J Bionic Eng 17 (2020) 55–63
DOI: https://doi.org/10.1007/s42235-020-0004-9 *Journal of Bionic Engineering*
https://www.springer.com/journal/42235

Flexible Piezoresistive Sensors based on Conducting Polymer-coated Fabric Applied to Human Physiological Signals Monitoring

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Abstract

This paper describes a flexible pressure sensor based on polypyrrole(PPy)-Cotton composites, in which PPy is grown on cellulose fibers of cotton pads *via* an *in situ* vapor growth method, which is beneficial to the homogeneity of the composites. The resulting devices exhibits rapid response and recovery speed, the response and recovery times are 220 ms and 240 ms, respectively. The optimal PPy-Cotton Pads (PCPs) sensor shows low detection limit, which is about 50 Pa. At the same time, it exhibits excellent durability in the measurement of repeated loading-unloading pressure over 1000 cycles. The resultant sensor can be attached on different positions of body and applied to recording physiological signals, such as wrist pulse, vocal cord vibration, respiration and eyes blinking. Finally, a 4 × 4 pressure sensor array shows that the PCPs sensor has capability in pressure distribution detection and represents great potential in the fields of wearable electronics and biomedical devices.

Keywords: flexible device, pressure sensor, vapor growth, physiological signals monitoring

1 Introduction

In recent years, with the development of wearable health monitoring devices, electronic skins (E-skins), medical diagnostics and soft robots[1–4], the demand for electronic devices with flexible, elastic and portable features has grown rapidly. Flexible pressure sensors as a vital part of wearable electronic devices have received extensive attention.

Generally, pressure sensors can be divided into four categories according to their different mechanisms: piezoresistive sensors[5–7], piezoelectric sensors[8,9], capacitive sensors[10] and field-effect transistor-based pressure sensors[11]. Among them, the flexible pressure sensors based on piezoresistive sensing mechanism have become one of the research hotspots in pressure sensor field because of their simple mechanism and structure, as well as outstanding sensing properties, such as high sensitivity, short response and recovery time, low detection limit and good working stability. In some recent works, the piezoresistive sensors fabricated by compositing fabrics with conductive materials exhibited good performance and gained wide attention.

Fabrics are flexible and porous materials which are knitted or naturally pressed by natural fibers[12]. Compared with synthetic flexible materials, fabrics own the features of renewable and degradable, which makes them as a good candidate in the fields of eco-friendly and disposable wearable electronics[13]. In recent years, many flexible pressure sensors based on the composite structure of conductive material and fabrics have been investigated and reported. For example, Ge *et al*. developed a rGO/polyaniline wrapped sponge which is prepared *via* rGO coating and the *in situ* synthesis of polyaniline nanowires on the backbones of sponge[14]. Luo *et al*. achieved high-repeatability and low-hysteresis flexible piezoresistive sensors by introducing carbon black particles and polyvinylidene fluoride in the knitted fabric as the electrical and mechanical interconnects between the fibers[15]. Yang *et al*. fabricated a wearable

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textile strain sensor by acting graphene oxide as a colorant to dye the polyester fabric, and then the graphene oxide is reduced at high temperature[16].

Till now, the conductive materials such as carbon black[17], silver nanowires (AgNWs)[13,18,19], carbon nanotubes (CNTs)[20–22], graphene[16,23] and some conductive polymers have been chosen and introduced into the porous skeleton of fabrics to build flexible pressure sensors. However, in most of the works, the composites were fabricated by the methods of dip-coating or drop-coating with suspension of conductive materials. The conductive materials could both attach on the surface of fabric fibers and fill in the porous structure leading to the distribution of conductive materials inhomogeneous, which is unfavorable to the consistency of sensors. Meanwhile, mechanical mixture method is difficult to guarantee the stability of composite structure.

