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3D braided yarns to create electrochemical cells

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Abstract

The demands for new configurations of electrochemical cells continue to grow and novel approaches are being enabled by the advent of new electromaterials and novel fabrication strategies. Wearable energy storage devices that can be seamlessly integrated into garments are a critical component of the wearable electronics genre. Recently, flexible yarn supercapacitors have attracted significant attention due to their ability to be integrated into fabrics, or stitched into existing textiles. Large-scale production of yarn supercapacitors using conventional manufacturing processes, however, is still a challenge. Here, we introduce the use of braiding technology to achieve a predetermined arrangement of fibre electrodes, the basis of a mass fabrication protocol to produce specific electrochemical cells: wearable supercapacitors. The resultant supercapacitors show a high capacitance of 1.71 mF cm^{-1} . The structure is highly flexible with a 25% capacitance loss recorded after 1000 bending cycles.

Keywords

create, yarns, cells, braided, electrochemical, 3d

Disciplines

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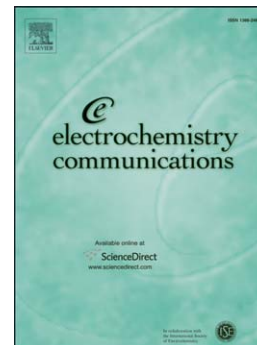
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3D Braided Yarns to Create Electrochemical Cells

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Abstract

The demands for new configurations of electrochemical cells continues to grow and novel approaches are being enabled by the advent of new electromaterials and novel fabrication strategies. Wearable energy storage devices that can be seamlessly integrated into garments are a critical component of the wearable electronics genre. Recently, flexible yarn supercapacitors have attracted significant attention due to their ability to be integrated into fabrics, or stitched into existing textiles. Large-scale production of yarn supercapacitors using conventional manufacturing processes, however, is still a challenge.

Here, we introduce the use of braiding technology to achieve a predetermined arrangement of fibre electrodes, the basis of a mass fabrication protocol to produce specific electrochemical cells: wearable supercapacitors. The resultant supercapacitors show a high capacitance of 1.71 mF cm^{-1} . The structure is highly flexible with a 25% capacitance loss recorded after 1000 bending cycles.

Keywords:

Wearable; supercapacitor; braiding; polypyrrole

1. Introduction

The advent of new electromaterials, particularly in the form of fibres, has enabled creative approaches to the fabrication of novel electrochemical cell configuration. Smart garments, are clothes with embedded functional electronic componentry including sensors and antennas [1, 2]. They may be used for physiological measurement and monitoring [3], hazard detection [4], and/or wireless communication [5]. This approach has found widespread application in personalized wearable medical monitors, and even in the military field [6]. Wearable energy storage devices must be seamlessly integrated into such garments [7]. Supercapacitors have been extensively studied for energy storage due to their high power density, fast charge-discharge and extended cycle life [8]. Recently, flexible yarn supercapacitors have attracted significant attention due to the ability to integrate them into fabrics, or stitch into existing textiles [9].

Long lengths of fibre electrodes are necessary to fabricate yarn supercapacitors. Such fibre electrodes may be formed on thin metal wires [10-12], or metal coated plastic wires [13]. Alternatively carbon fibres [14], reduced graphene oxide (rGO) fibres [15], carbon nanotube(CNT) fibres[16], or composites containing them have been used [17-23]. Several device architectures have been developed for yarn supercapacitors. These include two parallel fibres, two-ply yarns and coaxial yarns [24]. In the first two types, two fibre electrodes and a separator or solid polymer electrolyte are fixed in parallel or twisted. And in the third type, the core electrode, separator or solid polymer electrolyte and outer electrode are assembled layer by layer. These yarn supercapacitors have yielded double-layer capacitances in the range of 0.01-6.30 mF cm⁻¹, and pseudo-capacitance values up to 263 mF cm⁻¹[23].

Yarns are generally made by twisting fibres together. For large-scale production of yarn supercapacitors, simply twisting or grouping two fibre electrodes will cause direct contact of the electrodes. Wrapping fibre electrodes with separator membranes [22] or winding with

insulated wires [10] solves this short circuit problem, but limits large-scale manufacturing.

