



# Biomass-derived activated carbon materials with plentiful heteroatoms for high-performance electrochemical capacitor electrodes

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## ABSTRACT

Activated carbons for electrochemical capacitor electrodes are prepared from soyabean using chemical activation with KOH. The pore size is easily controllable by changing the mass ratio between KOH and carbonized product. The as-prepared materials possess a large specific surface area, unique structure, well-developed hierarchical porosity and plentiful heteroatoms (mainly O and N). Thus resulted in its high specific capacitance, good rate capacity and cycling stability. Moreover, attributing to worldwide availability, renewable nature and low-cost, activated carbon prepared from soyabean has a good potential in energy conversion and storage devices.

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## 1. Introduction

Electrochemical capacitor is a promising energy storage technology for applications where high power density and long-cycle life are required. Carbon materials have attracted intense interests as electrode materials for electrochemical capacitor, because of their high surface area, electrical conductivity, chemical stability and low cost [1]. Various carbonaceous materials, including carbon aerogels [2,3], carbon nanotubes [4,5], graphene [6,7], ordered mesoporous carbons (OMCs) [8,9] have been investigated as electrode materials of EDLC. However, the preparation for these carbons requires expensive and non-renewable raw materials, a lot of time and energy, and tedious preparation procedures [10]. By contrast, porous carbon materials derived from biomass are a potential choice for the construction of electrochemical capacitor electrodes owing to its worldwide availability, renewable nature and low-cost.

Up to now, many biomasses are converted into porous carbons, such as sunflower seed shell [10], broad beans [11], Rice husks [12] and collagen fiber [13,14]. However, the porous carbon derived from biomass usually suffers from limited capacitance. Incorporating heteroatoms (mainly N and O) into the carbon framework is an efficient approach for enhancing the capacitance performance of carbons [15]. The incorporated heteroatoms not only enhance the surface wettability of carbon material which would increase the surface area accessible to aqueous electrolyte, but also provide extra pseudo-capacitance

[16–19]. In order to improve the charge storage ability, an efficient strategy is to carbonize heteroatom-containing precursors directly.

Herein, a heteroatom-enriched biomass, soyabean, has attracted our attention. Soyabean, as a renewable, abundant and environmentally friendly resource, is rich in carbon, nitrogen and oxygen elements, and thus can be carbonized into heteroatom-enriched carbon materials. We employ KOH as activated agent, and the pore structure can be easily tuned by changing the ration of KOH to carbon material. Symmetrical supercapacitors have been constructed and exhibited good electrochemical performance with a specific capacitance of 248.1 F/g at current density of 0.1 A/g and excellent cycle stability with 98.75% retention of the initial specific capacitance after 10,000 cycles at a current of 1 A/g.

## 2. Experimental

### 2.1. Preparation of materials

Soyabean powders received from bean milk factory was put into a quartz boat in a tubular furnace and then heated to 400 °C and maintained for 2 h under N<sub>2</sub> atmosphere (at a ramp of 5 °C/min). The obtained black carbonized products were impregnated with KOH solution and then dried in an electric oven at 120 °C for 12 h. The dried mixture was activated in a muffle furnace under a flow of N<sub>2</sub> gas at 750 °C and maintained for 2 h (at a ramp of 5 °C/min). After cooling under nitrogen flow, the resultant was thoroughly washed with 0.1 M HCl, followed by deionized water until pH = 6.5. And then the materials were dried at 60 °C in an electric oven. The product finally obtained was designated as AC-X, where X represents the mass ratio between KOH and carbonized product.

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## 2.2. Characterization

Field emission scanning electron microscopy (NOVA NANO SEM230, JAPAN) was used to characterize the materials. Chemical analysis was performed using an EDAX energy dispersive spectroscopy (EDS) system interfaced to the FESEM. X-ray photoelectron spectra (XPS) were recorded using an X-ray photoelectron spectrometer (K-Alpha 1063) with a monochromatic Al  $K\alpha$  X-ray source. Nitrogen adsorption and desorption isotherms were obtained at 77 K using a Micromeritics ASAP 2020 system. Specific surface areas were determined according to the Brunauer–Emmett–Teller (BET) method. The pore size distribution (PSD) plot was recorded from the adsorption branch of the isotherm based on the non-local density functional theory (NLDFT) method.

## 2.3. Electrode preparation and electrochemical measurements

The as-obtained materials were mixed with acetylene black and polytetrafluoroethylene (PTFE) at the mass ratio of 80:10:10, dried to subarid, rolled to slice with 0.1 mm in depth and cut into disks with 10 mm in diameter. The electrodes were transferred into a vacuum oven and dried overnight at 120 °C. The mass of each electrode was about 5–8 mg. A sandwich type cell was constructed from two electrodes, with similar weights, facing each other and separated non-woven paper. Standard 2025 coin cell design was utilized for the electrochemical tests employing 1 M  $H_2SO_4$  as the electrolyte. Galvanostatic charging/discharging was carried out on LAND CT-2001A in the potential range of 0–0.9 V at different current densities of 0.1–20 A/g. Cyclic voltammetry (CV) measurements were performed at different constant scan rates (5–50 mV/s) from 0 to 0.9 V. The EIS

was characterized at open circuit potential in the frequency range from 100 kHz to 0.01 Hz with the amplitude of 5 mV.

