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Abstract

Wearable electronics offer the combined advantages of both electronics and fabrics. Being an indispensable part of these electronics, lightweight, stretchable and wearable power sources are strongly demanded. Here we describe a daily-used nylon lycra fabric coated with polypyrrole as electrode for stretchable supercapacitors. Polypyrrole was synthesized on the fabric via a simple chemical polymerization process with ammonium persulfate (APS) as an oxidant and naphthalene-2,6-disulfonic acid disodium salt (Na₂NDS) as a dopant. This material was characterized with FESEM, FTIR, tensile stress, and studied as a supercapacitor electrode in 1.0 M NaCl. This conductive textile could endure 1000 stretching cycles with 100% strain applied, and still retained its electrical conductivity and electrochemical properties. Interestingly, we also found that this material showed improved electrochemical properties when it was being stretched.

Keywords

supercapacitor, electrode, stretchable, applications, fabric, polypyrrole, lycra, nylon, coated

Disciplines

Engineering | Physical Sciences and Mathematics

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Polypyrrole coated Nylon Lycra fabric as stretchable electrode for supercapacitor applications

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ABSTRACT

Wearable electronics offer the combined advantages of both electronics and fabrics. Being an indispensable part of these electronics, lightweight, stretchable and wearable power sources are strongly demanded. Here we describe a daily-used Nylon Lycra fabric coated with polypyrrole as electrode for stretchable supercapacitors. Polypyrrole was synthesized on the fabric via a simple chemical polymerization process with ammonium persulfate (APS) as oxidant and naphthalene-2, 6-disulfonic acid disodium salt (Na_2NDS) as dopant. This material was characterized with FESEM, FTIR, tensile stress, and studied as a supercapacitor electrode in 1.0 M NaCl. This conductive textile could endure 1000 stretching cycles with 100% strain applied, and still retained its electrical conductivity and electrochemical properties. Interestingly, we also found that this material showed improved electrochemical properties when it was being stretched.

KEYWORDS: Polypyrrole, e-textile, supercapacitors, stretchable electrode.

1. Introduction

The incorporation of electronics into wearable items has demonstrated significant advances in terms of miniaturisation, functionality and comfort. These e-textiles have found broad applications in continuous personal health monitoring, high performance sportswear, wearable displays and a new class of portable devices [1-5]. Being an indispensable part of these applications, lightweight, stretchable and wearable power sources including batteries and supercapacitors are strongly demanded. The ideal wearable power sources would be made into breathable textile formats with stretchability (i.e. mechanical resilience) being conformal to the curved surface and sustain its function during the body movement.

As one type of power sources, supercapacitors possess the advantages of higher power densities, excellent reversibility and long cycle life [6]. Recently, there has been an

emerging interest in stretchable supercapacitors [7, 8]. Stretchable supercapacitor electrodes using single-walled carbon nanotubes (SWNT) integrated into poly (dimethylsiloxane) (PDMS) [7] or textile substrates [8] have been developed. Buckle-structured stretchable carbon nanotube macrofilms on PDMS exhibited a much similar specific capacitance with or without 30% strain applied (54 F g^{-1} to 52 F g^{-1} at a current density of 1 A g^{-1}). The specific capacitances did not change, even up to 1000 charge–discharge cycles [7]. The stretchable textile-based supercapacitor was fabricated by integrating SWNT into fabric via a “dipping and drying” process [8]. It exhibited a specific capacitance of 62 F g^{-1} at a current density of 1 mA cm^{-2} , and no obvious capacitance fade was observed after being subjected to 100 elongation cycles with 120% strain applied.

Inherently conducting polymers (ICPs) is one important member of the supercapacitor materials family. However, to the best of the authors’ knowledge, the application of ICPs coated fabric in stretchable supercapacitors has not been reported, though recently we have reported the stretchable buckle-structured polypyrrole integrated into a SIBS substrate for Mg batteries [9]. In particular, ICPs are interesting candidates for flexible energy storage devices due to their relatively high theoretical capacities, fast redox properties, lightweight and mechanical flexibility [10-14]. A flexible ultrafast all-polymer battery composed of PPy coated cellulose fibres electrodes could be charged with currents as high as 600 mA cm^{-2} with only 6% capacity loss over 100 subsequent charge and discharge cycles [15]. Flexible supercapacitor with polypyrrole/manganese dioxide composites coated on polypropylene fibrous films as electrodes delivered an optimal capacitance of $\sim 110 \text{ F g}^{-1}$ [16]. These two types of electrodes, PPy or PPy/MnO₂ coated fibre, were flexible but not stretchable at least in the reported structure. Moreover, ICPs are well-suited for the fabrication of conductive textiles [3, 17, 18], and ICPs coated textiles have been studied in energy storage [19-21].

