



# Bean dregs-based activated carbon/copper ion supercapacitors



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## ABSTRACT

Bean dregs-based activated carbon/copper ion redox active electrolytes supercapacitors (BDACCIS) were prepared by combining bean dregs-based activated carbon with copper ion redox active electrolytes, which were prepared by introducing  $\text{NH}_4\text{NO}_3$  (NN) auxiliary electrolytes into  $\text{Cu}(\text{NO}_3)_2$  (CN) solutions. The physical properties of obtained activated carbon materials are characterized by scanning electron microscopy, powder X-ray diffraction, nitrogen adsorption/desorption isotherm measurements. The electrochemical properties of the constructed supercapacitor are studied by electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), galvanostatic charge/discharge (GCD) and cycle life tests. The bean dregs-based activated carbon materials exhibit interestingly well-interconnected porous structure with the large specific surface area of  $2905 \text{ m}^2 \text{ g}^{-1}$ . As-prepared BDACCIS presents a high specific capacitance ( $492 \text{ F g}^{-1}$  at  $1 \text{ A g}^{-1}$ ) and high rate performance ( $285 \text{ F g}^{-1}$  at  $20 \text{ A g}^{-1}$ ). Simultaneously, it also shows the low equivalent series resistance ( $R_s$ ) and charge-transfer resistance ( $R_{ct}$ ) of 1.05 and  $0.75 \Omega$ , considerable energy and power densities of  $51.3 \text{ W h kg}^{-1}$  and  $855.1 \text{ W kg}^{-1}$ , and satisfactory cycling stability with 81.4% capacitance retention after 10000 cycles at  $1 \text{ A g}^{-1}$  in 2 M CN + NN redox active electrolyte, demonstrating that the BDACCIS would be a promising supercapacitor.

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## 1. Introduction

The escalating power demand has ensured tremendous emphasis being placed on the search of energy storage and conversion devices with high power, high energy as well as robust in withstanding hundreds of thousands of charge/discharge cycles without degrading. Thus, supercapacitors which are also referred to as electrochemical capacitors (ECs) or ultracapacitors are considered as most promising candidates due to its uniqueness that fills the gap between traditional dielectric capacitors and batteries [1]. As we all know, according to the different electrode materials, supercapacitors can be roughly categorized into three group namely carbon-based supercapacitors, metal oxides-based supercapacitors, conducting polymers-based supercapacitors. As electrode materials, various metal oxides, conducting polymers and carbon materials were tremendous studied in the past several years [2–27,29,40–42,44,47]. Metal oxide-based supercapacitors and conducting polymers-based supercapacitors for storing energy through rapid faraday redox reactions or phase changes on the surface of active materials always display high pseudo-capacitances, but bear the problems of limited resources, low

conductivity and poor stability, which hinder its application in large-scale production.

By contrast, carbon-based supercapacitors with the unique advantages of environmental benignity, natural abundance and long cycle life are widely favored. Various carbon materials, including carbon nanotube, carbon aerogel, carbon nanofiber and graphene, have been widely utilized as the electrode materials for supercapacitors [7]. However, most of these carbon electrode materials usually yield low capacitance and suffer from harsh preparation process. Among carbon materials, particularly, utilizing sustainable biomass derived activated carbon like yeast cells [16], bean dregs [18], hemp [19], seaweed [20], dead leaves [21], egg white [22], fungus [23], soybeans [24], waste coffee beans [25], olive pits [26] and eggplant [27] as the electrode materials in the supercapacitors applications have received much attention due to its natural abundance, easy preparation and low-cost. Generally, the electrical energy of carbon-based supercapacitors mainly stored through an electrical double-layer process (non-Faradaic process) of the adsorption/desorption of electrolyte ions at electrode/electrolyte interfaces. The large specific surface areas of carbon materials often can help to enhance the capacitive performance. However, specific capacitance is sometimes not directly proportional to the specific surface areas [17]. Large specific surface areas cannot be efficiently utilized, leading to unfavorable capacitance of carbon electrode materials in

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electrolytes. Namely, low effective specific surface areas and partly harsh synthesis process restrict the wide application of carbon-based supercapacitors [24]. Based on the above facts, the preparation of supercapacitors with excellent supercapacitive performance through simple synthesis process, combining with their inimitable advantages of metal oxide-based supercapacitors, conducting polymers-based supercapacitors and carbon-based supercapacitors, are urgent to be explored.

Here, we have reported an activated carbon/copper ion supercapacitor with superior performance by using bean dregs-based well-interconnected porous activated carbon as electrodes, combining with copper ion redox active electrolytes by introducing NN auxiliary electrolytes into CN solutions. Our research results show the constructed supercapacitor exhibits high specific capacitance, excellent rate performance, small  $R_s$  and  $R_{ct}$  values, considerable energy and power densities, and preeminent cycling stability. All these results prove that as-prepared BDACCIS holds great potential in energy storage and conversion.

