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Short Communication

Synthesis of Graphene and Electrochemical Performance

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We synthesize the graphene with the microwave method. Graphite oxide was synthesized via Hummers method with flake graphite as raw material. Graphene is demonstrated to behave as an excellent microwave absorbent, which shows a high specific surface area and good electrochemical performance. The structure of highly oxidized graphite shows a smooth surface with tightly packed sheets. The good structure and excellent performance suggest its promising application in supercapacitors.

Keywords: microwaves; sonicate; aqueous; exceptional

1. INTRODUCTION

In recent years, graphene has attracted tremendous attention for its remarkable electronic and thermal conductivity, large specific surface area, high mobility of charge carriers, and excellent chemical stability and mechanical strength due to its unique structure made of sp^2 carbon atoms tightly packed into a honeycomb lattice [1-3]. For the sake of practical application, it is critical to find versatile methods that can produce graphene abundantly and efficiently at low cost.

Microwaves, an alternative energy input source, have been widely used because of their internal and volumetric heating of materials, in the field of organic synthesis [4], environmental remediation [3], preparation of catalysts [5] and activated carbon[6-9]. In this process, microwave energy is transformed into heat by using a microwave absorbent. For this very reason, it is necessary to select a proper microwave absorbent to efficiently convert microwave energy into heat [10-13].

In the present work, we synthesize the graphene with the microwave method. Graphene is demonstrated to behave as an excellent microwave absorbent, which shows a high specific surface area and good electrochemical performance.

2. EXPERIMENTAL

2.1 Materials preparation

All the chemicals were of analytical reagent and used as received without further purification. In a typical preparation progress, 5 g graphite powder and 2.5 g sodium nitrate were mixed with 130 ml sulfuric acid in an ice bath under vigorous stirring for 2 h. Weighted potassium permanganate was then slowly added into the system while the temperature was kept from exceeding 20 ^{o}C. The temperature of the mixture was subsequently raised to 35 ^{o}C and maintained for 1h, then the mixture was heated to 98 ^{o}C for 30 minutes. 500 ml deionized water and 5 ml hydrogen peroxide was added into the mixture. After centrifugation and washing to remove residual salt, the wet graphite oxide was dried at 60 ^{o}C for 48 h. After the mixtures were subjected to sonication (40k Hz, 100 W) in 150 ml of deionized water for 1 h, a dark brown suspension was obtained. The suspension was centrifuged to remove water, and further dried in the oven. The production of graphene was achieved in a quartz reactor (30 cm in length, 18 mm in diameter) located in a microwave oven (800 W).

2.2 Materials characterization

The morphology and the structure of the sample were examined with a field-emission scanning electron microscopy (FESEM, JEOL JSM-6300F). The specific surface area of graphene was obtained on a Micromeritics ASAP 2020 nitrogen adsorption equipment.

2.3 Electrochemical measurements

The working electrode was prepared by 85 wt% of the active material, 10 wt% of conducting agent (carbon black), and 5 wt% of binder (polyvinylidene difluride, PVDF). This mixture was pressed onto the glassy carbon electrode and then dried at 60 ^{o}C. The electrolyte used was 3M KOH aqueous solution. The capacitive performance of the sample was tested on the CHI608A electrochemical workstation with cyclic voltammetry and chronopotentiometry functions using three-electrode system (a saturated calomel electrode (SCE) as the reference electrode, a Pt electrode as the counter electrode).

3. RESULTS AND DISCUSSION

To further investigate the image of the graphene, FESEM was adopted. In Fig. 1 the FESEM image of highly oxidized graphite shows a smooth surface with tightly packed sheets, while the exfoliated sample exists as transparent, wrinkled sheets, indicating these layers are exfoliated to a very large extent. They are well organized. The morphology is suited for the supercapacitor.

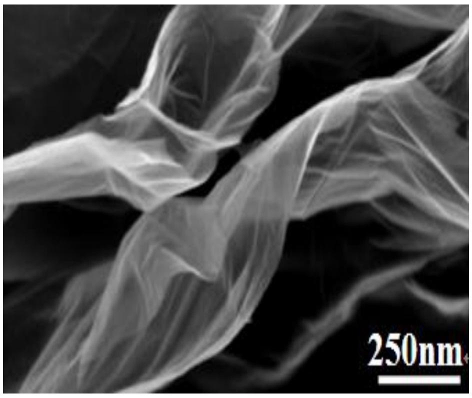

Figure 1. The SEM image of graphene particles

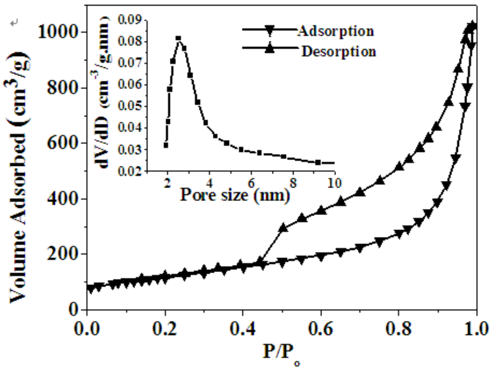

Figure 2. Nitrogen sorption isotherm of graphene at 77K, inset shows the pore size distribution

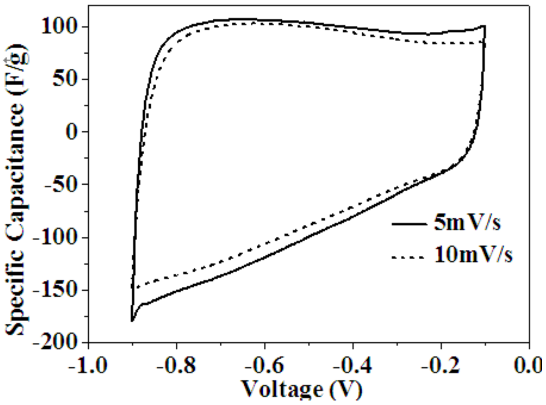

Figure 3. Cyclic voltammograms of the graphene-based supercapacitor

The as-prepared graphene sheets were characterized via nitrogen absorption technology in order to investigate the surface area and pore structure. As shown in Fig. 2, the isotherm exhibits a typical mesoporous shape. The graphene has a specific area of 415 m^2/g by BET calculation between 0.05 and 0.3 P/P_o. The total pore volume is 1.53 g/cm^3. From the BJH method, we know that the pore size distribution of the mesopores inset is mainly centered around 2~4 nm, indicating that the graphene layers interact with each other form a porous structure. The structural characteristics and high conductivity of graphene hold the potential as materials for energy storage based on the ion adsorption mechanism and have been widely demonstrated.

Fig. 3 shows the cyclic voltammograms of the graphene-based supercapacitor at scan rates of 5 mV/s and 10 mV/s, respectively. Cyclic voltammetry curves at scanning rates of 5 mV/s and 10 mV/s in a potential range of -0.1 V to -0.9 V. The electrochemical properties were tested in an aqueous KOH electrolyte, and relatively good supercapacitor performance was exhibited due to the large surface area, high electric conductivity, and high surface area. When evaluated for supercapacitive performance, the specific capacitance is between 185 F/g and 100 F/g at 5 mV/s [12].

4. CONCLUSIONS

In summary, graphite oxide was synthesized via Hummers method with flake graphite as raw material. graphene particles have been prepared by a simple and cost-effective approach. The structure of highly oxidized graphite shows a smooth surface with tightly packed sheets. The good structure and excellent performance suggest its promising application in supercapacitors.

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