



Natural sisal fibers derived hierarchical porous activated carbon as capacitive material in lithium ion capacitor



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HIGHLIGHTS

- Hierarchical porous activated carbon is derived from natural sisal fibers.
- Sisal fiber activated carbon (SFAC) is applied to LIC for the first time.
- Hierarchical porous structure benefits the electrochemical properties of SFAC.
- LIC displays high energy density of 41 Wh kg⁻¹ at the power density of 5.7 kW kg⁻¹.

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ABSTRACT

Lithium-ion capacitor (LIC) is a novel advanced electrochemical energy storage (EES) system bridging gap between lithium ion battery (LIB) and electrochemical capacitor (ECC). In this work, we report that sisal fiber activated carbon (SFAC) was synthesized by hydrothermal treatment followed by KOH activation and served as capacitive material in LIC for the first time. Different particle structure, morphology, specific surface area and heteroatoms affected the electrochemical performance of as-prepared materials and corresponding LICs. When the mass ratio of KOH to char precursor was 2, hierarchical porous structured SFAC-2 was prepared and exhibited moderate specific capacitance (103 F g⁻¹ at 0.1 A g⁻¹), superior rate capability and cyclic stability (88% capacity retention after 5000 cycles at 1 A g⁻¹). The corresponding assembled LIC (LIC-SC2) with optimal comprehensive electrochemical performance, displayed the energy density of 83 Wh kg⁻¹, the power density of 5718 W kg⁻¹ and superior cyclic stability (92% energy density retention after 1000 cycles at 0.5 A g⁻¹). It is worthwhile that the source for activated carbon is a natural and renewable one and the synthesis method is eco-friendly, which facilitate that hierarchical porous activated carbon has potential applications in the field of LIC and other energy storage systems.

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1. Introduction

With the rapid consumption of fossil fuel and changing of global climate, the urgent requirement for renewable resources and energy storage systems is necessary [1,2]. Lithium ion battery (LIB) and electrochemical capacitor (ECC) are the two most promising energy storage systems currently. LIB possesses much high energy density (100–250 Wh kg⁻¹) but limited power density (below 1 kW kg⁻¹). Conversely, ECC presents high power density (5–10 kW kg⁻¹) and excellent cyclic stability but suffers from low energy density (4–6 Wh kg⁻¹) [3]. However, the energy storage

systems with high energy density, high power density and long cycling life are required urgently in the field of electronic vehicle (EV) and hybrid electronic vehicle (HEV) [4,5].

Lithium ion capacitor (LIC) is a new type of energy storage system that bridges gap between LIB and ECC, which demonstrates higher energy density than ECC and higher power density than LIB [6]. LIC utilizes a capacitor-type material as positive electrode, a LIB-type anode material as negative electrode and a mixture of lithium salt and organic solvent as electrolyte. During charging/discharging of LIC, anions adsorption/desorption occur on the surface of positive electrode and lithium ions intercalation/deintercalation occur in negative electrode. In LIC, the pre-lithiated carbonaceous materials usually are served as negative electrode, such as graphite [7], hard carbon [8], soft carbon [9] and graphene [10,11], to reduce the potential of negative electrode and enlarge

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working voltage of LIC, thus improving the energy density. Additionally, activated carbon (AC) is widely used as positive electrode of LIC because of high specific surface area, low cost, superior cyclic stability and abundant source [12]. However, the measured specific capacitance of AC in real ECC ranges from 40 F g⁻¹ to 100 F g⁻¹ in organic electrolyte [13]. Therefore, it is necessary to develop new AC with high electrochemical performance.

There are mainly two sources for AC precursors, fossil-based carbon sources and biomass-based sources. Compared with traditional fossil-based carbon sources such as coal, pitch and coke, biomass-based sources exhibit huge advantages in practical application due to low cost and environment friendliness. In recent years, much work has been carried out to develop new carbon material with high electrochemical performance. Qian reported human hair-derived carbon used in ECC, which presented a specific capacitance of 126 F g⁻¹ in organic electrolyte containing lithium salt [14]. Ding reported peanut shell-derived carbon applied to both positive and negative electrode in sodium ion capacitor, which exhibited excellent energy density and power density [15]. As AC precursors, additionally, cellulose [16], wood [17], banana fibers [18] and so on were investigated in ECC.

As biomass materials, sisal fibers extracted from the leaf of sisal plant, are widely used in the field of transportation, petrochemical engineering and waterproofer, on account of such properties as low cost, high relative strength, biodegradability and so on [19]. Although sisal fiber-derived carbon used in LIB and lithium sulfur battery has been investigated [20,21], no one reported sisal fiber activated carbon (SFAC) served as positive electrode in LIC to my knowledge. Herein, we demonstrate that the hierarchical porous SFAC was prepared by hydrothermal and activation process. The structure and electrochemical properties of SFAC were characterized and studied in a half cell. In addition, LIC was assembled with SFAC as positive electrode and pre-lithiated mesocarbon microbeads (MCMB) as negative electrode and the electrochemical performance was investigated.

