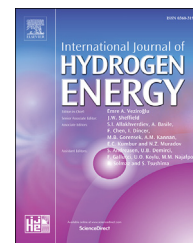




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Old-loofah-derived hard carbon for long cyclic anode in sodium ion battery

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ABSTRACT

Hard carbon was prepared via the carbonization of the old loofah sponge at 800 °C for 1 h in the inert N₂ atmosphere for sodium ion battery (SIB) anode. The resultant old-loofah-derived hard carbon was investigated by scanning electron microscope (SEM), transmission electron microscope (TEM), X-ray photoelectron spectroscopy (XPS), Raman, galvanostatic charge/discharge, cyclic voltammetry (CV) and alternating current (AC) impedance. The results suggested that the old-loofah-derived hard carbon powders consisted of many irregular micro-particles with the mean particle size of 12 μm. Furthermore, the old-loofah-derived hard carbon anode also delivered satisfactory electrochemical performances in SIB. For example, the initial discharge specific capacity was as high as about 695 mAh g⁻¹ at 25 mA g⁻¹, and the reversible discharge specific capability after 1000 cycles was still about 171 mAh g⁻¹ even at 1000 mA g⁻¹, indicating long cycle stability and the promising feasibility of the old-loofah-derived hard carbon anode. The disordered micro-structure and large interlayer distance may jointly contribute into the satisfactory electrochemical performances.

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Introduction

The irreversible depletion of fossil fuels in the earth greatly triggered the development of some promising energy storage and conversion devices such as fuel cell, solar cell, supercapacitor and lithium ion battery (LIB) [1–5]. Among them, LIB has been widely used to power the portable electronic devices due to its many advantages such as high voltage, high energy density and long cyclic life span since the successful

commercialization in 1990s [6–9]. However, there is still great concern about the potential shortage and uneven distribution of lithium resource, which may limit the further development and increase the cost of LIB [10]. Considering that Na ranks the 6th most abundant element on the earth's crust, Na⁺ may be a suitable alternative to Li⁺ due to the similar physicochemical properties and easy availability [11]. Therefore, sodium ion battery (SIB) regained the increasing attentions due to low cost, safety and environmentally benign [12]. However, it can't

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be neglected the fact that Na^+ is larger than Li^+ , which makes many current electrode active materials in LIB unsuitable for SIB any more, especially at the anode side [13]. For instance, the widely-used graphite anode in commercial LIB delivered poor Na-storage capacity in SIB because its interlayer distance (0.34 nm) was less than the critical minimum interlayer distance (0.37 nm) for Na^+ deinsertion/insertion [14]. Therefore, it is very necessary to pursue the suitable SIB anode material with larger interlayer distance for Na^+ deinsertion/insertion and accommodation [15].

It is widely acknowledged that carbon is recognized as one of the promising electrode active materials for energy storage system due to low cost, good electrical conductivity and stable physicochemical property [16]. Up to now, some carbonaceous materials such as hard carbon, carbon spheres, carbon nanofibers and carbon nanotubes have been tried to be the anodes in SIB [17–20], among which hard carbon is the most promising SIB anode material because of large interlayer distance and low degree of graphitization and disordered structure [21–23]. Generally, hard carbon can be prepared through the pyrolysis of various carbonaceous precursors in the inert atmosphere. These carbonaceous precursors mainly included the polymer, the sugar, the biomass, the petroleum pitch and the anthracite, among which the biomass-based precursors may be preferable due to their abundance, easy availability, low cost and renewability [24–28]. In other words, biomass-derived hard carbon is a promising SIB anode material in terms of sustainable development. For examples, hard carbon anode from argan shell delivered 333 mAh g^{-1} at 25 mA g^{-1} after 100 cycles [29]; and leaf-membrane-derived hard carbon anode displayed a reversible capacity of 288 mAh g^{-1} at 20 mA g^{-1} after 200 cycles [30].

