

Article

One-Pot Improvement of Stretchable PEDOT/PSS Alginate Conductivity for Soft Sensing Biomedical Processes

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Abstract

Hydrogels have immense potential in soft electronics due to their similarity to biological tissues. However, for applications in fields like tissue engineering and wearable electronics, hydrogels must obtain electrical conductivity, stretchability, and implantability. This article explores recent advancements in the development of electrically conductive hydrogel composites with high conductivity, low Young's modulus, and remarkable stretchability. By incorporating conductive particles into hydrogels, such as poly(3,4-ethylenedioxythiophene)/poly(styrenesulfonate) (PEDOT/PSS) researchers have enhanced their conductivity. This study presents a one-pot synthesis method for creating electrically conductive hydrogel composites by combining PEDOT/PSS with alginate. The hydrogel reveals changes in chemical composition upon treatment with dimethyl sulfoxide (DMSO). Additionally, surface morphology analysis via Field Emission Scanning Electron Microscopy (FESEM) and Atomic Force Microscopy (AFM) demonstrate the impact of DMSO treatment on PEDOT/PSS/alginate films. Furthermore, electrical conductivity measurements highlighted the effectiveness of the conductive hydrogels in Electromyography (EMG) and human motion detection. This study offers insights into the fabrication and characterization of stretchable, conductive hydrogels, advancing their potential for various soft sensing biomedical applications. The optimized PEDOT/PSS/alginate composite under dry condition shows a conductivity of 0.098 S/cm and can be stretched without significant loss in conductivity or mechanical stability. This one-pot method provides a simple and effective way to improve the properties of conductive hydrogel-based sensors.

Keywords: biomaterials; conductive hydrogel; PEDOT/PSS; sensors and electronics

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1. Introduction

Advancements in wearable bioelectronics are facilitating the shift towards patient-centric, personalized healthcare [1,2]. Traditional electronic systems are composed of rigid materials, such as metals and silicon, in a two-dimensional (2D) plane and are not suitable for interfacing with the human body [1,3–8]. Wearable bioelectronics adapt to the soft, curvilinear surfaces of the body in order to provide noninvasive, real-time monitoring of a patient's physiological state, including their heart rate, respiration, and blood oxygen levels, for in situ clinical monitoring and personal health management [2,9,10].

There has been significant progress in the development of stretchable conductive materials for point-of-care health monitoring through creative, structural organization and/or novel material selection [11–14]. The first strategy utilizes deterministic geometries

(e.g., wave/wrinkle, kirigami tessellations, cracks) to allow otherwise rigid materials to deform out of plane in response to stress [15–17]. The second strategy swaps rigid substrate materials for stretchable, conductive materials whose conformal properties are independent of their geometry [18–20]. In combination, these strategies provide options that significantly expand the interfacing capabilities of wearable bioelectronics with the human body [18,21,22].

Poly(3,4-ethylenedioxythiophene)/poly(styrene sulfonate) (PEDOT/PSS) is a highly promising conductive polymer for use in wearable sensing systems [23–25]. PEDOT/PSS boasts tunable conductivity, good transmittance, and excellent thermal stability, in addition to being compatible with a wide range of production processes [24–28]. Furthermore, the inherent biocompatibility of the polymer allows it to readily interface with the human body and sense signals such as body temperature, humidity, and strain [27,29–31]. Despite these advantages, native PEDOT/PSS has very limited stretchability due to its rigid conjugated backbone and strong interchain interactions that can lead to crack formation under strain [25,27]. To address this key limitation, it is necessary to incorporate other materials, such as elastomers, plasticizers, and hydrogels, that provide free volume for chain stretching while also enhancing the crystallinity of PEDOT regions within PEDOT/PSS substrates to compensate for potential reductions in conductivity [31].

Here, we present a one-pot method for improving the stretchability and conductivity of hydrogel films composed of PEDOT/PSS and sodium alginate. Sodium alginate is a naturally derived polysaccharide that forms a highly tunable, stretchable hydrogel in the presence of multivalent cations—most commonly calcium. When combined with PEDOT/PSS, the resulting hydrogel films retain the inherent stretchability of alginate while also gaining the conductive properties native to PEDOT/PSS substrates. Further modification of conductive hydrogel films via exposure to dimethyl sulfoxide (DMSO) removes insulative PSS groups, strengthening interchain interactions in PEDOT-rich regions of the substrate and significantly improving the conductivity of the overall films [25]. This simple method allows for the fabrication of highly conductive and stretchable films that can be readily incorporated into wearable bioelectronic platforms.