2. Experimental

2.1. Materials synthesis

Sisal fibers (SFs) were supplied by Guangxi Sisal Group Co., Ltd. The maize-yellow SFs were washed with deionized water to remove the impurities, cut into small pieces (~0.5 cm in length) and dried in the oven at 80 °C. Firstly, SFs were pre-treated by hydrothermal reaction. 3.0 g of SFs, 10 mL of 1 M sulphuric acid (H₂SO₄) solution and 140 mL of deionized water were placed in a 200-mL stainless steel autoclave. The mixture solution was heated at 180 °C for 12 h, and then cooled to room temperature. The brown prepared materials denoted as char precursor were collected after washing by deionized water, followed by centrifugation and drying in the oven at 80 °C. Secondly, the char precursor was activated using potassium hydroxide (KOH). The char precursor and different mass of KOH were grinded fully in an agate mortar. The mass ratio of KOH to char precursor (R_{KOH/C}) was designed as 0, 1, 2 and 3. The mixture was removed into a nickel boat and the process of carbonization and activation was carried out at 800 °C for 2 h under argon atmosphere and the heating/cooling rate kept at 3 °C min⁻¹. The resulting samples were thoroughly washed with 1 M hydrochloric acid (HCl) solution and deionized water until the pH decreased to 7, followed by centrifugation and drying at 80 °C in the oven. At last, sisal fiber activated carbon (SFAC) was collected for next experiment. Based on R_{KOH/C}, the corresponding SFAC was denoted as SFAC-0, SFAC-1, SFAC-2 and SFAC-3. The commercial activated carbon (YP-50F, Kuraray Chemical, Japan) signed as AC was purchased for comparison.

2.2. Materials characterization

X-ray diffraction pattern of samples was performed on X-ray diffractometer (XRD, Rint-2000, Rigaku) using Cu-K α radiation ($\lambda = 0.15406$ nm) in the range of $2\theta = 10\text{--}70^\circ$. Raman spectra was recorded by WiTec Alpha 300 system using 632.8 nm laser under ambient condition. The morphology, microstructure and energy dispersive spectroscopy (EDS) were carried out on scanning electron microscopy (SEM, Sirion 200) and transmission electron microscopy (TEM, Tecnai G12, 200 kV). The specific surface area and pore structure were measured by the nitrogen adsorption/desorption isotherms at 77.3 K using a Quadrasorb SI (Quantachrome Instruments). Prior to the measurement, all samples were degassed at 200 °C for 10 h. The specific surface area (SSA) was calculated by the Brunauer–Emmett–Teller (BET) equation. The total pore volume (TPV) was obtained at relative pressure $p/p_0 = 0.99$. The pore size distribution (PSD) was determined by a quenched solid density functional theory (QSDFT) model.

2.3. Electrochemical measurements

The positive electrode was prepared by coating the slurry of as-prepared material (80 wt%), Super P (10 wt%) as a conducting agent and poly (vinylidene fluoride) (PVDF, 10 wt%) as a binder dissolved in *N*-methyl pyrrolidinone (NMP) on aluminium foil, followed by drying overnight at 120 °C. The electrode was roll-pressed and punched into disks with 12 mm in diameter. The negative electrode was prepared by the same method, however, of which the mixture slurry was coated on copper foil containing MCMB (90 wt%), Super P (5 wt%) and PVDF (5 wt%).

The electrochemical performance of positive electrode and negative electrode (MCMB) were tested in CR2025 coin-cell with lithium metal as counter electrode, a polypropylene micro-porous film as the separator and a mixture of 1 M LiPF₆ in ethylene carbonate (EC), ethyl methyl carbonate (EMC) and dimethyl carbonate (DMC) (1: 1: 1, v/v/v) as electrolyte. Pre-lithiation process of MCMB (LMCMB) was carried out by galvanostatic discharging at 50 mA g⁻¹ after cycling (charging/discharging) at the same current density for three cycles, and terminated once the specific capacity of cell reached 270 mAh g⁻¹.

Button LIC was assembled with as-prepared material as positive electrode and LMCMB as negative electrode. The separate and electrolyte of LIC kept the same with that in half cells. The mass ratio of positive/negative electrode was designed as 1. The total active material mass of both electrodes was used for energy density and power density calculation. All cells were assembled in an argon-filled glove box. Based on different capacitive materials, the corresponding LIC was denoted as LIC-SC0, LIC-SC1, LIC-SC2, LIC-SC3 and LIC-AC. Cyclic voltammetry (CV) was performed in the potential range of 2–4 V by CHI1000C multi-channel potentiostat. Galvanostatic charging-discharging (GCD) was carried out using a Neware GCD system. Electrochemical impedance spectroscopy (EIS) was tested by a CHI 660A electrochemical workstation in the frequency range of 0.01–10⁵ Hz. All electrochemical measurements were performed at room temperature.

The specific capacitance and the specific capacity could be calculated according to the following Eq. (1) and Eq. (2):

$$C \text{ (F g}^{-1}\text{)} = I \text{ (A g}^{-1}\text{)} * \Delta t \text{ (s)} / \Delta V \text{ (V)} \quad (1)$$

$$Q \text{ (mAh g}^{-1}\text{)} = I \text{ (A g}^{-1}\text{)} * \Delta t \text{ (s)} / 3.6 \quad (2)$$

where I is current density, Δt is discharging time and ΔV is discharging voltage window except for IR drop.

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