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Pomelo peels-derived porous activated carbon microsheets dual-doped with nitrogen and phosphorus for high performance electrochemical capacitors

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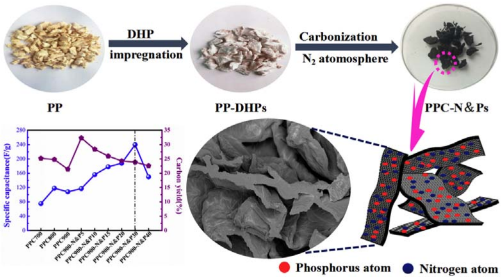
HIGHLIGHTS

- Porous activated carbon nanosheets fabricated by biowaste mass of pomelo peels.
- Dual-doped with nitrogen and phosphorus elements by using ammonium phosphate.
- High specific surface area of 807.7 m^2/g and large pore volume of 0.4378 cm^3/g.
- A capacitance of 240 F/g with 100% of capacitance retention after 10,000 cycles.
- Symmetric electrochemical capacitor device exhibits an energy density of 11.7 Wh/kg.

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, biomass pomelo peel is used to fabricate the porous activated carbon microsheets, and diammonium hydrogen phosphate (DHP) is employed to dual-dope carbon with nitrogen and phosphorus elements. With the benefit of DHP inducement and dual-doping of nitrogen and phosphorus, the prepared carbon material has a higher carbon yield, and exhibits higher specific surface area (about 807.7 m^2/g), and larger pore volume (about 0.4378 cm^3/g) with hierarchically structure of interconnected thin microsheets compared to the pristine carbon. The material exhibits not only high specific capacitance (240 F/g at 0.5 A/g), but also superior cycling performance (approximately 100% of capacitance retention after 10,000 cycles at 2 A/g) in 2 M KOH aqueous electrolyte. Furthermore, the assembled symmetric electrochemical capacitor in 1 M Na_2SO_4 aqueous electrolyte exhibits a high energy density of 11.7 Wh/kg at a power density of 160 W/kg.

1. Introduction

Since the early 1950s, there has been significant attention on the development of electrochemical capacitors due to their high charging and discharging efficiency, long cycle life, and high power density. [1–3]. The electrode material is an important component of electrochemical capacitors, including carbon material [4,5], transition metal oxide/nitride (such as NiO, VN [6,7]), and a conductive polymer [8].

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The metal oxide/nitride and conductive polymer provide a large pseudocapacitance, but the cycle stability is poor [9]. In contrast, carbon materials have excellent electrical properties, good physical and chemical stability, and high specific surface area, and thus they are currently the most commonly used electrode materials of electrochemical capacitors. Nowadays, there are some novel routes for fabricating electrode materials, such as crystal-structure modification [10], heterostructured nanocomposites [11], experimental, simulation-based [12], and so on. In addition, a study on C/Si_3N_4 composites [13] and some other applications of the materials such as capacitor, intelligent sensor [14–17] have been proposed, which further expanded the application of carbon composites materials.

The material sources for the preparation of carbon materials are quite extensive, such as polymers [18,19], which expand the usage of recycled polymers for multifunctional nanocomposites preparation with various fillers [20–22] and different hosting polymer matrices [23–26]. Due to the shortage of fossil resources such as oil and coal, the use of waste biomass sources prepared carbon materials for energy applications, have become the focus of researchers due to the low cost and environment-friendly properties. There are many works that have utilized various waste biomass to derive carbon materials, such as fungi [27], pistachio shells [28], banana peels [29,30], cassava peels [31], seaweed [32], firewood [33], and so on, which have exhibited excellent electrochemical properties as electrode materials for electrochemical capacitors. In comparison, the pomelo peel is a widely distributed biomass precursor material. Up to now, there are some reports have utilized the abandoned pomelo peel to synthesize carbon materials by KOH activation for energy applications [34–36]. The prepared carbon materials have a high specific surface area, however, the yield of carbon materials is always ignored in these works. Furthermore, the research showed that cellulose was a linear homopolymer of the glucopyranose residue linked by the β-1, 4-glycosidic linkage, which was the most important component of the biomass [37]. It was confirmed that levoglucan was the main product in the cellulose pyrolysis process (accounting for more than 50% by weight), and it was easy to volatilize at high temperatures [38]. Hence, it needs to be pointed out that the carbon yield was crucial for utilized various waste biomass to derive carbon materials. Recently, some reports showed that addition of acid would inhibit the formation of levoglucose during the pyrolysis process [39,40].