Here we report a stretchable supercapacitor electrode material based on polypyrrole (PPy) coated Nylon Lycra fabric via chemical polymerization. We chose stretchable Nylon Lycra fabric as substrate due to its stretchability and wearing comfort. Although in our group J. Wu *et al.* have reported some preliminary results about the resistance change of PPy coated Lycra fabric under strain of up to 60% [22], this resistance change during or after the elongation-relaxation cycles was not characterized. Also its electrochemical properties were not investigated. In this work, PPy coated Lycra fabric electrode demonstrated excellent stretchability. It could endure 1000 stretching cycles whilst retaining its electrical conductivity and electrochemical properties. Compared with the as-synthesized conductive fabric, the fabric after being subjected to 1000 stretching cycles with 100% strain applied delivered a nearly unchanged specific capacitance. In addition, the electrochemical performance was improved while this PPy coated fabric was being stretched.

2. Experimental section

2.1 Reagents and materials

Pyrrole, naphthalene-2, 6-disulfonic acid disodium salt (Na₂NDS) and ammonium persulfate (APS) were obtained from Sigma-Aldrich. Pyrrole was freshly distilled, while all other chemicals were used as-supplied. All aqueous solutions were prepared with Milli-Q water (~18 MΩ). The textile used was a stretchable knitted fabric with 80% Nylon and 20% Lycra purchased from Spotlight Stores Pty Ltd.

2.2 PPy polymerization on fabric

Before polymerization, Nylon Lycra fabric was washed with an aqueous solution of scouring agent and sodium hydroxide for 1 h to remove grease and other impurities attached on them, followed by soaking and rinsing with H₂O till neutral pH. Deposition of pyrrole on textiles has been carried out by oxidation of pyrrole in the presence of textiles, following the

method described in the previous reports [23, 24]. Briefly, the fabric samples (2.08g) were immersed in APS and Na₂NDS solution and kept at 4°C for 30 minutes, followed by adding pyrrole solution dropwise to initiate polymerization. The polymerization reaction was performed at 4°C for 2 hours. The coated fabrics were then washed with water and dried at ambient temperature. The concentration of monomer, oxidant and dopant was 0.02M, 0.02M and 0.009M, respectively. The textile-liquor ratio was kept at 1:50 (w/v, g: ml). The amount of PPy deposited on the fabric was determined by the weight increase of the fabric after coating. The content of PPy in such composite was about 6.36%.

2.3 Characterization of PPy coated fabric

The surface morphologies of the conductive fabric were investigated by means of a cold-field-gun field emission microscope (FESEM, JEOL JSM7500FA). FT-IR spectra were recorded on a FT-IR spectrometer (IRpretige-21, SHIMADZU) over the range from 800 cm⁻¹ to 2000 cm⁻¹. The surface resistance was determined according to the American Association of Textile Chemists and Colorists Test Method 76-1995. Two rectangular copper electrodes were placed on the fabric sample and the surface resistance (R_s) was given by:

$$R_s (\Omega/\square) = \frac{W}{D} R'$$

Where, R' is the resistance measured by the Multimeter, and W and D are the width of the sample and the distance between the two copper electrodes, respectively.

2.4 Tensile test

Stretching tests were conducted using a Shimadzu EZ mechanical tester. Substrate size was 50 mm (length) × 10 mm (width). The fabric was elongated / released at a controlled speed of 3.3% s⁻¹. The concomitant resistance was measured by an Agilent 34410A digital multimeter during the stretching cycles.

2.5 Electrochemical properties of PPy coated fabric

Cyclic voltammetry and electrochemical impedance spectroscopy were carried out in a standard three-electrode system using a Pt mesh counter electrode, a Ag/AgCl (3M NaCl) reference electrode, and a PPy coated Nylon Lycra fabric attached onto reticulated vitreous carbon (RVC) as working electrode. The electrolyte was 1.0 M NaCl. Cyclic voltammetry was performed using a Solartron SI 1287 and scanned between -0.4 V to 0.5 V. Electrochemical impedance spectra were measured potentiostatically using a PCI4/750 Potentiostat/ Galvanostat/ ZRA (Gamry Instruments, Inc. USA). The frequency range was from 100 kHz to 0.1 Hz with an AC perturbation of 10 mV at open circuit potential.

Galvanostatic charge-discharge cycling was carried out in 1.0 M NaCl using a battery-testing device (Neware Electronic Co., China). PPy coated Nylon Lycra fabric was attached onto RVC and used as electrodes. The cells were charged galvanostatically at 1.0 A g⁻¹ to 0.85 V, and then discharged using the same current to a cut-off voltage of 0 V.

3. Results and discussions

3.1 Surface morphology

The surface morphology of the blank and PPy coated Nylon Lycra fabrics are shown in Fig. 1. After chemical polymerization, PPy was successfully coated on each individual fibre. Some loosely attached aggregates were also observed (Fig. 1b and c). These aggregates could withstand vigorous washing indicative of their strong adherence to the PPy coating layer. The PPy film exhibited a rough surface composed of PPy nodules in different sizes at high magnification (Fig. 1d).