2. Materials and Methods

2.1. Materials

PEDOT/PSS aqueous solution (PH1000, Heraeus Clevios) was purchased from Hanau, Hessen, Germany. Dimethyl sulfoxide (DMSO) (purity $\geq 98\%$), D-(+)-gluconic acid δ -lactone (GDL), and calcium carbonate (CaCO_3) were obtained from Sigma Aldrich (Burlington, MA, USA). Sodium alginate (Na-ALG, viscosity 80–120 cp) was purchased from FUJIFILM Wako Pure Chemical Corporation (Osaka, Japan). All chemicals were purchased and used without further purification. All aqueous solutions were prepared using deionized water (DI) unless otherwise stated.

2.2. PEDOT/ALG Hydrogel Film Preparation

In order to make hydrogels with various mechanical and electrical properties, three different PEDOT/PSS/ALG precursor solutions were prepared at room temperature. First, alginate (ALG) 10% was made by vigorously mixing 4 g of sodium alginate in 40 mL deionized water using a magnetic stirrer (Corning™, Corning, NY, USA) until it completely dissolved. This alginate was used in all samples, and the remaining amount was kept in the fridge at 4 degrees Celsius. To prepare PEDOT/PSS films, we added the pristine aqueous PEDOT/PSS solution into the alginate precursor solution. To investigate the properties of the whole range of PEDOT/PSS/ALG compositions, we prepared three different concentrations of alginate. We made PEDOT/PSS/ALG 1:1 by mixing equal

volumes of 1.3 wt% PEDOT/PSS and 1.3 wt% alginate, PEDOT/PSS/ALG 1:3 by mixing equal volumes of 1.3 wt% PEDOT/PSS and 3.9 wt% alginate, and PEDOT/PSS/ALG 3:1 by mixing three times the volume of 1.3 wt% PEDOT/PSS with respect to that of 1.3 wt% alginate. We then added 16 mg/mL gluconic acid and 4.5 mg/mL calcium carbonate to accelerate the gelation of films. The solutions were then strongly mixed using vortex. Stirring the solutions between each step assured proper bonding. The precursor solutions were then transferred into a substrate, usually petri dishes, and were kept under fume hood for minimum 12 h at room temperature. The petri dishes were left open to accelerate the drying sequence. Hydrogel films made with this method were referred to as 3:1, 1:1, or 1:3 PEDOT/ALG. Figure 1a depicts this procedure.

