

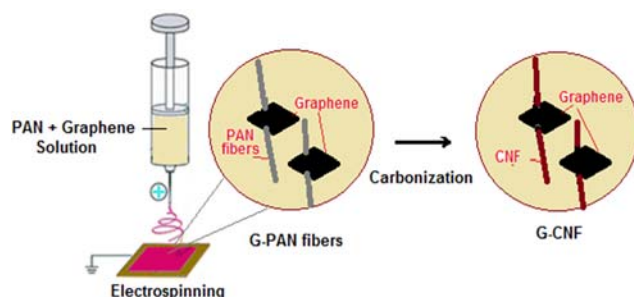
## Improved electrochemical performance of free standing electrospun graphene incorporated carbon nanofibers for supercapacitor

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### ABSTRACT



In this paper, we report the fabrication of carbon nanofibers (CNFs) by electrospinning of polyacrylonitrile (PAN) solution in N,N-dimethylformamide (DMF) with different concentrations followed by stabilization and carbonization in a tubular quartz furnace. To improve the electrochemical performance, graphene nanosheets have been used to prepare porous graphene/carbon nanofibers (G-CNFs). The morphology of the porous G-CNFs were characterized by means of scanning electron microscopy (SEM). Diameter of CNFs and G-CNFs were found to be in the range of 400 – 500 nm reveals the fibers in nanoscale with high porosity. The electrochemical performance of as-synthesized CNFs and G-CNFs was studied by cyclic voltammetry (CV), galvanostatic charge discharge (GCD) and electrochemical impedance spectroscopy. The CV curve of the pure CNFs show distorted rectangular shape whereas CV curve of G-CNFs exhibit a nearly rectangle-shaped profile which is the characteristic of an ideal electric double-layer capacitor. The improved electrochemical performance of G-CNFs is due to the improved internal electrical conductivity of G-CNFs via graphene nanosheets interlaying.

*Keywords: Carbon nanofibers; graphene; electrospinning; electrochemical performance; supercapacitor*

### INTRODUCTION

Among the variety of nanostructure carbon materials, graphene and carbon nanofibers (CNFs) have received rapidly growing attention for supercapacitors in recent years. Graphene is a two dimensional (2D) graphitic nanosheets of carbon material made of

monolayered or multilayered.<sup>1-3</sup> Graphene, the latest addition to the family of two dimensional (2D) materials, is distinguished by its unusual band structure, rendering the quasi particles in it formally identical to mass less, chiral fermions. Graphene carries high tensile strength, superior mechanical properties, and very large specific surface area (up to 3100 m<sup>2</sup>/g for individual graphene nanosheet). Electrical and thermal conductivities of graphene are exceptional. Graphene can also be conveniently fabricated and functionalized.<sup>4-11</sup> To date, exfoliated graphene nanosheets derived from natural graphite have been produced in large scale at low cost, and the process does not seek the aid of extensive chemical oxidation. These graphene nanosheets with the average thickness of 3-5 nm consist typically of a few graphene monolayers and have demonstrated attractive physical as well as chemical behaviors as a new family of carbon nanomaterials for

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electrochemical energy conversion and storage. It has been reported that a number of graphene-based nanomaterials have been integrated into porous electrodes of supercapacitors with high specific capacitance, which are based mainly on the configuration of graphene paper in aqueous electrolyte.<sup>12</sup> Interestingly, owing to different stacking manners of carbon orbitals on the surface, carbon nanofibers (CNFs) have much larger functionalized surface area compared to that of carbon nanotubes (CNTs). Recently electrochemical studies have shown that CNFs are capable of promoting the kinetics of electron transfer reaction, minimizing electrode surface fouling and enhancing electrocatalytic activity.<sup>13</sup> On the other hand, electrospinning technique as a low-cost, scalable, top-down nonmanufacturing tool has been commonly considered for producing continuous nanofibers of a broad spectrum of polymers and polymer-derived carbon, metals, metal oxides, ceramics, etc. Electrospinning also provides versatile routes of fabricating low-cost, porous, nanofibrous electrodes for promising use in supercapacitors and rechargeable batteries. CNFs synthesized via carbonization of fabricated electrospun polymer nanofibers carry excellent electrical and thermal conductivities and high connectivity for transport of electrical and thermal currents, and thus have raised particular interests of materials scientists and engineers worldwide.

