



Rich nitrogen-doped ordered mesoporous phenolic resin-based carbon for supercapacitors



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ARTICLE INFO

Article history:

Received 5 September 2014

Received in revised form 12 October 2014

Accepted 12 October 2014

Available online 17 October 2014

Keywords:

Nitrogen doping

Ordered mesoporous carbon

Phenolic resin

Electrochemical performance

Supercapacitors

ABSTRACT

Nitrogen-doped ordered mesoporous phenolic resin-based carbon (N-OMC) with a high nitrogen content (11.64 wt.%) and 2D hexagonal mesostructure is synthesized by the carbonization of 2D hexagonal ordered mesoporous phenolic resin with melamine as the nitrogen source. The effect of nitrogen doping on structure and porosity, and the doping configuration of nitrogen are investigated in detail. Compared with the 2D hexagonal ordered mesoporous phenolic resin-based carbon (OMC) without nitrogen doping, N-OMC shows superior capacitive performance in 1 M H₂SO₄ electrolyte. The specific capacitance of N-OMC is up to 216 F g⁻¹ at a current density of 0.1 A g⁻¹, attributed to the co-contribution of double-layer capacitance and pseudocapacitance, which is much higher than that (127 F g⁻¹) of OMC. Moreover, N-OMC shows a good rate capability (114 F g⁻¹ retained at a high current density of 20 A g⁻¹) and an excellent cycling stability (no capacitance loss over 10 000 cycles). The good electrochemical performance of N-OMC is ascribed to the nitrogen doping and the ordered mesostructure, and makes it a promising electrode material for supercapacitors.

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1. Introduction

Supercapacitors have received significant attention due to their high power density and long cycle life, and have been considered to be a promising high power energy source for portable electronic devices and electric vehicles etc [1–3]. Based on the charge-storage mechanism, supercapacitors can be divided into two classes: pseudocapacitors and electrical double-layer capacitors (EDLCs). Pseudocapacitors, using metal oxides and conducting polymers as the main types of electrode materials, store electrical energy faradaically by redox reactions, but often suffer from poor electrical conductivity and cycling stability [2,4]. EDLCs, which usually use the porous carbons as the electrode materials, store energy based on the charge separation at electrode-electrolyte interfaces [1]. Today, commercial supercapacitors are dominated by EDLCs with activated carbon as the electrode material due to the high microporosity and low cost [5,6]. However, the specific

capacitance is relatively low and an evident decrease in capacitance is observed at high current density, resulting in low energy density, which limits their further application [7,8].

Various porous carbon materials have been studied as the candidates for activated carbon [9–11]. Among them, ordered mesoporous carbons have been considered to be promising electrode materials for supercapacitors, owing to their uniform pore size and specific channels [7,12]. Among the ordered mesoporous carbons, 2D hexagonal ordered mesoporous phenolic resin-based carbon (OMC) synthesized by using triblock copolymer F127 as a template and phenolic resin as a carbon precursor through a solvent evaporation induced self-assembly (EISA) method, followed by thermopolymerization and carbonization, has attracted extensive attention, due to the simple synthesis, low cost and 2D hexagonal mesoporous structure [13–15]. Previous studies has demonstrated that 2D hexagonal ordered mesoporous structure is more favorable for ion diffusion than the isolated 3D cubic and disordered wormlike pore structure [7,16]. However, the surface of OMC is highly hydrophobic, thus only a few specific active sites are available for charge storage as the electrode material for supercapacitors [17], resulting in an unsatisfied electrochemical capacitive performance.

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Nitrogen doping is a facile and effective approach to enhance the capacitance of carbon materials, for nitrogen doping can 1) introduce pseudocapacitance through redox reaction, 2) improve the surface wettability of carbon materials to electrolyte, ensuring a complete utilization of the exposed surface for charge storage, and 3) increase the electric conductivity, acquiring a high rate performance [18–22]. Various carbon morphologies have been treated with nitrogen doping and exhibit improved electrochemical capacitive performance [23–26].

Herein, we report a facile method to prepare nitrogen-doped ordered mesoporous phenolic resin-based carbon (N-OMC) with a high content of nitrogen (11.64 wt.%) and 2D hexagonal mesostructure by direct pyrolysis of 2D hexagonal ordered mesoporous phenolic resin with melamine as the nitrogen source. Electrochemical studies show that the as-prepared N-OMC exhibits a much improved electrochemical capacitive performance in comparison with that of OMC without nitrogen doping. The effects of nitrogen doping on structure, porosity and the capacitive performance of the obtained N-OMC are analyzed in detail.

2. Experimental

2.1. Sample preparation

The synthesis process of N-OMC and OMC is illustrated schematically in Fig. 1. First, 2D hexagonal ordered mesoporous phenolic resin was synthesized by a process similar to that reported by Zhao et al. [7,13]. Then, N-OMC was prepared by pyrolysis of 2D hexagonal ordered mesoporous phenolic resin with melamine as the nitrogen source, while OMC was prepared by direct pyrolysis of 2D hexagonal ordered mesoporous phenolic resin without melamine.

