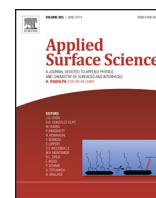




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Preparation of polyaniline/graphene oxide nanocomposite for the application of supercapacitor

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ABSTRACT

Graphene oxide was synthesized by an improved Hummers method. Three polyaniline (PANI)/graphene oxide (GO) nanocomposite electrode materials were prepared from aniline (ANI), GO, and ammonium persulfate (APS) by chemical polymerization with the mass ratio ($m_{\text{ANI}}:m_{\text{GO}}$) 1000:1, 100:1, and 10:1 in ice water, respectively. The crystal structure and the surface topography of all materials were characterized by means of X-ray diffraction (XRD), Fourier transform infrared spectrum (FT-IR) and scanning electron microscopy (SEM). The electrochemical properties of the composite were evaluated by cyclic voltammetry, galvanostatic charge/discharge, and the impedance spectroscopy, respectively. The test results show that the composites have similar and enhanced cyclic voltammetry performance compared with pure PANI based electrode material. The PANI/GO composite synthesized with the mass ratio ($m_{\text{ANI}}:m_{\text{GO}}$) 1000:1 possessed excellent capacitive behavior with a specific capacitance as high as 355.2 F g^{-1} at 0.5 A g^{-1} in $1 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ electrolyte due to the unique morphology of Mace-like PANI/GO composite, and after 1000 cycles, the specific capacitance of the composite still has 285.8 F g^{-1} . These results demonstrate exciting potentials of the composite for high performance supercapacitors or other power source system.

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1. Introduction

Electrochemical capacitors (often called supercapacitors) are novel energy storage systems that have been applied in many fields because of their long cycle-life, excellent reversibility and high power density [1–3]. However, their widespread use is limited by their lower energy storage density and relatively higher effective series resistance than that of batteries. Therefore, many researchers in this area have focused on the development of different electrode materials such as various forms of carbon, conducting polymers, and transition metal oxides [4–6]. Electrically conducting polymers have attracted worldwide research interest because of their high flexibility and relatively high specific capacitance as one of the main electrode materials for supercapacitors. Among various polymers, polyaniline (PANI) is extremely attractive as electrode materials for supercapacitors due to high electrochemical activity, chemical stability and ease of synthesis as well as relatively low cost [7–9]. But it can produce volume expansion and contraction sometimes, low intensity of cycle stability, easy to collapse when in charge and

discharge [10,11]. So it is needed to be studied further to improve the cycle performance of PANI electrode materials.

In order to overcome this drawback, all kinds of carbon materials, such as porous carbon [12], mesoporous carbon [13] and carbon nanotube [14], have been investigated due to their good conductivity, stable physicochemical properties, low cost, and long cycle life [1]. Graphene oxide (GO) surface contains a large number of functional groups (carboxyl, hydroxyl, epoxy group, etc.) [15,16]. These unique functional groups make it good dispersion and hydrophilic in the water, which make it as a promising candidate for the fabrication of supercapacitor electrode materials [17,18]. In addition, graphene oxide also has larger specific surface area, wide chemical potential, excellent chemical stability, and rich drape morphology [19]. It will improve the conductivity and cyclic stability of the electrode after compound graphene oxide to polymer. Therefore, combining nanometer-sized and nanostructured graphene oxide with PANI has been extensively studied. For instance, Wang et al. [20] prepared the nanocomposite with a mass ratio of aniline/graphite oxide, 100:1 and found that the specific capacitance was 531 F g^{-1} at 0.2 A g^{-1} in aqueous electrolyte. Xu et al. [21] prepared PANI/GO nanocomposite by in situ polymerization with the assistance of supercritical carbon dioxide (SC CO_2) and PANI/GO nanocomposite with aniline concentration at 0.1 mol L^{-1} exhibited high specific capacitance (425 F g^{-1}) at a current density of 0.2 A g^{-1} .

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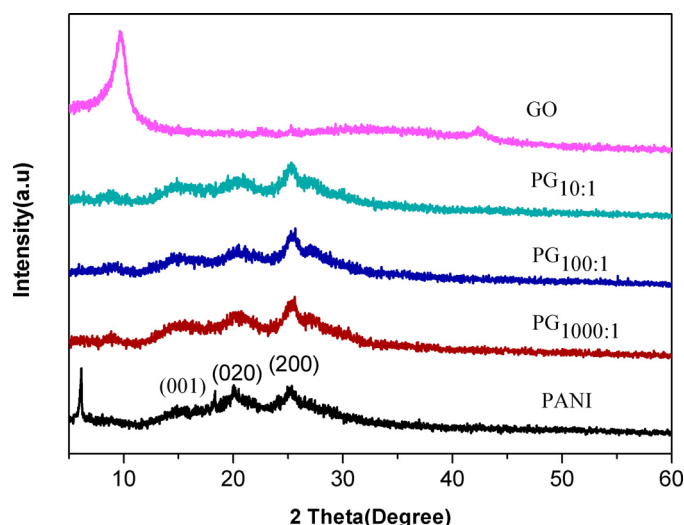


Fig. 1. XRD pattern of graphene oxide, PANI, PG_{100:1}, PG_{10:1}, and PG_{1000:1}.

In the present work, synthesis, chemical structure, and morphology of PANI/GO nanocomposite and electrochemical performances as electrode materials for supercapacitors are investigated.

