



Preparation and supercapacitive performance of clew-like porous nanocarbons derived from sucrose by catalytic graphitization



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ABSTRACT

Highly graphitic clew-like porous nanocarbons (GCPNs) were prepared by using sucrose and nickel acetate as the carbon source and nickel catalyst precursor, respectively. The influences of the amount of nickel acetate on the microstructure, morphology, degree of graphitization, surface area and pore texture of GCPNs were investigated by scanning and transmission electron microscopy, power X-ray diffraction, Raman spectroscopy and Brunauer-Emmett-Teller measurement, respectively. Electrochemical performance of GCPNs was studied by cyclic voltammetry, galvanostatic charge-discharge, and electrochemical impedance spectroscopy measurements in 6 molL^{-1} KOH aqueous electrolytes. GCPNs exhibit interestingly novel clew-like nanostructures, which are composed of interconnected and curled carbon nanowires with sizes ranging from 10 to 25 nm. Especially, GCPN-2 (the mass ratio of nickel acetate to porous activated carbon was 0.22) shows the largest specific surface area of $691\text{ m}^2\text{ g}^{-1}$, the highest degree of graphitization (0.81) and the lowest equivalent series resistance (ESR) of $0.53\ \Omega$, which ensures sufficient electro-active sites and rapid charge-discharge rates. In addition, GCPN-2 exhibits the hierarchical micro-meso porous architecture which favors the fast diffusion of electrolyte ions. Consequently, GCPN-2 delivers a high specific capacitance (248 F g^{-1} at 0.5 A g^{-1}), a high rate performance (210 F g^{-1} at 5 A g^{-1}) and an excellent cycling stability (94.3% retention after 5000 cycles at 0.5 A g^{-1}), demonstrating that GCPN-2 would be a promising electrode for supercapacitors.

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1. Introduction

In recent years, promising researches have been conducted in the fields of supercapacitors [1–2]. Because of their higher charge-discharge efficiency, faster charge capability, long cycle life, environmental friendliness, and product safety, supercapacitors have been considered as attractive power sources for digital communication, memory backup devices, electric vehicles, and a number of day-to-day electronic equipment [3–5].

Carbon materials are well known as the first candidate electrode materials for supercapacitors due to their high specific surface area accompanied with relatively high electrochemical stability and excellent electronic conductivity [6–9]. Various carbon materials, including activated carbons (ACs), carbon nanotubes (CNTs), and carbon gels (CGs), have been utilized as the electrode materials for supercapacitors. Among these, ACs with their lower cost, higher specific surface area, and larger production

volumes are considered as the choice for commercial supercapacitors [10]. Sucrose is a particularly attractive AC precursor due to its uniform structure, environmentally friendly, very low price and high chemical purity, which might be considered as the new generation of commercial supercapacitors [11].

Generally, the electrical energy of carbon supercapacitor mainly stored between the carbon electrode and electrolyte interface. The large surface area of carbon can help to enhance the capacitive performance. However, specific capacitance is sometimes not directly proportional to the surface area [12]. The small micropores of carbons limit their accessibility to electrolytes during the charge-discharge process, especially in the case of a relatively high loading current density. Therefore, surface areas of micropores cannot be efficiently utilized, leading to undesirable capacitance of carbon electrode materials in electrolytes. While mesoporous carbons are favorable for the high transportation speed of electrolyte ions due to their larger pore sizes, but they have insufficient capacitance compared with microporous carbons. Thus, a carbon with hierarchical micro-meso porous structures is expected to be a superior electrode material for supercapacitors [13].

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Porous carbons with graphitic or highly graphitic structure attract extensive attentions in electrode materials because of their high surface area, efficient electronic conductivity, and excellent mass transport. Conventional methods for the preparation of graphitic carbon often require high temperature treatment, normally higher than 2000 °C [14]. However those processes with high energy consumption lead to a significant reduction in the surface area and pore volume of the porous carbon. Graphitic carbon can be also prepared by means of catalytic pyrolysis of carbon precursors in the presence of transition metals at relatively low temperature (<1000 °C). This method is called catalytic graphitization, which reduces energy loss and makes amorphous carbons transform into crystalline materials at moderate temperature [15]. Therefore, porous carbon with graphitic character prepared by catalytic graphitization is highly desirable to enhance their electrochemical performance.

In this paper, highly graphitic clew-like porous nanocarbons (GCPNs) were prepared by using sucrose and nickel acetate as the carbon source and nickel catalyst precursor, respectively. The effects of the mass ratios of nickel acetate on graphitization, pore feature and electrochemical performance were intensively investigated. As-prepared GCPN-2 combines the features of high specific surface area ($\sim 691 \text{ m}^2 \text{ g}^{-1}$), hierarchical micro-meso porous architecture, a twisted clew-like nanostructure and a highly graphitic structure with a low equivalent series resistance (ESR) of 0.53Ω , which exhibits sufficient electrochemical activity and rapid charge-discharge feature. The GCPN-2 presented a high specific capacitance of 248 F g^{-1} at 0.5 A g^{-1} , a high rate performance of 210 F g^{-1} at 5 A g^{-1} and an excellent cycling stability of 94.3% retention after 5000 cycles at 0.5 A g^{-1} , showing a promising application in supercapacitors.

