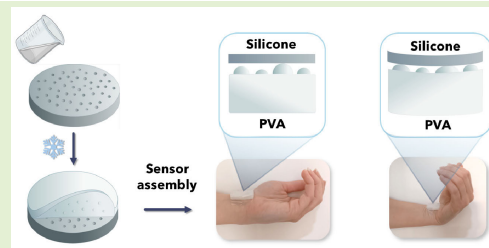


PVA-Based Hydrogels With Engineered Surface Roughness for Triboelectric Wearable Sensors

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Abstract—The rapid growth of wearable devices for health-care monitoring and portable consumer electronics has driven the demand for new sensors that are lightweight, flexible, and capable of real-time, continuous monitoring. Wearable sensors must conform to the human body and enable personalized, accessible health diagnostics beyond traditional point-of-care testing. The wearable sensor market is expected to grow significantly, from U.S. \$1.6 billion in 2023 to U.S. \$4.2 billion by 2028. Hydrogel-based wearable sensors, particularly those using polyvinyl alcohol (PVA), are promising candidates due to their flexibility, biocompatibility, hydrophilicity, and tunable electrochemical and mechanical properties. Triboelectric nanogenerators (TEGs) convert low-frequency mechanical energy into electrical energy by contact electrification between materials with different electronegativities. Hydrogel-based TEGs combine hydrogels' mechanical and ionic properties with triboelectric effects to create self-powered, wearable sensors for monitoring human motion, pressure, and physiological signals. Surface roughness of PVA hydrogels significantly affects triboelectric sensor performance by increasing the contact area and enhancing charge generation, thus improving sensitivity and output. Optimizing surface roughness, hydrogel composition, and mechanical properties is crucial for developing high-performance wearable triboelectric sensors. In this study, PVA hydrogels with optimized surface roughness were fabricated using silicone rubber templates with varying porosity. The resulting single-electrode TEGs (S-TEGs) were tested as pressure sensors, with the best sensor achieving a sensitivity of 12.78×10^{-3} nC/Pa in the 20160 Pa range. For wearable applications, safer hydrogels were developed by replacing sulfuric acid (H_2SO_4) with potassium chloride (KCl), making the devices more suitable for biomedical uses. A wearable sensor prototype based on this optimized PVA hydrogel demonstrated effective human motion detection, showcasing its potential for healthcare monitoring.



Index Terms—Conductive hydrogel, pressure sensor, single electrode triboelectric nanogenerator (TEG), wearable sensor.

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I. INTRODUCTION

THE development and spread of wearable devices arises from the necessity of affordable and versatile technology in healthcare and consumer electronics. Similar devices, namely wearable sensors, must fit the human anatomy, be lightweight, and allow for real-time and, in some applications, continuous, fast-responsive monitoring of the considered parameter [1], [2], [3], [4], [5]. The growing interest in these technologies is highlighted when considering that in 2023, the estimated market size of wearable sensors was U.S. \$1.6 billion and, in 2028, is expected to rise to U.S. \$4.2 billion [6]. To further advance these technologies and accelerate their transition from laboratory research to real-world applications, it is essential to prioritize the use of simple and biocompatible materials during fabrication. Hydrogel-based wearable sensors seem to be good candidates for this purpose, as they are flexible and lightweight, while their electrochemical and mechanical properties can be tuned to fit the requirements of the intended application [7].

Among the different wearable electronic technologies, triboelectric nanogenerators (TEGs) stand out as devices able to convert random, low-frequency mechanical energy, called *high-entropy energy* [8], [9], [10], [11], [12], [13], [14], into electrical energy. By putting in contact two materials with different electronegativity, opposite charges build up on their