Supercapacitors (SCs) are one of the important energy storage devices due to their high capacity, high power density, long life cycle and low internal resistance makes them able to store and deliver energy at relatively higher rates. Therefore, supercapacitors are considered as prospective energy storage system when compared to the conventional capacitors and secondary batteries.<sup>14-20</sup> The performance of supercapacitors is generally depend on the physical and chemical properties of electrode materials used in supercapacitors.<sup>21-23</sup> Since the electrochemical performance of supercapacitors is mainly governed by the electrode materials, the development of materials with high capacitance and power density is an important task to achieve the industrial desire. The electrochemical performance of electrodes relies on the high surface area, average pore diameter, surface functionalities to ensure the fast oxidation-reduction reaction, and high electrical conductivity. Currently, considerable effort has been put to develop carbonaceous materials as electrodes because of good electrical conductivity, low cost, non-toxicity, high chemical stability, and simplicity of preparation.<sup>24,25</sup> Among them, electrospun carbon nanofibers have large surface area and porosity, seem to be an excellent candidate for electrode materials. However, the use of carbon nanofibers for supercapacitors is still restricted to the low power and energy density. To improve the electrochemical properties of carbon nanofibers, the modification of carbon nanofibers is carried out. Kim et al utilized the electrospinning technique to prepare nonwoven carbon webs.<sup>26</sup> It was obtained that the increased activation time during carbonization facilitated the development of microporous structural fibers. This in turn increased the surface area of fibers and improved the specific capacitance. Carbon nanofibers with a microporous structure could also be generated by the addition of polymethyl hydrosiloxane (PMHS) in PAN precursor solution.<sup>27</sup> With this background, we have prepared the

electrodes for supercapacitors by using continuous graphene-beaded CNFs (G-CNFs). The aim of study was to present the advantages of electrospun CNFs as continuity and electrical conductivity and graphene as large specific surface area and high electrical conductivity for the purpose of electrical energy storage. The porous G-CNFs were produced via electrospinning of polyacrylonitrile (PAN)/N,N-dimethylformamide (DMF) solution dispersed with oxidized graphene nanosheets followed by stabilization and then carbonization. The chemical structure and electrochemical properties of the novel G-CNFs based electrodes were characterized and compared with pure electrospun CNFs.

## EXPERIMENTAL

### *Materials and methods*

Polyacrylonitrile (PAN, purity 98.5%, Mw = 150,000) and N,N-dimethylformamide (DMF, purity 99%) were purchased from Sigma-Aldrich. The oxidized graphene nanosheets with an average thickness of 6-8 nm were supplied by National Physical Laboratory, New Delhi, India. All the chemicals were used as received without further purification or modification. The morphology and structure of the prepared CNFs and G-CNFs were investigated by a SEM (Zeiss EVO18). Electrochemical performance by means of cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS) was studied on CH Instruments (Model-CHI6112D) electrochemical workstation with the typical symmetrical three-electrode system. 1 M KOH was used as electrolyte in electrochemical measurement. The specific capacitance of electrode was calculated from galvanostatic charge-discharge.