In this study, a pressure-sensitive composite with piezoresistive performance was obtained by *in situ* growing polypyrrole (PPy) on cotton pads *via* vapor growth method. The sensitive layer was packaged by medical tapes with silver electrodes to fabricate a flexible pressure sensor. Through vapor growth method, PPy could coat on cellulose fibers uniformly and firmly. Comparing to insoluble materials, $FeCl_3$ as oxidant that promotes the polymerization of pyrrole, could completely dissolve in water and form a homogeneous solution. By immersing the cotton in $FeCl_3$ solution, $FeCl_3$ could uniformly distribute in cotton fibers, which guarantees the uniform polymerization of pyrroleon on the surface of cotton fibers. It is beneficial to the homogeneity of pressure-sensitive composite. The developed flexible sensors could be applied in physiological signals monitoring, such as wrist pulse, respiration, speech recognition and blink recognition, which indicates the sensors have great potential for the applications in wearable flexible devices and health-care. Moreover, the pressure sensor arrays based on PCPs can realize the detection of spatial distribution of the applied pressure.

2 Experimental

2.1 Materials

Cotton pads were purchased from the Unicharm, with thickness of 1.5 mm. $FeCl_3$ anhydrous (> 97%) and Pyrrole (> 98%) were purchased from Sinopharm Chemical Reagent Co., Ltd. Conductive Silver Paste (SP1012) was purchased from the Guangdong Nanhai Qiming Everbright Technology Co., Ltd. The deionized water used throughout all experiments was purified through a Millipore system.

2.2 Fabrication of PPy-Cotton pressure-sensitive layer

The fabrication process of PCPs is shown in Fig. 1a. The PPy grows on cotton pads using a method described elsewhere[24]. The blank cotton pads were washed by deionized water to remove impurities and dried at 60 ˚C before using. $FeCl_3$ aqueous solution with different concentrations (0.01 M, 0.03 M, 0.05 M, 0.07 M, 0.1 M and 0.2 M) were prepared by adding $FeCl_3$ in deionized water under magnetic stirring until completely dissolved. The blank cotton pads were soaked into $FeCl_3$ solution in different concentrations for 2 min, then, sealed together with 1 mL pyrrole in beakers of 500 mL for 12 h (Fig. 1b) and to achieve PCPs. Finally, the PCPs were washed by deionized water to remove $FeCl_3$ and unreacted pyrrole, and dried at 60 ˚C. The PCPs fabricated by different concentrations of $FeCl_3$ aqueous solutions from 0.01 M to 0.2 M were named from PCP-1 to PCP-6, respectively.

2.3 Fabrication of PCPs pressure sensors

The schematic illustration of the PCPs pressure sensors is shown in Fig. 1d. The PCPs were cut into squareness (1 cm × 1 cm), and sandwiched between two pieces of medical tapes with silver electrodes. The silver electrodes were fabricated by screen-printing technique. Then, two copper bands were fixed at the edges of two electrodes for following measurements. Finally, the devices were aged under a fixed pressure of 1 kPa for 24 h.

2.4 Fabrication of pressure sensor array

The 0.07 M $FeCl_3$ solution were dropped on blank cotton pads to form a 4 × 4 array (50 μL per drop). The array was sealed together with 1 mL pyrrole into a beaker of 500 mL for 12 h. Then the array was washed by deionized water and dried at 60 ˚C. Then fixing four medical tapes with silver electrodes on top of the array, and another four were fixed under the array, the upper

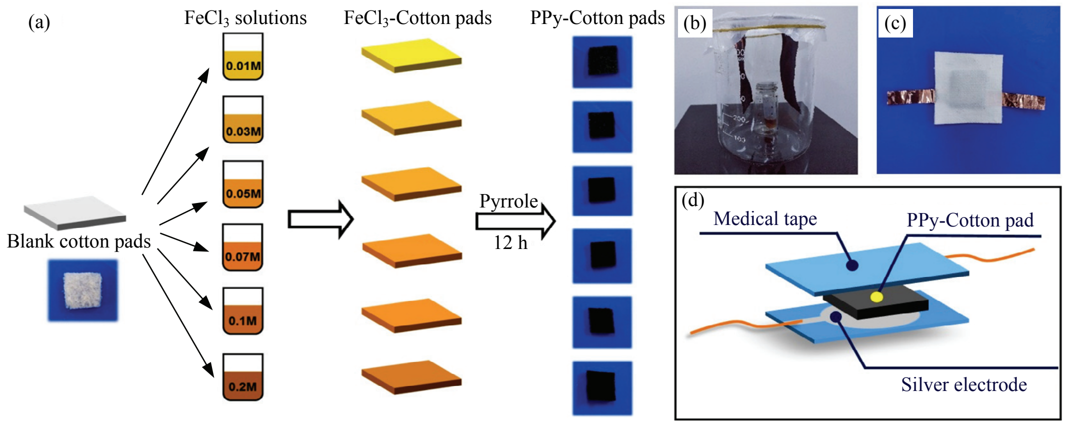

Fig 1 (a) Schematic illustration of PCPs; (b) photograph of the cotton pads with $FeCl_3$ solutions sealed with pyrrole in a beaker; (c) photograph of the PCP sensor; (d) schematic illustration of the structure of the PCP sensor.

and lower electrodes are perpendicular to each other. Finally, fixed copper bands at the edges of the electrodes for further measurement.

