Materials Research Bulletin 64 (2015) 6–11

Contents lists available at ScienceDirect

Materials Research Bulletin

journal homepage: www.elsevier.com/locate/matresbu

Enhanced supercapacitive performance of porous activated carbon derived from polyaniline prepared by electrochemical synthesis

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ARTICLE INFO

Article history:
Received 17 March 2014
Received in revised form 21 November 2014
Accepted 1 December 2014
Available online 3 December 2014

Keywords:
A. Carbides
B. Chemical synthesis
C. Electrochemical measurements
D. Electrochemical properties

ABSTRACT

The activated carbon was prepared from polyaniline by using electrochemical synthesis, subsequent carbonization and activation under the condition of mixed oxygen and nitrogen. The electrochemical measurements result showed that the activation led to better capacitive performances. The carbon reached a high specific gravimetric capacitance of 285 $F\,g^{-1}$, with rectangular cyclic voltammetry curves at a scan rate of 2 $mV\,s^{-1}$ in 6.0 $mol\,L^{-1}$ KOH solution, and it remained 83.7% even at a high scan rate of 50 $mV\,s^{-1}$. Furthermore, the porous carbon electrodes exhibited durable operation. After 10,000 cycles, the specific capacitance is still about 98.6% of the initial specific capacitance. These demonstrated that the activated carbon would be a promising electrode material for supercapacitors.

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1. Introduction

Supercapacitors are recognized as an important alternative to the present power sources as a result of their long cycle life, high specific power and quick charge–discharge capability. These particular properties make supercapacitors suitable for many commercial applications such as power electronics, aircrafts, cranes, elevators, tramways and so on [1–3]. It is a diverse class of devices which can incorporate a variety of active electrode materials like activated carbon, conducting polymers and metal oxides [4–6]. Among the various types of materials investigated as electrode active materials of supercapacitors, activated carbon is studied extensively because of their large surface area, feasible porous structure, relatively high electrical conductivity and excellent electrochemical stability [7–9].

In general, the specific capacitance of carbon electrodes increases with carbon specific surface area. However, there is no linear relationship between the capacitance and specific surface area [10]. To improve the performance of supercapacitors, it is crucial to enhance the kinetics of ion and electron transport at the electrode/electrolyte interface [11].

Polyaniline (PANI) has been attracting significant interest due to its high conductivity, good redox reversibility and stability in aqueous solutions and air for its applications in rechargeable batteries, electrochromic displays, electrocatalysis, and sensors. It could be prepared by usual methods, such as chemical oxidation polymerization [12], self-assembly method [13], emulsion polymerization [14], template polymerization [15]. Compared with these methods, electrochemical synthesis has been extensively used to prepare functional conducting PANI due to their simple manipulation and low cost. Recently, PANI has been used as an attractive precursor for synthesis of activated carbon. Li et al. prepared the activated carbon from PANI showing a specific capacitance of 205 $F\,g^{-1}$ in 6 M KOH [16]. Xiang et al. reported a specific capacitance of 210 $F\,g^{-1}$ at a scan rate of 2 $mV\,s^{-1}$ for the activated carbon prepared from PANI base by K_2CO_3 activation for the application in supercapacitor electrodes [17]. In this paper, we prepared PANI by electrochemical synthesis, subsequent carbonization and activation under the condition of mixed oxygen and nitrogen. All the electrochemical tests demonstrated that the prepared porous activated carbon possessed the excellent electrochemical performance.

2. Experimental

2.1. Preparation of PANI

All reagents used were of analytical grade, and distilled water was used in the experiment. The PANI was electrodeposited using HDV-7C transistor potentiostatic apparatus connected to a simple two-electrode electrochemical cell. The two-electrode configuration contains two Ti sheets as the working electrode and the counter electrode. The electrodeposition was carried out in a mixed solution of 0.2 M aniline and 0.75 M H_2SO_4 by potentiostatic

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http://dx.doi.org/10.1016/j.materresbull.2014.12.002
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electrolysis with the potential of 2.20 V at room temperature. Before the electrodeposition, the Ti sheets were polished by SiC abrasive paper from 300 to 800 grits and then dipped in HCl (5%) for 10 min. After that, they were washed by distilled water and rinsed with acetone in ultrasonic bath before each experiment.