Braiding is a process that involves intertwining three or more sets of yarns over and under each other [25]. Two or more fibres can be braided into one yarn without contacting each other. Here, we introduce the use of braiding technology to enable production of yarn supercapacitors. Stainless steel (SS) wires and polyester fibres are braided into one yarn, and the SS wires play the role of current collectors. The SS wires in the yarn are fixed and separated by the insulated polyester fibres, so no additional separators are needed. Braiding is a fast continuous method of fabrication. To illustrate the use of this approach to create devices, polypyrrole (PPy) was electrodeposited onto the SS wires to produce active electrodes. The fabricated yarn supercapacitors showed good flexibility for application in wearable electronics. Other fine metal wires are commercially available including nickel, copper, titanium and platinum and would be amenable to this braiding method of fabrication. Apart from PPy, other conducting polymers or a variety of metal oxides/hydroxides such as γ -MnO₂ or Ni(OH)₂ can be simply electroplated on to such wires. Alternatively a number of new organic conducting fibres have recently emerged [26]. All such materials would be suitable for use in these novel electrochemical devices.

2. Experimental

2.1 Reagents and materials

Polyester fibres (100D) and nylon/SS fibres were obtained from China (Shijiazhuang Yunchong Trading Co., Ltd.). Pyrrole was purchased from Merck, sodium *p*-toluenesulfonate and lithium sulphate monohydrate were obtained from Sigma-Aldrich. Pyrrole was freshly distilled, whereas other chemicals were used as supplied. All aqueous solutions were prepared using Milli-Q water (~18 M Ω).

2.2 Fabrication of yarn supercapacitor

3D yarns were braided from polyester and nylon/SS fibres using a Trenz-Export braiding machine. The nylon fibres were removed using formic acid after braiding. Electrodeposition of PPy was achieved at constant current (0.05 and 0.1 mA cm⁻¹ for yarns with 2 and 4 SS wires, respectively) for 60 min using an aqueous solution containing 0.1 M pyrrole and 0.1 M sodium *p*-toluenesulfonate. During the deposition process, all the SS wires in the yarn were connected together. The samples were rinsed with water and dried in a fume hood overnight. Then the yarn samples were sealed in a transparent plastic tube injected with 1.0 M Li₂SO₄ aqueous solution to test capacitor performance. The quasi-solid state supercapacitor was fabricated using poly(vinyl alcohol) (PVA)/H₃PO₄ gel electrolyte prepared as described previously [27, 28].

2.3 Characterization

The morphology of SS electrodes extracted from the yarn were characterized by FE-SEM (JEOL JSM-7500FA). Cyclic voltammetry (CV) was performed over the range of 0 to 0.8 V using a CHI 650D electrochemical workstation (CHI Instruments, USA). Electrochemical impedance spectra (EIS) were measured using a Gamry EIS 3000™ system over the frequency range of 100 kHz to 0.01 Hz with an AC perturbation of 10 mV at open circuit potential. Galvanostatic charge–discharge tests were performed using a battery test system (Neware electronic Co., China) between 0 and 0.8 V. The cyclic bending test of the yarn supercapacitor was carried out using Shimadzu EZ mechanical tester.

3. Results and discussion

The 3D braided yarns can be produced continuously without limitation in length. Two SS wires formed a double helix structure in the yarn (Fig. 1a). The SS wires in the yarn were fixed by the polyester fibres with a separation of ~2 mm to avoid direct contact. Electrodeposition of PPy on the SS wires was readily achieved using constant current. SEM

image confirmed a coherent PPy film with a cauliflower morphology composed of large nodules as previously described [27, 28].

Fig.1 Schematic diagram to illustrate the 3D braiding process. (a-1) Polyester fibre and SS wire bobbins, (a-2) Braiding head, (a-3) As-prepared braid structure (a-4) Braided supercapacitor.