2.5 Characterizations

Field Emission Scanning Electron Microscopy (FESEM, JEOL 7500F microscope) was used to observe the surface of blank cotton pads and PCPs.

2.6 Measurement

The Digital push and pull gauge (HP-50 model, Shanghai Yunjin electrical Co. Ltd., China) was used to provide the required pressure. The CHI660E electrochemical analyzer (CH Instruments, Inc., Shanghai) was used to measure the current *v.s.* applied pressure relationship and current-voltage (I-V) curves, and the current measurements were carried out at a constant applied voltage of 1 V. In this work, the response and recovery times were defined as the time to reach 90% of the total current change when the pressures are applied and released, respectively.

3 Results and discussions

3.1 Piezoresistive characteristic and mechanism of pressure sensors

First of all, in order to investigated the final content of PPy in resultant PCPs materials, the weight of cotton pads before and after growing PPy were recorded. As shown in Fig. 2a, the PPy content increases with the raise of the concentration of $FeCl_3$ aqueous solution, and presents a linear relationship. The PPy content is defined as $(m - m_0)/m$, where m represents the mass of PCPs and m_0 represents the initial mass of cotton pads without PPy. The similar phenomenon could be observed in Scanning Electron Microscopy (SEM) images of PCPs with different PPy contents (shown in Figs. 2b–2i). There is no obvious difference between the cellulose fibers of different samples under low magnification (Figs. 2b–2e). Under high magnification (Figs. 2f–2i), the surface of blank fibers is rough, but the surface of PCP-6 fibers is smooth. With the increase of the PPy content, the smooth degree of the surface of cellulose fibers increases obviously. This indicates that PPy coated on the surface of the fibers uniformly by *in situ* vapor growth.

The piezoresistive characteristics of the devices made of PCPs were measured and systematically studied. Fig. 3a shows the $\Delta I/I_0$-pressure relationship and the linear fitting curves. Herein, the current variation ratio is defined as $\Delta I/I_0 = (I_p - I_0)/I_0$, where I_p represent the final current under applied pressure and I_0 represent the initial current. It can be observed that the fitting curves of PCP-3 and PCP-4 exhibit better linearity, and the current change of other devices turns to saturate when the pressure exceeded 2.5 kPa. The pressure sensitivity is defined as $S = (\Delta I/I_0)/\Delta P$, where ΔP represents the pressure change. The relationship of sensitivity *v.s.* PPy content relationship is shown in Fig. 3b, the sensitivity of the devices increases until the PPy content reaches 3.81%, and then decreases with further increase of PPy content. The reason for this phenomenon is as follow: when the PPy content is too low, the conductivity of the PCP-fibers is poor, which results in a high contact