Fresh loofah is known as one kind of delicious vegetable. When the loofah is old, it usually changes into the sponge consisting of the hemicelluloses and the celluloses and isn't edible any more. Sometimes, the old loofah sponge can be reused for kitchen cleaning due to its soft resilient fibers and high water uptake [31]. In principle, high content hemicelluloses and celluloses may make old loofah as an appropriate biomass precursor for hard carbon anode in SIB. However, for the moment, there is still no report about the old-loofah-derived hard carbon for SIB anode. Therefore, it is very instructive to explore the carbonization of the old loofah sponge and investigate the corresponding feasibility for SIB anode. For this purpose, the old loofah sponge was carbonized in the tubular furnace at 800 °C for 1 h under the inert N_2 atmosphere. Furthermore, the old-loofah-derived hard carbon was investigated by methods of SEM, TEM, XPS, Raman, galvanostatic charge/discharge, CV and AC impedance. Fortunately, the old-loofah-derived hard carbon anode delivered the satisfactory reversible capacity even after 1000 cycles at 1000 mA g^{-1} .

Experimental

Carbonization of the old loofah

Firstly, the old loofah was cut into small pieces and washed with deionized water. Then the old loofah pieces were

immersed into 0.1 M ZnCl_2 solution for 24 h at room temperature and dried at 60 °C. The old loofah pieces were carbonized in tubular furnace at 800 °C under the inert N_2 flow for 1 h at 5 °C \cdot min $^{-1}$. Finally, the obtained black old-loofah-derived hard carbon was washed with 2.9 M HCl and deionized water, respectively. And the clean black carbon powders can be obtained after being dried and ground.

Structure and morphology characterization

The morphology of the sample was probed in terms of SEM (TESCAN VEGA3 SBH, Czech) and TEM (JME-2100 JEOL, Japan). The corresponding chemical element component was characterized by X-ray photoelectron spectroscopy (XPS PHI5000 Versaprobe-II ULVAC-PHI, Japan). Raman spectrum was recorded on Laser Raman spectrometer (785 nm, Renishaw invia, UK).

Electrochemical measurements

The old-loofah-derived hard carbon was fully mixed with acetylene black and polyvinylidene fluoride in a weight ratio of 7:2:1 in *N*-methyl-2-pyrrolidinone for the electrode slurry, which was coated onto copper foil and then vacuum dried at 60 °C for 12 h. The corresponding loading density of the old-loofah-derived hard carbon in the anode was about 0.46 mg cm^{-2} . The electrochemical performances of the anode were investigated by assembling CR2032 coin cell with Na foil as the counter electrode and reference electrode, in which the porous glass fiber membrane was used as the separator, and the electrolyte was 1.0 M NaClO_4 and 5.0% fluorinated ethylene carbonate in the mixture of ethylene carbonate with diethyl carbonate (1:1 in volume). The coin cell was assembled in an argon-filled glove box (Mikrouna Super 1220/750/900, China) with the levels of O_2 and H_2O less than 0.1 ppm and measured by galvanostatic charge/discharge from 0.01 V to 3.0 V (vs Na/Na $^+$) on a battery testing system (Neware CT-3008W, China) at room temperature. The charge/discharge current densities were 25 mA g^{-1} , 50 mA g^{-1} , 200 mA g^{-1} , 500 mA g^{-1} , 1000 mA g^{-1} and 2000 mA g^{-1} , respectively. The corresponding electrochemical performances were evaluated on the electrochemical workstation (Parstat 4000, Princeton Applied Research, USA) in terms of CV from 0.01 V to 3.0 V at 0.5 mV s^{-1} and AC impedance from 0.01 Hz to 100 kHz with a perturbation of 10 mV.

Results and discussion

SEM

The digital photo of the old loofah was shown in Fig. 1a. Obviously, the old loofah seems like a yellow cylindrical sponge consisting of the reticular fibers. After high temperature pyrolysis in the inert N_2 atmosphere, the old loofah was carbonized and became into black carbon (in Fig. 1b). After further fully being ground, black carbon was broken into the irregular micro-particles with the mean particle size of about 12 μm , as shown in Fig. 1c.

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