Enhancement of electrochemical performance of textile based supercapacitor using mechanical pre-straining

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ABSTRACT

Recently developed textile and paper based supercapacitors are well-suited for wearable energy storage devices due to their mechanical flexibility and stretchability. In this study, the effects of mechanical straining on carbon nanotube coated textiles are studied, and we demonstrate the pre-straining of the textile can result in an enhanced specific capacitance as well as power and energy densities. In-situ resistance measurement during mechanical straining of the textile capacitors show a decrease in resistance with tensile straining, which contributed directly to the enhancement of electrochemical performance. Two different carbon nanotube textiles based on polyester and cotton with different mechanical behaviors are examined, where the polyester textile show an increase in specific capacitance from 53.6 F g⁻¹ to 85.7 F g⁻¹ after the textile is pre-strained to 30% permanent elongation prior to electrochemical testing, constituting a 37% enhancement. Similarly, the cotton textile shows an enhancement in specific capacitance from 122.1 F g⁻¹ to 142.0 F g⁻¹ after 30% permanent elongation, which is a 22% enhancement. Specific capacitance, energy density, and power density are increased further by electroplating of MnO₂ nanoparticles on the carbon nanotube coated cotton textile and then imposing a permanent elongation of 30%. Our results indicate that the simple mechanical pre-straining of the textile fibers contribute to significant enhancements in the electrochemical performance of the supercapacitors.

1. Introduction

Supercapacitors are receiving much interest due to their high power density and high reliability compared to other rechargeable...
energy storage systems. The power density of a supercapacitor is higher than that of the conventional rechargeable batteries due to fast charging–discharging times between the electrode and electrolyte interface that is characteristic of an electrical double layer capacitor (EDLC). Carbon based materials including active carbon, carbon nanotube (CNT), and graphene with high surface areas are currently being developed as electrode materials for EDLCs. Since only physical adsorption of ions occurs in supercapacitors without any faradic reactions, supercapacitors have longer cycle life than conventional rechargeable batteries. A typical rechargeable battery significantly loses a large portion of its energy efficiency after a few hundred charge–discharge cycles, whereas a supercapacitor maintains its energy efficiency over ~98% of the initial specific capacitance for more than 3000 cycles [1]. However, supercapacitors have a drawback of having a lower energy density (1–10 Wh kg⁻¹) than rechargeable batteries (10–100 Wh kg⁻¹). One way to overcome the low energy density limitation is to incorporate pseudocapacitor materials such as MnO₂, RuO₂, or electrically conducting polymers combined with a carbon based electrode [2–4]. Although the energy density is still lower than the level that could be provided by a battery, the supercapacitors, on the basis of the advantages noted above, are currently being considered as a promising candidate for wearable energy storage devices that could power small devices such as the smart mobile systems, wireless electronics, and electric cars [5,6].

A notable advance in wearable supercapacitors was achieved by Hu et al., who introduced and demonstrated the potential of textile [7] and paper [8] based supercapacitors. CNT coated textile supercapacitors are especially well-suited for wearable energy storage device applications due to their double porous structure, which aids ion diffusion to enhance the specific capacitance compared to the CNT deposited on a flat polyester substrate [7]. Another benefit of using textile based supercapacitor is the inherent stretchability of the underlying textile such that the device can withstand the mechanical motion of the human body when used as a wearable energy storage device. Another advantage of the CNT textile supercapacitor is that the fabrication process is extremely simple where a dip coating process is used to coat the CNTs on the textile since the underlying textile can easily absorb the aqueous based CNT ink [7,8]. Hu et al. also tested the mechanical behavior of textiles and noted that the conductance of the CNT coated textile can be enhanced with tensile loading [7], therefore, supporting the feasibility of this technology for wearable energy device applications that will be subjected to mechanical strains during operation.

In this work, a systematic study of the performance of CNT coated textiles for two different textile choices of polyester and cotton textiles were explored. The two textiles have different mechanical behavior, which could lead to different conditions for reduction in conductance after tensile elongation is imposed. We propose an optimized condition for the pre-straining prior to fabrication of a supercapacitor that is directly linked to the enhancement of specific capacitance, power density, and energy density. Incorporation of MnO₂ pseudocapacitor nanoparticles was also explored together with mechanical pre-straining to further enhance the energy density of the CNT coated textile while maintaining a high power density for optimized electrochemical performance.