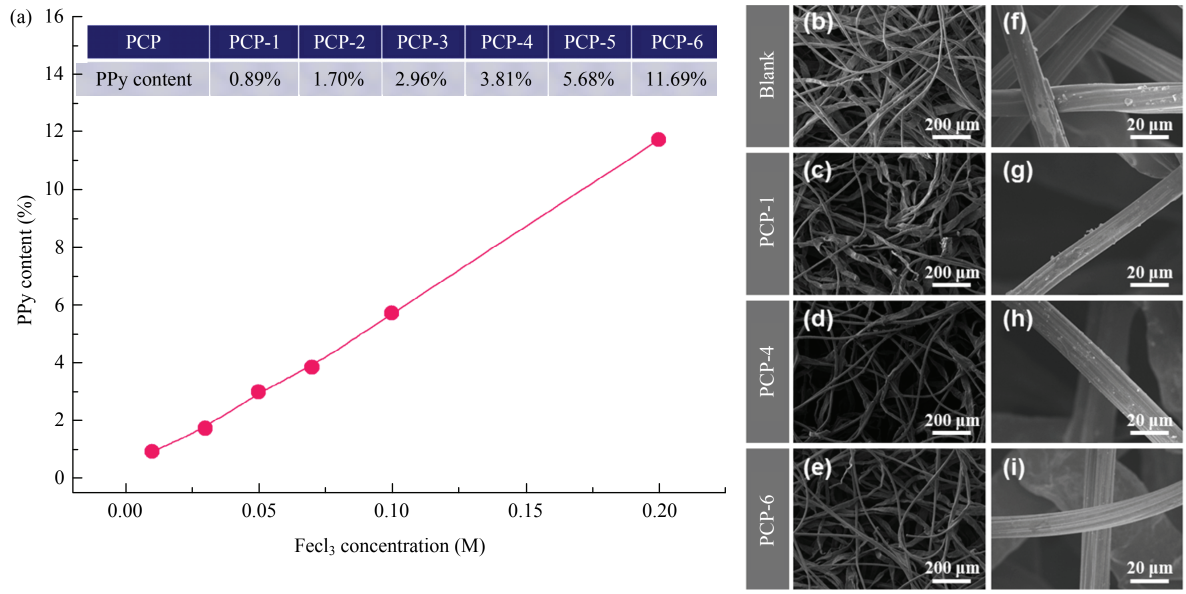

Fig. 2 (a) The relationship curve between PPy content in PCPs and concentration of $FeCl_3$ aqueous solution; (b–e) low magnification and (f–i) high magnification SEM images of blank cotton pads and PCPs.