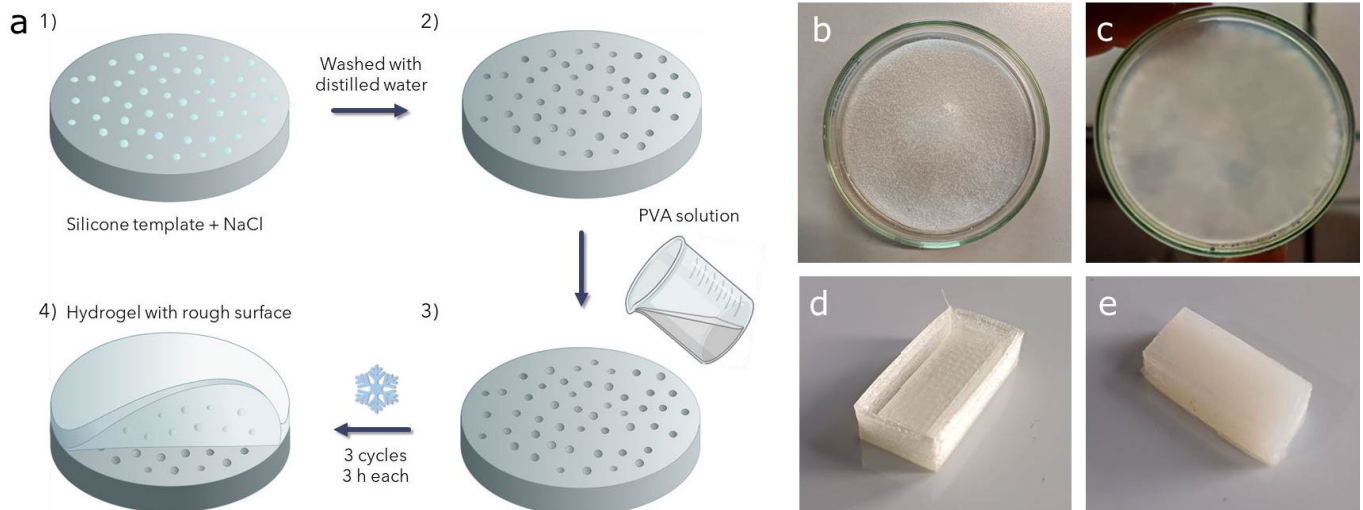


Fig. 1. (a) Schematic process of the silicon template fabrication and hydrogel preparation. (b) Silicone template after removing NaCl. (c) PVA hydrogel after a freeze–thaw cycle. (d) TPU 3-D-printed negative scaffold for the silicone capsule. (e) Final prototype of silicon-supported PVA-based triboelectric motion and pressure sensor.

surfaces, which is then discharged when the materials are separated, thus generating an electron flow through an external circuit and a consequential triboelectric potential [15]. The development of a device that can convert mechanical energy into electrical energy by coupling the effects of contact electrification and electrostatic induction allows the fabrication of novel sensors and power sources [11]. Hydrogel-based TENGs are emerging as versatile platforms for energy harvesting and self-powered sensing, combining hydrogels' unique mechanical and electrical properties with triboelectric principles [16], [17]. Their key features and applications include self-powered sensors for human motion and health monitoring [18]. Hydrogel TENGs can be used for motion tracking by detecting and converting human motion into electrical signals, making them suitable for gesture recognition and gait analysis, or as pressure sensing for tactile pressure detection [8], [9], [15], [17], [19], in robotics [20] and pressure feedback for artificial limbs, or structural health. Triboelectric effects can also be exploited in hydrogel-based devices for sensor applications, by tracking the movement of different limbs [21], [22], [23] and distinguishing between muscular traction and relaxation.

The investigation for similar technology focuses first on the hydrogel material. Polyvinyl alcohol (PVA) hydrogels are widely used in triboelectric sensors due to their excellent hydrophilicity, biocompatibility, flexibility, and ionic conductivity, which are critical for wearable and flexible sensor applications [21], [24], [25], [26], [27]. The roughness of the surface in PVA hydrogel-based triboelectric sensors plays a significant role in their performance, as it can influence the contact area between the hydrogel and the opposing triboelectric material. This interaction directly affects the triboelectric charge generation and thus the sensor's sensitivity and output [11], [28]. The optimization of surface roughness alongside hydrogel composition and mechanical properties plays a key role in the development of high-performance PVA

hydrogel-based triboelectric sensors for wearable and flexible electronics.