2. Experimental

2.1. Sample preparation

CNT coated textile electrodes for electrical double layer (EDLC) supercapacitors were prepared from two different textiles composed of polyester (4Science) and 100% pure cotton. The polyester and cotton textiles have different stretchability that allowed us to study the effect of the mechanical response of the textile on the electrochemical performance. The polyester and cotton textiles were coated with CNTs (Carbon Ins.) by a simple dip coating process by immersing the textile in CNT ink made of 0.032 g of single-walled nanotube (SWNTs), 0.4 g of 4-dodecylbenzenesulfonic acid (SDBS) surfactant (Sigma Aldrich), and 40 ml of deionized water (DI) water. Since strong Van der Waals interactions are present between CNTs and textile fibers, the CNTs are adhered well to the textile during this simple dipping process. The cotton textile also reported to have a strong hydrogen bonding to the CNTs to yield a stronger adhesion. The prepared CNT ink was then sonicated for 1 h to attain an even suspension prior to dip coating, and the textile was dipped in CNT ink to deposit 0.065 mg cm⁻² that prevented excess loadings of the solvent molecules. Mass of the coated CNTs was recorded using an electronic scale with accuracy down to 10⁻⁶ g. After the dip coating process, the sample was dried at 120°C for 10 min in a box furnace to evaporate the remaining solvents.

In order to enhance the energy density of the CNT coated textiles, MnO₂ nanoparticles were coated using electrochemical oxidation driven by a three electrode electroplating system that consisted of the CNT-polyester or the CNT-cotton electrode as the working electrode, Pt wire (Fisher Scientific) as the counter electrode, and Ag/AgCl (Fisher Scientific) as the reference electrode. Current and voltage controls were provided by a potential static system (VMP3-BioLogic Science Ins). MnO₂ nanoparticles were deposited on to the CNT coated textiles by an oxidation reaction of Mn from a solution with a concentration of 0.02 M Mn(NO₃)₂ (Sigma Aldrich) and 0.1 M NaNO₃ (Sigma Aldrich) at a constant current of 0.5 mA cm⁻² for 1 h by utilizing a potential static equipment. The SEM image in Fig. 1 shows that MnO₂ nanoparticles with an average size of 300 nm were electroplated on to the CNT coated textile.

![Fig. 1. SEM images of MnO₂ nanoparticles deposited by three-electrode electroplating on to the CNT-cotton textile.](image-url)
2.2. Electrochemical measurement

Electrochemical properties were measured by using a potential static system in a coin cell testing setup to measure the specific capacitance (F g⁻¹). The anode and the cathode consisted of the same CNT coated textiles, which are separated by an insulating polymer textile layer. A Na based electrolyte was used in this study with a concentration of 1 M Na₂SO₄ 8.06 g (Sigma Aldrich) solution in DI water (60 ml). Electrochemical performance was measured using both the charge–discharge and the cyclic voltammetry (C–V) methods. The charge–discharge measurement was performed in a voltage range of 0 to 0.85 V, as is common for water based electrolytes, at different current densities of 0.2 A g⁻¹, 1 A g⁻¹, 2 A g⁻¹, 4 A g⁻¹, and 8 A g⁻¹. Specific capacitance \( C_s \) was then calculated from the measured charge–discharge curve using the following equation for a symmetric measurement system [9].

\[
C_s = 2I/(\Delta V/\Delta t)
\]  

where \( \Delta V \) is the working voltage, \( \Delta t \) is change of time and \( I \) is current density that was normalized by the weight of the active materials. C–V results were also obtained at varying voltage scan rates of 100 mV s⁻¹, 50 mV s⁻¹, and 20 mV s⁻¹ to examine the enhancement of specific capacitance across the samples. In addition, electrochemical impedance spectroscopy measurements were carried out using a sinusoidal voltage input with a frequency in the range of 10 kHz to 10 mHz with a voltage amplitude of 10 mV. The equivalent series resistance (ESR) of electrodes was then determined by the real part of the impedance and was then used to calculate the energy and power densities by using the following equations:

\[
E = 1/2C_e(\Delta V)^2
\]  
\[
P = (\Delta V)^2/4m(ESR)
\]

where \( \Delta V \) is the working voltage, \( C_e \) is the total capacitance of the entire device, \( m \) is the total mass of the total device, and ESR is the total resistance. Total capacitance of the entire device is expressed as

\[
C_T = I/(\Delta V/\Delta t)
\]