In this work, we used the biomass pomelo peel to prepare porous activated carbon microsheets and used diammonium hydrogen phosphate (DHP) to introduce dual-doping of nitrogen and phosphorus via one-step carbonization under N_2 atmosphere. The decomposition of DHP could cause phosphorylation reactions [41] in pyrolysis process, which can effectively reduce the formation of levoglucose and enhance the formation of char [40]. Therefore, the carbon yield could be effectively improved. Meanwhile, the prepared carbon material would exhibit high specific surface area and uniform pore size distribution due to the inducement of the DHP-incorporated carbonization. Furthermore, DHP is an inorganic salt containing abundant nitrogen and phosphorus elements, which could effectively dope the carbon framework with nitrogen and phosphorus atoms. Which would largely improve the wettability of the carbon-based material in the aqueous electrolytes. Therefore, as the electrode material of electrochemical capacitors, the prepared carbon microsheets would exhibit good electrochemical performance.

2. Experimental

2.1. Chemicals and materials

Pomelo peel (PP) was recycled from pomelo after being washed with the mixed solvent of deionized water and alcohol and dried under vacuum at 60 °C overnight. Diammonium hydrogen phosphate (DHP) of analytical grade was purchased from *Sinopharm Chemical Reagent Co. Ltd.*

2.2. Preparation of dual-doped carbon

i) In a typical synthesis, the obtained dry PP (4 g) was heat-treated under N_2 atmosphere in a tube furnace at the temperatures of 700–900 °C for 2 h in a flow of 70 sccm (the abbreviation of standard cubic centimeter per minute). Then the sample was naturally cooled down to room temperature to obtain normal carbon material.
ii) For further modification, the dry PP (4 g) was treated with DHP aqueous solution for 24 h, followed by wringing and then drying under a vacuum at 60 °C for 48 h. The DHP treated pomelo peel was heat-treated at 900 °C for 2 h in a tube furnace with an N_2 flow of 70 sccm. Finally, the dual-doped porous activated carbon was fabricated. The ratio of the remaining carbon of the samples was calculated according to the following equation:

$$r_{\text{carbon}}\ (\%) = W_b/W_a \times 100\% \qquad (1)$$

Where W_b (g) is the quality of final samples after carbonization, and W_a (g) is the quality of pomelo peel after being washed and vacuum dried.

2.3. Materials structural characterization

The morphology and microstructure of the fabricated materials were characterized using a HITACHI S-4800 scanning electron microscope (SEM) and transmission electron microscope (TEM, JEOL, JEM-2010, Japan). The X-ray photoelectron spectroscope (XPS, physical Electronics UK) was used to analyze the chemical composition of the samples. The Raman spectroscopy (Horiba Scientific, France, excitation-beam wavelength = 532 nm) was used to characterize the properties of materials. The specific surface area and pore structure of the samples was characterized by Nitrogen adsorption and desorption experiments at 77 K (Micromeritics, ASAP 2010M,USA). The specific surface area was calculated from the nitrogen adsorption isotherm using Brunauer-Emmett-Teller (BET) method and the Non Local Density Functional Theory (NLDFT) model was used to analyze the pore size distribution of samples.

2.4. Electrochemical performance

The working electrodes that were used for electrochemical measurements were prepared as follows: the fabricated carbon samples, carbon black, conductive graphite, and PTFE were mixed at a weight ratio of 80: 7.5: 7.5: 5 to make a homogeneous black paste and then coated on the surface of nickel foam, which was used as a current collector. The electrode was dried under vacuum at 60 °C for 6 h, and compressed at a pressure of 10 MPa to minimize the loss of electroactive materials during the electrochemical testing process. The performance of electrochemical properties was tested using cyclic voltammetry (CV), galvonostatic charging-discharging (GCD) and electrochemical impedance spectroscopy (EIS) in an electrochemical workstation (CHI660E, Shanghai, China). The cycling stability test was performed using LAND CT2001A instrument.

*Regarding the Single Electrodes Test:*Electrochemical measurement was conducted at room temperature in a traditional three-electrode configuration containing the working electrode, a platinum gauze counter electrode, and a saturated calomel reference electrode. The alkaline solution (2 M KOH) was used as the electrolyte for electrochemical tests. Cyclic voltammetry was recorded between −1.0 and 0 V at different scan rates from 5 to 100 mV/s. For galvonostatic charging-discharging measurement, the current densities were varied from 0.5 to 10 A/g within the same potential range as in cyclic voltammetry measurements. The electrochemical impedance spectroscopy spectrum was acquired with a frequency range from 10^{-2}–10^5 Hz.

Regarding the Symmetric Electrochemical Capacitor Test: The solid-

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