The CV curves of the yarn supercapacitor retained a nearly rectangular shape at scan rates up to 50 mV s^{-1} (Fig. 2a). As the scan rate reached 100 mV s^{-1} , the CV curve became distorted. This can be explained by the slower inclusion/ejection and diffusion of counter ions compared to the electron transfer in PPy at high scan rates [27, 28]. Consequently, the length specific capacitance of the yarn supercapacitor decreased with an increase in scan rate as presented in Fig 2b. The length specific capacitances were calculated to be 0.78 to 1.71 mF cm^{-1} . This value exceeded that of the reported yarn supercapacitors based on pen ink/nickel wire (0.504 mF cm^{-1}) [10], Chinese ink/SS wire (0.1 mF cm^{-1}) [11], rGO/Au wire (0.01 mF cm^{-1}) [12], MnO_2/ZnO nanowire (0.2 mF cm^{-1}) [13], rGO fibre (0.02 mF cm^{-1}) [15], CNT/PEDOT fibre (0.47 mF cm^{-1}) [17], CNT fibre (0.018 mF cm^{-1}) [18], CNT/graphene fibre (0.027 mF cm^{-1}) [19], MnO_2/rGO fibre (0.143 mF cm^{-1}) [20], and CNT sheet (0.029 mF cm^{-1}) [29].

The charge-discharge curves of the yarn supercapacitor at different current densities ranging from 5 to $50 \text{ } \mu\text{A cm}^{-1}$ are presented in Fig. 2c. The curves showed a nearly symmetrical triangular shape, indicative of good capacitor behaviour. It delivered a length specific capacitance of 1.79 mF cm^{-1} at a current density of $5 \text{ } \mu\text{A cm}^{-1}$. Even at the current density of $50 \text{ } \mu\text{A cm}^{-1}$, the capacitance was still as high as 1.25 mF cm^{-1} (Fig. 2d).

The Nyquist plot of the yarn supercapacitor is shown in Fig 2e. A semicircle in the high frequency region and a straight line in the low frequency region were obtained. The x-

intercept of the Nyquist plot represents the equivalent series resistance (ESR) for the device [30]. The ESR of our yarn supercapacitor was measured to be $\sim 11 \Omega \text{ cm}^{-1}$. The straight line at low frequencies indicates capacitive behaviour [31].

The cycling stability of the yarn supercapacitor was tested at a current density of $20 \mu\text{A cm}^{-1}$. After 1000 cycles, 30% of the initial capacitance was retained. PPy film swells and shrinks during the charge-discharge cycles, resulting in mechanical degradation [32].

Fig. 2 (a) CV curves and (b) length-specific capacitance of the yarn supercapacitor at different scan rates. (c) Charge-discharge curves and (d) length-specific capacitance of the yarn supercapacitor at different current densities. (e) Nyquist plot of the yarn supercapacitor. (f) Capacitance retention of the yarn supercapacitor for 1000 charge-discharge cycles.

For wearables, the yarn supercapacitor should possess excellent flexibility and maintain electrochemical performance under bending. CV tests were conducted at 5 mV s^{-1} while the yarn supercapacitor was held at different bending angles. The shapes of the CV curves only changed slightly up to 180° bending (Fig. 3a), reflecting that the yarn supercapacitor is highly flexible. The capacitance decreased by 13% as the device was bent from 0 to 180° . Furthermore, the yarn supercapacitor was subjected to repeated bending-relaxation to 90° for up to 1000 cycles and it suffered 25% loss (Fig. 3b).

Fig 3 CV curves of the yarn supercapacitor at different bending angles (a), and after different cycles of bending (b).

Our single yarn supercapacitor shows good electrochemical performance, which makes it promising for integration with other micro electronic device. However, the energy of a single yarn supercapacitor may not meet the requirement of those micro devices. The

common approach is to connect two yarn supercapacitors or more in parallel. However, it will greatly increase the device dimension and also involve the package challenge. With the braiding technology, we can enhance the energy storage in the same yarn by simply incorporate more fibre electrodes. As a proof of concept, 4 SS wires were braided into the yarn. After PPy deposition, two SS wires were connected as positive electrode and the other two as negative electrode. This configuration equals to two single yarn supercapacitors connected in parallel. The output current doubled within the same voltage window as shown in the CV curves (Fig. 4a). The charge or discharge time was more than two times of the value of the 2-wire device at the same applied current density (Fig. 4b), since half of the total current is applied to each single device. According to the previous results of the capacitances at different current densities (Fig. 2d), the capacitance increased with the decreased current density.