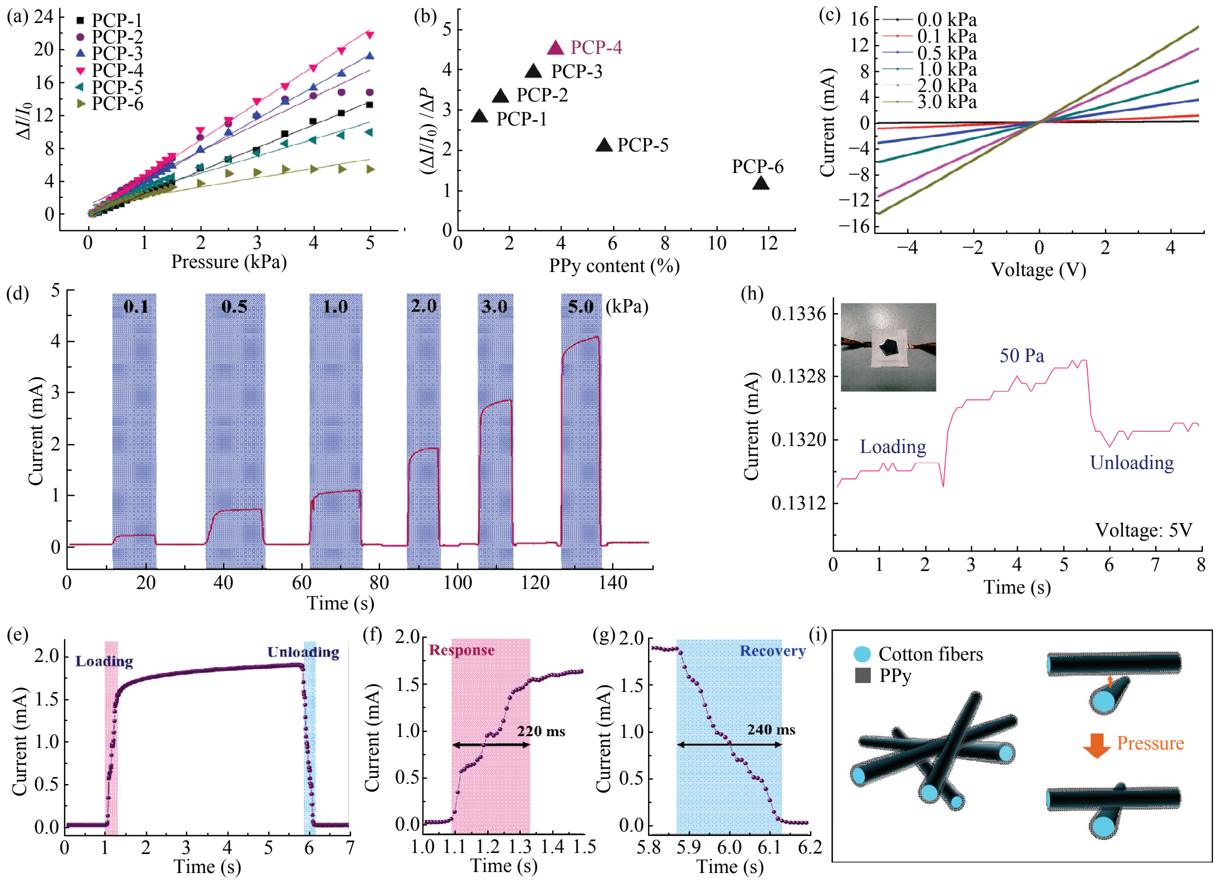

Fig. 3 (a) The $\Delta I/I_0$-pressure relationship curves of the PCP sensors with linear fitting; (b) the relationship between sensitivity of PCP sensors and PPy content; (c) the current-voltage (I-V) curves of the PCP-4 sensor under a given pressure range from 0 kPa to 3.0 kPa; (d) dynamic current-time curve of the PCP-4 sensor under different pressures; (e) the response and recovery curve of the PCP-4 sensor under pressures of 2.0 kPa; (f, g) the enlarged view of response and recovery curves of PCP-4 sensor, respectively; (h) the minimum detection limit of the PCP-4 sensor; (i) the mechanism diagram of the PCP sensitive material.

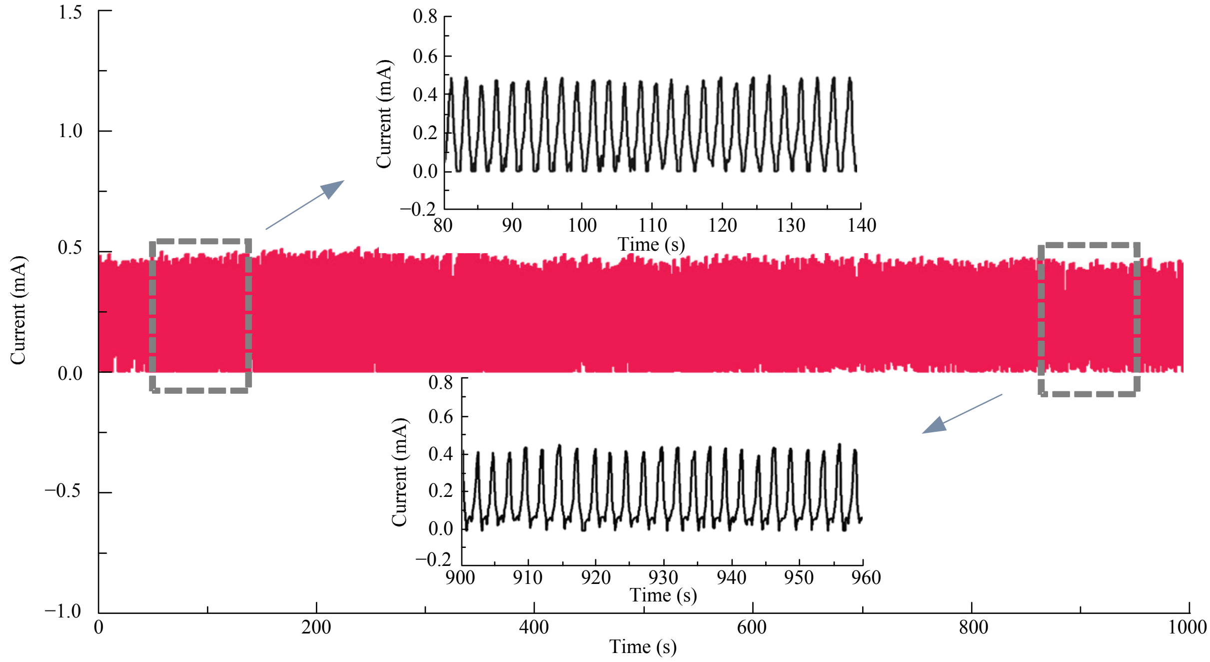

Fig. 4 Current-time curves of the PCP-4 sensor for 1000 loading/unloading cycles with an applied pressure of 0.3 kPa.