Herein, a PVA hydrogel, obtained via a facile fabrication method previously widely investigated in our group [29], [30], was employed, and its surface roughness was further optimized in this study. The material was implemented in a single-electrode TENGs (S-TENGs) and subsequently employed as a pressure sensor and wearable sensor to monitor human motion. The PVA hydrogel surface roughness was optimized using a deposition method via silicone rubber templates prepared with different porous sizes and distributions. The different sensors were prepared by sealing the rough PVA hydrogels in a food-grade silicon rubber support, categorized by porous sizes and salt quantities in the template. The pressure tests were performed by observing current variations in a single-electrode set-up, while applying calibrated weights of different masses on the surface of the S-TENGs. As regards the wearable sensor application, the replacement of H_2SO_4 with KCl in the rough PVA hydrogels was carried out before sealing [26]. As a proof-of-concept, a wearable hydrogel sensor was designed for human motion detection and consequently tested on a volunteer's hand [31], [32]. The investigated sensors have the advantages of a simple and efficient preparation method, being lightweight and portable, and can be customized according to the required size.

II. EXPERIMENTAL PART

A. Chemicals

Poly (vinyl alcohol) (PVA), degree of hydrolysis = 99+%; molecular weight = 89 000–98 000; H_2SO_4 : 95.0%–98.0%w/w; Potassium chloride: 99.0%–100.5%; Sodium chloride: >99% were purchased by Sigma–Aldrich; Cyclohexane: pure; >99% was purchased by Across Organics; Reschimica purchased the precursor of the silicone resin was purchased by Reschimica (base A and hardener B).

B. Preparation of the Rough-Surfaced PVA Hydrogel

To increase the rough surface of the hydrogel used for sensor preparation, a porous silicon template was prepared by mixing a two-component silicone solution with a known salt quantity. Dimension and density of the pores on the silicone surface have been modulated by adding different amounts and different size fractions of NaCl salt, which was subsequently removed by dissolution in distilled water. This causes the presence of pores of various sizes and amounts on the silicone template, as shown in the schematic process in Fig. 1(a).

NaCl was sifted through three different sieves to obtain two size fractions, which were named 425–355 and 355–300, regarding the salt diameter range. Then, silicone templates were prepared by mixing 3.0 g of each component of a silicone bicomponent solution in a beaker. NaCl of the desired size fraction (425–355 or 355–300 μm) or amount (1.0 or 2.0 g) was then added to the silicone and mixed thoroughly. The mixture was placed in the ultrasound bath at an intermediate frequency for 3 min at room temperature and then poured into a 7.0 cm diameter Petri dish, where it was left to cure for 3–4 h at room temperature. NaCl was removed by leaving the silicone template in a thermostatic distilled water bath at 60 °C for 5 h. Finally, the silicon template was removed, dried on tissue, and placed in a Petri dish [Fig. 1(b)].

The hydrogel was prepared via the freezing–thawing method, as shown in Fig. 1(c), as reported in our previous paper [29]. Briefly, 1.5 g of PVA was added to 15.0 mL of aqueous 1.0 M H_2SO_4 solution and heated at 70 °C–90 °C in a water bath under stirring to obtain a transparent solution. The solution was then treated in an ultrasonic bath to remove the remaining bubbles and transferred into a Petri dish containing the desired silicone templating [Fig. 1(a)]. Three freeze–thaw cycles were made, consisting of 3 h of freezing and about 1 h of thawing. Four PVA hydrogel samples were prepared and called as follows (based on the porous size and porous amount): 425–355/1, 425–355/2, 355–300/1, 355–300/2 (Table S1).