### *Preparation of Carbon Nanofibers (CNFs) and Graphene incorporated Carbon Nanofibers (G-CNFs)*

PAN nanofibers with different weight percent (6, 8 and 10 wt%) were produced by electrospinning. For PAN nanofibers of 6 wt%, solution consisting of PAN in DMF was prepared as follows. PAN powder was first dissolved in DMF to prepare 6 wt% PAN solutions; stir it for 1 hr at 60 °C. During the electrospinning process, the as-prepared solution was then placed in a 10 ml plastic syringe installed with a stainless spinneret, which was connected to a positive high voltage DC power supply. A laboratory made rotary aluminium disc with the diameter of 3.3 cm was electrically grounded and used as nanofibers collector. High DC positive voltage 18 kV was applied between the needle of the syringe and collector. The distance between the tip of the syringe needle and collector was fixed at 22 cm. The flow rate of the electrospinning solution was fixed at 0.6 ml/h through a computer control syringe pump.<sup>28,29</sup> After electrospinning, the PAN nanofibers were peeled off from the aluminium foil wound on the rotary aluminium disc, and then dried in an oven at 80 °C for 10 hr prior to the stabilization treatment. The stabilization and carbonization of the as-prepared PAN nanofibers were performed in a tubular quartz furnace. The electrospun PAN nanofibers were first heated up at a rate of 1°C min<sup>-1</sup> and maintained at 250°C for 1 hr in air for the purpose of oxidative stabilization of PAN. The fibers were then carbonized as the temperature was increased from 250 to 800 °C in N<sub>2</sub> atmosphere at a rate of 5°C min<sup>-1</sup> and annealed

at 800°C for 30 min. Thereafter, the furnace was cooled down to 400 °C in N<sub>2</sub> and then kept at this temperature by introducing the air for 1 hr for activation of the carbonized PAN nanofibers. For the purpose of comparison, CNFs with 8 wt% and 10 wt% PAN/DMF solutions were also synthesized under the same processing parameters.<sup>30</sup> For the preparation of G-CNFs, functionalized graphene (0.2 wt%) was mixed in PAN (10 wt%)/DMF solution, stirred for 1 hr at 60 °C and transferred to syringe for electrospinning. The electrospinning processing parameters and heat treatment for stabilization and carbonization process were maintained as that of pure CNFs.

#### Characterizations

The morphology and structure of the prepared CNFs and G-CNFs were investigated by a SEM (Zeiss EVO18). Electrochemical performance was studied on a CH Instruments (Model-CHI6112D) electrochemical workstation with the typical symmetrical three- electrode system. In this case, a 1 M KOH was used as electrolyte in electrochemical measurement. The electrochemical behaviors of samples included cyclic voltammetry (CV), Galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS) were detected on a CHI 6112D electrochemical workstation. The specific capacitance of electrode was calculated from Galvanostatic charge-discharge.

## RESULTS AND DISCUSSION

#### Scanning electron microscopy (SEM)

Figure 1 (a), (b), (c) and (d) represents the typical SEM images of the CNFs with 6 wt%, 8wt%, 10 wt% and G-CNFs respectively. These carbon fibers are found to be very smooth with diameter in the range of 400 – 500 nm. The average diameter was found to be increased by increasing the weight percentage of polyacrylonitrile from 6 wt% to 10 wt%. It is clearly observed from SEM micrograph of G-CNFs that the fiber diameter has been increased than that of CNFs (10 wt%) due to graphene sheets somewhere interconnected with CNFs as seen in Figure 1(d). TEM image of G-CNF is shown in Figure 1(e). It is seen that fibers are interconnected by graphene sheets forming composite of graphene and CNF in the form of nanofibers structures.