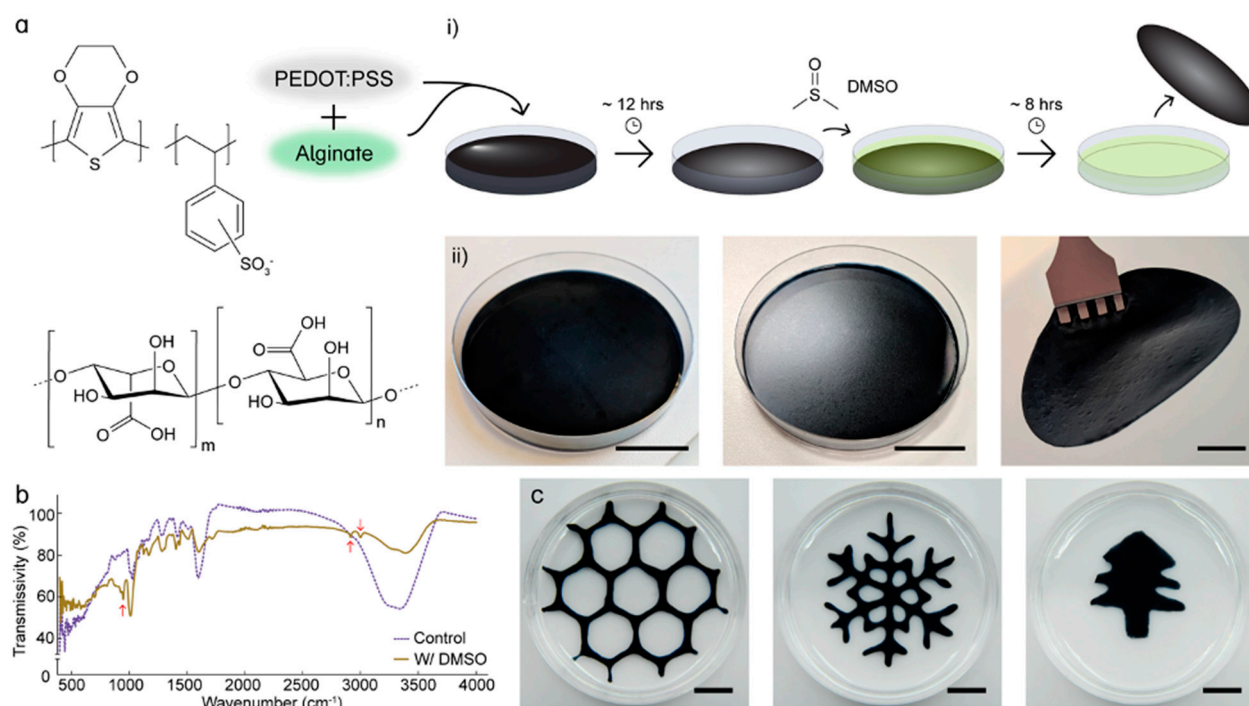


Figure 1. (a) A schematic illustration of preparing PEDOT/ALG films. (b) FTIR spectra of 1:1 PEDOT/ALG films. Characteristic bands are shown by arrows. (c) Simple patterning of 1:1 PEDOT/ALG films in various shapes. Scale bar: 1 cm.

2.3. DMSO Post-Treatment

In order to form a conductive and mechanically stable film using secondary doping method, DMSO > 99% was poured on the dried 1:3, 1:1 and 3:1 PEDOT/PSS, and the thoroughly submerged films, together with the petri dishes, were left at room temperature for 8 h after sealing the cap using parafilm to avoid the solution from evaporating. The films then easily detached from the substrate and could be kept in DMSO for over two months without any change in their mechanical and/or electrical properties for further characterization and analysis.

2.4. Resistance Measurements

PEDOT/ALG films were cut into $1 \times 2 \text{ cm}^2$ rectangles and left to dry completely under ambient conditions (~10 min). Copper wires were attached to each end of the rectangles using conductive silver epoxy adhesive (8331D, MG Chemicals (Ontario, Canada)). A low resistivity meter (Loresta-GP MCP-T600, Mitsubishi (Tokyo, Japan)) was used to measure the electrical conductivity of the films with the probes attached to the copper wires.

2.5. Dynamic Mechanical Analysis (DMA)

Uniform rectangular sections of 1:3, 1:1, and 3:1 PEDOT/ALG films were cut from larger films for tensile testing. For each PEDOT/ALG ratio, two conditions were tested: a control condition without DMSO treatment and an experimental condition with DMSO treatment. Since the control films would disintegrate when in contact with water, they were strained as dry films. The experimental samples were lightly dried using a delicate task wipe (Kimwipes, Kimtech™ (Busan, Republic of Korea)) before straining. Stress/strain curves for each film were obtained using a DMA Q800 (TA Instruments (Dallas, TX, USA)) equipped with a tensile clamp set to perform a force ramp at 0.1 N/min until sample failure.

3. Results

3.1. Fourier-Transform Infrared Spectroscopy (FTIR)

Several methods, such as conductivity measurements, spectroscopy, microscopy, and mechanical testing, may be used to examine the obtained films. Figure 1b shows the Fourier Transform InfraRed (FTIR) spectra of 1:1 PEDOT/ALG as the control and 1:1 PEDOT/ALG with DMSO treatment using a FTIR spectrometer (JASCO Inc., Jeddah, Saudi Arabia). As well as the absorption bands at 1564 cm^{-1} for the C=C stretching in the thiophene ring, at 1270 and 1122 cm^{-1} for the vibrations of the fused dioxane ring, and at 862 cm^{-1} for the stretching of the C–S bond in the thiophene ring [8] associated with PEDOT/PSS in both spectra, the absorption bands at 980, 2900 and 3000 confirm the presence of DMSO in films after treatment with DMSO, while in the control sample, before adding DMSO, those peaks disappeared. Based on the FTIR spectra, adding DMSO did not damage the PEDOT/ALG chains, and it allowed the re-orientation of the carboxylate groups and maintained the mechanical stability of the film [9].

3.2. PEDOT/ALG Film Patterning

The patterning of conductive hydrogels is an important step in making bioelectronics. Due to their water content, it is challenging to pattern hydrogel-based films. Figure 1c shows different patterning of our stretchable and conductive hydrogel films in different shapes, including honeycomb, snowflake, and Christmas tree, to confirm the stability, patternability, and homogeneity of the films, making them perfect options for use in a wide spectrum of applications in flexible sensors and actuators [10]. These films are stable in different media, and no change in their physical condition has been noticed in DI water, PBS, and ethanol after 6 months.

