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Self-adhesive and conductive hydrogels and their applications in bioelectronics

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Abstract. At present, the market pressure strain sensors mainly include intrinsic conductivity, composite conductivity, and flexible metal conductivity. Most of the strain sensors can met various basic requirements such as strain sensitivity and mechanical properties. However, biocompatibility and the ease of use are still areas to be explored and improved. This paper proposes a novel strategy to fabricate a kind of self-adhesive and conductive hydrogel using natural biomacromolecule gelatin, tannic acid (TA) and conductive polymer PEDOT: PSS. The sensor is biocompatible, nontoxic, and can be directly attached to the skin without the use of adhesive tape, and sufficiently large electrical signal response can be collected under stress. The presence of tannic acid forms a cross-linking network with the gelatin in the organic hydrogel, which can effectively increase the toughness (which can reach 203.62 kPa/m^3). PEDOT: PSS provides the conductivity for the hydrogel, and hydrogel with 0.3wt% PEDOT: PSS content have a conductivity of about 2.1ms/cm. Therefore, this study found the possibility of making a flexible electronic element by using a composite of PEDOT: PSS, gelatin, and TA.

Keywords: strain sensor, flexible electronic element, composit materials, PEDOT: PSS, tannic acid.

1. Introduction

In the last decade, researchers have made great efforts to develop flexible electrical devices. Typically, traditional sensor networks are made out of flexible metal circuits (such as gold, silver nanowires) on a flexible substrate polymer (consisting of polyvinyl alcohol, thermoplastic polyurethane, etc.), mounted on the skin by additional adhesives or straps [1]. However, the deployment of traditional sensors is significantly hampered by their low biocompatibility and substrates' and electrical conductors' delamination [2]. With the increasing popularity of smart terminals, wearable flexible electronic sensor devices show a wide range of potential applications in areas such as electronic skin, and human-computer interaction systems, and the preparation of gel devices with adhesion, excellent mechanical properties, and electrical conductivity has become a hot spot for research in this field [3].

Wearable electronics must be mechanically flexible in order to be able to fit the curved contour of biological tissue while still delivering effective electrical connections. Compared to stretchable electronics based on inorganic materials, hydrogels typically have mechanical properties that more closely resemble those of biological soft tissues, meaning they can easily adapt to strains and deformations caused by body movements. Hydrogel is a kind of gels with a hydrophilic three-dimensional cross-linked network structure, which can store water inside the network. Thus it

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has both solid and ionic conduction properties. In addition, tough and stretchable hydrogels can be repeatedly deformed without affecting other functions such as electrical conductivity and adhesion. The self-adhesive hydrogel has sufficient adhesive strength and repeatable adhesion properties for long-term use. It maintains strong adhesion in humid environments. These characteristics make it possible to be widely used in wearable bioelectronic products [3].

Gelatin, as one of the basic bases commonly used for hydrogels, it is a natural polymer of collagen after hydrolysis. It contains a large amount of active functional groups in its molecular chain, which is biodegradable and biocompatible and can avoid toxic degradation products. Gelatin has a reversible sol-gel transition during heating and cooling due to the disruption and restoring of the triple-stranded helical structure. These characteristics make it an excellent biomass material for the construction of hydrogels.

To construct a conductive hydrogel, this study needs to incorporate a conductive material framework. Conductive polymer Poly (3,4-ethylene dioxythiophene): polystyrenesulfonate (PEDOT: PSS) is an important framework for materials of interest to researchers in the field of bioelectronics [4-6]. PEDOT: PSS is a core-shell structured conductive polymer (presented in Figure 1.) consisting of a positively charged hydrophobic PEDOT and a negatively charged hydrophilic PSS, where the conductive PEDOT core is embedded in an insulating PSS shell to form an aqueous dispersion. Tests have shown that PEDOT: PSS films prepared directly from aqueous PEDOT: PSS solutions have low conductivity (typically below 1 S/cm), but the conductivity of specially treated films can be increased to the 10^3 S/cm [7-9]. Composite hydrogel material can be improved by adding elastomer or fiber to PEDOT: PSS, making it soft and stretchable [10]. The tensile-fracture ratio of this material can be up to several hundred [11-13].

