductive hydrogels with different PANI contents indicate that, within a certain range, higher PANI content leads to greater maximum tensile strength and elastic modulus (Figure 5b). When the PANI content is 30 mg, the 3-PANI/CS/PVA/GL composite conductive hydrogel achieves a maximum tensile strength of 468.57 kPa and a compressive storage modulus of 326.35 kPa.

Having understood the basic tensile stress-strain properties of the hydrogel, the energy dissipation of the 2-PANI/CS/PVA/GL composite conductive hydrogel during tensile deformation was explored by analyzing its mechanical behavior under tensile loading-unloading cycles at different strain values. The tensile strength of the hydrogel gradually increases with strain (Figure 5c). However, the stress-strain curves of the tensile cycles do not exhibit hysteresis under low tensile conditions. Yet, when the tensile elongation reaches 80%, the hydrogel shows a certain degree of hysteresis. As shown in Figure 5d, the 2-PANI/CS/PVA/GL composite conductive hydrogel can stably cycle 300 times under 100% elongation conditions, and data from the 1st, 2nd, 60th, 100th, 200th, and 300th cycles were recorded. Although the hydrogel curve shifts at the beginning of stretching, it becomes increasingly stable and gradually converges into overlapping curves as the number of stretching cycles increases. The stability of the hydrogel remains relatively unchanged, indicating that the hydrogel possesses certain fatigue resistance and cyclic stability.

3.4 Analysis of the Dehydration and Dry Resistance of Hydrogels

Since we replaced the solvent of the composite conductive hydrogel with a binary solvent mixture of GL/H2O, GL, being a highly hygroscopic substance, can absorb and retain moisture from the environment. When GL/H2O permeates into the CS/PVA-based hydrogel in place of H2O, it effectively reduces water evaporation, thereby delaying the drying process of the hydrogel and endowing it with excellent dry resistance and water retention properties.

The PANI/CS/PVA/GL composite conductive hydrogel was placed in a constant temperature air oven at 25 ºC to test its water loss and water retention properties. As shown in Figures 6a and 6b, the Sr of the 3-PANI/CS/PVA/GL composite conductive hydrogel containing GL gradually decreases over time, stabilizing after about 48 hours, and reaches 41.5% after 72 hours. In contrast, the Sr of the 3-PANI/CS/PVA composite conductive hydrogel without GL decreases rapidly within 24 hours and

ISSN: 2414-1895 DOI: 10.6919/ICJE.202505_11(5).0003

stabilizes at 24 hours, with an Sr of only 6.1%. This indicates that the introduction of GL significantly enhances the dry resistance of the hydrogel.

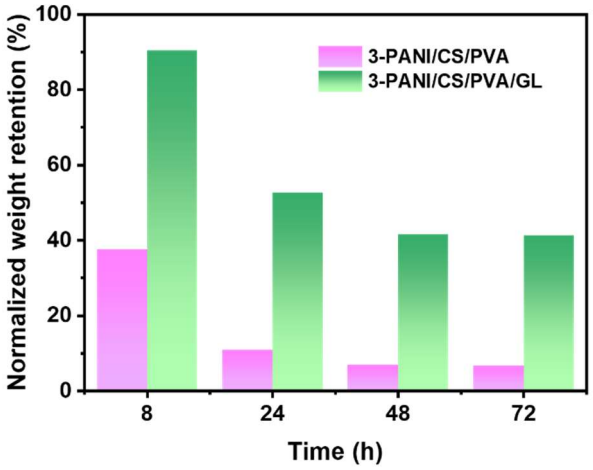

Figure 6. Water loss and Water retention of PANI/CS/PVA/GL Composite Conductive Hydrogel.

3.5 Analysis of the Electromechanical Sensing Properties of Hydrogels

Through tensile and compressive performance tests of the composite conductive hydrogel, we have learned that both CS and PANI can enhance the mechanical properties of the PANI/CS/PVA/GL composite hydrogel. This composite conductive hydrogel exhibits excellent mechanical flexibility, cyclic stability, and low hysteresis, thereby laying a solid performance foundation for the subsequent design of strain sensors and related applications in human physiological motion detection.