C. Single-Electrode TENG-Based Sensor Preparation

Food-grade silicone rubber support was prepared by thoroughly mixing 1.5 g of silicone base and 1.5 g of hardener in a beaker. The mixture was poured into a 3-D-printed TPU scaffold previously prepared, with an internal area of 3.2×1.5 cm. After 3–4 h, the curing was complete, and the silicone was removed from the scaffold [Fig. 1(d)]. PVA hydrogel, properly cut to fit the silicone support, was placed inside it with the rough face in contact with the bottom of the support. The sensor prototype was subsequently assembled by pouring silicone solution over the hydrogel, thereby sealing the capsule [Fig. 1(e)]. The replacement of H_2SO_4 with KCl was carried out by soaking the 425–355/1 PVA hydrogels in distilled water for 3 days, then in 1.0 M KCl solution for one day, and then sealing into the silicone support as previously described (PVA/KCl-based 425–355/1 S-TENG).

D. Characterizations

SEM, ATR-FTIR, and porosity measurements have been conducted on the freeze-dried PVA hydrogel.

A Renishaw field-emission scanning electron microscope, equipped with an InLens detector, operating at 10 kV, and a current of 80 pA was used. No metal coating was applied to acquire the presented image. The ATR-FTIR spectra were collected using a Perkin Elmer Spectrum Two spectrometer, equipped with a Universal ATR accessory, with a resolution of 0.5 cm^{-1} in the range 4000–400 cm^{-1} using 40 scans. Moreover, the pore diameters of the silicone template and the pore distribution were obtained with ImageJ software from SEM images.

The optical images of the hydrogels were taken with an Olympus BX53 equipped with a plan achromatic 5X objective, NA 0.10, and a working distance of 20 mm. The images were acquired by using the module NavSharp¹ of the EasyNav¹ package in LabSpec6 software from Horiba. The module allows for navigation on a sample image with any topography, keeping the field of view focused on the sample surface.

For porosity measurements [33], the samples were carefully weighed (M_d). A pycnometer of 25 mL was filled to the brim with cyclohexane and rapidly closed with the stopper. The excess of cyclohexane that leaked from the little capillary on the stopper was dried out with tissue, and a glass watch was placed on the stopper to reduce the loss of cyclohexane due to evaporation. The filled pycnometer was then weighed (M_2). The freeze-dried PVA samples were put inside the pycnometer and weighed again (M_1). After 30 min, the PVA sample was removed from the cyclohexane, and the remaining liquid on its surface was roughly dried with some tissue before being weighed (M_w). The porosity was then estimated through the following equation:

$$\text{Porosity} = \frac{M_w - M_d}{(M - 1 - M_2) + M_w} \cdot 100 \quad (1)$$

where M_w is the mass of the sample imbibed with cyclohexane, M_d is the mass of the freeze-dried sample, M_1 is the mass of the pycnometer and the cyclohexane after insertion of the sample, and M_2 is the mass of the pycnometer and the cyclohexane before the insertion of the sample.

Electrochemical impedance spectroscopy (EIS) measurements were conducted on a 1.0 cm hydrogel diameter sample in a Swagelok cell connected to an Autolab GSTAT128N potentiostat/galvanostat (Metrohm–Autolab).

Mechanical tests were conducted using an INSTRON TESTING MACHINE 4465 dynamometer connected to the “Bluehill Universal” software. The hydrogel samples were cut into 3.0×1.0 cm rectangles. The applied deformation rate is 5 mm/min using a 100 N load cell. The width and thickness of each sample were measured using a digital caliper (sensitivity of 0.01 mm).

E. Single-Electrode TENG-Based Sensor Applications

The measurements for the pressure sensor were carried out by manually placing different standard weights (1, 2, or 5 g) on the sensor surface. After the placement, the standard weight was left still for 5 s on the sample. To ensure system stabilization, a 10-s interval was observed between the consecutive

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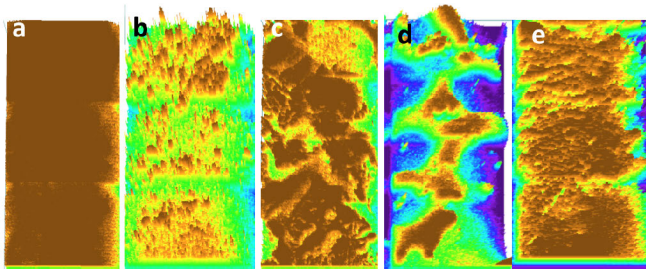