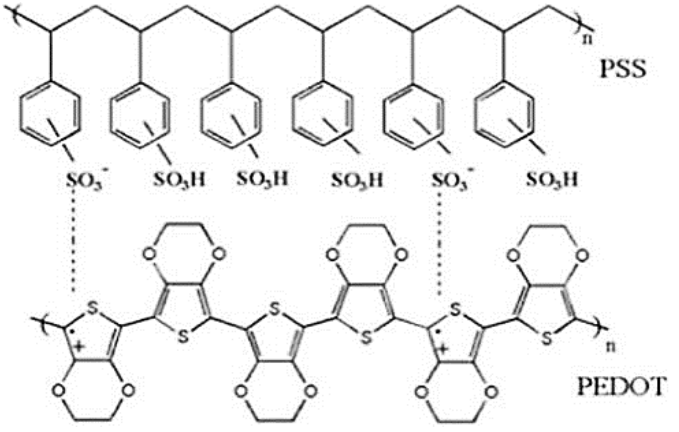

Figure 1. Structure of PEDOT: PSS [8].

Here, this study uses a simple one-pot method to prepare a Gelatin / PEDOTPSS / Tannic acid hydrogel. Among them, PEDOTPSS acts as the main conductivity provider and improves the conductivity of the hydrogel. TA modified the structure of the gelatin network in the organic hydrogel to achieve better mechanical properties such as low modulus and high toughness, as well as to improve the electrical properties of strain sensing [14]. So this study founds the possibility of making a kind of flexible electronic elements using a composite of PEDOT: PSS, gelatin and TA, which has great potential for development.

2. Method

2.1. Research design

This study uses one pot method to prepare hydrogels. The gelatin (type A from porcine skin) and tannic acid were dissolved in distilled water (10 wt%) separately, and the gelatin solution was heated to 50℃ and stirred to ensure complete dissolution. PH of tannic acid solution and PEDOTP:SS solution was adjusted to 7.0 by NaOH (5M) solution. Then slowly add PEDOT: PSS solution to the

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gelatin solution at predetermined gelatin/PEDOT: PSS ratio, and stand for 50℃ for ten minutes and then full stirring to form a uniform solution. Then, using a specified TA to gelatin ratio, the TA solution was gradually added to the gelatin solution at 50 °C. Full stirring to present the mixture as a uniform translucent dark blue liquid. Solution casting was used to create gelatin films with a thickness of around 1 mm, which were then sealed in an area for 24 hours to allow full gelation.

2.2. Mechanical and adhesive properties

The mechanical and adhesive properties of the hydrogels were measured by an INSTRON tensile tester 5500 at room temperature and strain rate of 5 mm/min. The Shear strength were measured on pig skin.

2.3. Conductivity and preparation of sensor

The resistance and conductivity of the hydrogel were measured using the source measure unit KEITHLEY DMM6500 by four-wire resistance method. The conductivity values can be calculated as $\delta = \frac{1}{\rho} = \frac{L}{RA}$. The sensor is achieved by combining hydrogel and DMM6500. The hydrogel was attached to the finger or wrist and connected to the instrument wire by the copper plate at both end to reduce the loss of voltage at the junction. The test was performed at room temperature, 40 μA current.