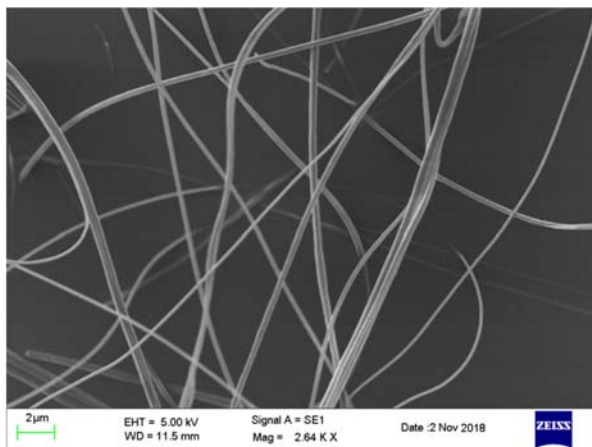


Figure 1 (a): SEM micrograph of 6 wt % CNF

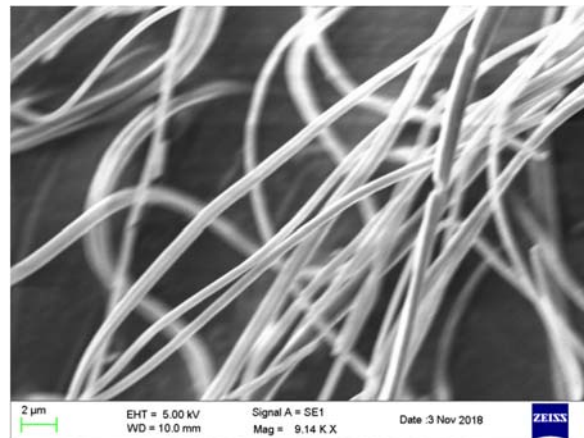


Figure 1 (b): SEM micrograph of 8 wt % CNF



Figure 1(c): SEM micrograph of 10 wt % CNF

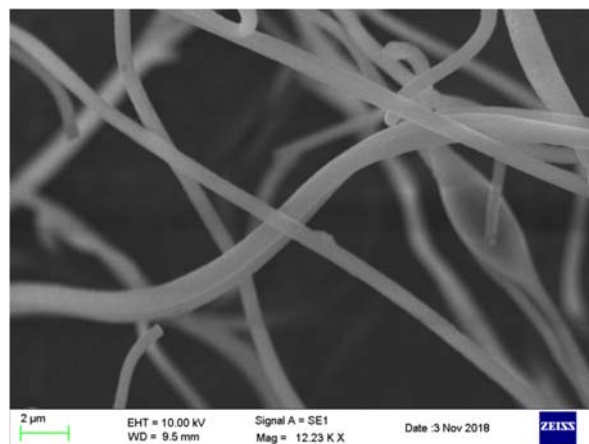
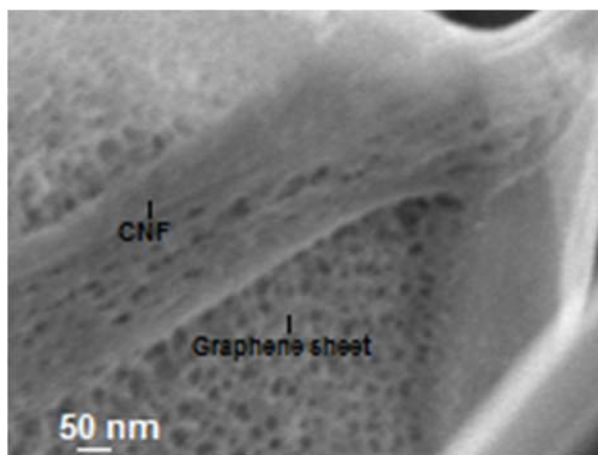


Figure 1 (d): SEM micrograph of G-CNF

With considering the unique topological connectivity and high electrical conductivity of the G-CNFs network, the synthesized G-CNFs are suitable for potential use as electrodes for supercapacitors. In general, graphene nanosheets can attain a large surface area and very high electrical conductivity, which may eliminate the need of conductive fillers and result in thinner electrodes.<sup>31</sup>