3.3. Scanning Electron Microscopy (SEM)

To examine the surface morphology of the PEDOT/ALG films, Field Emission Scanning Electron Microscope (FESEM, FEI Magellan 400 XHR Scanning Electron Microscope (Hillsboro, Oregon)) images are used. Figure 2a shows the surface morphologies of all films with different ratios of PEDOT to alginate and before and after being emerged in DMSO. Calcium chloride (CaCl_2) is used in control samples before tests to maintain consistent ionic conditions and ensure that any observed effects in experimental samples are due to the variable being tested, not the presence or absence of calcium, plus it helps with structural stability. The control samples (before adding DMSO) show a homogeneous structure with an irregular structure and variable pore size. On the other hand, FESEM pictures of the films that were submerged in DMSO exhibit a uniform interconnected structure due to the creation of long chains on the surface across the films. These long chains accelerate electron transfer and, hence, increase the conductivity, which also have improves the mechanical responses of these films [11]. Moving from one side to the other side for electrons via these

fibrous chains is easier if there are more PEDOT/PSS nanoparticles in the material rather than insulating polymers like hydrogels [12]. This belief is clearly validated in Figure 2a through increasing the percentage of alginate in the film. Moreover, using 1:3 PEDOT/ALG in control films demonstrates more nanoparticles than 1:1 and more than 3:1. This also has been shown for the same films after treatment with DMSO overnight.

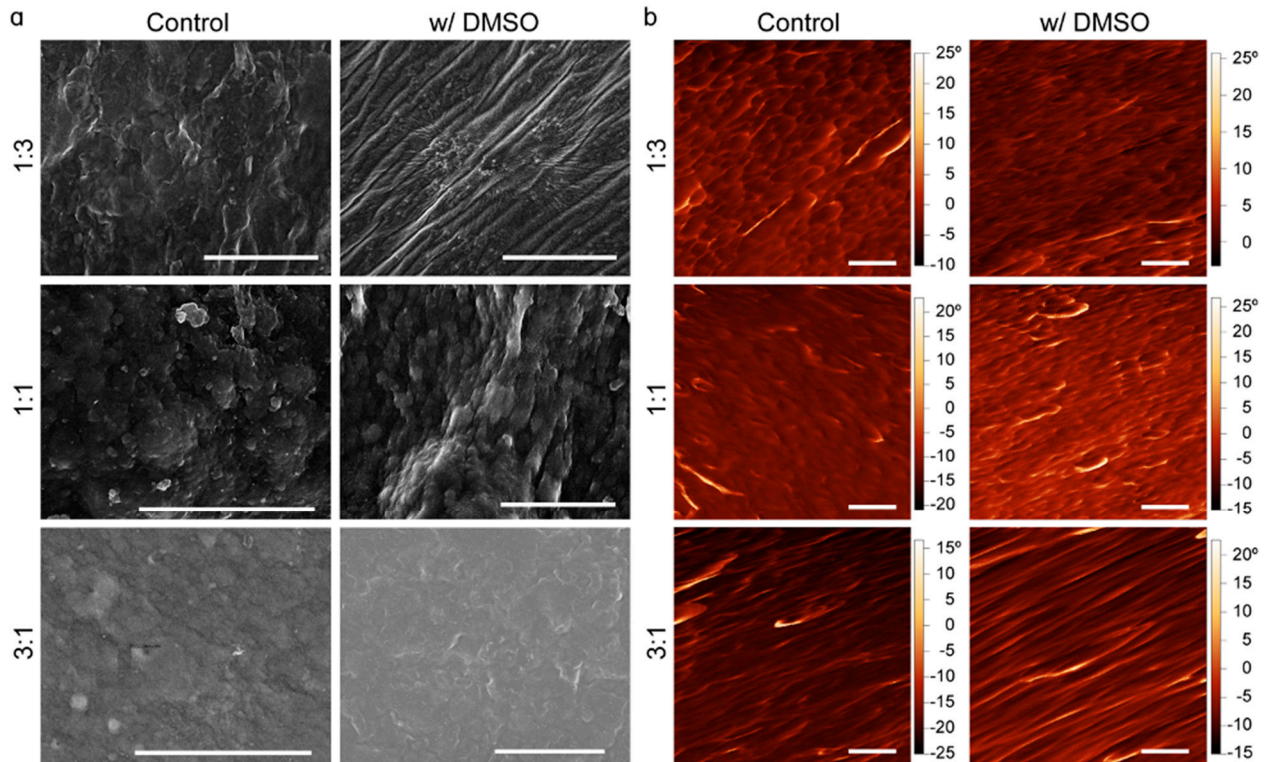


Figure 2. (a) SEM images of 1:3, 1:1, and 3:1 PEDOT/ALG films in CaCl_2 (control) and after they had been emerged in DMSO. Samples were dried before we took the images. (b) Corresponding AFM topography images. Scale bar: 10 μm .