The use of gel or polymer electrolyte can minimize electrolyte leakage, which is highly desirable for the development of wearable yarn supercapacitor. Thus, we also tried to use PVA/H₃PO₄ gel electrolyte to fabricate a quasi-solid state supercapacitor. As shown in Fig. 4c, the CV curves became more distorted at high scan rates compared with that in liquid electrolyte, which can be ascribed to the lower conductivity of the gel electrolyte. Nevertheless, the quasi-solid state supercapacitor exhibited comparable performance at low scan rates (1.95 mF cm⁻¹ at 5 mV s⁻¹). It also showed good flexibility with ~10% of capacity decay as it was bent to 180° (Fig. 4d).

Fig. 4 (a) CV curves at 5 mV s⁻¹ and (b) charge-discharge curves at 20 μA cm⁻¹ of yarn supercapacitors with 2 and 4 SS wires in 1.0 M Li₂SO₄ aqueous electrolyte. CV curves of yarn supercapacitor at (c) different scan rates, and (d) different bending angles at 5 mV s⁻¹ in PVA/H₃PO₄ gel electrolyte.

4. Conclusion

We introduce braiding technology to arrange two fibre electrodes for fabrication of a yarn supercapacitor without separator. PPy was electrodeposited as the electroactive material. The fabricated yarn supercapacitor delivers a length specific capacitance of 1.71 mF cm^{-1} at 5 mV s^{-1} , and 1.79 mF cm^{-1} at $5 \text{ } \mu\text{A cm}^{-1}$ in $1.0 \text{ M Li}_2\text{SO}_4$ aqueous solution. It can be bent to 180° with a 13% loss in capacitance and retained 75% of its initial capacitance after 1000 cycles of bending-relaxation at 90° . Unlike other yarn supercapacitors which need to be connected in parallel to increase the output energy, our braiding technology can embed more fibre electrodes in the same yarn to meet the requirement. Our strategy provides a new direction for large-scale manufacturing of yarn supercapacitors.

Conflict of interest

There is no conflict of interest.

Acknowledgements

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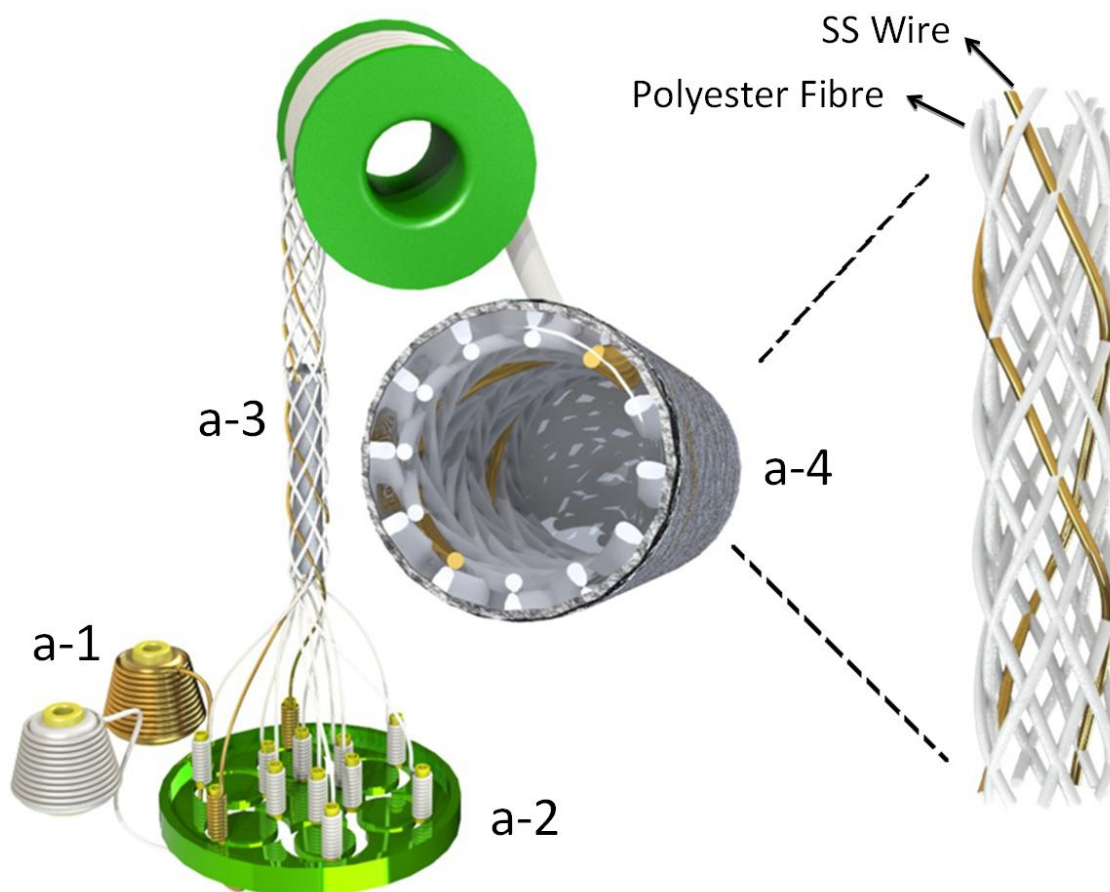