2.4. Characterization of the hydrogel

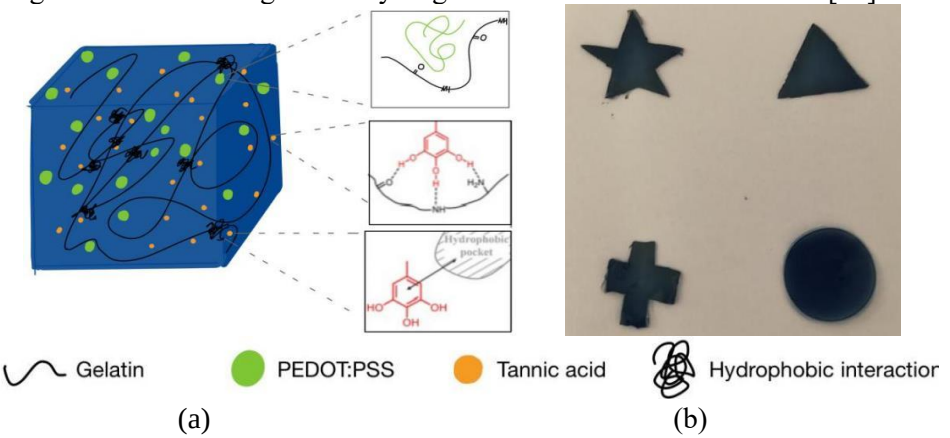
The microstructure and pore morphology of gelatin/PEDOT: PSS(0.1)/TA (0.5) hydrogel were observed by scanning electron microscopy (ZEISS SMI0784 SEM). For the microstructural analysis of the hydrogel, the samples were freeze-dried by CHRIST freeze dryer (Alpha 1-4 LSCbasic) in a vacuum at -55.2 ℃.

Fourier transform infrared (FT-IR) spectra were obtained by using FT-IR spectrometer. The collection ranged from 400-4600cm-1.

3. Results

3.1. Structure and mechanism of gelatin/PEDOT: PSS/TA hydrogel

In order to prevent strong H bonding, the PH is adjusted to 7, and this is a reasonable value in the PH range of human skin. As shown in Figure 2(a), Tannic acid, a kind of macromolecular polyphenolic compound, contains many polymothiol and catechol groups that can interact with various polymers through hydrogen bonds. TA and gelatin can produce physical cross-linking to form a more robust hydrogel due to hydrogen bonding and hydrophobic interactions. The conductive polymer PEDOT: PSS is embedded in the gelatin network, and the final hydrogel appears dark blue. The end product of this process is gelatin-based homogeneous hydrogel with PEDOT: PSS embedded [15].



(a) (b)

Figure 2. Structure. (a) Schematic representation of Gelatin/PEDOT: PSS/TA hydrogel; (b) appearance display.

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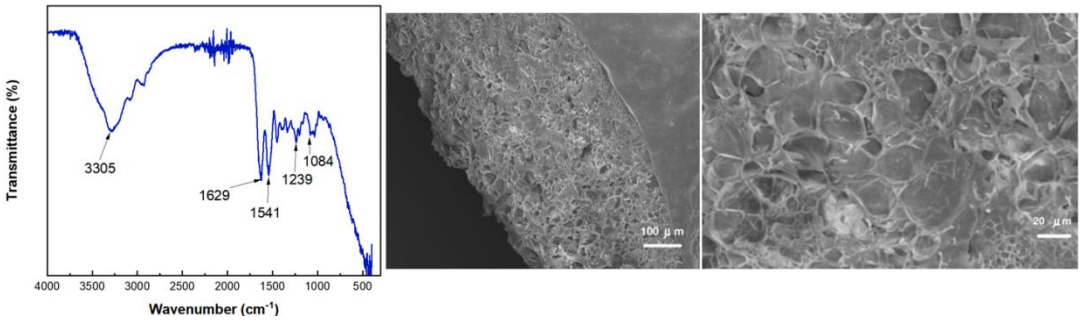
The dark blue liquid can form hydrogels with different appearances in the mold (Figure 2b), and the films can be directly cut into various shapes with scissors.

To identify the chemical bonds and interactions present in the hydrogel gelatin/PEDOT: $PSS_{(0.1)}/TA_{(0.5)}$, it was characterized by Fourier transform infrared spectroscopy (FTIR) in Figure 3(a). The spectrum indicate that the hydrogel has distinct absorption bands at 1629 and 1541cm^{-1}, corresponding to C=O stretching (amide I) and N-H (amideII) in gelatin, respectively. There are characteristic absorption from OH stretches in gelatin and TA (3305cm^{-1}). Since the sample and probe heads were exposed to air when using FTIR to do characterization, miscellaneous peaks presented (1700-2400cm^{-1}) from the influence of different molecules in air.