2. Experimental

2.1. Materials preparation

All reagents in the experiment were of analytical grade and were used without further purification. In a typical procedure, first, 10.2 g of sucrose was dissolved in 40 ml distilled water and transferred into a 80 mL Teflon-lined stainless-steel autoclave for a hydrothermal treatment at 180 °C for 12 h. Afterwards, the resulted hydrothermal carbon was carbonized at 800 °C for 2 h with a heating rate of $5 \text{ }^\circ\text{C min}^{-1}$ in a purified nitrogen flow, and further activated in mixed gas of oxygen and nitrogen (the volume percent of oxygen is 5%) at 450 °C for 1 h with a heating rate of $4 \text{ }^\circ\text{C min}^{-1}$ to obtain porous activated carbon (PAC). The prepared PAC was then impregnated with aqueous solution of nickel acetate (the mass ratios of nickel acetate to PAC were 0.12, 0.22, and 0.32) and then dried at 100 °C. Then, the impregnated PAC was heated to 900 °C ($5 \text{ }^\circ\text{C min}^{-1}$) under N_2 flow to obtain Ni/graphitized nanocarbons composite. Finally, the Ni/graphitized nanocarbons composite was washed with hydrochloric acid in order to eliminate Ni particles. The obtained graphitized nanocarbons were denoted as GCPN-1, GCPN-2 and GCPN-3, respectively.

2.2. The characterization of materials

Scanning electron microscopy (SEM) observations were obtained using a JEOL JEM-3010 SEM microscope. Transmission electron microscopy (TEM) images were examined by using a JEOL 2010 microscope (200 kV). X-ray diffraction (XRD) patterns were carried out using a D/MAX-3C diffractometer using Cu Ka

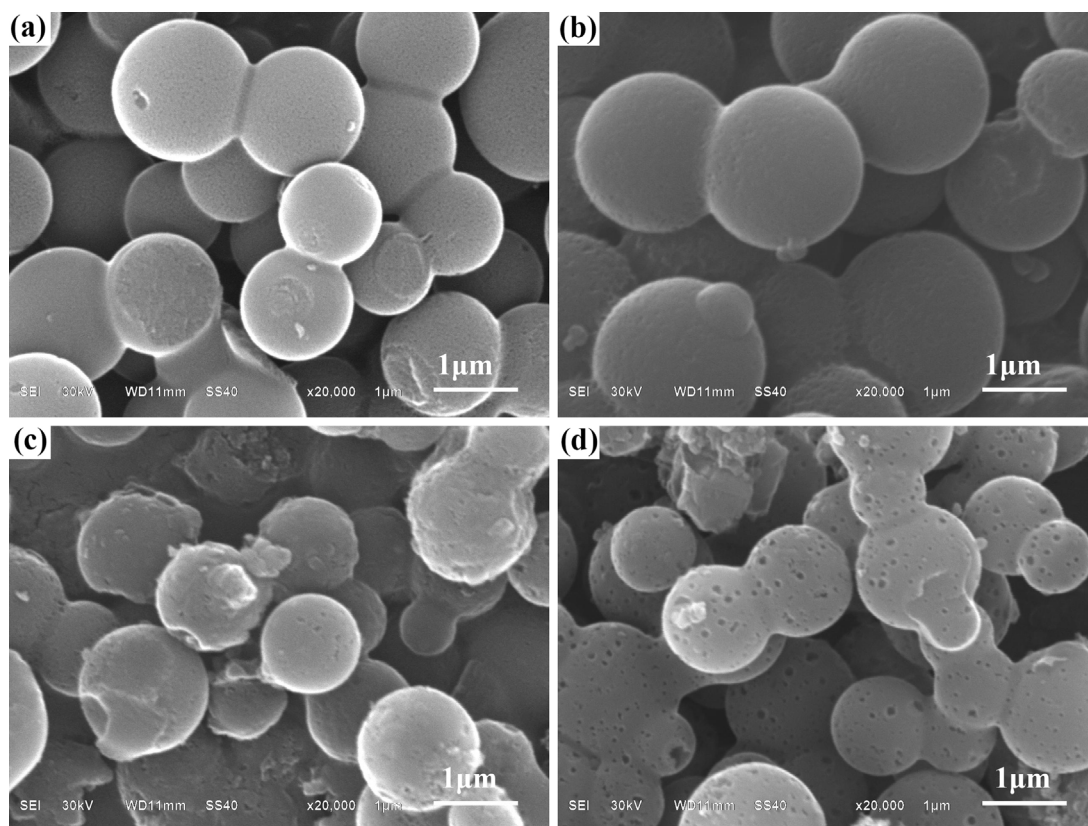


Fig. 1. SEM images of: (a) PAC; (b) GCPN-1; (c) GCPN-2 and (d) GCPN-3.

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