Figure 1

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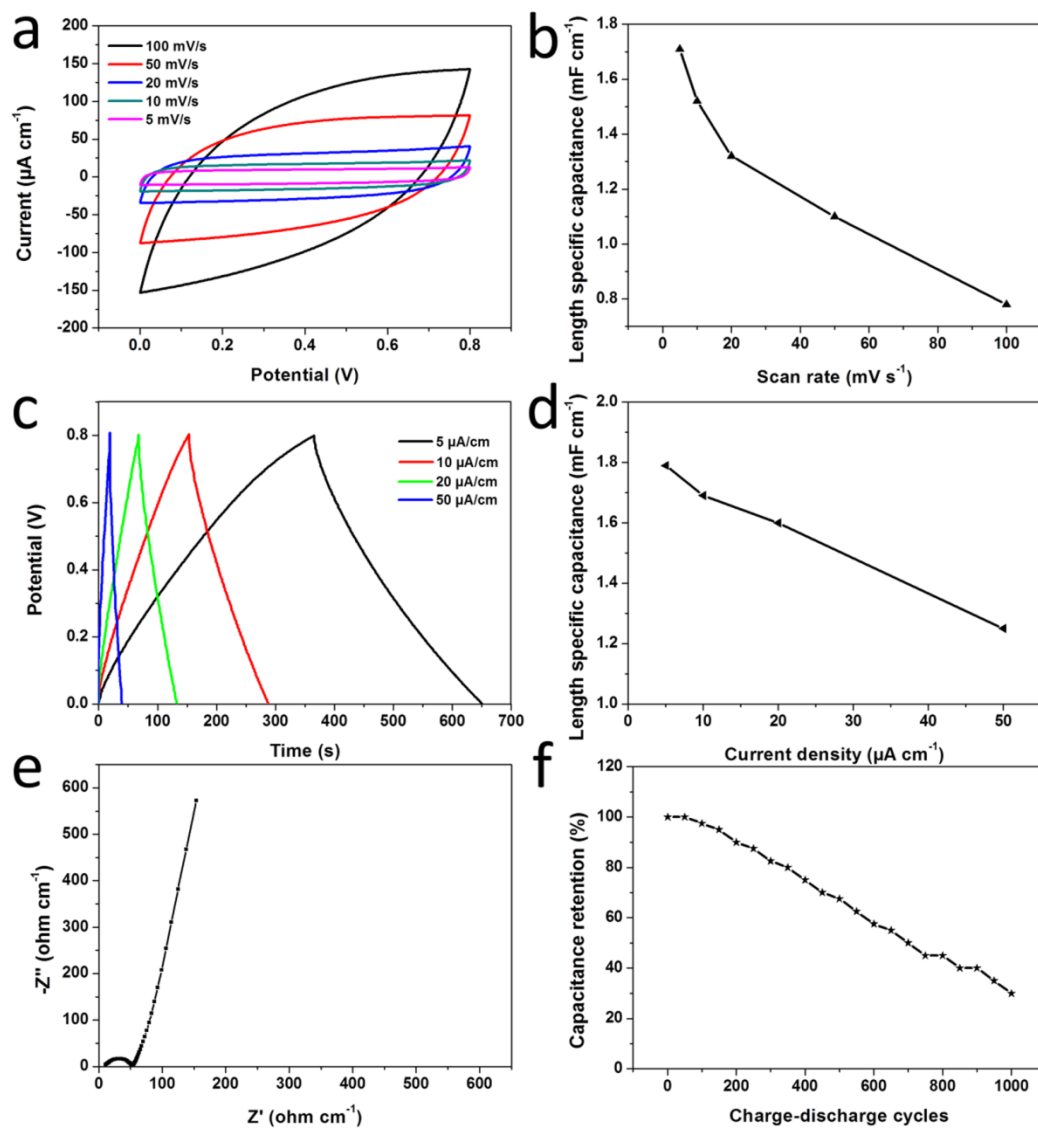