Fig. 2. Topographic images obtained using the module NavSharp of the EasyNav package in LabSpec6 software from Horiba on optical images of: (a) not-template PVA hydrogel, (b) 425–355/2, (c) 425–355/1, (d) 355–300/2, and (e) 355–300/1 surfaces.

measurements. For each weight, a total of 30 measurements were recorded.

For human motion detection measurements, the PVA hydrogel sensors were affixed to different human joints, specifically a finger and wrist, using adhesive tape. The selected point was bent to induce a mechanical deformation of the sample. Five bending cycles were performed for each joint to observe the reproducibility.

In both experimental setups, electrical connections were established by inserting a platinum wire into the hydrogel through the silicone capsule. This configuration allowed the triboelectric sensor to function as the working electrode, while the reference and counter electrodes were short-circuited and connected to a 10×10 cm aluminum square [34]. Electrical measurements were performed with an Autolab GSTAT128N potentiostat/galvanostat (Metrohm–Autolab) controlled by NOVA 2.10 software.

III. RESULTS AND DISCUSSION

A. Fabrication and Characterization of PVA Hydrogel

The silicone template morphology was observed using optical and SEM microscopy (Fig. S1), confirming the attainment of pores in selected dimensions corresponding to the implemented salt fraction. Through ImageJ software, the pore size distribution results as $74 \pm 15 \mu\text{m}$ for the NaCl 425–355 fraction used and $53 \pm 10 \mu\text{m}$ for the NaCl 355–300 fraction, with a surface pore coverage of 91% and 73%, for 425–355 fraction with 2 and 1 g, and 85% and 66% for 355–300 fraction with 2 and 1 g, respectively.

The prepared hydrogels were subjected to structural and morphological characterization.

The topography reconstruction of the PVA hydrogel surfaces obtained by software elaboration of optical microscopy images is reported in Fig. 2. A change in surface roughness appears evident in all templated hydrogels when compared to the untemplated one. Moreover, the roughness can be modulated according to the silicone templating used.

The ATR-FTIR spectrum of the PVA is reported in Fig. 3(a). The broad peak at 3293 cm^{-1} (highlighted in light blue) relates to the stretching of the OH bond. In contrast, the peaks at 2939 , 2909 , and 1423 cm^{-1} (highlighted in light red) relate to the CH_2 asymmetric stretching, symmetric stretching, and bending, respectively. The peak at 1326 is associated with OH rocking with CH wagging, and the peak at 1142 cm^{-1}

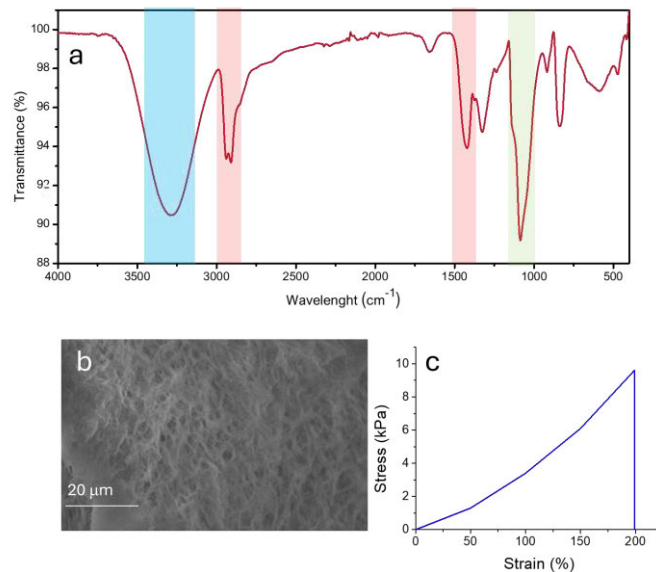


Fig. 3. (a) ATR-FTIR spectrum, (b) SEM images of the PVA hydrogel sample's cross section, and (c) tension stress-strain curve of the PVA hydrogel sample.