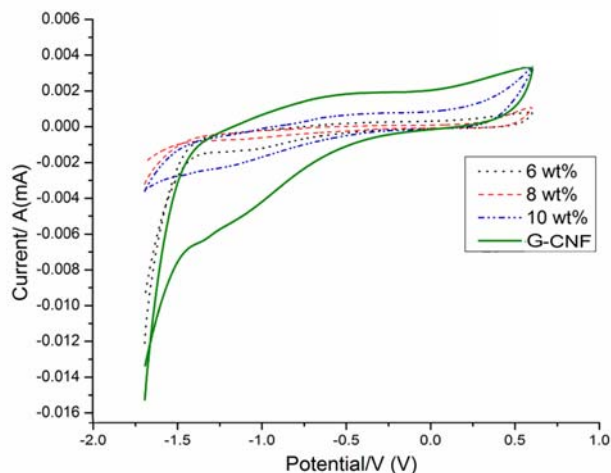




**Figure 1 (e):** TEM micrograph of G-CNF

### Electrochemical properties

The electrochemical properties of CNFs and G-CNFs were evaluated in 1 M H<sub>2</sub>SO<sub>4</sub> aqueous electrolyte. Figure 2 shows the cyclic voltammetry curves of CNFs and G-CNFs at the scan rate of 5 mV/s. In cyclic voltammetry experiment, the working electrode potential was ramped linearly versus time. Unlike in linear sweep voltammetry, after the set potential is reached in a CV experiment, the working electrode's potential was ramped in the opposite direction to return to the initial potential. These cycles of ramps in potential may be repeated as many times as desired. The current at the working electrode is plotted versus the applied voltage (i.e., the working electrode's potential) to give the cyclic voltammogram trace. Cyclic voltammetry is generally used to study the electrochemical properties of carbon based materials. Cyclic voltammetry provided information regarding the reversible nature of the electrodes. The shape of the curves represent the rectangular frame type indicating as an ideal electrode materials form electric double layer rapidly and evenly in the topography of the electrode/electrolyte interface. When the voltage scanning direction was changed, the current can reach steady state rapidly. Therefore, the cyclic voltammetry curve of the ideal electrode material is shown as a nearly rectangular shape.<sup>32</sup> At 5 mV/s scan rate, the integral area of the cyclic voltammetry curve of the G-CNFs is more than CNFs. This shows that graphene/carbon nanofibers have more capacitance than pure carbon nanofibers.

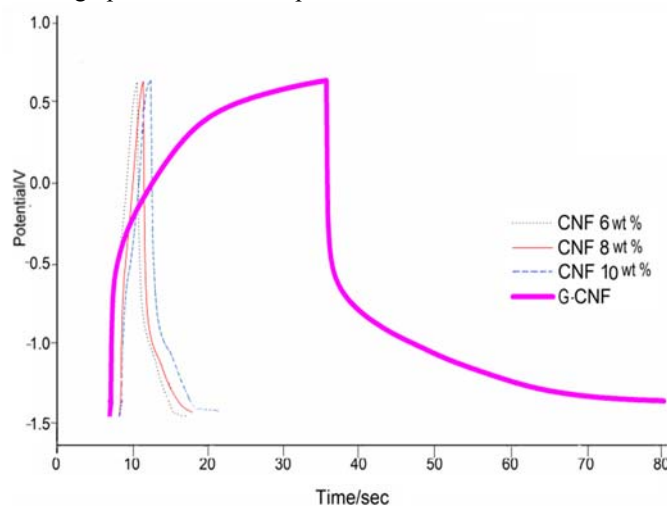


**Figure 2:** Cyclic voltammetry of CNFs and G-CNFs at the scan rate of 5 mV/s

Galvanostatic charge-discharge (GCD) measurement was carried out to evaluate the rate performance of the pure CNFs and G-CNFs. The ability to retain high capacitance during fast charge/discharge processes is a crucial requirement for high performance and practical EDLCs. The galvanostatic charge and discharge curves of CNFs and G-CNFs at the current density of 1 A/g are shown in Figure 3. The figure indicate that the charge/discharge curves exhibit a shape like the triangle, indicating its electric double-layer capacitive behaviour, in accordance with the CV curves. The specific capacitance ( $C_s$ ) of the electrode material can be calculated based on the discharge curves using equation (1);