Figure 2

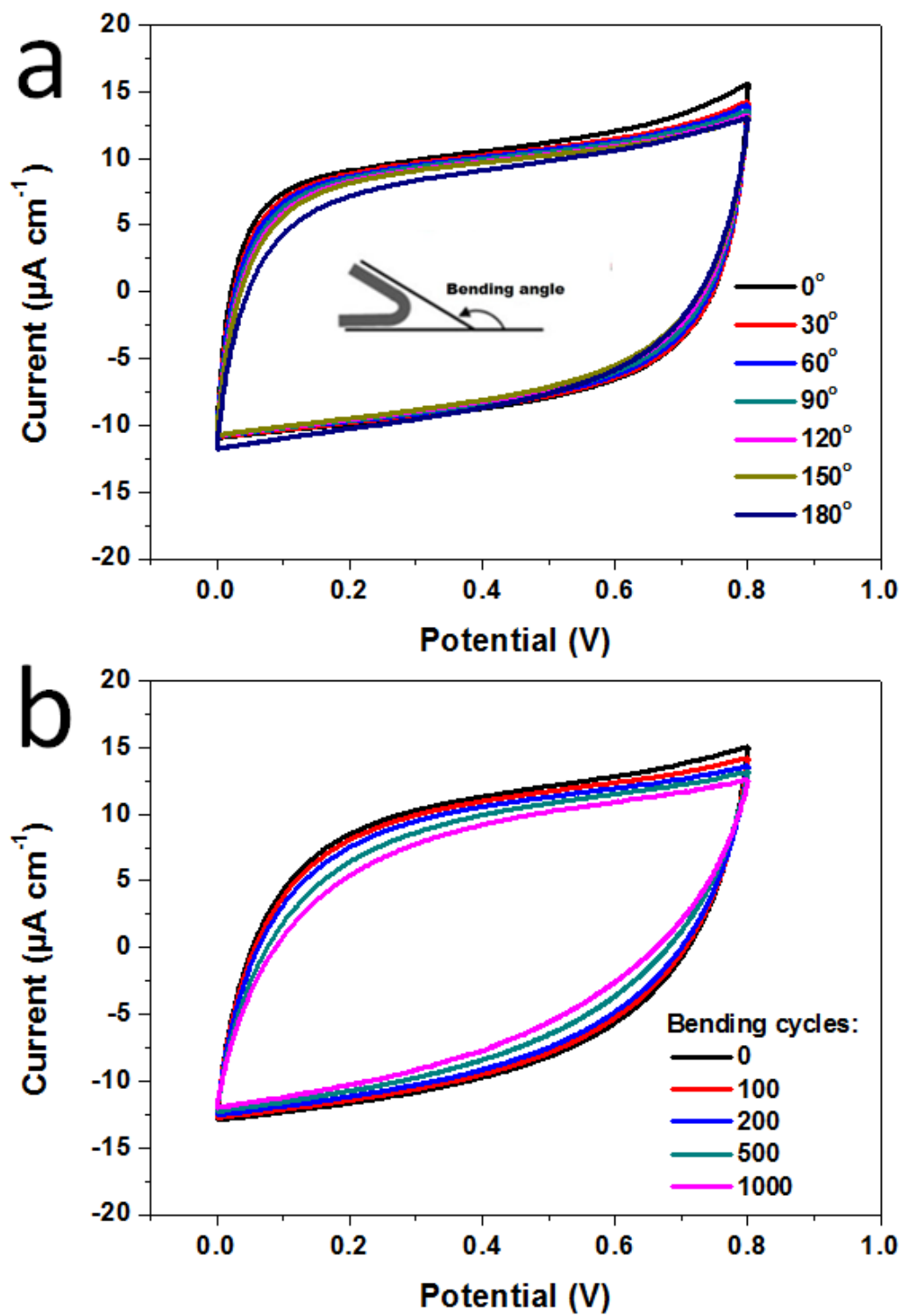


Figure 3

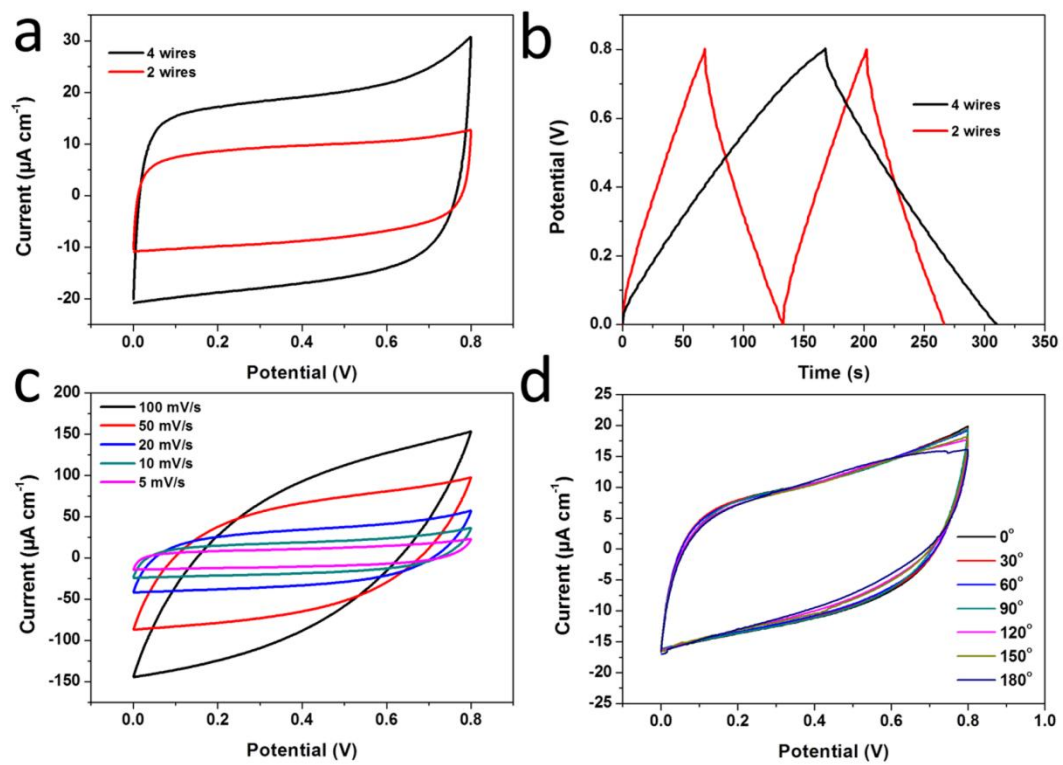


Figure 4

Highlights

- First demonstration of braiding technology in yarn supercapacitor fabrication.
- Easy integration of multiple fibre electrodes in one yarn for high capacitance.
- The yarn supercapacitor demonstrates a high capacitance of 1.71 mF cm^{-1} .
- A widely used industry technology makes sale up feasible.
- A new direction for large-scale manufacturing of electrochemical devices.

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