resistance between PCP-fibers. The sensors cannot exhibit a significant resistant change even under a high applied pressure. When there is too much PPy grown on the cotton fibers, the PCP-fibers exhibit good conductivity, which leads to the PCP materials show a low resistance even without applied pressure. The sensors also could not exhibit an obviously resistance change when pressure is applied. Hence, there is a moderate PPy content which could ensure the sensors have the optimal sensitivity to pressure. The sensitivity of the devices based on PCP-4 is highest, hence, the PCP-4 sensor was selected as the optimal sensor for the following measurements.

The current-voltage (I-V) curves of the PCP-4 sensor under the applied pressure from 0 kPa to 3 kPa are shown in Fig. 3c (test voltage from −5 V to 5 V). It can be observed that the I-V curves exhibit good linear behavior. The slope of the I-V curves increased with increasing pressure, implying a decrease in resistance.

As shown in Fig. 3d, the current response of PCP-4 sensor under different pressures from 0.1 kPa to 5.0 kPa are measured. The device exhibit rapid response and recovery speed, the current increases immediately and then remains stable, and the current response increases with the increase of pressure, which was in accordance with the result shown in Fig. 3c. Figs. 3e–3g show the single response and recovery processes of the PCP-4 sensor under a pressure of 2.0 kPa, the response time is about 220 ms and the recovery time is about 240 ms. Besides, the minimum detection limit of the PCP-4 sensor was measured and shown in Fig. 3h. The device can detect a pressure as low as 50 Pa.

The mechanism schematic diagram of the PCP sensor is shown in Fig. 3i. In PCP materials, the surface of cellulose fibers is covered by conductive PPy, however, due to the loose structure of cotton, which is formed by cellulose fibers stacking results in less contact points between conductive fibers without applied pressure, only a few conductive paths could form in the PCPs and lead to extremely high resistance. When pressure is applied on the device, the structure of PCPs becomes denser and contact points between conductive fibers increase, causing the resistance decreases rapidly. The resistance decreases continuously with the increase of pressure.

Repeatability is a significant parameter for pressure sensors. Here, the repeatability of the PCP-4 sensor was further explored, the 1000 times of loading-unloading repeat-cycles with an applied pressure of 0.3 kPa was measured and shown in Fig. 4. The device can maintain its sensing performance during the 1000 cycles of the loading-unloading process, and exhibits high

Table 1 Comparison of the pressure sensing parameters for previous and this work

Materials	Sensing mechanism	Sensitivity	Detection limit	Response/Recovery time	References
Gold nanoparticles	Piezoresistive	0.08 kPa^{-1}	25 Pa	270 ms/110 ms	Ref. [25]
Hierarchical rGO wrinkles	Piezoresistive	178 kPa^{-1}	42 Pa	261 ms/131 ms	Ref. [26]
Mxene/rGO	Piezoresistive	22.56 kPa^{-1}	< 10 Pa	245 ms/212 ms	Ref. [27]
rGO-Ag NW@cotton fiber	Piezoresistive	4.23 kPa^{-1}	–	220 ms/420 ms	Ref. [28]
PPy-Cotton	Piezoresistive	4.48 kPa^{-1}	50 Pa	220 ms/240 ms	This work