$$C_s = \frac{i\Delta t}{m(V_f - V_i)} \quad (1)$$

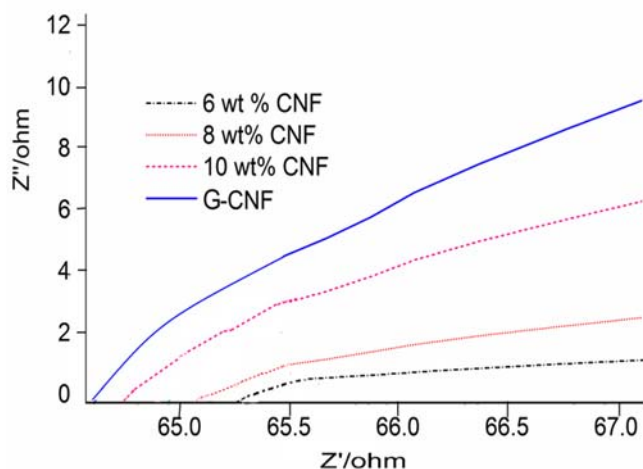
Where,  $i$  is the discharge current,  $\Delta t$  is the time for a full discharge,  $m$  is the mass of active material in the electrode,  $V_f$  and  $V_i$  are the potential limits of a full discharge, respectively.<sup>33</sup> G-CNFs electrode exhibits a high specific capacitance of 450 F/g at 1 A/g, which is more than that of CNFs of 90.6 F/g, 120 F/g and 157.5 F/g at 6 wt%, 8 wt% and 10wt% respectively. The higher supercapacitance of G-CNFs is owing to the synergistic effects of graphene and carbon nanofibers. The graphene might be linked or formed beads for the carbon nanofibers with which more surface area of graphene leads to 3D porous network.



**Figure 3:** Galvanostatic charge-discharge of CNFs and G-CNFs at the current density 1 A/g

Electrochemical impedance spectroscopy was employed to analyze the impedance behaviors of CNFs and G-CNFs. Figure 4 shows the Nyquist plots of CNFs with 6 wt%, 8 wt% and 10wt% and G-CNFs electrodes. Each curve contains nearly vertical line in the low-frequency region. The intercept of the line at the real axis represents the internal resistance, which is related to the intrinsic electrical resistance of the active materials ( $R_\Omega$ ), the electrolyte resistance, and the contact resistance at the interface between the active material and current collector. The change in intercept was largely governed by the electrical resistance of the active materials because the other two factors are similar conditions.<sup>31,34</sup> The impedance spectra are fitted using the complex nonlinear least squares method. The intercept of the real axis in the high-frequency region corresponds to the contact resistance, which originates from the ionic resistance of electrolyte, intrinsic

resistance of electrode. The slope of the straight line in the low-frequency region is inversely correlated with the ion diffusion resistance within the electrodes.<sup>35</sup> The line of G-CNFs electrode is more vertical compared to that of CNFs revealed a better capacitance characteristic. The resistance of CNFs with 6 wt%, 8 wt%, 10wt% and G-CNFs were found to be 65.3  $\Omega$ , 65.1  $\Omega$ , 64.9  $\Omega$  and 60  $\Omega$ . This shows that the resistivity of G-CNF is low as compared to that of CNFs and hence G-CNFs is a potential candidate for the electrodes of supercapacitors. The incorporation of graphene into CNFs leads to tremendously increase in specific capacitance indicates the novel method for the preparation of EDLC electrodes for supercapacitors.



**Figure 4:** Nyquist plots of CNFs with 6 wt%, 8 wt% and 10wt% and G-CNFs electrodes.

## CONCLUSIONS

Carbon nanofibers (CNFs) and graphene incorporated carbon nanofibers (G-CNFs) have been successfully fabricated by electrospinning and carbonization. The diameter of CNFs and G-CNFs are in the range of 400-500 nm. The specific capacitance of the CNFs was found to be increased with the weight percentage of PAN. The higher specific capacitance of G-CNFs is owing to the interconnectivity effect of graphene and carbon nanofibers. The excellent connectivity of the graphene with CNFs significantly improved the specific surface area, electrical conductivity. The higher specific capacitance of G-CNFs as compared to CNFs represents the suitability of graphene incorporated carbon nanofibers to be utilized in performances of supercapacitors.

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