3.4. Atomic Force Microscopy (AFM)

Additionally, Figure 2b provides the corresponding AFM topography images of PEDOT/PSS/ALG 1:3, 1:1, and 3:1 before and after submerging them in DMSO. The roughness and height of the 1:3 PEDOT/PSS/ALG samples clearly show that the compositions of the samples are different, and exposing the samples to DMSO creates stronger chains and, therefore, non-uniform and more stable films [13]. Upon adding more PEDOT/PSS, the roughness increases, as does the average height.

3.5. Resistance Measurements

The addition of PEDOT/PSS into alginate-based hydrogel films imparts the films with conductive properties, enabling them to function as substrates for biosensing applications [14]. Upon the addition of PEDOT/PSS, a notable decrease in resistance was observed in the control samples, corresponding to an increase in the film's conductivity (Figure 3a). Furthermore, it was observed that films with a higher ratio of PEDOT/PSS exhibited increased conductivity, implying a correlation between the concentration of PEDOT/PSS and the enhancement of electrical conductivity in the films.

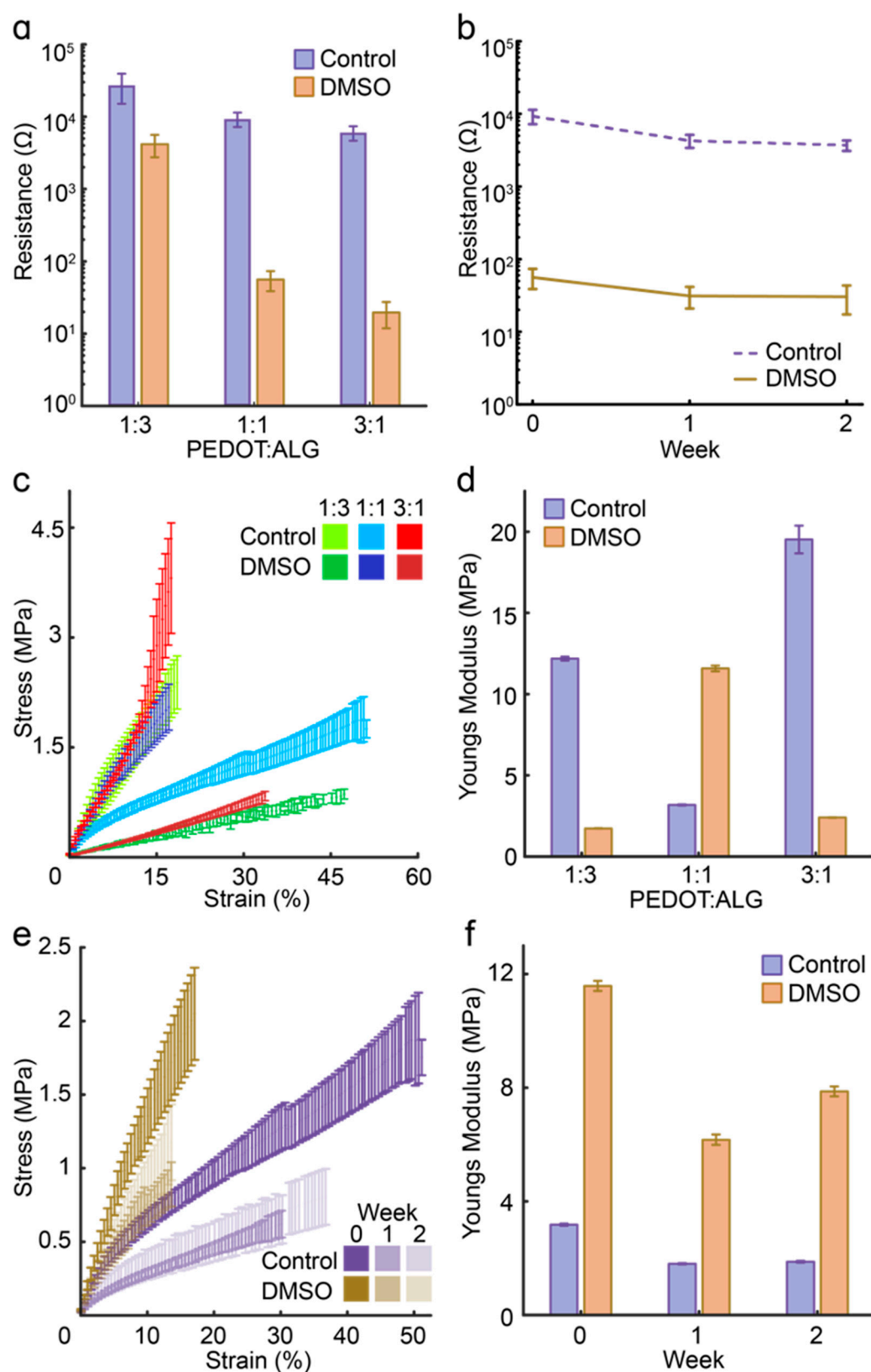


Figure 3. (a) Electrical conductivities of 1:3, 1:1, and 3:1 for PEDOT/ALG films in CaCl₂ (control) and DMSO. (b) Resistance of 1:1 for PEDOT/ALG films in CaCl₂ (control) and DMSO over time. (c,d) Mechanical properties of 1:3, 1:1, and 3:1 for PEDOT/ALG films in CaCl₂ (control) and DMSO. (e,f) Mechanical properties of 1:1 for PEDOT/ALG films in CaCl₂ (control) and DMSO over time.

Exposure to DMSO further increases the conductivity of the PEDOT/ALG films due to improved chain alignment [15]. The conductivity of the films after DMSO exposure increases by a single order of magnitude for 1:3 PEDOT/ALG, two orders of magnitude for 1:1 PEDOT/ALG, and almost three orders of magnitude for 3:1 PEDOT/ALG. DMSO-treated PEDOT/ALG films retain this improvement over the course of two weeks and exhibit a similar slight increase in conductivity, corresponding to the decrease in resistance, as shown in Figure 3b, as that for the non-treated films. The conductivity values were measured as 0.055 S/cm for the 1:3 film, 0.098 S/cm for the 1:1 film, and 0.124 S/cm for the 3:1 film.

The observed decrease in resistance could be attributed to various factors such as increased intermolecular interactions, improved polymer chain alignment, or enhanced charge carrier mobility within the film structures [16]. The enhancement of electrical conductivity in PEDOT/PSS films through the incorporation of dimethyl sulfoxide (DMSO) is attributed to significant morphological and structural modifications within the polymer matrix. DMSO functions as a secondary dopant, inducing phase separation between the conductive PEDOT domains and the insulating PSS component. This