Synthesis of 2D hexagonal ordered mesoporous phenolic resin: in a typical experiment, 1.0 g of F127 was dissolved in 20.0 g of ethanol, then 5.0 g of the resol precursor in ethanol solution containing 0.61 g of phenol and 0.39 g of formaldehyde [7,13] was

added under stirring. The resulting homogeneous solution was transferred to a dish and the ethanol evaporated at room temperature over 8 h. The sample was heated at 100 °C for 24 h in an oven to thermopolymerize the phenolic resin and then calcined at 350 °C under nitrogen atmosphere for 5 h with a heating rate of 1 °C min⁻¹ in the tube furnace to obtain the 2D hexagonal ordered mesoporous phenolic resin.

Synthesis of N-OMC and OMC: 1.5 g melamine was dissolved in 15 mL dimethylsulfoxide at 100 °C, then 100 mg 2D hexagonal ordered mesoporous phenolic resin was added and stirred for 10 h at 100 °C. Subsequently, the obtained homogeneous mixture was dried in a vacuum oven for 12 h at 100 °C. Resulting powder was finely ground and then calcined at 200, 350, 500, 700 °C for 2 h with a heating rate of 1 °C min⁻¹ under nitrogen atmosphere, respectively. After cooled under nitrogen atmosphere to room temperature, N-OMC was obtained. OMC without nitrogen doping was prepared by direct calcination of the 2D hexagonal ordered mesoporous phenolic resin at 200, 350, 500, 700 °C for 2 h with a heating rate of 1 °C min⁻¹ under nitrogen atmosphere, respectively, without the mixing with melamine.

2.2. Sample characterization

Powder X-ray diffraction (XRD) patterns were obtained on a D/MAX 2550VB/PC diffractometer (Rigaku, Japan) using Cu K α radiation ($\lambda = 0.154$ nm). N₂ adsorption/desorption isotherms were obtained on an ASAP 2020 (Micromeritics, USA) instrument at 77 K. Prior to the measurements, the samples were degassed at 180 °C for 8 h. The specific surface area was determined by BET method, and pore size distribution calculation was derived from the adsorption branches of the isotherms by BJH theory. Transmission electron microscopy (TEM) images were obtained on a JEOL 2100 microscope (Japan). Elemental analysis was conducted on a CHNS Vario EL instrument (Elementar Analysensysteme GmbH, Germany) by a conventional CHN combustion method. XPS analysis was performed on an ESCALAB 250Xi spectrometer

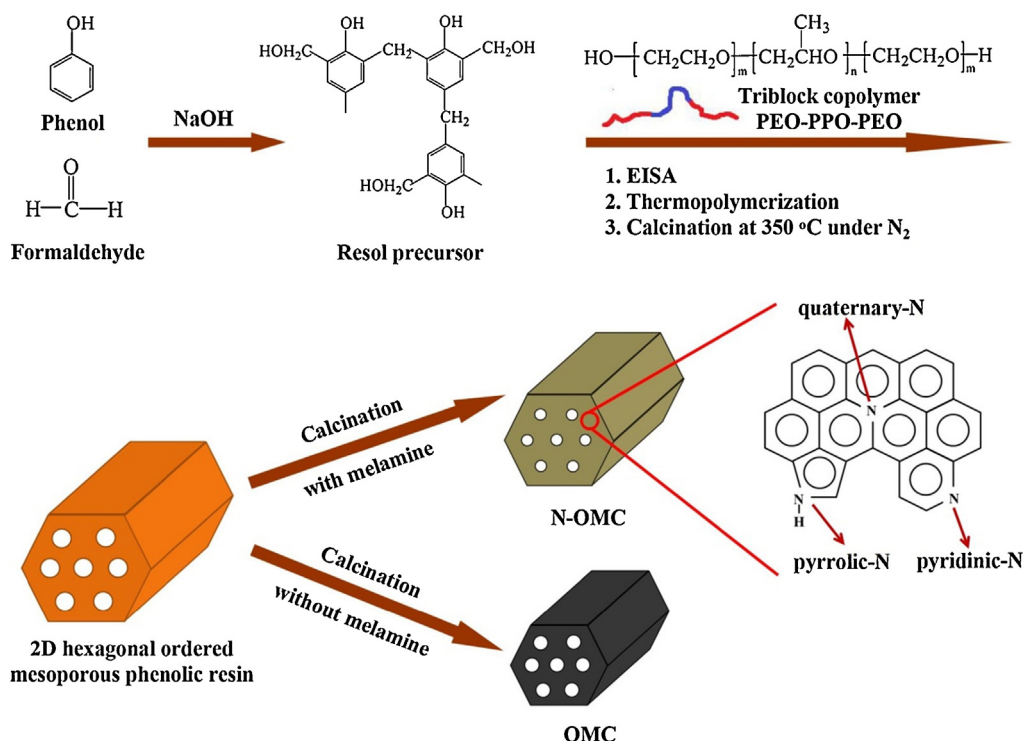


Fig. 1. Illustration of the synthesis process of N-OMC and OMC.

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