



Short communication

Graphene-coated pyrogenic carbon as an anode material for lithium battery

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HIGHLIGHTS

- Graphene-coated pyrogenic carbon was synthesized through pyrolysis.
- Gaps between separated graphene sheets were “soldered” by “glue” molecules.
- Graphene-coated carbon has excellent Li storage capacity cycle performance.

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ABSTRACT

In this work, cotton fibers and pyrene-dispersed graphene sheets were used to produce graphene-coated pyrogenic carbon as an anode material for lithium battery. The graphene sheets were wrapped around the cotton fibers by simply dipping the fabric in a graphene/pyrene-derivative suspension. And then the cotton/graphene textile was annealed at 700 °C in a quartz tube furnace under Ar flow conditions. During the annealing process, the gaps between separated graphene sheets were “soldered” by “glue” molecules (aromatic molecular surfactant) to form graphene-coated pyrogenic carbon. Because of the unique electric properties of the graphene “skin” on the pyrogenic carbon, the flexible graphene-coated pyrogenic carbon showed relatively large storage capacity to lithium. Galvanostatic charge–discharge experiments also showed that the graphene-coated pyrogenic carbon electrode provided a reversible discharge capacity as high as 288 mA h g⁻¹ even after 50 cycles and thus can be used as an anode material in lithium battery.

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1. Introduction

Graphene is a one-atom thick material made up of sp²-bonded carbon atoms and has unique physicochemical properties, including high electrical and thermal conductivity, quantum Hall effect, massless transportation properties, good mechanical properties, and so on [1–11]. As a result, graphene has been used in many applications to benefit the society and improve life quality. Among all the applications, the use of graphene-based materials as electrodes for lithium battery is a very promising one and has advantages over that of other carbonaceous materials because graphene has relatively low-cost and is easy to access. Anode with graphene alone, however, can only provide relatively low lithium storage capacity and unstable/poor cycle performance. A number of graphene-based composites, using various carbonaceous materials and nanosized metal/oxides as templates, thus have been developed to enhance the lithium storage capacity and the cycling

performance of graphene anode [12–14]. Nevertheless, large-scale production and application of the graphene composites for electrodes are still unrealized mainly because of the high cost, inaccessibility, and especially potential health risks associated with the template materials.

Recent studies showed that highly electro-conductive carbon nanotube (CNT) based nanocomposites for electrodes can be simply prepared by dipping cotton fleece in CNT solution because of the strong binding between carbon nanotube and cotton fiber [15,16]. This makes it possible to envision a simple way to produce graphene based nanocomposites using similar method due to the similarity between graphene and CNTs. To date, however, little attention has been paid to the fabrication of graphene-coated textiles, which probably is due to technical obstructions of pre- and post-treatment of the composites, particularly with respect to difficulty in preparing stable graphene solutions and wrapping seamless graphene layers on the fibers. Recently, Su et al. reported that pyrene molecules can be used to prepare stable graphene solutions, in which pyrene can not only act as nanographene molecules to heal the possible defects in the graphene oxides but also act as

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electrical “glue” soldering adjacent graphene sheets during annealing [17]. Similarly, Zhang et al. developed a simple and scalable exfoliation approach to produce high-quality graphene suspension by sonicating graphite in an aqueous solution of hydrophilic pyrene molecules [18]. It is anticipated that the pyrene dispersed graphene can be used in many applications to produce graphene based nanocomposites, including the fabrication of graphene-coated textiles.

In this work, a simple synthesis was developed to produce graphene-coated pyrogenic carbon as an anode material for lithium battery. As shown in Scheme 1, natural cotton fibers were used as the templates where graphene sheets were wrapped on the fibers (i.e., graphene-coated textiles) by dipping the cotton fleece in pyrene-dispersed graphene suspensions and further “soldered” into graphene-coated pyrogenic carbon after annealing (pyrolysis). The morphological structures, thermal and electrical properties of the graphene-pyrogenic-carbon nanocomposites were examined in details with atomic force microscopy (AFM), fluorescent measurement, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) and Galvanostatic charge–discharge experiments. The main objective of this work is to develop a facile, cost-effective, and environmental friendly method to produce graphene-coated pyrogenic carbon materials for lithium battery and other energy storage and electronic applications.

