



Hierarchical nitrogen-doped porous carbon with high surface area derived from endothelium corneum gigeriae galli for high-performance supercapacitor



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ARTICLE INFO

Article history:

Received 27 December 2013

Received in revised form 28 February 2014

Accepted 2 March 2014

Available online 15 March 2014

Keywords:

Endothelium corneum gigeriae galli

Nitrogen-doped porous carbon

Supercapacitor

High specific surface area

ABSTRACT

Endothelium corneum gigeriae galli derived 3D hierarchical nitrogen-doped porous carbon was for the first time prepared by preliminary carbonization at 450 °C and final KOH activation at high temperatures. The surface and structural properties of the as-synthesized samples are analyzed with Brunauer–Emmett–Teller surface analyzer apparatus, X-Ray Diffractometer, scanning electron microscopy, transmission electron microscopy, X-ray photoelectron spectrometer. The electrochemical performances are analyzed by cyclic voltammetry, galvanostatic charge/discharge cycling and electrochemical impedance spectroscopy. The obtained results show that the sample carbonized at 900 °C possesses the SSA of 2149.9 m² g⁻¹, average micropore diameter of 1.78 nm, and exhibits the highest initial specific capacitance of 198.0 F g⁻¹ at current density of 1 A g⁻¹ in 6 M KOH solution. It retains good specific capacitance retention of 91.6% after 3000 charge/discharge cycles at current density of 2 A g⁻¹.

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1. Introduction

The current and future demands for green energy production and proper storage systems are higher than ever for sustainable development owing to severe environmental issues and the fossil energy crisis. Renewable, sustainable energy harvesting normally relies on non-continuous power sources such as solar, wind, hydro or geothermal. Therefore, the successful implementation of such technologies is therefore strongly depend on the development of more efficient energy storage systems.

Supercapacitors are electrochemical capacitors that store electricity through reversible adsorption of positive and negative ion onto active electrode materials that have high specific surface area (SSA) [1,2]. Supercapacitors have attracted increasing attention due to their long life cycle (> 10⁵), highly reversible charge storage process and specific power density along with increased

concern over the exhaustion of natural resources [3,4]. Moreover, supercapacitors are found wide application in the recovery of braking energy in numerous vehicles (cars, buses, trains, etc.) [5], portable electronics and the emergency exits of the Airbus A380 [6,7].

For a high-performance supercapacitor, electrode materials with high SSA are required to accommodate a large number of electrolyte ions at the electrode/electrolyte interface thereby promoting the electrical double-layer capacitance [8,9]. Although graphene-based materials have been recognized as promising active materials for supercapacitors due to its outstanding electrical conductivity and large surface area [10], present mainstream of electrode materials in commercial supercapacitors is almost entirely dominated by biomass-derived porous carbon because of its low manufacturing costs, abundant raw materials, and energy/environmental concerns [11]. These porous carbons are generally produced by pyrolysis or carbonization of various biomass followed by physical (CO₂, steam, microwave) and chemical activation (KOH, K₂CO₃, ZnCl₂, etc.) to enhance the surface area and introduce porosity for making the materials functionally more effective [12–15].

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To date, various biomass-derived porous carbons have been investigated for supercapacitor electrodes with high capacities and good capacity retentions. Among these research, plant-based biological wastes (phytomass) such as coconut [16], corn grain [17], lignocellulosic materials [18], seaweeds [17,19], tea leaves [20], celuce leaves [21], tree leaves [22], watermelon [23], rice husk [24], are the primary raw sources for porous carbon materials. More recently, researchers have started utilizing animal biomass (zoomass) for the synthesis of hierarchical porous carbons for high-performance supercapacitor electrodes. Fish scale was used to prepare hierarchical lamellar porous carbon material with attractive electrochemical properties, which demonstrates that fish scale is a promising candidate precursor to prepare low cost but high performance porous carbon material [25]. Another novel hierarchical porous carbon material was obtained from the carbonization of the animal bone after KOH activation [26]. Electrochemical studies revealed superior electrochemical performances of the porous carbon in KOH electrolyte. Interestingly, a high-capacity carbon for the first time was prepared from renewable chicken feather for supercapacitor electrode materials, which provides an economical and environmentally friendly synthesis route for porous carbon [27].

However, until now there have been only a few reports in which phytomass or zoomass are used as the precursors to synthesize nitrogen-doped porous carbons for enhancing the specific capacitance via the additional pseudo-capacitance of nitrogen functionalities [28,29]. Endothelium corneum gigeriae galli (ECGG) is a sort of biowaste from inner wall of chicken gizzard. It mainly contains pepsin, amylase, types of keratin, glutamic acid and kinds of amino acid, etc. [30]. World-widely, most people eat the fleshy chicken gizzard whereas the ECGGs are usually discarded, although it may be collected for traditional chinese medicine in China.