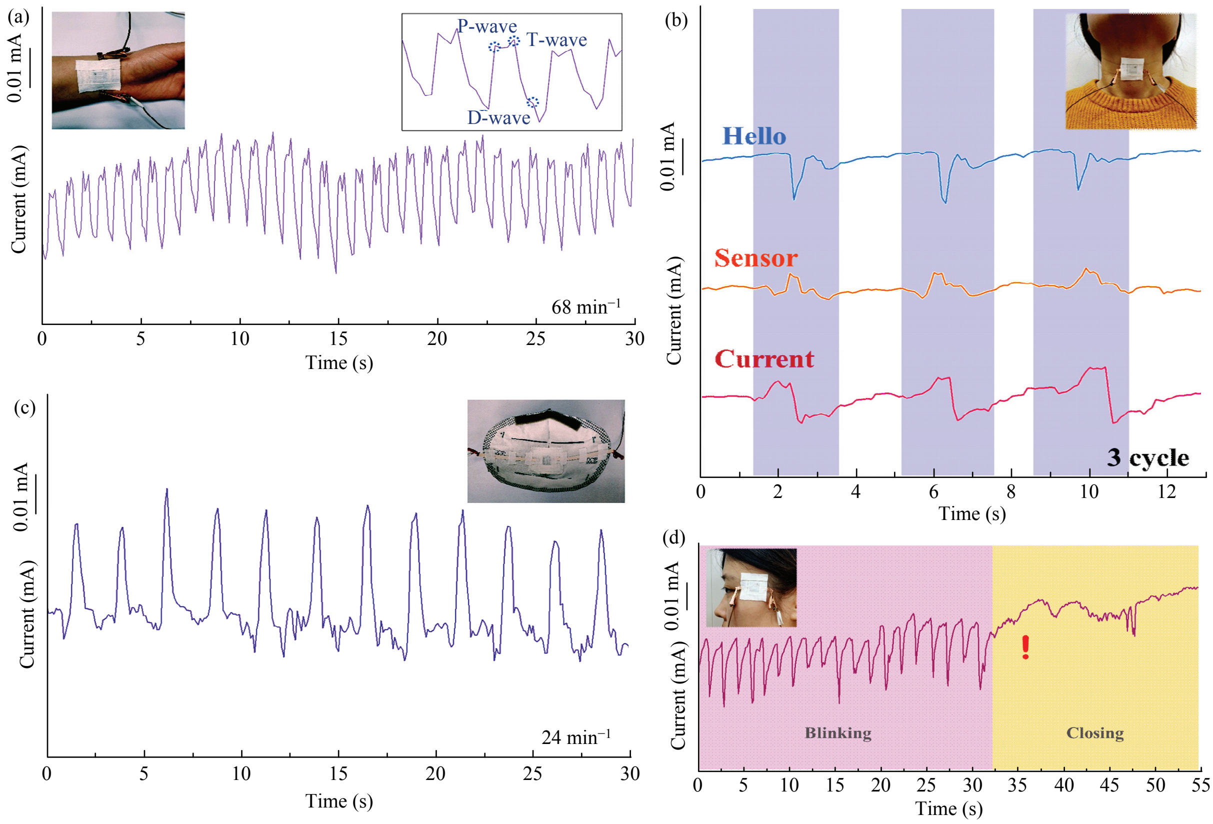

Fig. 5 (a) Current-time curves of PCP-4 sensor for wrist pulses monitoring; (b) current-time curves of the PCP-4 sensor during the speech-recognition measurement; (c) current-time curves of the PCP-4 sensor during the respiration rate monitoring; (d) current-time curves of the PCP-4 sensor for monitoring blink of the tester.

repeatability and stability. The wonderful repeatability of PCPs sensors is owing to the uniform distribution of PPy in the composite structure.

For comparison, the pressure sensing parameters of some recent literatures are presented in Table 1. The PCP sensors in this work exhibit comparable pressure sensing behavior, no matter in sensitivity, detection limit or response speed. Moreover, in this work, PCP sensors were fabricated by *in situ* growing PPy on cotton pads *via* vapor growth method, which guarantees the resultant sensors have a homogeneous and stable structure.

3.2 Application of the pressure sensors for human physiological signals monitoring

In order to further explore the applications of the PCP-4 sensor for wearable devices, the resultant devices were used to detect human physiological signals from weak vibrations such as pulse, vocal cord vibration and blink.

Firstly, the PCP-4 sensor was attached to the wrist of a tester to monitor wrist artery pulse signal. The resultant curve is shown in Fig. 5a. The current signal of the sensor changed periodically with the pulsation rhythm of the tester. The pulsation rhythm measured by the PCP-4 sensor was 68 min^{-1}, which was in accordance with that measured from a commercial sphygmomanometer. Furthermore, three waves can be distinguished from the single response cycle clearly: percussion wave (P-wave), tidal wave (T-wave) and diastolic wave (D-wave), which are related to heart rate, ventricular pressure, and systolic and diastolic blood pressure[29]. The results can reflect the state of blood circulation and monitor physiological indexes such as arterial blood pressure and heart rate. Therefore, the PCP-4 sensor is suitable for the application of portable and disposable pulse monitoring, which is potential to be a simple and low-cost method to observe heart state.