2.3. Mechanical tensile test and unloading test

A more systematic study to determine the optimized conditions for improved conductance—and therefore specific capacitance—with imposed tensile strain was conducted. In order to determine the change in resistance with tensile loading, a desktop tensile tester (Titron, MTS) was modified to track the resistance in-situ during tensile testing. A Keithley voltmeter (2182A) and a source meter (2400) were used to measure the voltage while current was being supplied to determine the resistance in-situ. The grips were specially designed to allow for proper electrical contact with the textile for resistance measurement while maintaining an even pressure on the specimen, as shown in Fig. 2.

Both the CNT-polyester and the CNT-cotton textiles were tested with an in-situ electro-mechanical tester to determine the conditions for optimized conductance for both types of textiles. Textile specimens with dimensions of 20 mm in width, 60 mm in length, and 0.1 mm in thickness were tested at a constant displacement rate of 2 mm min⁻¹. Normalized load and percent elongation or strain were then determined by:

\[
\text{Normalized load} = P/wt
\]

\[
\text{Percent elongation} = z/L_0
\]

3. Results & discussion

3.1. Electrochemical properties of CNT based electrodes and MnO₂ coated electrodes

The CNT-polyester and the CNT-cotton textiles without any mechanical straining were first evaluated for their electrochemical properties, and the cyclic voltammetry and the charge–discharge results are shown in Fig. 3. The current density (A g⁻¹) vs. voltage tested between 0 and 0.85 V at a scan rate of 100 mV s⁻¹ are shown in Fig. 3a that indicate that the MnO₂ coated CNT-cotton specimen has a higher specific capacitance than the CNT-polyester and CNT-cotton textile. The maximum current densities of the CNT-polyester and CNT-cotton were determined to be 4.0 A g⁻¹ and 5.9 A g⁻¹, respectively, and the corresponding area under the C–V curve was larger for the CNT-cotton than for the CNT-polyester textile. The higher specific capacitance and energy density of the CNT-cotton are consistent with the C–V measurements in Fig. 3a. The specific capacitance measured here for the CNT textiles are higher than the values commonly reported for conventional EDLCs and this enhancement is attributed to an easier access of Na ions to the surfaces of CNTs as a result of the underlying porous structure of the textile [10].
Although CNT coated textile can provide high specific capacitance and power density, EDLCs typically suffer from low energy density. While a high specific capacitance has previously been reported with RuO$_2$ (1000 F g$^{-1}$), RuO$_2$ is expensive and harmful to the environment [11,12] while MnO$_2$ is a cost effective and environmentally compatible alternative [13-15]. MnO$_2$ has a theoretical specific capacitance of 1370 F g$^{-1}$ [16], and Mn$^{3+}$ and Mn$^{4+}$ are known to provide a high charge storage. Therefore, the usage of MnO$_2$ pseudocapacitor nanoparticles in conjunction with a CNT coated textile was used in this study to significantly enhance the energy density. MnO$_2$ nanoparticles are known to possess higher specific capacitance than their bulk counterparts due to their larger surface area to volume ratio, and for this reason the MnO$_2$ nanoparticles were deposited onto a CNT-cotton textile by the electroplating process described above to further enhance the specific capacitance and energy density [16-20]. The CNT-cotton textile was chosen over the polyester textile for MnO$_2$ coating since the overall specific capacitance was higher than that of the CNT-polyester due to hydrogen bonding and large Van der Waals interactions [21]. The resulting C–V and the charge–discharge plot for the MnO$_2$ coated CNT-cotton textile is shown in Fig. 3a, b. The C–V curve in Fig. 3a indicates a max in current density of 13.4 A g$^{-1}$, which is significantly higher than that of CNT-cotton electrodes. Therefore, the area under the C–V curve is larger for the case of adding MnO$_2$ nanoparticle.