3.2 Infrared spectroscopy

Fig. 2 shows the FTIR spectra of uncoated Nylon Lycra fabric and PPy coated fabric. The spectrum of the coated fabric after being stretched for 1000 cycles with 100% strain

applied is also shown. The spectrum of uncoated Nylon Lycra demonstrated a stable nylon crystalline structure (Fig. 2 (i)) [25]. Bands at 1640 cm^{-1} and 1547 cm^{-1} could be attributed to C=O stretching vibrations (Amide I band) and N-H bending vibrations (Amide II band) [26, 27], respectively. The spectra of the coated fabric before and after stretching were characterized by typical features of PPy [28, 29]. Those Amide bands attributed to Nylon structure could not be observed, indicating that the fabric fibres were completely coated with a layer of PPy [30]. The band at 1539 cm^{-1} and 1454 cm^{-1} corresponded to the C-C and C-N stretching vibrations in the pyrrole ring, respectively. The broad band from 1400 to 1250 cm^{-1} was attributed to C-H or C-N in-plane deformation modes and had a maximum at around 1300 cm^{-1} . In the region from 1250 to 1100 cm^{-1} corresponding to the breathing vibration of the pyrrole ring, the peak was situated at around 1155 cm^{-1} . The peak at 1083 cm^{-1} was corresponded to the mode of in-plane deformation vibration of NH_2^+ formed on the PPy chains by protonation. The band of C-H and N-H in plane ring deformation vibration was situated at 1026 cm^{-1} . The peaks at 964 cm^{-1} and 920 cm^{-1} could be attributed to C-C out of plane ring deformation vibration and C-H out of plane deformation vibration of the ring, respectively [29].

3.3 Tensile test

Such PPy coated Nylon Lycra exhibited a surface resistance of $149\ \Omega/\square$. The electrical resistance of this conductive fabric was investigated with elongations of 25%, 50% and 100% applied at a rate of $3.3\% \text{ s}^{-1}$ at wale direction. Cyclic resistance changes were observed during the mechanical elongation-relaxation process (Fig. 3). The electrical resistance decreased during the elongation process and increased during the relaxation process with 25% strain applied (Fig. 3a). This could be attributed to the better surface-surface contacts within yarns on stretching, which made the polymer penetrate into the yarn and improved the conductivity [31]. It is well-recognized that the contact resistance at the

overlapped points which decreased with loading governed the resistance change of the knitted fabric, for the contacting resistance was much higher than the filament resistance itself [32, 33]. Interestingly, in our work, the electrical resistance appeared in a double-peak phenomenon under the high strain of 50% and 100%. When the fabric was stretched, the resistance first slightly increased and then decreased forming a peak. This phenomenon also occurred during the strain release process forming the second peak, and it has also been reported by P. Calvert *et al.* [34]. The formation of this double peak might be attributed to the slow strain recovery of the fabric at large elongation [34], which is consistent with the hysteresis between the elongation and relaxation induced in the strain stress curves (Fig. 3d, 3e and 3f). Higher hysteresis was introduced with higher applied elongation. Our results are different from the previous results reported by J. Wu *et al.* [22]. In that report the resistance of PPy coated Nylon Lycra fabric decreased significantly at low strains (up to 20%), followed by slow decrease until a nearly steady state (60% strain) was attained. These differences might be explained by that PPy used in these two investigations were synthesized under different conditions (e.g., different oxidant, different dopant and different reaction temperature) leading to different electrical properties.

The surface resistance of PPy coated conductive fabric was irreversibly increased after being subjected to elongation-relaxation cycles. Higher irreversible resistance was induced with the higher strain applied. After 1000 cycles of stretching, the resistance was increased by 15%, 32% and 66% with 25%, 50% and 100% elongation applied, respectively. This can be explained by the large elongations resulting in severe damage to the PPy film, leading to lower conductivity. Nevertheless, PPy on Nylon Lycra still sustained its conductivity under such high elongation of 100%. It was much higher than 10%, the commonly recognized mechanical strain at which PPy films can endure and still retain their conductivity [35]. This high stretchability might be due to its homogenous distribution as

nanoparticles on the fabric, which was in agreement with the result obtained with PEDOT-PSS on Spandex fabric by Y. Ding *et al.* [36].

3.4 Electrochemical properties

Cyclic voltammetry (CV) was used to determine the electrochemical properties of the PPy coated Nylon Lycra fabric. Fig. 4a shows the CV curves of PPy coated Nylon Lycra fabric attached onto RVC in 1.0 M NaCl at a scan rate of 10 mV s⁻¹. The blank RVC was also investigated over the same potential range for comparison, and it exhibited a negligible response. PPy exhibited a nearly rectangular CV behaviour which indicated that the charge-discharge responses of the electric double layer were highly reversible and kinetically facile [37]. However, the rectangular CV shape became distorted when the scan rate reached 25 mV s⁻¹ and above (Fig. 4b). This might be explained by the entering into/ejecting and diffusion of counter ions being too slow compared to the transfer of electrons in the PPy matrix at high scan rates [38].