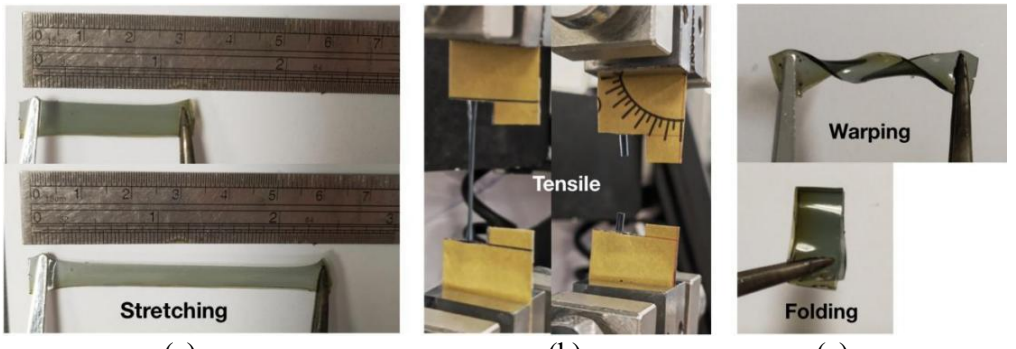
(a) (b)

Figure 3. Characterization. (a) FTIR spectra of hydrogel gelatin/PEDOT: PSS (0.1)/TA(0.5); (b) SEM images of hydrogel gelatin/PEDOT: PSS(0.1)/TA(0.5) at low-magnification image and high-magnification image.

Furthermore, this study uses scanning electron microscopy (SEM) analysis to perform and explore the network in the hydrogel gelatin/PEDOT: $PSS_{(0.1)}/TA_{(0.5)}$ (Figure 3(b)). Gelatin has a large porous microstructure with about 20 microns in size and an uneven shape. It was observed that there were dense hierarchical uniform microporous structures of about 5 micron size, which is the result of cross-linking of the TA-gelatin network. This structure with holes and channels allows the stable dispersion of PEDOT: PSS in the hydrogel. And these porous hydrogels are beneficial for making comfortable and wearable electronic devices.

3.2. Mechanical properties

Hydrogel gelatin/PEDOT: PSS/TA has good mechanical properties and excellent mechanical flexibility, can be easily stretched, rotated and folded (Figure 4), in order to meet the wearable requirements of flexible electronic devices.



(a) (b) (c)

Figure 4. Measurements of mechanical properties. Illustration of (a) Stretching, (b) Tensile stress–strain tests, (c) Warping and folding.

To see the role of PEDOT: PSS and TA in modulating the mechanical properties of the gel network, we investigated the mechanical properties of the prepared Gelatin/PEDOT: PSS/TA hydrogels by tensile stress–strain tests. The typical tensile stress-strain curves of hydrogels are prepared by using

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different PEDOT as well as TA contents are as below in Figure 5. The tensile strength and elongation at break of the hydrogel increase significantly and then decrease when the TA content increases in the range of 0-1wt%. Gelatin/PEDOT: $PSS_{(0.1)}/TA_{(0.5)}$ present the highest toughness (203.62 kPa/m^3). In other words, it has the highest tensile strength (156 kPa) as well as the largest elongation at break (385%), which is much greater than gelatin/PEDOT: $PSS_{(0.1)}/TA_{(0)}$ (34 kPa, 153%).