(highlighted in green), related to vibrations in the crystalline phase of PVA [35], [36], [37].

Fig. 3(b) reports the SEM image of the PVA hydrogel. The low porosity, confirmed even by pycnometer measurements ($28\% \pm 8\%$), is attributable to the higher crosslinking due to the H bonds derived from the acid media employed.

PVA hydrogel reached a tensile modulus of $9 \pm 1 \text{ kPa}$ without fracture at a strain of $(2.0 \pm 0.3) 10^2\%$.

From the intercept with the x -axis with the Nyquist plot obtained by EIS measurements (Fig. S2), a resistance value of 186Ω was calculated, with an ionic conductivity of 0.49 S/m .

To prepare the single-electrode TENG-based sensor first, a silicone-based support was first fabricated by pouring the silicone mixture into the 3-D-printed TPU negative scaffold [Fig. 1(d)]. Subsequently, the PVA hydrogel was cut with a stamp in a rectangular shape equal to the area of the internal part of the scaffold, namely $3.0 \times 1.0 \text{ cm}$, and inserted with the rough surface in contact with the silicone. The flat side of the hydrogel sample was sealed by pouring the silicone solution, thus producing a simple encapsulation for the electrode material, as in Fig. 1(e).

B. TENG Applications

1) *Pressure Sensor*: The single-electrode TENGs were tested as pressure sensors by following the change in current overtime, while different weights were placed and removed from the sensors' surfaces, with the employed set-up presented in Fig. 4(a). The transient current generated over time in response to the applied pressure is measured by plotting the current (i) versus time (t) in Fig. 4(b) and was consequently integrated to obtain charge variations (q) versus time (t) [Fig. 4(c)]. When the weight is placed, a negative current peak is generated; when it is removed, a positive current is observed. Namely, by integrating the signal, the weight placement is visible, since its application induces a charge movement,

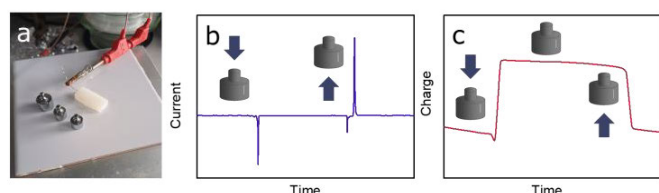


Fig. 4. (a) Setup for the electrochemical measurements. (b) Weight application protocol with current versus time. (c) Charge versus time response.

caused by both the triboelectric effect and the hydrogel ionic conductivity. With the weight removal, the relaxation of the hydrogel material, and the separation of the silicone from the hydrogel electrode, a new charge movement is allowed, opposed to the previous one.

The tested pressures, calculated from weight values and contact areas, are 32.7, 65.4, and 163.5 Pa, respectively. As TENG sensors' response is based on the friction between the surfaces of two materials, as previously described, we investigated the effect of the different roughness of the hydrogel surfaces on the pressure tests. The triboelectric-generated voltage induces a current that is measured versus time and is the signal used by the sensor. A change in current value is observed during the placement and removal of weight [Fig. 4(b) and (c)]. The electrical responses, transformed into charge (Coulombs) by the integration of the electrochemical signal areas (Fig. S3), are reported in Table S1. The data represent the average of 30 measures. Fig. 5 reports the graphics obtained by plotting the charge versus pressure. Pressure application and pressure release data graphics are reported, since the resulting signals highlighted a different output in terms of charge values. To observe the effect of the amount of template porosity on the hydrogel surface, the response of 425–355/1, 425–355/2, 355–300/1, and 355–300/2 S-TENGs has been compared [Fig. 5(a), (b), (e), and (f)]. A greater number of large pores could lead to their collapse, resulting in a loss of roughness in the template hydrogel and a decrease in signal. In contrast, when the pore diameter is smaller, a greater quantity leads to an increase in the roughness of the template hydrogel. The effect of the template porous size on hydrogel surface was investigated by comparing the responses obtained with 425–355/1, 355–300/1, 425–355/2, and 355–300/2 S-TENGs [Fig. 5(c), (d), (g), (h)]. In all cases, higher modulation and more accurate linear electrochemical responses were obtained with 425–355 samples.