redistribution leads to the formation of a PEDOT-enriched network, which facilitates more efficient charge transport. Furthermore, DMSO promotes a conformational transition of PEDOT chains from a coiled to a more linear and expanded structure, thereby increasing carrier mobility. Concurrently, the partial removal or rearrangement of PSS reduces the density of insulating regions, further enhancing the film's electrical conductivity. These findings suggest that while DMSO treatment initially impacts conductivity adversely, the long-term trend reflects an overall enhancement in electrical properties for both control and treated samples. These films can be stored at room temperature for up to two months without significant change in their conductivity. Further analysis is warranted to elucidate the underlying mechanisms driving this phenomenon and to optimize the fabrication process for desired conductivity outcomes.

3.6. Dynamic Mechanical Analysis (DMA)

Tensile tests were performed on rectangular sections of PEDOT/ALG films with varying PEDOT/PSS-to-alginate ratios (1:3, 1:1, 3:1) to assess the impact of DMSO on their mechanical properties. The DMSO-treated samples exhibited an increase in stretchability, as shown by their increased strain-to-break relative to the dry control samples, in exchange for a decrease in stiffness (Figure 3c,d). This is likely attributed to the lateral association of alginate chains caused by exposure to DMSO and additional solvent-induced gelation within the alginate network [17]. The solvent acts as a plasticizer, reducing intermolecular rigidity and increasing the film's ability to deform under strain. Enhanced chain alignment and increased crystallinity, facilitated by DMSO, result in a more mechanically robust and stretchable network. These combined effects render DMSO-treated PEDOT/PSS films highly suitable for application in next-generation stretchable and wearable electronic devices. The underlying mechanisms have been substantiated by multiple studies, including the detailed structural and electrical characterizations reported in the recent literature. Long term, the PEDOT/ALG films show minimal mechanical degradation within a week of synthesis, followed by consistent mechanical behavior afterwards (Figure 3e,f). Stretchability is found to be 45% strain for 1:3, 68% for 1:1, and 53% for 3:1. The corresponding Young modulus values were 0.46 MPa (1:3), 0.33 MPa (1:1), and 0.52 MPa (3:1).

DMSO treatment decreased the Young modulus for the 1:3 and 3:1 films, indicating a softening effect, whereas for the 1:1 film, the modulus increased, suggesting stiffening. This contrasting result may arise from the unique balance between PEDOT/PSS and alginate

content in the 1:1 formulation, which allows for a different interaction mechanism upon DMSO exposure.