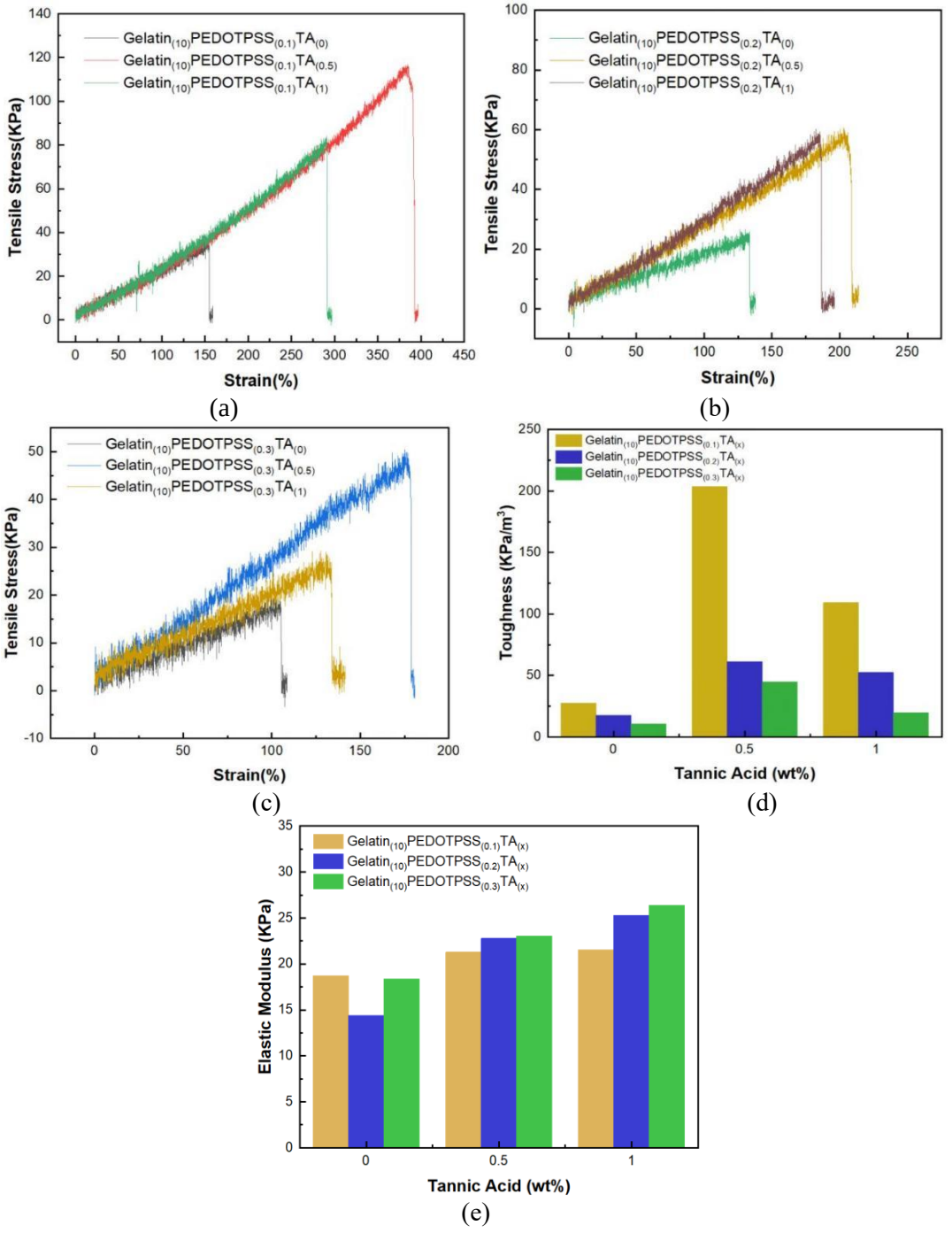

Figure 5. Tensile test for hydrogels with different gelatin/PEDOT: PSS/TA content ratios. Typical tensile stress-strain curves as a function of TA content of hydrogel containing: (a) 0.1wt%; (b) 0.2wt%; (c) 0.3wt% PEDOT: PSS; (d) Toughness; (e) Elastic modulus a function of TA and PEDOT: PSS content.