This can be justified by a greater roughness in the surface of the hydrogel due to the increased salt particle diameter, which facilitates the contact points with the insulating material. The graphs obtained during pressure application and release show a similar trend with linear behavior for 425–355 hydrogels S-TENGs. Higher sensitivity is obtained with a 425–355/1 sensor under pressure release conditions, and the responses of the 425–355 sensors are reported in Table I and Fig. S4.

2) *Human Motion Sensor*: Due to the confirmed operation of the hydrogel sensor prototype, we attempted real-time human motion monitoring employing PVA/KCl-based 425–355/1 S-TENG. This wearable sensor was comfortably attached to

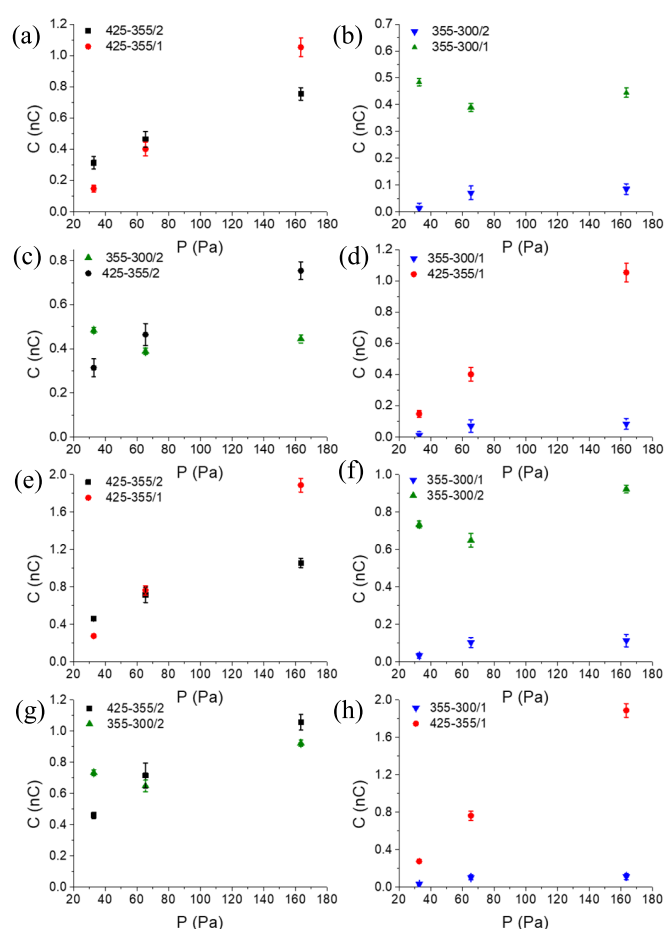


Fig. 5. Charge versus pressure graphics (a) 425–355/2 and 425–355/1 application, (b) 355–300/2 and 355–300/1 application, (c) 425–355/2 and 355–300/2 application, (d) 425–355/1 and 355–300/1 application, (e) 425–355/2 and 425–355/1 release, (f) 355–300/2 and 355–300/1 release, (g) 425–355/2 and 355–300/2 release, and (h) 425–355/1 and 355–300/1 release.