The specific capacitance of PPy coated Nylon Lycra was calculated using the following equation [39]:

$$C = \frac{A/2}{f \times v \times m}$$

Where C is the specific capacitance, A is the integral area of the cyclic voltammogram loop, f is the scan rate, v is the voltage window, and m is the mass of electroactive material (PPy). It delivered a specific capacitance of 123.3, 100.7, 69.7 and 39.4 F g⁻¹ at a scan rate of 10, 25, 50 and 100 mV s⁻¹, respectively. PPy coated fabric, after being stretched to 100% for 1000 cycles, exhibited a much similar electrical response during the cyclic sweeps (not shown). Its specific capacitance slightly decreased by less than 10% to 113.9, 92.9, 67.8 and 38.2 F g⁻¹ at scan rates of 10, 25, 50 and 100 mV s⁻¹, respectively (Fig. 5). This is further

proof that this conductive fabric could endure such large mechanical stretching and still sustain its electrical and electrochemical properties.

PPy coated Nylon Lycra fabric was also investigated with *in situ* strain applied in the electrolyte of 1.0 M NaCl, and the corresponding cyclic voltammograms at a scan rate of 50 mV s⁻¹ are shown in Fig. 6a. It should be pointed out that the fabric used for this investigation was stretched from its initial or as-synthesized state only, no elongation-relaxation cycle was applied during the measurement. They exhibit a distorted CV shape with no strain applied but with increasing applied strain the electrical response was gradually enhanced; suggestive of promoted electronic transport. A nearly ideal rectangular shaped cyclic voltammogram (CV) was observed when 60% of strain was applied, indicative of highly reversible and kinetically facile charge-discharge responses of the electric double layer. This might be due to the easy accessibility of ions at the electrolyte-electrode interface and the increased electrical conductivity of PPy coated fabric induced by the applied strain. PPy coated Nylon Lycra was also investigated with strain at a scan rate of 100 mV s⁻¹, and similar phenomenon was observed (not shown). The specific capacitance of PPy coated fabric with different strains applied was calculated and shown in Fig. 6b. The capacitance increased sharply with increasing strain, up to 40% elongation. It delivered a specific capacitance of 69.7 and 39.4 F g⁻¹ with 0% elongation at a scan rate of 50 and 100 mV s⁻¹, respectively. Much higher capacitance of about 101.9 and 88.2 F g⁻¹ could be achieved when a 60% strain was applied. Moreover, only a 28% capacitance loss was observed when the scan rate reached to 100 mV s⁻¹ from 10 mV s⁻¹ (Fig. 6b inset), compared to 68% loss with 0% elongation (Fig. 4b). The capacitance obtained at 50 mV s⁻¹ with 60% strain applied might be comparable to or lower than that of 235.35 F g⁻¹ reported for PPy coated activated carbon at a scan rate of 20 mV s⁻¹ [40]. In their work a conductive stainless steel current collector was used and PPy was electrochemically synthesized.

Fig. 7 shows the Nyquist plots of PPy coated Nylon Lycra at an equilibrium open circuit potential with or without strain applied. At the high frequency region, the diameter of compressed semicircle afforded the charge-transfer resistance (R_{ct}) in the electrochemical system [41], which decreased with increasing applied strain. The lower electrical impedance was indicative of the improved conductivity. This result was consistent with the improved charge/discharge responses shown in cyclic voltammograms for PPy coated Nylon Lycra.

The charge/discharge profiles of the symmetric supercapacitors composed of PPy coated Nylon Lycra fabrics electrodes at a current density of 1.0 A g^{-1} with different strain applied are presented in Fig. 8. As can be seen, the $E-t$ responses did not follow a linear relationship (Fig. 8a). This might be due to the pseudo-capacitance contributed from the surface-confined Faradaic reaction of PPy. It is also noticed that the duration of one full charge/discharge cycle became longer, indicative of higher capacitance, with increasing strain applied (up to 60%). These results are consistent with those obtained with CV. The discharge capacitance per electrode can be calculated from the following equation [42]:

$$C_m = \frac{2Q}{\Delta V \times m} = \frac{2I \times t}{\Delta V \times m}$$

Where C_m , I , t , ΔV and m are the discharge capacitance per electrode, the current of charge–discharge, time of discharge, charge/discharge potential windows and the amount of active materials on one electrode, respectively.

