



Polystyrene activated linear tube carbon nanofiber for durable and high-performance supercapacitors

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ABSTRACT

With increasing demand for sustainable energy, it is essential to develop low cost, high performance, and environment-friendly materials for energy storage application. Metal oxides and sulfides are mostly being used as electrode materials for energy storage devices. However, their wide applications are precluded due to their higher cost, low stability, and adverse effect on the environment. Therefore, development of environment-friendly supercapacitors with low cost, high performance, and stable performance is a big challenge. Here, we report surface engineered carbon nanofibers for durable and high-performance supercapacitor. Surface engineered carbon nanofibers showed the highest specific capacitance of 277 F/g (at 1 mV/s), along with superior flexibility and cyclic stability. Moreover, they showed high energy and power density of 30.5 Wh/kg and 8.3 kW/kg, respectively. The cyclic stability showed almost 100% retention in charge storage capacity up to 5000 cycles. Electrochemical properties of a fabricated symmetrical supercapacitor device using these carbon nanofibers showed improved charge storage capacity at elevated temperatures. The charge storage capacity improved by over 150% by increasing temperature from 10 to 60 °C. Our results suggest that surface engineered carbon nanofibers could be a potential candidate for higher performance and durable supercapacitors.

1. Introduction

Increasing popularity of portable electronic devices and transition towards greener approach by using electric vehicles have triggered scientists to develop high performance, low cost, and environment-friendly materials for energy storage applications [1,2]. Supercapacitors, Li-ion batteries are some of the well-known energy storage devices [2,3]. High power density, rapid charging/discharging and considerably longer life cycles are some of the advantages of supercapacitors over Li-ion batteries [1–6]. However, energy density obtained from Li-ion batteries (~150 Wh/kg) is much higher than that of supercapacitors (~5 Wh/kg) necessitating a scope of improvement for future energy devices [2,3,5]. Various materials such as metal oxides, sulfides, selenides, and carbides are mostly being used for energy storage applications [7–10]. However, commercial applications of these materials are limited due to their high cost, low performance, electrochemical instability, and detrimental effect on the environment

[11,12]. Therefore, it is a challenge to develop energy storage material which has low cost, higher performance, durable and is environment-friendly.

Supercapacitors based on the electric double layer (EDL) which store the charge using active surface area of the materials are very promising due to their stable performance over the prolonged period. Various materials such as activated carbon, graphene, carbon nanotube, and carbon nanofiber are some of the exciting EDL electrode materials [13–20]. Low cost, longer charge-discharge life cycle, thermal stability, high surface area, cyclic stability, tunable physical structures, and high power density compared to the unstable pseudocapacitive material (metal oxides and conducting polymers) favors the use of carbon-based EDLCs, however, their charge storage capacity needs to be improved [21].

Among the various carbon-based materials, carbon fibers are attracting considerable research interest for energy storage applications due to their high electrical conductivity, high-temperature stability,

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higher surface area, and facile producibility [22]. Moreover, they can be electrospun into an aligned array of fibers/fiber web [23,24], yarn [25], and freestanding paper [22] sustaining their potentials for scalable industrial production. Polyacrylonitrile (PAN) is one of the highly used polymer precursors for carbon fibers [26]. Carbon fibers derived from PAN show high conductivity and the specific surface area which makes them very suitable for energy application [20,27,28]. Most of the time, carbon fibers need to be activated either physically or chemically to further improve their energy storage capacity [24]. However, these activation processes involve control over several steps and possess difficulties over controlling pore size and surface properties of the obtained carbon. Chemical activation processes (such as by KOH) destroy most of the oxygen and nitrogen-based functionalities present inherently in the structure of carbon fiber, thus adversely affecting the electrochemical properties [29].

In this study, we use a facile electrospinning process to fabricate nitrogen and oxygen dual doped carbon nanofibers (CNF) using PAN precursor for supercapacitor applications. The surface of the carbon nanofibers was engineered by using another low-temperature degradable polymer such as polystyrene (PS) along with PAN. Linear tube carbon nanofibers (LTCNF) were fabricated by electrospinning PAN and PS polymer blend followed by thermal treatment. Carbon nanofibers prepared using pure PAN showed a surface area of about $56 \text{ m}^2/\text{g}$ which improved to $212 \text{ m}^2/\text{g}$ for carbon fibers made using PAN-PS blend. This improved energy storage property of CNF significantly after surface modification. Furthermore, the electrode showed almost 100% retention in charge storage capacity up to 5000 cycles of charge-discharge study with over 150% improvement in charge storage capacity on increasing temperature from 10 to 60°C .