In the 1:3 film, the high alginate concentration results in a dominant hydrogel matrix, and DMSO treatment likely disrupts hydrogen bonding and slightly swells the gel, leading to a softer and more elastic network. In the 3:1 film, the PEDOT/PSS is more dominant, and DMSO, known to enhance chain mobility and plasticization, reduces the rigidity of the film by weakening intermolecular interactions among PEDOT/PSS chains.

In contrast, the 1:1 film presents a more balanced composition where both PEDOT/PSS and alginate are sufficiently present to form an interpenetrating network. In this formulation, DMSO treatment may enhance interactions at the interface of PEDOT/PSS and alginate, possibly through improved chain alignment or partial densification, leading to a more tightly packed and crosslinked structure. This can result in an increase in Young's modulus and a more rigid stress/strain response.

Thus, the mechanical outcome of DMSO treatment appears to be composition-dependent, and the 1:1 film is a unique case where intermediate ratios allow DMSO to enhance structural integrity rather than induce softening.

Among the three PEDOT/ALG formulations investigated (1:3, 1:1, and 3:1), the ratio of PEDOT/PSS to alginate played a critical role in determining the electrical and mechanical properties of the hydrogel films. As the concentration of PEDOT/PSS increased, the electrical conductivity of the films improved significantly, with the 3:1 formulation achieving the highest conductivity. However, this enhancement came at the cost of reduced stretchability and increased stiffness, as evidenced by a higher Young modulus and diminished mechanical compliance. Conversely, increasing the alginate content (as in the 1:3 formulation) improved the stretchability and softness of the hydrogel but resulted in lower electrical conductivity and reduced performance stability under repeated mechanical deformation. The 1:1 formulation demonstrated a favorable balance, achieving a conductivity of 0.098 S/cm while maintaining excellent mechanical flexibility, moderate Young's modulus, and good stability during cyclic strain tests. This combination of properties makes the 1:1 PEDOT/ALG hydrogel the most promising candidate for bioelectronic applications, particularly for use in flexible and stretchable EMG sensors where both conductivity and mechanical conformity are essential.

3.7. Electromyography (EMG)

One of the main applications of conductive films is in electronic circuits and EMG. Firstly, a simplified circuit model was utilized to characterize the RC time constant of the material, as illustrated in Figure 1a. The schematic of the parallel plate capacitor, featuring planar copper plates separated by a PEDOT/PSS conductive film, was employed to understand the electrical behavior of the material. The fabricated conductive films were placed in between two copper layers to make a capacitor (Figure 4a) and then connected to a voltage source.

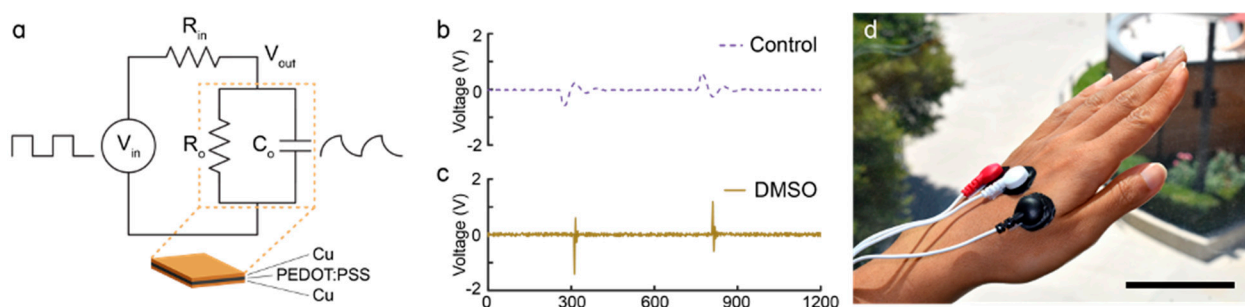


Figure 4. Cont.