## 2. Experimental section

### 2.1. Materials preparation

All chemical reagents in the experiment were of analytical grade and were used without further purification. Distilled water was used in this experiment. Firstly, BD were dried for removing the intrinsic moisture and then ground into fine particles in the mortar. Next, 8 g BD fine particles were dispersed in 45 ml distilled water under vigorous stirring. After 30 min, liquid pastes were sealed into a Teflon-lined stainless steel autoclave and followed by hydrothermal treatment at 180 °C for 12 h to form bean dregs-based hydrochar (BDH). The BDH was filtered, washed with distilled water several times, and then dried in a vacuum at 100 °C for 12 h. The resulting products were treated with 2 M  $K_2CO_3$  solution (the mass ratios of BDH to  $K_2CO_3$  was 1:2) with vigorous stirring 4 h at room temperature and then drying at 100 °C until a constant weight was reached. The obtained dry powders were heated to 700 °C with a rate of 5 °C min<sup>-1</sup> in nitrogen flow and maintained at the desired temperature for 1 h. The obtained carbon materials were washed with 2 M hydrochloric acid and abundant distilled water to pH = 7 by filtration. Then it was dried at 100 °C for 12 h to obtain the bean dregs-based activated carbon by  $K_2CO_3$

activation (BDACK). As-obtained BDACK was further activated in mixed gas of oxygen and nitrogen (the volume percent of oxygen is 5%), the temperature at 420 °C with a heating rate of 4 °C min<sup>-1</sup> was held for 1.5 h to obtain bean dregs-based activated carbon using mixed gas activation (BDACG).

### 2.2. Material characterizations

The morphologies and microstructures of samples were observed on a JEOL JEM-3010 scanning electron microscope (SEM). Powder X-ray diffraction (XRD) patterns were recorded on a D/MAX-3C diffractometer using Cu K $\alpha$  radiation ( $\lambda=1.541 \text{ \AA}$ ). The surface structure and porosity of samples were estimated from the isotherms of nitrogen adsorption/desorption at 77 K. The specific surface area was calculated from N<sub>2</sub> adsorption/desorption isotherm using the Brunauer–Emmett–Teller (BET) equation. The pore size distribution of the samples was calculated based on density function theory (DFT) method.

### 2.3. Electrode preparation, supercapacitor assembly and electrochemical measurements

The electrode was prepared by mixing 80 wt.% of sample with 10 wt.% acetylene black (AB) conductor and 10 wt.% polyvinylidene fluoride (PVDF) emulsion to form slurry. The slurry was filled onto stainless steel net (diameter, 10 mm). The mass of the active material was approximate 3.0 mg for all single electrodes (the thickness of active layer on the electrode is about 0.9 mm). All the electrodes were dried in a vacuum oven at 100 °C overnight before each experiment. Then different concentrations (1 M, 2 M, 3 M, 4 M) of CN+NN redox active electrolytes were prepared by introducing NN auxiliary electrolyte to CN solutions (the molar ratio of NN to CN was 1). Similarly, the different molar ratio CN+NN redox active electrolytes with fixed 2 M CN were prepared as well (the molar ratio of NN to CN was 0.5, 1, 2, 3). Sandwich-type supercapacitors were set up with two symmetrical carbon electrodes separated by a nylon separator in copper ion redox active electrolytes.

Electrochemical tests for the supercapacitors were performed by using a symmetrical two-electrode system without a reference electrode on a CHI 660A electrochemical workstation system (CHI Inc. USA) under ambient conditions. EIS, CV, GCD and cycle life

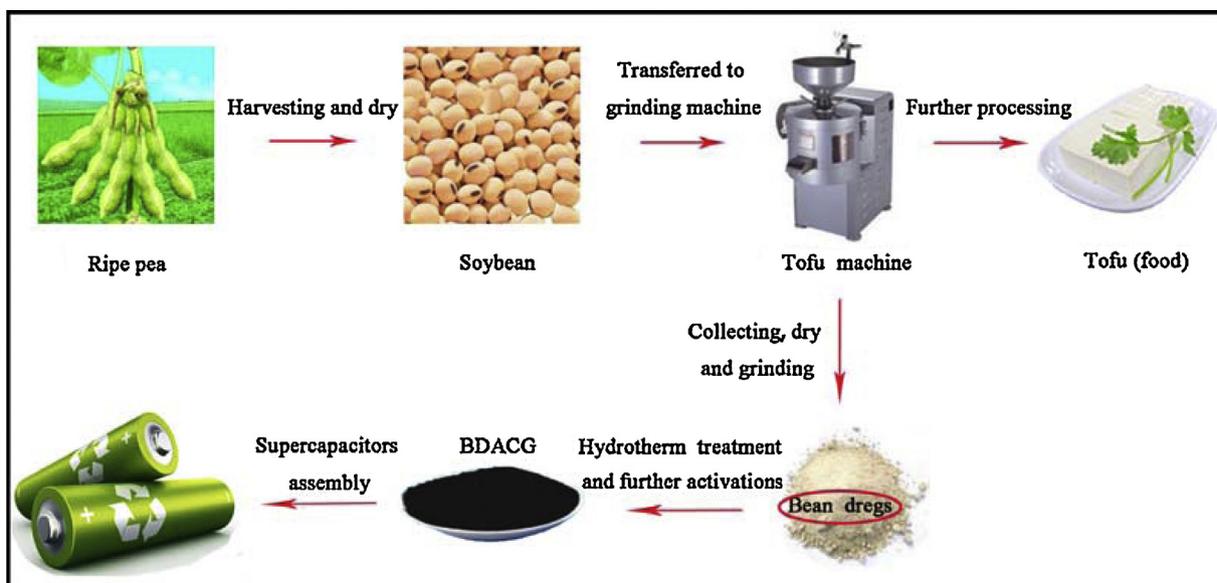


Fig. 1. Schematic illustration of the preparation routes of the supercapacitors.

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