In order to calculate the specific capacitance and energy density, the charge–discharge tests were performed and the results are shown in Fig. 3b. The time required to charge and discharge with the same working voltage in the range of 0–0.85 V at a current density of 200 mA g$^{-1}$ was measured for both the CNT-cotton and CNT-polyester textiles. From the slope of the charge–discharge plots, the specific capacitance and energy density of the CNT-polyester were calculated to be 53.6 F g$^{-1}$ and 2.7 Wh kg$^{-1}$, respectively, and the corresponding values for the CNT-cotton textiles were calculated to be 122.1 F g$^{-1}$ and 4.8 Wh kg$^{-1}$. With the addition of MnO$_2$ nanoparticles, a smaller slope in the charge–discharge graph was measured that resulted in higher value for specific capacitance and energy density of 282.0 F g$^{-1}$ and 14.2 Wh kg$^{-1}$, respectively. The addition of MnO$_2$ nanoparticles to the CNT-cotton textile, therefore, constituted a 230% enhancement in the specific capacitance compared to that of the CNT-cotton textile electrode without MnO$_2$ addition.

For all three specimens of CNT-polyester, CNT-cotton, and MnO$_2$ coated CNT-cotton, the specific capacitances were also measured at varying current densities of 200 mA g$^{-1}$ to 8 A g$^{-1}$, as shown in Fig. 3c. Across all current densities, the MnO$_2$ coated CNT-cotton textile had the highest specific capacitance, followed by the CNT-cotton electrode and then the CNT-polyester. The specific capacitance was measured to decrease with an increase in current density above 200 mA g$^{-1}$, where the MnO$_2$ coated CNT-cotton, CNT-cotton, and CNT-polyester were measured to have specific capacitances of 282.0 F g$^{-1}$, 122.1 F g$^{-1}$, and 53.6 F g$^{-1}$, respectively.
3.2. Electro-mechanical testing of CNT coated polyester and cotton

Hu et al., in their previous study of a CNT textile supercapacitor, reported that the conductance of CNT textile electrodes can be enhanced by imposing a mechanical tensile strain. The enhanced conductance was attributed to alignment of the CNTs as the underlying fibers stretch in the direction of the tensile loading that could result in more efficient contact between the CNTs that in turn can result in an enhancement in conduction pathways or improved conductance. In order to utilize this enhancement of conductance through mechanical straining, a systematic study was conducted where the mechanical behavior of the CNT coated textiles was tested while collecting the resistance of in-situ.

The results of the in-situ electro-mechanical testing are shown in Fig. 4, where the normalized load and the corresponding resistance measurements are plotted against percent elongation for both CNT-cotton and CNT-polyester textiles. The normalized load vs. percent elongation shown in Fig. 4a indicates that the CNT-polyester can withstand a larger amount of normalized load, where failure occurred at 52.0 MPa at 80% elongation. In comparison, the CNT-cotton textile has a lower mechanical resilience, where failure occurs at a lower normalized load of 22.0 MPa at 140% elongation. The corresponding reduction in normalized resistances for both the CNT-polyester and CNT-cotton textiles are plotted together with the mechanical behavior in Fig. 4a and b. A reduction in resistance with mechanical loading occurred in the early stages of tensile straining, but an increase in resistance was apparent at larger strains due to a reduction of the cross-sectional area of the textile. The degree of reduction in resistance was larger for the CNT-polyester specimen, and a minimum in

Fig. 4. In-situ resistance measurement during mechanical tensile straining of CNT-polyester (CPE) and CNT-cotton (CCT). The normalized load and normalized resistance vs. percent elongation for CPE and CCT are shown in (a, b). The SEM images of CPE and CCT before straining are shown in (c, d) and the SEM images after straining for CPE and CCT are shown in (e, f).
The CNT-polyester specimen in Fig. 5a resulted in permanent elongations was observed at larger strains. For example, at smaller elongation, but the deformation mostly consisted of Fig. 5a and b, respectively. A larger amount of recovery is observed unloadings at elongation displacements of 2 mm, 5 mm, 10 mm, 15 mm, 20 mm, and 25 mm were performed to determine the strain upon unloading.

Tensile tests with intermittent deformation more efficiently without much recovery, intermittent unloadings were performed. Tensile tests with intermittent unloadings at elongation displacements of 2 mm, 5 mm, 10 mm, 15 mm, 20 mm, and 25 mm were performed to determine the amount of permanent strain upon unloading.