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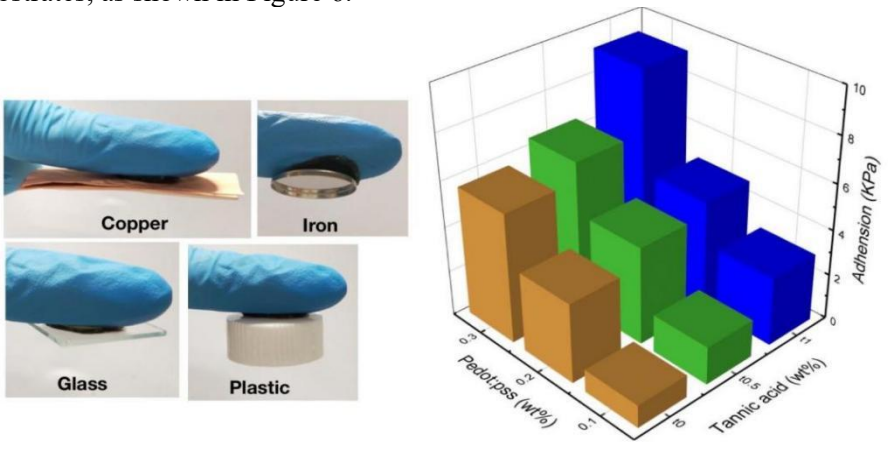
The rationale given is that with the addition of TA, the majority of the triple helix joints in the gelatin network vanished, and more hydrogen bonds and hydrophobic interactions developed between the two substances, so that the hydrogel network was stronger when the gelatin-TA formed reversible physical crosslinks. With the increase of TA content, the number of crosslinking sites in the hydrogel increases simultaneously, which strengthens the hydrogel and gives it higher tensile strength and elongation. However, gelatin/PEDOT: $PSS_{(X)}/TA_{(1)}$ displayed a lower tensile properties by further increasing the TA content but still somewhat better than that hytyogel without TA addition. This can be attributed to excess TA creates a large amount of cross-linking points to hinder the shifting of the gel molecular chains, while excessive cross-linking and the potential for inhomogeneous phase separation can reduce the hydrogel's mechanical properties [16]. Figure 5(e) shows that the elastic modulus of the hydrogel was gradually proportional to the TA content in the hydrogel. This is the result of increased hydrogen bonding between TA-gelatin as well as hydrophobic interactions. When the cross-linking point increases, the movement of the gel polymer chains is subjected to greater resistance. So the gel molecular chains become more resistant to strain when the hydrogel is subjected to tensile stress, resulting in the increase of elastic modulus.

Different PEDOT: PSS contents' effect on the mechanical properties of the hydrogels has also be investigated. Compared to gelatin/TA hydrogels (tensile fracture ratio can be up to 1500%) [17, 18], the addition of PEDOTPSS decreases the toughness of the material. The tensile strength and tensile fracture ratio of gelatin / PEDOT: PSS / TA gradually decreased by increasing the PEDOT: PSS content (from 0.1wt% to 0.3wt%) in hydrogels. At a TA content of 0.5, the hydrogel containing 0.1wt% PEDOT: PSS had the highest tensile strength, tensile fracture ratio and toughness, higher than that of hydrogels containing 0.2wt% (or 0.3wt%) PEDOT: PSS (58 kPa, 205%, 61.24 kPa/m^3). This behavior can be attributed to the rigid polymer chain that PEDOT: PSS has, which makes it brittle and stiff, at the same time, as a conductive polymer, it behave as a nucleation site for hydrogel network assembly. Thus hydrogels with higher PEDOT: PSS content have a smaller tensile fracture ratio and toughness. The elastic modulus of the hydrogel increased with the PEDOT: PSS content. When the TA content in hydrogels was 1wt%, the elastic modulus of the hydrogel containing 0.3wt% PEDOT: PSS was 26.39KPA, which was much larger than the elastic modulus of the hydrogel with 0.1wt% PEDOT: PSS (21.56 kPa). The mechanical properties improvement is because of the penetration network formed by the large number of benzene and thiophene rings contained in PEDOT: PSS and the gelatin polymer chain, resulting in strengthening the hydrogel and increasing its resistance under stress.

3.3. Adhesion performance

The Gelatin/PEDOT: PSS/TA hydrogels exhibited efficient adhesive behavior on various organic and inorganic substrates, as shown in Figure 6.



(a) (b)

Figure 6. Adhesive performance of gelatin/PEDOT: PSS/TA hydrogels. (a) Photographs of gelatin/PEDOT: PSS/TA adhering to metals and polymers; (b) The adhesive strength on pigskin.