2.2. Preparation of activation carbon

The PANI was heated to 700 °C at a heating rate of 7 °C min^{-1} under nitrogen flow and maintained at this temperature for 1 h. Afterwards, the carbon material was activated by heating at a rate of 10 °C min^{-1} to 450 °C for 1 h by oxygen and nitrogen (the volume percent of oxygen is 5%). In order to facilitate the description in this paper, carbon from carbonizing PANI at 700 °C under nitrogen was marked as CC and activated carbon as AC.

2.3. The characterization of materials

Scanning electron microscope (SEM) measurements were performed on a JEM-6700F scanning electron microscope. Transmission electron microscope (TEM) measurements were performed on a JEOL JEM-20100F transmission electron microscope at all acceleration voltage of 150 kV. X-ray diffraction (XRD) patterns were recorded on a D/MAX-3C diffractometer with Cu Kα radiation ($\lambda = 1.541$ Å). The surface structure and porosity of the carbon was examined by N_2 adsorption at 77 K. The specific surface area was calculated from the N_2 adsorption isotherm using the Brunauer–Emmett–Teller (BET) equation. The pore size distribution of the samples was determined by Barrett–Joyner–Halenda (BJH) method. The surface characteristics of the samples were finished by X-ray photoelectron spectroscopy (XPS) (K-Alpha 10 63, Thermo Fisher Scientific, Britain).

2.4. Electrochemical testing

The electrode was prepared by mixing 80 wt% as-prepared samples, 10 wt% acetylene black, and 10 wt% polyvinylidene fluoride in *N*-methyl pyrrolidone to form homogenous slurry. The slurry was painted on stainless steel net, with typically 4–6 mg of activated carbon applied to each electrode. Then the electrodes were dried at 100 °C for 24 h in a vacuum oven. A sandwich-type cell was constructed from two electrodes, with similar weights facing with each other, and separated by nylon film, and the 6 mol L^{-1} KOH solution was used as electrolyte.

Electrochemical measurements were performed by CHI 660 A electrochemical workstation (CHI Inc., USA) at room temperature, and the capacitive performances were evaluated by cyclic voltammetry (CV) and galvanostatic charge–discharge cycles (GCD). Electrochemical impedance (EIS) was performed on a fully discharged cell by sweeping frequencies from 100 kHz to 0.01 Hz at amplitude of 5 mV. The cycling performance was charged and discharged in the potential range from 0.001 to 1.0 V at a constant current density of 0.5 A g^{-1} on a Neware BTS cell test apparatus.

3. Results and discussion

Fig. 1 shows the SEM and TEM images of the prepared samples. It can be seen that pristine PANI exhibits a bamboo-like structure with rough surface. From the characterization image of TEM, it can be observed that the lengths of PANI rods are between 1 and 1.5 μm, and diameters range from 250 to 300 nm. PANI nanorods cross each other, and some inner paths are joined together. As shown in Fig. 1(b), it can be seen that sample CC still exhibits the bamboo-like structure with the diameters of about 200 nm, which are smaller than that of the pristine PANI nanorods. After activation at 450 °C by oxygen and nitrogen, the activated carbon (AC) retains the general morphology of the CC. It is obvious that sample AC has the smallest diameter of about 80 nm (inset of Fig. 1(c)). After carbonization and activation, the diameter of the nanorods becomes smaller. The reasons may be attributed to the breakage of some chemical bonds and partial oxidation of carbon surface.

Fig. 2 shows the XRD patterns of the obtained carbons. The broad peak of the (0 0 2) (24°) diffraction of both carbons indicates the typical pattern of amorphous structure. For CC, the (1 0 0) peak

Fig. 1. SEM and TEM images of: (a) PANI (b) CC and (c) AC. Inset figures are the corresponding TEM images.

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