The results for CNT-polyester and CNT-cotton are shown in Fig. 5a and b, respectively. A larger amount of recovery is observed at smaller elongation, but the deformation mostly consisted of permanent elongations was observed at larger strains. For example, the CNT-polyester specimen in Fig. 5a resulted in permanent elongation of 30% when unloaded from a total elongation of 47%. A similar amount of permanent elongation in the CNT-cotton textile could be observed when unloaded after a larger amount of total elongation of 42%. Since a larger amount of permanent deformation is required in the CNT-cotton textile to maintain the benefit from the aligned CNTs, the effect of mechanical straining on enhancing the overall performance as an electrical double layer is expected to be more pronounced for the CNT-polyester textile.

Mechanical straining can enhance the conductance of the textile capacitor, which can in turn enhance the specific capacitance, but the tensile strain imposed has to be maintained upon removal of the tensile load for fabrication of the cell. Therefore, the choice of the textile should consider how much the imposed strain can be retained upon removal of the load to sustain the CNT and the fiber alignment. To determine which textile can retain permanent deformation more efficiently without much recovery, intermittent unloading tests were performed. Tensile tests with intermittent unloadings at elongation displacements of 2 mm, 5 mm, 10 mm, 15 mm, 20 mm, and 25 mm were performed to determine the amount of permanent strain upon unloading.

The charge-discharge plot for CNT-polyester before and after tensile straining is shown in Fig. 6a. The CNT-polyester and CNT-cotton electrodes were tested at a working voltage of 1V, and a clear difference in specific capacitance was observed upon application of mechanical pre-straining. The calculated specific capacitance of the CNT-polyester at 200 mA g\(^{-1}\) was increased from 53.6 F g\(^{-1}\) to 85.7 F g\(^{-1}\), an increase of 37%. Similarly, the CNT-cotton showed that tensile pre-straining resulted in the increase in the specific capacitance from 122.1 F g\(^{-1}\) to 142.0 F g\(^{-1}\), an increase of 20%. The degree of enhancement is smaller for that of the CNT-cotton textile compared to that of the CNT-polyester due to the geometry of the pore structure, as explained previously. The two textiles have different three-dimensional fiber network structures, and the tight fiber network of the CNT-polyester electrode resulted in maximized conductance with a smaller imposed strain of 24% in comparison with the CNT-cotton electrode. The conductance of the CNT-cotton electrode was maximized at a large elongation of 140%, and therefore the imposed 30% permanent elongation was not enough to maximize the textile’s conductance, resulting in a smaller degree of enhancement of the specific capacitance. However, it should be noted that the overall specific capacitance is still higher for that of the CNT-cotton, presumably due to the stronger hydrogen bonding and Van der Waals forces between the CNTs and the cotton fibers [21].

A further enhancement of the energy density was demonstrated by using MnO\(_2\) nanoparticles. The MnO\(_2\) coated CNT-cotton textile was mechanically pre-strained and the resulting enhancement of the charge-discharge behavior with mechanical pre-straining is shown in Fig. 6c. The charge-discharge slope of the mechanically strained MnO\(_2\) coated CNT-cotton is lower than that of the other electrodes, and the corresponding specific capacitance of the strained MnO\(_2\) coated CNT-cotton is 351 F g\(^{-1}\), which is higher than that of the other electrodes.

The change in specific capacitance of the textiles before and after pre-straining at different current densities was evaluated, and

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**Fig. 5.** Normalized load vs. percent elongation with unloadings taken at displacements of 5 mm, 10 mm, 15 mm, 20 mm, and 25 mm for (a) CNT-polyester (CPE) and (b) CNT-cotton (CCT) electrodes.
3.4. Impedance and Ragone plot of electrodes