## 3. Results and discussion

The as-prepared materials are first characterized by SEM. As shown in Fig. 1(a), the sample AC-2 shows plenty cavities on surface. Fig. 1(b) shows the morphology of AC-3. It was obvious that the hole size of AC-3 is bigger than that of AC-2, which might attribute to the higher mass ratio of KOH and carbonized product. As shown in Fig. 1(c), the sample AC-4 is composed of fewer holes but micrometric curves within the texture. The sufficient amount of KOH etch carbon atom to make the holes disappear and curves remain. The specific architecture can provide electrolyte for nanoscale pore on these curves and therefore reduce the diffusion distances of charges to the interior.

Typical nitrogen adsorption–desorption isotherms at 77 K for the as-prepared carbon materials are depicted in Fig. 2(a). All the samples exhibit the type I isotherm, indicating that all samples are typical microporous carbon. It is obvious that the pore volume of AC-4 is much higher than those of AC-2 and AC-3. In addition, the isotherms of AC-4 show gradual increase at relative pressure  $P/P_0 > 0.2$ , and a small hysteresis loop extending from  $P/P_0 = 0.4$  to 0.9, indicating the co-existence of mesopores [19].

Pore size distributions obtained from the adsorption branch by the BJH model of the as-prepared porous carbon are shown in the Fig. 2(b). As shown in Fig. 2(b), all samples possess plentiful micropore size distribution (below 2 nm) but AC-4 also possesses considerable mesopores with pore size in the range of 2–5 nm. As demonstrated in the previous reports, the co-existence of small mesopores

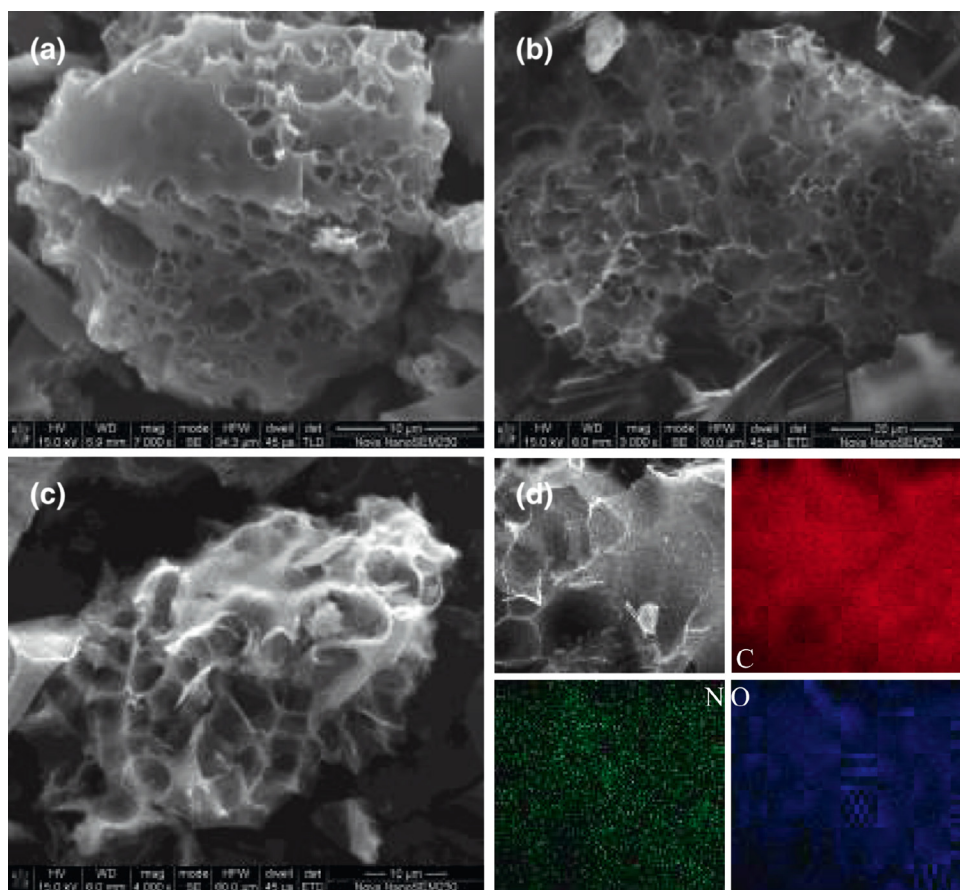


Fig. 1. (a–c) SEM images of AC-2, AC-3 and AC-4, (d) SEM images and corresponding elemental mapping images of AC-4.

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