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In this hydrogel system, the catechol groups in the TA molecular chain were successfully grafted into the gelatin molecular chain. The dynamic interactions generated between the hydrogel and the various substrates are responsible for the adhesive force. We analyzed that hydrogen bonds were important contributors to adhesion forces in hydrogel-substrate contact. The prepared hydrogel forms strong hydrogen bonds through the hydroxyl group (main) on the catechol group in TA and the amide group in the gelatin to promote its adsorption on the substrate surface. Moreover, the benzene ring contained in TA and PSS can interact with other aromatic rings through π - π overlapping, providing the adhesion force between the hydrogel and the substrate, especially the organic tissue. And second, interactions that can provide adhesion forces may also include covalent bonds, dynamic complexation and cation-π interactions formed by the benzene ring with the cations in the substrate etc [19, 20].

It could be seen from the Figure 6, the adhesion of hydrogel is proportional to the content of PEDOT: PSS and TA. The adhesion force of gelatin / $PEDOTPSS_{(0.3)}/TA_{(1)}$ can reach 9.2 kPa. Compared with the TA, PEDOT: PSS had larger impact to the adhesion property in the range of the proportions studied [12, 13].

3.4. Tunability of electrical properties of gelatin/PEDOT: PSS/TA hydrogels for strain sensing

In the hydrogel fabricated, PEDOT: PSS act as a conductive polymer to provide the conductivity. The PEDOT molecular chains have a large amount of Π electrons with a huge conjugated structure, and in this structure, the carriers (electrons) can move across bonds, that is, Π electrons can move around two nuclei without the action of other external forces, and under the action of the electric field, these carriers are able to make localized directional movements. The whole polymer chain of PEDOT is conjugated and delocalized capable, so when it is subjected to an external electric field, electrons are directionally transmitted on the chain, thus achieving the purpose of electrical conduction. Thus, the conductivity of the hydrogel gelatin/PEDOT: PSS/TA is directly proportional to the PEDOTPSS content.

Thanks to its good tensile properties and electrical conductivity, the finished gelatin/PEDOT: PSS/TA hydrogels can be used as strain sensors. Measured the resistance value at both ends of the sensor as the sensor response, as shown in the Figure 7. The resistance measured through its ends increases as the strain increases when the polymer sensor is subjected to tensile stress and it then decreases by decreasing the strain value until when it returns to its original shape so that it returns to its initial resistance. The reason this gelatin/PEDOT: PSS/TA hydrogel can be used as a strain sensor is the tunability of the electrical properties of the PEDOT: PSS. The electrical conductivity of a homogeneous mixed conductive hydrogel depends on the volume fraction of defects and the permeation threshold. In mathematical expressions, $\sigma \propto (Vf - Vc)^s$, where Vf stands for the volumetric fractions of the material, and Vc stands for critical volumetric fraction of the filler. The fitting parameter is presented as s. In the hydrogel, PEDOTPSS is uniformly dispersed in the gelatin-TA network, so that Vf can represent the area fraction of PEDOT: PSS. When the hydrogel material is subjected to applied stress, the channel in the gelatin-TA frame will deform, Vf becomes smaller, resulting in a decrease in conductivity [21]. In other words, the channel expansion reduces the effective volume fraction of the conductive molecular chain under stress, leading to electrical discontinuities and defects in the sensor material. The conductive path in the channel is reduced, resulting in decline of the conductivity. When the stress is unloaded, the gelatin/PEDOT: PSS/TA hydrogel quickly returns to its original shape due to its inherent elastic properties and the memory of its original position [22]. At the same time, the microchannels contract back to the previous state and the PEDOT: PSS polymer rearrange to produce more conductive paths.

After linking to the test system, the gelatin/PEDOT: $PSS_{(0.1)}/TA_{(0.5)}$ hydrogel was tested on the human finger and the wrist as shown. Figure 7 (b) and (d) shows the sensor responses from the system. At different holding times, namely holding the wrist in the bending conditions in time periods were also detected to check the retention response of the sensor. In this case, the sensor response was relatively stable. Moreover, the strain sensor can produce a rapid and timely response to rapid expansion, while producing a stable and sustained response to a state of continuous bending.