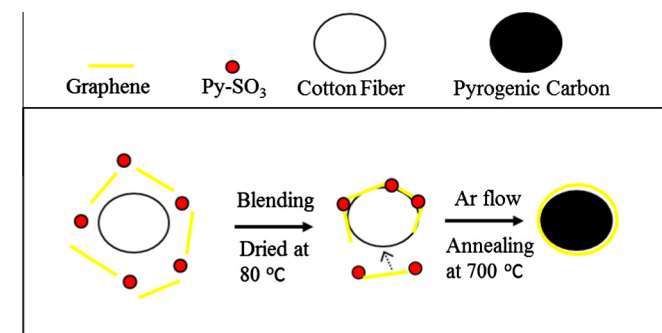
2. Experimental section

2.1. Materials

Synthetic graphite powder (<20 nm particle size) and 1,3,6,8-pyrenetetrasulfonic acid (Py-SO₃) tetrasodium salt hydrate were purchased from Sigma–Aldrich and used as received. Cotton fleece fabric (233 g/m²) was produced by Cotton Inc. (Cary, NC).

2.2. Preparation of graphene suspension

A graphene suspension was synthesized from the natural graphite flakes by a method similar to that of Zhang et al. [18]. A stock solution of Py-SO₃ at a concentration of 0.5 mg mL⁻¹ was prepared in deionized water by vigorous stirring for 1 h. The graphite powder was added to the pyrene solution with a weight ratio between the pyrene derivatives to the graphite powder at 2:1. Direct exfoliation of graphite to graphene sheets was performed by bath sonication of the obtained mixture solution for 1 h. The exfoliation process was monitored by comparing the fluorescence spectra of the suspension at the beginning and end of exfoliation period. In addition, a drop of the graphene solution was applied to a mica plate and dried at 80 °C for AFM analysis.



Scheme 1. Illustration of procedures to produce graphene-coated pyrogenic carbon from cotton fibers.

The obtained grey graphene solution was dried directly to prepare the graphene-coated textiles.

2.3. Preparation of graphene-coated pyrogenic carbon

It has been reported that CNTs can be coated on cotton fibers due to the mechanical flexibility and large binding energy [15,16]. Similar method was applied in this work to produce the graphene-coated pyrogenic carbon. Briefly, a cotton fleece was first dipped into the graphene suspension (1 mL/cm²), and then the wet grey cotton/graphene composites were dried at 80 °C for two hours. A slice of the cotton/graphene composites measuring 7 cm in length and 4 cm in width was then annealed for 20 min at 700 °C under inert gas (Ar) flow to produce the graphene-coated pyrogenic carbon for the electrodes.

2.4. Characterization

The dry Py-SO₃ exfoliated graphene samples on the mica surface were imaged with the tapping mode Nanoscope IIIa AFM instrument (Veeco instrument, Santa Barbara, CA, USA) in air. The fluorescence spectra of the graphene solution were determined using a Cary-Eclipse fluorescence spectrophotometer (Varian, Inc., Palo Alto, CA). The morphology, microstructures, and surface element compositions of the graphene-coated pyrogenic carbon were characterized using field emission gun SEM (Philips XL-30) equipped with an energy-dispersive X-ray analyzer. Electrochemical charge/discharge performance of the graphene-coated pyrogenic carbon was evaluated using 2032 button coin cells (Hohsen Corp.) [19]. Coin cells were assembled in a high purity argon filled glove box. Charge (lithium insertion) and discharge (lithium extraction) were conducted using an Arbin automatic battery cycler at a constant current density of 50 mA g⁻¹ between cut-off potentials of 0.01 and 2.8 V.

3. Results and discussion

Fig. 1A shows a typical tapping-mode AFM image of graphene/pyrene hybrids deposited on a freshly cleaved mica surface. The size of the graphene patches was in the micrometer range. The thickness of a single-layer hybrid ranged from 0.5 to 1.3 nm with an average of 0.9 ± 0.4 nm, measured from cross-sectional images (Fig. 1B). The variation of thickness was attributed to the possible inhomogeneous coverage of Py-SO₃ molecules on the graphene surface or simply due to the AFM system noises [18]. Holes with diameters ranging from 2 nm to 500 nm were found randomly distributed in the graphene sheets (Fig. 1A). Those holes might be caused by the sonication and could be partly healed during annealing and facilitate the capture of Li-ions.

Fig. 2 shows the fluorescence spectra (excited at 340 nm) of a graphene/pyrene suspension for different sonication periods. Prior to the sonication, the spectrum of the graphene solution showed a big peak at 500 nm, which could be ascribed to the excimer emission of pyrene derivatives [20,21]. The intensity of this peak decreased dramatically after one hour of sonication. In addition, we also observed a dramatic increase in the peak at 374 nm. This fluorescence behavior was virtually the same as that of the pyrene aqueous solutions alone when the concentration of pyrene is below its critical micelle concentration, suggesting that the fluorescence of the graphene/pyrene solution is controlled by the non-bound (free) pyrene monomers in the solution. Findings from Figs. 1 and 2 confirmed that graphene suspension was successfully synthesized by sonication of graphite in the pyrene solution.

Fig. 3A and B shows the pictures of the graphene-coated pyrogenic carbon sample, which is flexible and metallic. A circuit was

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