To determine the enhancement of the conductance by applied mechanical strain, the impedance measurements using AC current at a frequency range of 100 kHz to 10 mHz was performed for the CNT-cotton textile with and without MnO₂ nanoparticles, before and after tensile stretching. Fig. 7a shows the Nyquist plots to confirm the resistance change of the pre-stained MnO₂ coated CNT-cotton textile with respect to the unstrained MnO₂ coated CNT-cotton, and the results for the CNT-cotton electrode is also shown as a reference. Equivalent series resistance (ESR) was obtained by determining the real part of the impedance measurements that are calculated using Eqs. (2) and (3), and the results are shown in comparison with those of other electrodes in Fig. 7b. The CNT-polyester initially yielded power density of 0.8 kW kg⁻¹ and energy density of 2.7 Wh kg⁻¹, and an increase to 1.4 kW kg⁻¹ and 4.8 Wh kg⁻¹ was observed with mechanical pre-straining of 30% permanent elongation. CNT-cotton provided the power and energy densities of 6.4 kW kg⁻¹ and 6.1 Wh kg⁻¹, respectively, and these values were improved to 11.4 kW kg⁻¹ and 7.1 Wh kg⁻¹, respectively, after 30% pre-straining was imposed. Therefore, an enhancement in power and energy densities were clearly observed in all samples with mechanical pre-straining. Secondly, the CNT-cotton textiles were measured to have the highest power and energy densities compared to the CNT-polyester specimens, therefore confirming that the choice of the textile is an important factor that determines the electrochemical performance of the textile supercapacitors.

For the case of MnO₂ coated CNT-cotton textile, the energy density was calculated to be 14.2 Wh kg⁻¹ (~200% increase), where as the power density of the MnO₂ coated CNT-cotton electrode was determined to be 4.9 kW kg⁻¹ (~250% decrease). Our results indicate that the energy density is significantly enhanced by the addition of MnO₂, as expected, but the power density is decreased as also indicated by the increased ESR value. MnO₂ coated CNT-cotton textile upon applying mechanical pre-straining of 30% permanent elongation, however, showed a significantly enhanced power density of 14.2 kW kg⁻¹ and the energy density was also enhanced to 17.6 Wh kg⁻¹. The initial reduction in power density with the addition of MnO₂ nanoparticles was all recovered and even higher power density than the initial power density of the CNT-cotton textile without MnO₂ addition was achieved by imposing a permanent elongation of 30%.
The energy and power densities from the CNT-polyester, CNT-cotton, and MnO2 coated CNT-cotton are shown in the Ragone plot in Fig. 7b. Our results are compared here with other reported power and energy densities of other supercapacitors and pseudocapacitors. Energy and power densities of the reduced graphene oxide (RGO) sheets modified with ruthenium oxide (RGO–RuO2) supercapacitor was reported to have energy density of 26.3 Wh kg\(^{-1}\) but has less than 0.2 kW kg\(^{-1}\) of power density [24]. Other examples of supercapacitors that yielded high energy density but a low power density include RuO2–carbon or Ni(OH)\(_2\)–carbon electrode pairs [25], polycarbonate-modified reduced graphene oxide (PL:RGO) electrode [26], and carbon nanoparticles–MnO2 nanorod hybrid structure [27]. Our MnO2 coated CNT textile after pre-straining has a similar level of energy density 17.6 Wh kg\(^{-1}\), but the power density is significantly enhanced to 14.2 kW kg\(^{-1}\). Therefore, the power density and energy density were optimized by the addition of a pseudocapacitor material and applied mechanical pre-strain.

Our studies indicate that the performance of the textile based supercapacitors can be enhanced by a simple mechanical pre-straining procedure that resulted in an enhancement of the power density of more than 200% for the case of MnO2 coated CNT-cotton electrode, arising from the fact that the reduction in resistance occurred as the CNT and textile fibers align in the direction of tensile loading. Textile based supercapacitor when used as wearable energy storage device will experience a large mechanical strain during its operation, and the mechanical behavior analysis in this study suggests that the performance of the textile supercapacitor would be sustained with high reliability. In addition, the conductance increase of the CNT textile by the tensile straining is 26.3 Wh kg\(^{-1}\) for the case of MnO2 coated CNT-cotton textile that was mechanically pre-strained to 30% elongation. By addition of MnO2 nanoparticles, the energy density could be enhanced, but the power density was decreased. However, the mechanical straining resulted in a recovery of the lost power density and beyond such that the power density was higher than that of the CNT-cotton textile with no MnO2 addition. Therefore, the mechanical pre-straining can significantly enhance the electrochemical performance, and thus validating that the textile based supercapacitors are well-suited for wearable energy storage systems that will face mechanical strains during operation.

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