2. Experimental

2.1. Preparation of carbon nanofibers

PAN (average molecular weight of 130,000), PS (average molecular weight of 35,000) and *N,N*-dimethylformamide were purchased from Sigma, USA. CNF were prepared by electrospinning 10 wt% of PAN in *N,N*-dimethylformamide. LTCNF were fabricated by electrospinning of 10 wt% of PAN-PS (1:1 wt ratio) in *N,N*-dimethylformamide. During the electrospinning process, the fabricated fibers were collected over aluminum foil. The distance between the tip of the syringe and collector foil was 15 cm. A high voltage of 15 kV was applied, and a flow rate of 1 ml/h was maintained. The obtained fibers were carbonized in a tube furnace at 800°C without any pre-stabilization, at a rate of $5^\circ\text{C}/\text{min}$ for 2 h in an argon atmosphere. Fig. 1 shows the schematic diagram for the

preparation of carbon fibers.

2.2. Characterization

The crystallinity and phase purity of the prepared carbon fibers were studied by using powder X-ray diffraction technique (Shimadzu X-ray diffractometer) using 2θ scan with $\text{CuK}\alpha_1$ ($\lambda = 1.5406 \text{ \AA}$) radiation. Fingerprint identification of obtained carbon fibers was carried out using Raman spectroscopy using 514.5 nm wavelength as an excitation source (Model Innova 70, Coherent). Fourier transform infrared spectroscopy (FTIR) (Shimadzu IR Affinity-1 spectrometer) was used to analyze the chemical composition of the carbon nanofibers. X-ray photoelectron spectroscopy (XPS) was used to detect the nitrogen and oxygen doping and chemical bonding in the carbon nanofibers. A Thermo Scientific K α XPS system was used to record XPS spectra. The x-ray power of 75 W at 12 kV was used for the experiment with a spot size of 400 mm^2 . The XPS data acquisition was performed using the “Advantage v5.932” software provided with the instrument. The porosity and morphological properties of carbon fibers were examined using scanning electron microscopy (Philips XL 30 environmental scanning electron microscope). Micrometrics, USA, ASAP 2020 volumetric adsorption analyzer was used to analyze nitrogen adsorption/desorption isotherm of the carbon fibers at 77 K . Brunauer-Emmett-Teller (BET) absorption displayed specific surface area. Thermogravimetric analysis was carried out using TA instrument (TGA Q500) to understand thermal transitions for the electrospun fibers.

2.3. Electrochemical measurements

Electrocatalytic activities of the carbon nanofibers were studied using standard three electrode system using a Versastat 4–500 electrochemical workstation (Princeton Applied Research, USA). A platinum wire and saturated calomel electrode (SCE) were used as a counter and a reference electrode, respectively. These carbon nanofibers were used as working electrode. Electrochemical properties were studied using cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS) in 3 M LiOH electrolyte. EIS measurements were performed in a frequency range of 0.05 Hz to 10 kHz with an applied 10 mV of AC amplitude. Symmetrical supercapacitor was fabricated in 3 M LiOH electrolyte using an ion-transporting layer sandwiched between two identical carbon fiber electrodes. The active area of the device was 0.25 cm^2 and material loading of about 4 mg. Electrocapacitive performance of the device was assessed via cyclic voltammetry, galvanostatic charge-discharge, and electrochemical impedance spectroscopy.

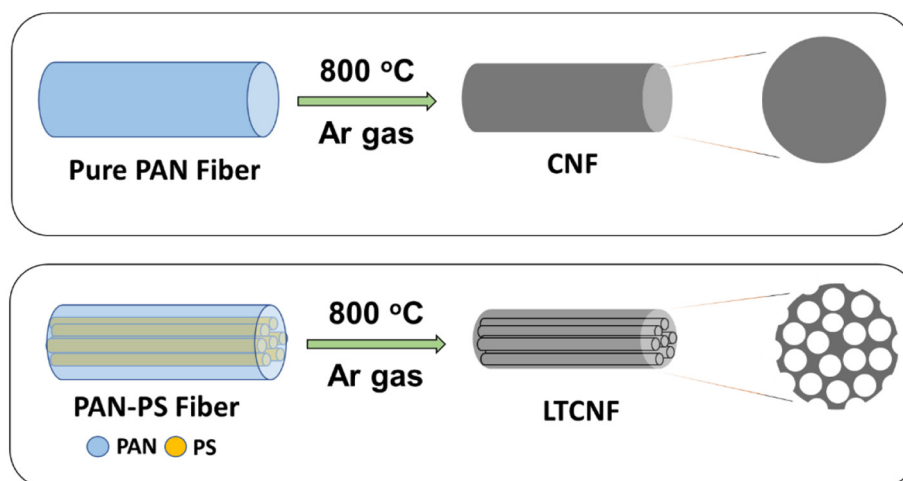


Fig. 1. Schematic showing electrospinning process and formation of CNF and LTCNF.

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