Moreover, the PCP-4 sensor was fixed on a person's throat to monitor the weak pressure caused by vocal cord vibration when the speaker spoke. The current signals of the PCP-4 sensor in the speech-recognition test are presented in Fig. 5b. The current-time curve represented specific patterns when the tester spoke different words such as "Hello", "Sensor", and "Current". It should be noticed that the signals of each word were measured for three times to show the repeatability. The current-time curves exhibit similar characteristic peaks and valleys when the tester speaks the same word, indicating the sensor possesses high sensitivity and excellent speech-recognition ability.

Additionally, the PCP-4 sensor can also be applied in the measurement of respiration rate. The device was fixed on a mask and put on the tester's face to detect the weak pressure caused by exhaled airflow during breathing. From the current-time curve shown in Fig. 5c, the tester's respiration rate is recorded as about 24 min^{-1}, which is conforms to the practical level. Breathing is a significant physiological index of human body, which can reflect condition of health. Many diseases could be diagnosed from breathing state. The pressure sensor can realize real-time monitoring of breathing state, which is of great significance in the wearable and medical fields[30].

Finally, the PCP-4 sensor was attached on the temple of tester to detect the signal of the eyes blinking. As shown in Fig. 5d, the current signal of sensor changes periodically with the tester normal blink during the first 30 s. And the curve turns flat when the tester closed eyes in following 25 s. The phenomenon is owing to the skin

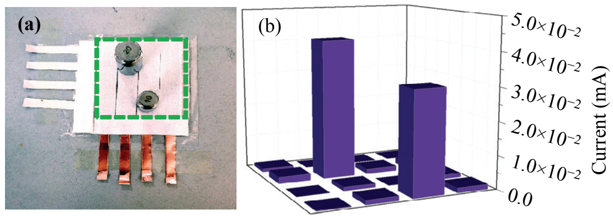

Fig. 6 (a) Photograph of the 4 × 4 pressure sensor array based on PCP-4; (b) the distribution of the relative resistance changes with various weights (10 g and 20 g).

and muscles around the eyes will tighten or relax during blink, and the pressure sensor could show response to the microvariations around temple. Hence, the PCP-4 sensor shows significant potential application for monitoring the fatigue driving. If the driver is driving under sober state, the normal blink will produce periodic pulse signals. When the driver eyes close for more than three seconds, the sensor cannot receive obvious pulse signal. At this time, the driver should be warned. This makes the PCP-4 sensor as a wearable device of great significance in practical application.

In the application of electronic skin, it is necessary to monitor the spatial pressure distribution by arraying sensor elements[31–33]. As shown in Fig. 6a, a 4 × 4 pressure sensor array based on PCP-4 was prepared (the green dotted area). Fig. 6b shows the distribution of the current change when various weights (10 g and 20 g) were placed on different pixels of the array. The signals were recorded and plotted as three-dimensional (3D) bar graph. It can be observed that only the pixels corresponding to positions which were loaded weights exhibits current increase, the other pixels only show slight change of current. The result indicates the array based on the PCP-4 sensor has the capacity to monitor the distribution of the pressure and distinguish the pressure magnitude roughly. The array shows the potential applications in electronic skin devices and wearable electronics.

4 Conclusion

In this study, the flexible pressure sensors with piezoresistive performance based on PCPs were fabricated. The *in situ* vapor growth method ensures the pyrrole could polymerize and coat on the cellulose fibers uniformly and firmly, so that the PCPs have stable electrical properties. The PCP-4 sensor is capable of monitoring a pressure as low as 50 Pa, and exhibits steady and fast response to pressures. The response and recovery times were 220 ms and 240 ms, respectively. Moreover, the sensor can be applied to monitor various human physiological signals, such as real-time wrist pulse, vocal cord vibration, respiration rate and eye blinking. In addition, the pressure-sensitive array based on PCP-4 exhibits good resolution in the detection of pressure distribution. In view of the characteristics and applications of the PCPs pressure sensor, it has potential application prospects in wearable electronics and biomedical monitoring.

Acknowledgment

This study was supported by the National Natural Science Foundation of China (Nos. 61773178 and 61671218), the Natural Science Foundation Committee (NSFC, Nos. 61674066 and 61903150), the China Postdoctoral Science Foundation (No. 801191010412).

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