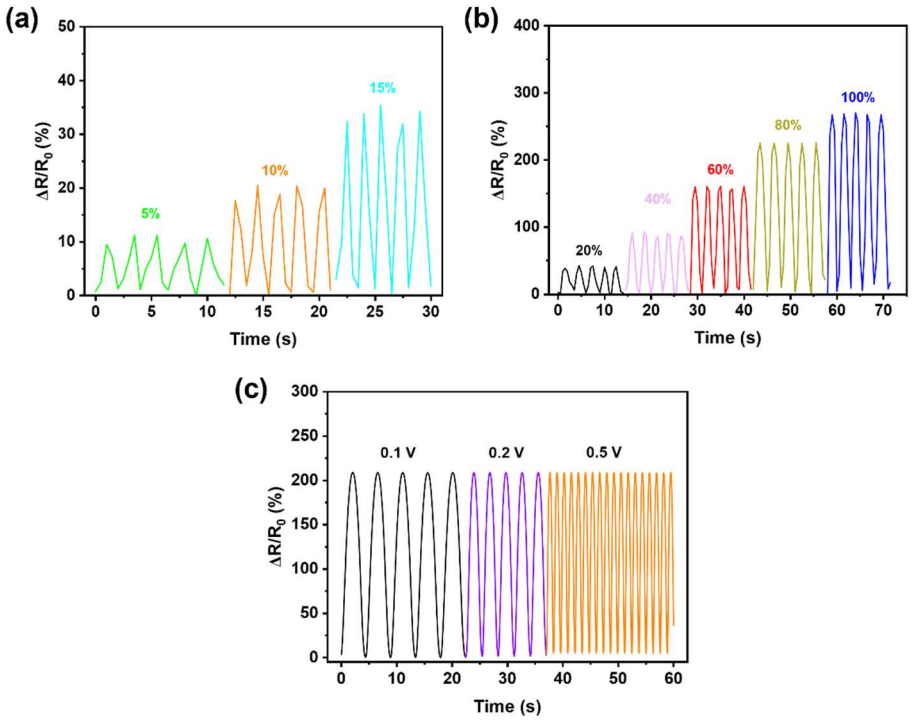

Figure 7. Characterization of electromechanical sensing properties of 3-PANI/CS/PVA/GL composite conductive hydrogel. (a-b) Variation of relative resistance of 3-PANI/CS/PVA/GL hydrogel sensor under strain of 5-100%. (c-d) The change in $\Delta R/R_0$ under repeated finger bending at 45° and 90° under different temperature conditions.

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Therefore, we investigated the electromechanical properties of the 3-PANI/CS/PVA/GL composite conductive hydrogel under different tensile state. Similar to Chapter 2 of this study, we focused on understanding the relationship between strain and $\Delta R/R_0$ (where $\Delta R=R-R_0$, and R_0 and R represent the resistance of the original hydrogel and the loaded hydrogel, respectively). The gauge factor (GF), defined as $GF=(\Delta R/R_0)/\varepsilon$, is a common parameter for evaluating sensitivity, with a higher GF value indicating greater sensor sensitivity.

Due to the successful incorporation of the conductive polymer, PANI effectively enhances the electrical conductivity of the hydrogel. Figure 7a and b illustrate the resistance changes of the 3-PANI/CS/PVA/GL hydrogel sensor under various cyclic strains at a speed of 10 mm/s. Notably, the sensor can operate within a range of 5% to 100%, demonstrating excellent reproducibility and reliability. In Figure 3.9-e, when the hydrogel sensor was tested at three different stretching rates (10 mm/s, 20 mm/s, and 30 mm/s), the electrical signals clearly reflected frequency-dependent characteristics. The superior electromechanical performance of the 3-PANI/CS/PVA/GL hydrogel primarily stems from its dynamically entangled network, which is an inherent property of the composite conductive hydrogel after polymerization. When the hydrogel sensor is deformed, the uniformity of the conductive network is disrupted, leading to changes in conductivity. However, when the hydrogel returns to its original state, the resistance can recover due to the restoration of the conductive network.

Subsequently, we will demonstrate the application of the 3-PANI/CS/PVA/GL composite conductive hydrogel sensor in detecting human motion across different scenarios. When the hydrogel sensor is attached to a volunteer's finger or wrist, it can accurately identify the resistance signals generated during finger and wrist bending(Figure7c). This provides a solid foundation for the diagnosis of joint-related diseases.

4. Conclusion

In this chapter, a PANI/CS/PVA/GL composite conductive hydrogel with excellent mechanical properties, electrical conductivity, and environmental stability was successfully prepared by incorporating conductive polyaniline (PANI) into the chitosan (CS)/polyvinyl alcohol (PVA) hydrogel system, along with the plasticizing and anti-freezing effects of glycerol (GL). Fourier transform infrared spectroscopy (FTIR) analyses confirmed the hydrogen bonding interactions among PANI, CS, and PVA, as well as the successful introduction of PANI. The experimental results demonstrated that the incorporation of PANI not only enhanced the mechanical properties of the hydrogel but also endowed the composite hydrogel with outstanding electrical conductivity. Additionally, the inclusion of glycerol imparted excellent freeze resistance and dehydration resistance to the hydrogel, enabling it to maintain stable mechanical and electrical performance even under extreme environmental conditions.

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