The specific capacitance obtained for PPy coated fabric without strain applied was 108.5 F g^{-1} , with an energy density of 6.7 Wh kg^{-1} and power density of 753.4 W kg^{-1} . The capacitance increased to 117.6, 119.6 and 125.1 F g^{-1} with an elongation of 20%, 40% and 60%, respectively. The corresponding energy density increased to 8.7, 9.4 and 11.1 Wh kg^{-1} , and power density increased to 763.0, 765.1 and 799.2 W kg^{-1} with 20%, 40% and 60% strain applied, respectively. PPy coated Nylon Lycra fabric with strain applied also exhibited an

improved cycling stability (Fig. 8b). The capacitance decreased severely to 13.6 F g^{-1} after 500 charge/discharge cycles, only $\sim 12.5\%$ of the initial capacitance. The retained capacitance was 52.9, 63.4 and 68.8 F g^{-1} , about 45%, 53% and 55% of the initial capacitance with 20%, 40% and 60% strain applied, respectively. PPy coated Nylon Lycra fabric delivered a much higher capacitance than stretchable SWCNT integrated into PDMS electrodes (54 F g^{-1} at 1 A g^{-1}) [7] or fabric electrodes (62 F g^{-1} at 1 mA cm^{-2}) [8] but with poorer cycling stability. The capacitance reported here is poorer than PPy coated unwoven ceramic fabrics (208.6 F g^{-1} at 1 mA cm^{-2}) [43]. The different substrate and different PPy synthesis condition might lead to this difference. It also means that the electrochemical properties of this stretchable PPy coated Lycra electrode can be improved by optimizing or changing the synthesis parameters.

4. Conclusion

In this study, we reported the use of a daily-used Nylon Lycra fabric as a platform for stretchable supercapacitor applications. Via chemical polymerization of PPy, a highly conductive textile with a sheet resistance of $149 \text{ } \Omega/\square$ was produced. Such a conductive fabric showed outstanding flexibility and stretchability, and demonstrated strong adhesion between the PPy and the fabric of interest. This fabric exhibited a nearly rectangular CV behaviour indicative of a capacitor property with a specific capacitance of 123.3 F g^{-1} at a scan rate of 10 mV s^{-1} . During stretching at wale direction, the resistance of this PPy coated Nylon Lycra fabric decreased with increasing elongation. After being stretched for 1000 times, the resistance of this conductive fabric was irreversibly increased. Higher irreversible resistance increase was induced with higher strain applied. Nevertheless, PPy coated Nylon Lycra still sustained its electrochemical properties with less than 10% specific capacitance loss after being stretched to 100% for 1000 times. Interestingly, its electrochemical properties could be improved with *in-situ* strain applied and exhibited a higher specific capacitance. The capacitance in a three-electrode system increased from 69.7 and 39.4 F g^{-1} with 0%

elongation to 101.9 and 88.2 F g⁻¹ with 60% strain applied at a scan rate of 50 and 100 mV s⁻¹, respectively. In a two-electrode system at a current density of 1.0 A g⁻¹ the specific capacitance increased from 108.5 F g⁻¹ with 0% strain to 117.6, 119.6 and 125.1 F g⁻¹ with 20%, 40% and 60% elongation, respectively. Also the cycling stability was improved with the applied strain. However, we should point out the cycling stability need to be improved for practical applications. One commonly applied strategy used to overcome this drawback is the formation of composites with carbon-based materials (e.g. carbon nanotubes, graphene etc.). Currently we are working on it.

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Captions

Fig. 1 FESEM images of (a) blank and (b, c and d) PPy coated Nylon Lycra fabric.

Fig. 2 FTIR spectra of blank Nylon Lycra fabric (i), as-synthesized PPy coated Nylon Lycra fabric (ii), and PPy coated Nylon Lycra fabric after being stretched to 100% for 1000 times at wale direction (iii).

Fig. 3 (a, b and c) Normalized electrical resistances under cyclic elongations and (d, e and f) strain-force curves for PPy coated Nylon Lycra fabric at wale direction with different strains applied. Strain applied was (a, d) 25%, (b, e) 50% and (c, f) 100%.

Fig. 4 (a) Cyclic voltammograms of blank RVC (i), and PPy coated Nylon Lycra fabric attached onto RVC in 1.0 M NaCl at a scan rate of 10 mV s⁻¹ (ii). (b) Cyclic voltammograms of PPy coated Nylon Lycra fabric attached onto RVC in 1.0 M NaCl at different scan rates: 10 (i), 25 (ii), 50 (iii) and 100 mV s⁻¹ (iv).

Fig. 5 The specific capacitance of the as-synthesized PPy coated fabric (i), and the fabric after being stretched to 100% for 1000 times (ii) at different scan rates.

Fig. 6 (a) Cyclic voltammograms of PPy coated Nylon Lycra fabric with different strains applied at a scan rate of 50 mV s⁻¹ in 1.0 M NaCl: 0% (i), 20% (ii), 40% (iii) and 60% (iv). (b) Specific capacitance of PPy coated Nylon Lycra fabric obtained at a scan rate of 50 mV s⁻¹ and 100 mV s⁻¹ with different strains applied. Inset: cyclic voltammograms of PPy coated fabric with 60% elongation applied in 1.0 M NaCl at different scan rates: 10 (i), 25 (ii), 50 (iii) and 100 mV s⁻¹ (iv).