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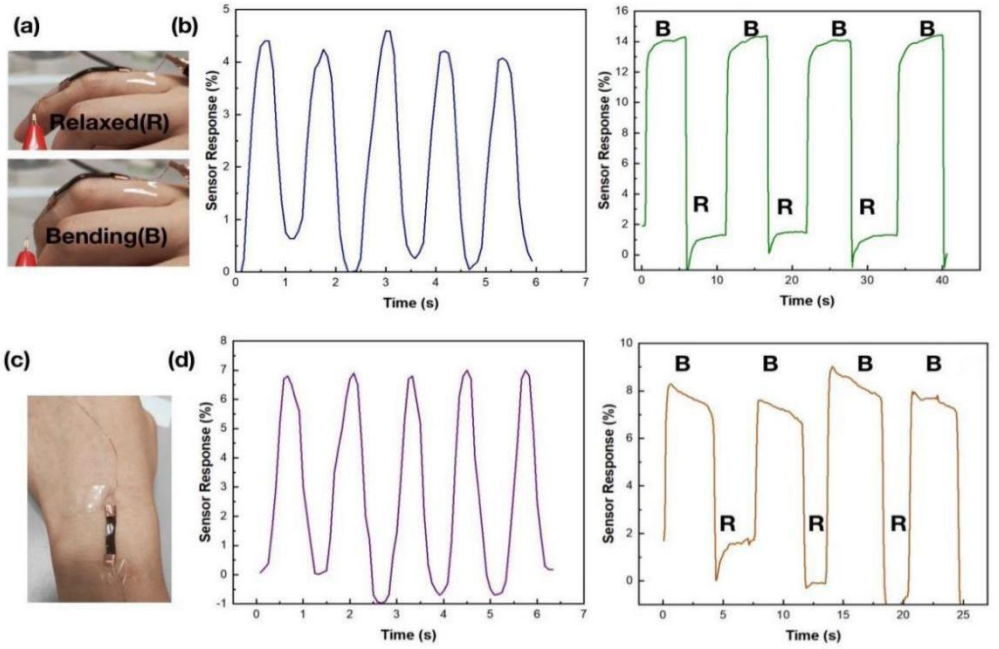

Figure 7. Demonstration of sensors. (a) Experimental condition of relaxed (R) and bending (B) condition on finger; (b) temporal and continuous response of the sensor due to bending of the finger; (c) visual representation of the wrist bent; (d) the sensor's temporal and continuous response as a result of the wrist bend.

4. Conclusion

This paper studied on gelatin/PEDOT: PSS/TA hydrogel, a kind of self-adhesive and conductive hydrogel, which can be used as strain sensor made of composite material and its resistance can change according to the strain and thus respond to the stress. The flexible strain sensor can adhere to our skin without the need of adhesive tape.

Hydrogels containing three materials, gelatin, tannic acid and PEDOT: PSS were made. Since conductive polymer PEDOT: PSS has excellent electrical properties, it is used to construct conductive networks. To make this electrical element more flexible, we used the soft gelatin as elastomer. At the same time, we found that adding tannic acid can build a stronger hydrogel network through hydrogen bonds and hydrophobic interactions, resulting in improving the overall mechanical properties of material. Moreover, due to the presence of catechol in tannic acid and the benzene ring in PEDOT: PSS, the hydrogel prepared can interact with the surface of the substrate through hydrogen bonding and Π overlapping, thus making it adhesive capable. It has been detected that this hydrogel can adhere to substrates such as skin, metals and polymers, and sufficiently large electrical signal response can be collected under stress.

So this study founds the possibility of making a kind of flexible electronic elements using a composite of PEDOT: PSS, gelatin and TA. And the sensor made is biocompatible, non-toxic, and can adhere directly to the skin. Although the performance of the new sensor in this experimental stage is still insufficient compared with the published ones, this material has great potential for development.

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