2. Experimental

2.1. Materials

All chemical reagents used in this study were of analytical laboratory grade. Aniline monomer was purchased from Tianjin Damao Chemical Reagent Co. Ltd. (Tianjin, China) and vacuum distilled before use to remove impurities. Graphite powder (325 mesh, carbon content 99.95 wt%) was obtained from Aladdin Chemical Reagent Co. Ltd. (Shanghai, China). Hydrochloric acid (HCl), sulphuric acid (H₂SO₄), nitric acid (HNO₃), potassium permanganate (KMnO₄), hydrogen peroxide (H₂O₂) and ammonium peroxydisulfate (APS, (NH₄)₂S₂O₈) were procured from Tianjin Da Chemical Reagent Co. Ltd. (Tianjin, China) and used as received without further purification.

2.2. Graphene oxide (GO) synthesis

Graphene oxide was synthesized by an improved Hummers method [22]. In a typical reaction, 1 g of graphite powder, 24 mL of HNO₃ (64 wt%), and 94 mL of H₂SO₄ (98 wt%) were stirred together in an ice bath (Below 10 °C). Next, 6 g of KMnO₄ was slowly added. At the same time, control the reaction temperature below 20 °C for 2 h. Once mixed, the solution is transferred to a 35 ± 5 °C water bath and stirred for about 1 h. Then the solution was stirred for 30 min while the temperature was raised to 85 °C. At this time, the solution can occur with color changes. 100 mL of distilled water was added, and the solution was stirred for 30 min under 85 °C. Finally, 10 mL of H₂O₂ (30 wt%) was added when the color of the solution turns from dark brown to yellow. After cooling to room temperature, the black homogeneous dispersion was separated by centrifugation. The prepared graphene oxide was dried in a vacuum at 60 °C for 12 h.

2.3. Preparation of graphene oxide/polyaniline nanocomposite

1 g of pure aniline (ANI) and 12.5 mL of HCl (1 mol L⁻¹) solution were stirred together in an ice bath. Next, a certain amount of GO was added. Last, 30 mL acid saturated solution of ammonium persulfate (APS) was slowly dropped and the ratio of amount of

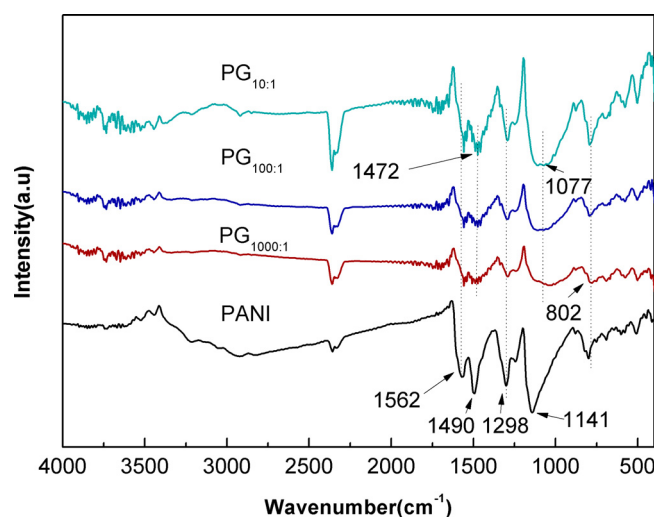


Fig. 2. FTIR spectra of PANI, PG_{100:1}, PG_{10:1} and PG_{1000:1}.

substance ($n_{\text{ANI}}:n_{\text{APS}}$) was 1:1. After the mixture was stirred for about 6 h in an ice bath, the precipitate product was collected, and washed several times with distilled water until the cleanout fluid was neutral. Finally, the obtained PANI/GO composites were dried in air at 80 °C for 12 h. The composites were signed as PG_{1000:1}, PG_{100:1}, and PG_{10:1} with the mass ratio ($m_{\text{ANI}}:m_{\text{GO}}$) 1000:1, 100:1, and 10:1, respectively. For comparison, the pure polyaniline was synthesized using the same method with no graphene oxide.

2.4. Materials characterization

Samples were characterized using powder X-ray diffraction with Cu KR radiation ($\lambda = 1.5418 \text{ \AA}$) at a step size of 2° per second. Scanning electron microscope (SEM) measurements were performed on a Hitachi S-3400N scanning electron microscope operated at 15 kV. The chemical structure of different PANI/GO composites and PANI were characterized by Fourier transform infrared spectrum (FTIR) (Affinity-1 instrument with a resolution of 4 cm⁻¹). Sample for FTIR measurement was homogeneous mixture onto a KBr disk and scanning from 400 to 4000 cm⁻¹.

2.5. Electrochemical measurement

The electrochemical performances were investigated using the supercapacitor test cells, which were fabricated with two-electrode cells configuration. The fabrication of working electrodes was carried out as follows. Briefly, the as-prepared materials, acetylene black and poly(tetrafluoroethylene) (PTFE) were mixed in a mass ratio of 85:10:5 and dispersed in ethanol. Then the resulting mixture was pressed on a stainless steel mesh current electrode ($\Phi = 15 \text{ mm}$) with a pressure of 18 MPa for 1 min in order to assure a good electrical contact, and dried at 80 °C for 24 h. The mass of active material was 6 mg. The electrodes, which were soaked with 1 mol L⁻¹ H₂SO₄ aqueous electrolyte solution for 24 h, were separated by a polypropylene film and were sandwiched in a stainless steel (SS) cell with a pressure of 170 MPa. Cyclic voltammograms (CV) were recorded from -0.2 to 0.8 V at various sweep rates, and the scan rate was 2, 5, and 10 mV s⁻¹. Galvanostatic charge/discharge measurements were done from -0.2 to 0.8 V at current densities of 1 A g⁻¹. Electrochemical impedance spectroscopy (EIS) measurement was carried out at open circuit potential with AC voltage of 10 mV amplitude in the 100 kHz to 0.1 Hz frequency range. All cyclic voltammetry (CV), galvanostatic charge/discharge and electrochemical impedance spectroscopy

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