Fig. 7 Nyquist plots of PPy coated Nylon Lycra fabric in 1.0 M NaCl with different strains applied.

Fig. 8 (a) Charge/discharge curves of PPy coated fabric with different applied strain: (i) 0%, (ii) 20%, (iii) 40% and (iv) 60% at a current density of 1.0 A g⁻¹. (b) Discharge capacitance

versus cycle number at 1.0 A g^{-1} for PPy coated fabric with different strain applied: (i) 0%, (ii) 20%, (iii) 40% and (iv) 60%.

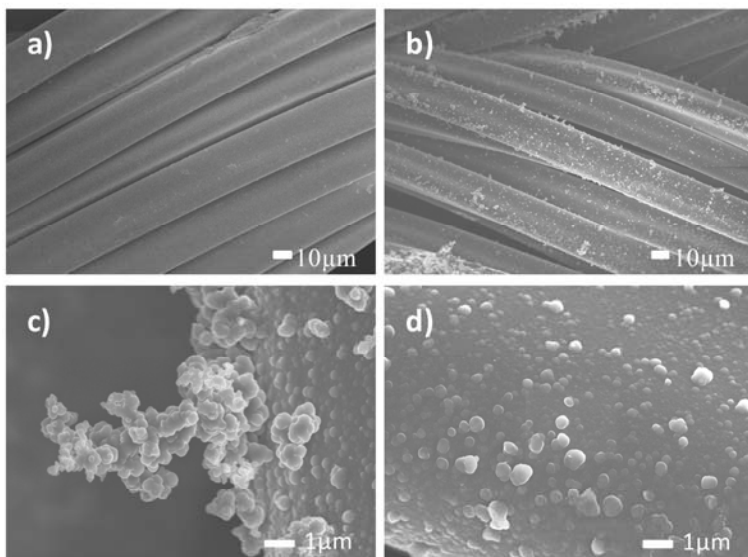


Figure 1

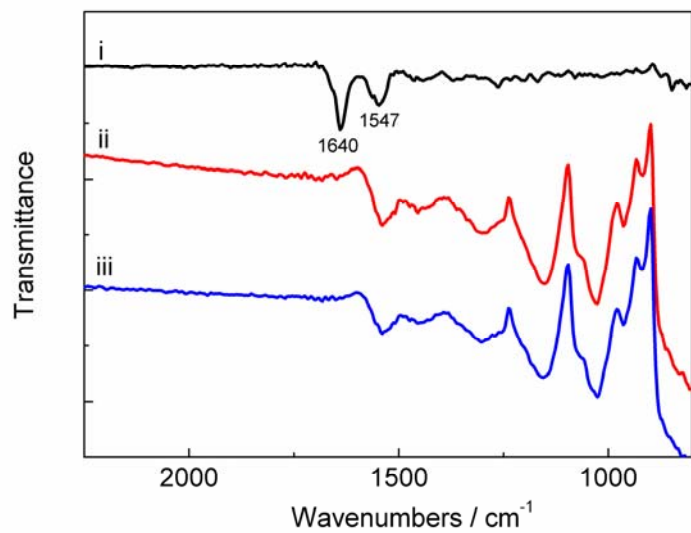


Figure 2

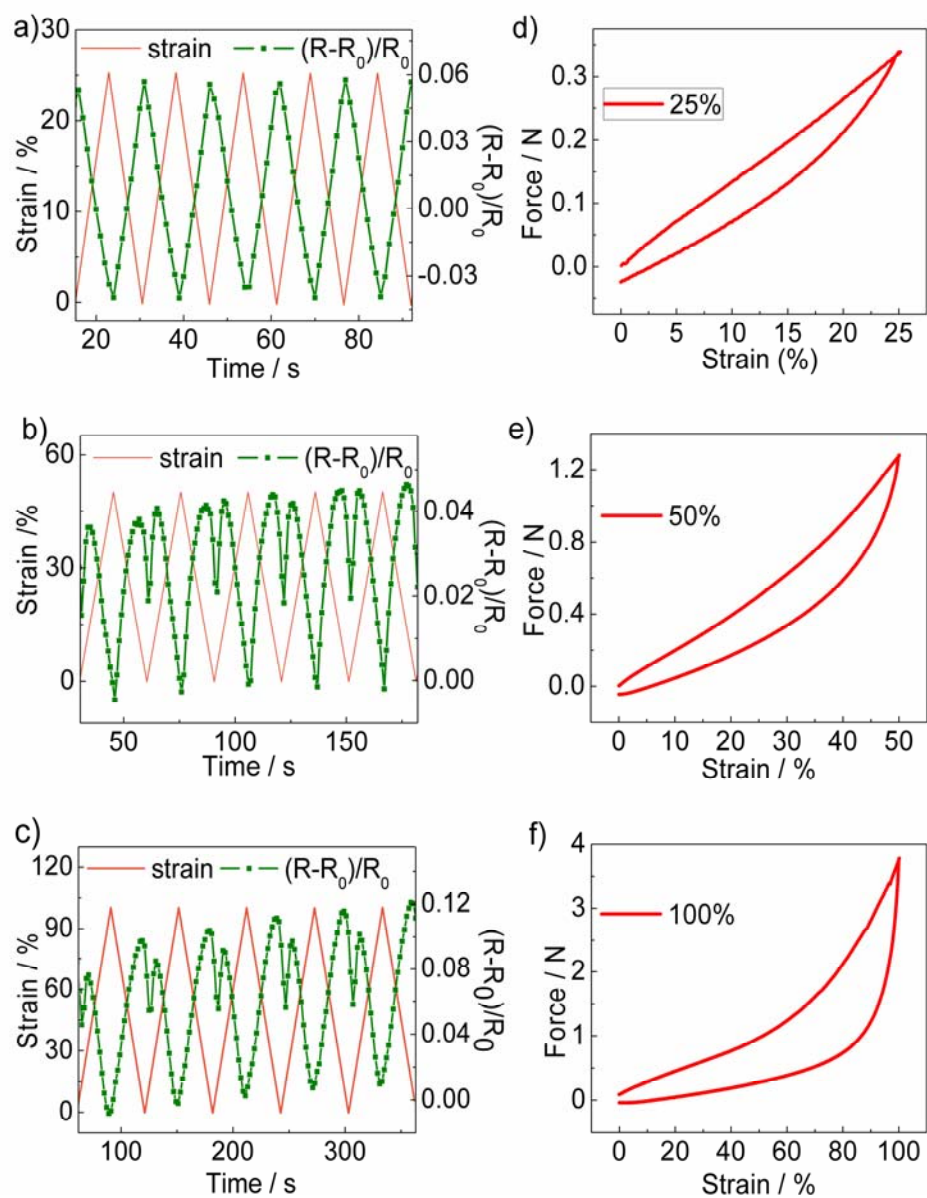


Figure 3

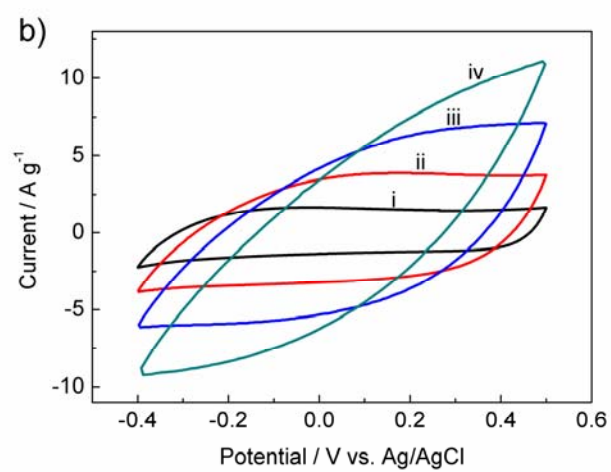
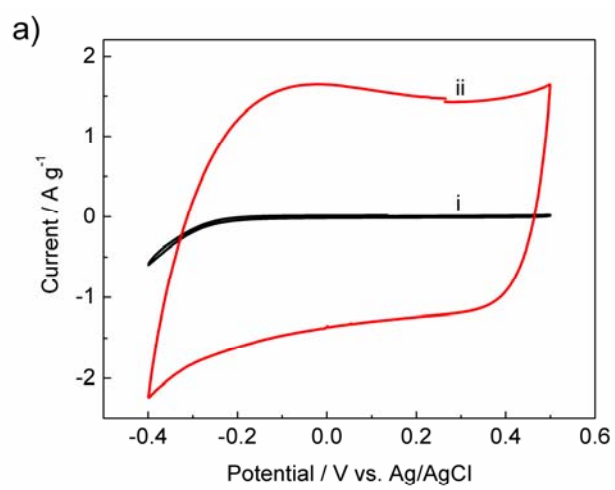