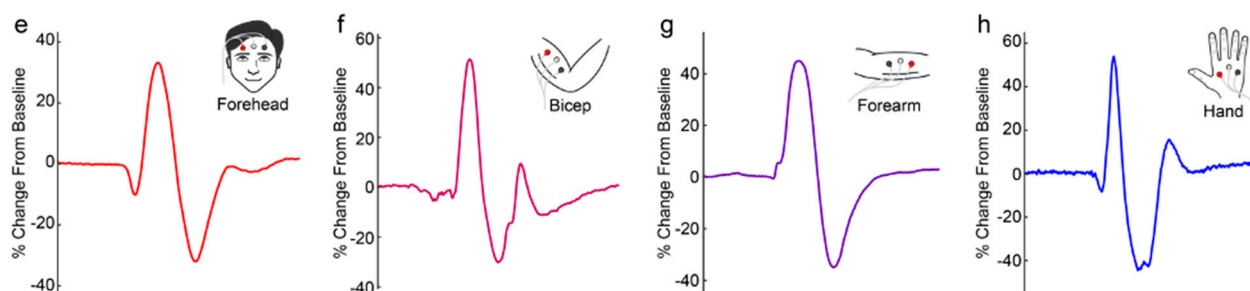


Figure 4. The applications of conductive stretchable PEDOT/ALG films (a) A simplified circuit model for characterizing the RC time constant of the material (top). A schematic of the parallel plate capacitor, wherein planar copper plates are interceded by a PEDOT/PSS conductive film (bottom). (b,c) Output signals for (d) EMG measurement using the PEDOT/PSS conductive electrodes from the hand. The electrodes were prepared and then commercial three leads were connected to a portable Arduino microcontroller to collect the data. Scale bar (5 cm). (e) The EMG signal for facial expression collected from the forehead. (f–h) The EMG signals collected from the biceps, forearm, and hand during exercise.

Figure 4b,c show real-time recording voltage signals for control and DMSO-treated samples, respectively. The PEDOT/PSS conductive electrodes were prepared and then commercial three leads were connected to a portable Arduino microcontroller to collect the data indicating the potential of PEDOT/ALG films in wearable healthcare devices. In electrochemical property tests for skin-conformable bioelectronic devices, a three-electrode system is commonly used to accurately characterize the electrochemical behavior of the working material: (a) Working electrode (WE)—This is the electrode made of the material being tested (in this case, the PEDOT/ALG hydrogel). This is where the electrochemical reactions of interest occur. The performance of this electrode reflects the material's charge storage, conductivity, and electrochemical stability. (b) Reference electrode (RE)—This provides a stable and known potential against which the working electrode's potential can be measured. It does not pass current and ensures accurate and reproducible voltage control. Common examples include Ag/AgCl or saturated calomel electrodes. (c) Counter electrode (CE)—Also known as the auxiliary electrode, it completes the circuit by allowing the current to flow through the system. It balances the current at the working electrode so that potential control and measurement are not influenced by the electrode's resistance. The electrodes are placed on hand (Figure 4d), and upon closing or opening, a spike will happen on the output signal. By comparing these two diagrams, we show that the PEDOT/PSS films that are treated with DMSO are more sensitive to motion than those without DMSO post-treatment, and they are considered better motion sensors, benefiting many applications.

Figure 4e showcases EMG signals for facial expression collected from the forehead, while Figure 4f–h display EMG signals acquired from the biceps, forearm, and hand during exercise, highlighting the adaptability and reliability of PEDOT/ALG films in physiological monitoring applications. Overall, these findings underscore the promising prospects of PEDOT/ALG films in the realm of flexible electronics and biomedical engineering.

4. Conclusions

In this study, a one-pot synthesis strategy was employed to enhance the electrical conductivity of stretchable PEDOT/PSS/alginate hydrogels without compromising their mechanical compliance or biocompatibility. The proposed formulation demonstrated significantly improved conductivity, rendering it highly suitable for applications in soft, skin-conformable bioelectronic devices. This work addresses a critical challenge in the

development of stretchable electronics by presenting a facile and scalable fabrication method that does not require complex post-processing or chemical doping.

Future work will involve evaluating the long-term mechanical stability and electrical performance of the hydrogel under cyclic loading and varied environmental conditions to establish its reliability in real-world applications. In vivo biocompatibility studies and degradation assessments will also be conducted to ensure clinical safety and efficacy. Furthermore, integration with wireless modules for data transmission and power supply will be explored to facilitate the development of autonomous wearable or implantable devices. Expanding the functionality of the hydrogel to include the multiplexed sensing of biochemical or biomechanical signals is also envisioned to broaden its application scope in personalized healthcare and soft robotics.

Author Contributions: Conceptualization, S.Z. and P.T.; methodology, S.Z. and A.R.E.; validation, S.Z. and A.R.E.; formal analysis, S.Z. and A.R.E.; resources, H.C. and P.T.; data curation, S.Z. and A.R.E.; writing—original draft preparation, S.Z. and A.R.E.; writing—review and editing, S.Z. and A.R.E.; H.C. and P.T.; visualization, S.Z.; supervision, H.C. and P.T.; project administration, H.C. and P.T. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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