TABLE I

LINEAR REGRESSION FITS AND R^2 VALUES FOR 425–355 S-TENGs

S-TENGs hydrogel	Linear fit	R^2	Pressure
425–355/2	$y = 3.29 \cdot 10^{-3} x + 0.22$	0.996	application
425–355/2	$y = 4.57 \cdot 10^{-3} x + 0.32$	0.993	release
425–355/1	$y = 6.96 \cdot 10^{-3} x - 0.08$	0.999	application
425–355/1	$y = 12.54 \cdot 10^{-3} x - 0.12$	0.998	release

the surface of a volunteer's wrist skin and finger with the assistance of adhesive tape [Fig. 6(a) and (c)]. The measurements were conducted in triplicate by applying a series of controlled deformations of the volunteer's wrist and finger, by bending and relaxing the joint after a selected time interval. As shown in Fig. 6(b) and (d), the movement of the wrist and the finger produced a change in the current response, thus inducing a charge movement due to the deformation of the hydrogel and the contact between silicon and PVA. The working principle is that when the sensor that fits the human skin is subjected to external stress caused by human movement, it changes the contact between the hydrogel and

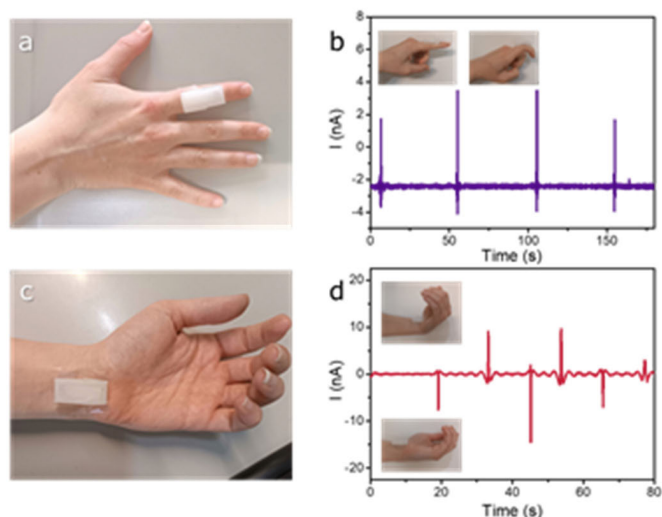


Fig. 6. (a) PVA/KCl-based sensor attached to a volunteer's finger, (b) current responses versus time of finger bending, (c) PVA/KCl-based sensor attached to a volunteer's wrist, and (d) current responses versus time of wrist bending.

the opposing triboelectric silicone layers. The change of the potential differences between the two materials occurs and causes an electron flow in the external circuit to flow (current measured). When the two materials lose contact, the system returns to its initial state. This process proceeds cyclically whenever there is an external stress [38].

The same fast-responsive signals could be stably obtained for repetitive movements. This behavior makes this S-TENG competitive with similar systems recently reported in the literature, such as the PTC-1 [38], the PVA/ NaNO_3 hydrogel [39], and glycerol/poly(vinyl alcohol) (GL/PVA) hydrogel and silicone rubber (GH-TENG) [24].

IV. CONCLUSION

PVA hydrogel samples with the same composition but different surface roughness were prepared by implementing a silicone template in the fabrication process, with the roughness modulated through various porous amounts and sizes. The PVA hydrogel preparation and consequential sensor assembly were conducted in a few steps, involving a simple yet efficient freeze-thaw cross-linking and silicon curing at room temperature. The morphology, mechanical, and electrical properties were evaluated on PVA hydrogels with H_2SO_4 .

S-TENG sensors have been assembled using a food-grade silicone scaffold, with the aim of both encapsulating the material to prevent solvent leakage and inducing the triboelectric effect between the silicone and PVA hydrogel. These devices have been tested as pressure sensors or real-time sensors of human motions. The pressure tests have been conducted with the application and removal of standard weights on the S-TENG surface. A linear response in the range 30–170 Pa stress applied was observed for all the S-TENGs, with a higher sensitivity observed for the 425–355/1 sensor. The selected S-TENG was then implemented as a human motion sensor, following the current variations during the bending and

relaxing of a finger or a wrist, providing fast and reproducible responses.

With the confirmation of human motion sensing, this work explores the horizon of simple, biocompatible, and cheap sensor fabrication for medical employment and wearable electronics.

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