Figure 4

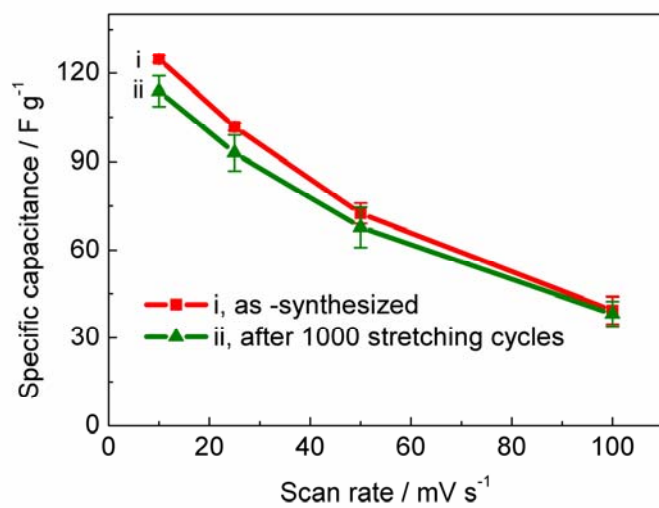


Figure 5

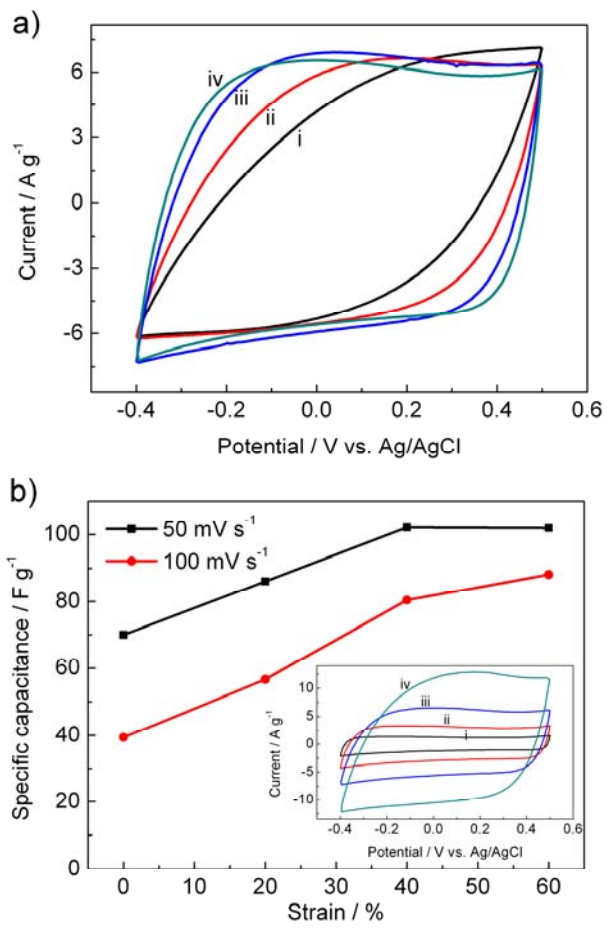


Figure 6

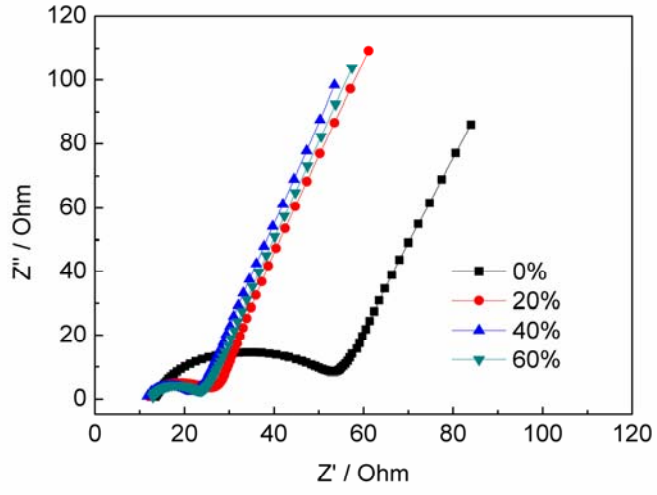


Figure 7

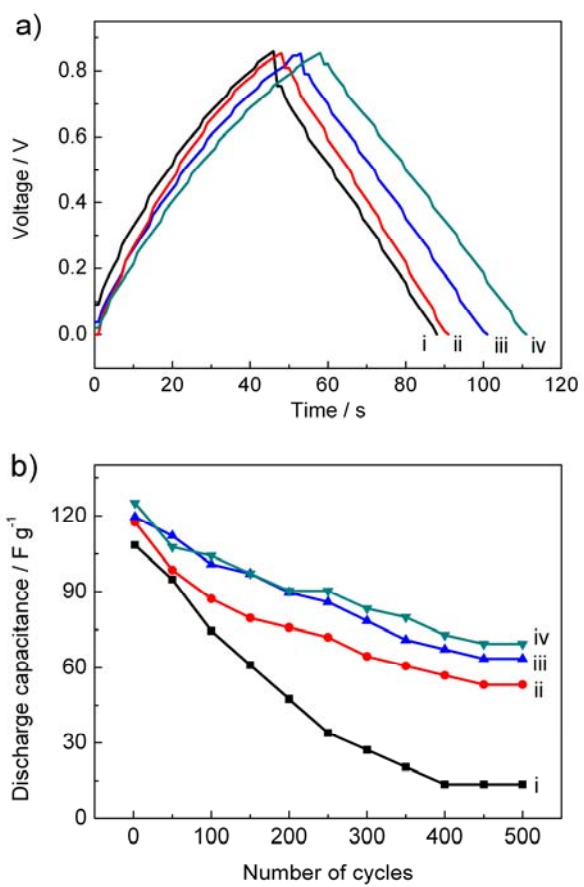


Figure 8