In this work we for the first time demonstrate the synthesis of ECGG-derived nitrogen-doped porous carbons with high SSA and abundant porosity. The physical properties such as SSA, pore size distribution, nanostructure, and surface functionalities, were analyzed as a function of the carbonization temperature in detail. The electrochemical performances of the resultant porous carbon based supercapacitor electrodes were also systematically investigated in 6 M KOH electrolyte.

2. Experimental

2.1. Preparation of ECGG-derived porous carbons.

ECGG used in the experiments was collected from poultry market in Guangzhou higher education mega center. Firstly, ECGG was washed with distilled water and dried at 100 °C for 12 h under air atmosphere. Subsequently, the ECGG was pulverized by an electric disintegrator. The resultant ECGG powder was placed in ceramic corundum boat, then transferred into an atmosphere furnace, and preliminarily carbonized at 450 °C for 2 h with a heating rate of 3 °C min⁻¹ and an Ar₂ flow rate of 25 mL min⁻¹. Preliminary carbonized products were mixed with solid KOH with a weight ratio of 1:1 and homogeneously gridded in an agate mortar. Thereafter, the mixture was loaded into a nickel combustion boat, then transferred into the atmosphere furnace, and activated at 800, 900, and 1000 °C for 2 h with a heating rate of 3 (< 600 °C) and 5 °C min⁻¹ (> 600 °C). The activated samples were alternatively washed by 15% HCl and deionized water and dried at 110 °C for 12 h. The final ECGG-derived porous carbons were denoted as ECGG-800, ECGG-900, and ECGG-1000, respectively.

2.2. Characterization of ECGG-derived porous carbons

SSAs and pore size distribution of the resultant carbons were investigated by using N₂ adsorption–desorption measurement on the Brunauer–Emmett–Teller (BET) apparatus (Micromeritics ASAP 2020 M) at liquid nitrogen temperature of 77 K. Pore size distributions were evaluated from the nitrogen adsorption by applying a hybrid nonlocal DFT (NLDFT) method, assuming a slit pore geometry for the micropores and a cylindrical pore geometry for the mesopores. The powder X-ray diffraction (XRD) patterns were recorded using a Bruker D8 advance diffractometer with monochromatic Cu K α radiation (40 kV, 20 mA) in the 2 θ angular regions between 10 and 90 °. Field emission scanning electron microscopy (FE-SEM) (ZEISS Ultra 55) and transmission electron microscopy (TEM) (JEM-2100HR) were applied to investigate the nanostructure of the ECGG-derived porous carbons. The surface functional groups of the ECGG-derived porous carbons were characterized by X-ray photoelectron spectroscopy (XPS, ESCALAB 250, Thermo-VG Scientific) with a monochromatic Al K α X-ray source (1486.6 eV photons). The binding energy was calibrated by means of the peak energy of C 1s at 284.8 eV, and the elemental compositions were determined from peak area ratios after the correction for the sensitivity factor for each element.

2.3. Electrochemical performance of ECGG-derived porous carbons

Cyclic voltammogram (CV) curves at different scanning rates and galvanostatic charge/discharge tests at various current densities were carried out on a CHI 606A electrochemical workstation (Shanghai Chenhua Instruments Co., China) using a three-electrode electrochemical cell with a Pt counter electrode and a reference electrode of Hg/HgO electrode in 6 M KOH solution between -1 and 0 V at room temperature. The working electrodes were prepared by loading a slurry containing ECGG-derived porous carbons (80 wt %, 12 mg), poly(vinylidene fluoride) (10 wt %, PVDF), and acetylene black (10 wt %) in N-methylpyrrolidone onto a nickel foam. After the electrode materials were loaded, the working electrode was pressed and dried in a vacuum oven at 60 °C for 24 h. The electrochemical impedance spectroscopy (EIS) measurements were carried out on uAUTOLABIII (Metrohm) in a frequency range from 100 kHz to 0.1 Hz with ac perturbation of 5 mV. The EIS data were analyzed using Nyquist plots consisted by Y axis of the imaginary part (Z'') and X axis of real part (Z') of impedance.

3. Results and discussion

FE-SEM images of the surface morphologies of the as-prepared ECGG-derived porous carbons are shown in Fig. 1. It can be seen from Fig. 1a, at low carbonization temperature of 800 °C, the ECGG-800 is of a relatively larger particle size composed of interstitial macropores decorated with a number of meso/micropores on the surface. For the sample of ECGG-900, it is apparently of hierarchical nanostructure where the inner wall of macropores decorated with abundant honeycomb-like interconnected mesopores. As the carbonization temperature reaches 1000 °C, heat treatment leads to the collapse of nanopores while hierarchical meso/micropores maintained. Obviously, the carbonization temperature significantly affects the porosity and surface structure of the resulting carbons, indicating that the pore structure development and pore size distribution can be tailored by varying the final heat temperature. TEM image (Fig. 1d) reveals that the amorphous porous structure of the ECGG-900 with meso/macropores which consistent with the observation of SEM images. HR-TEM image (the insert of Fig. 1d) exhibits a number of white spots implying abundant micropores.

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