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## Facile synthesis of carbon nanofibers-bridged porous carbon nanosheets for high-performance supercapacitors



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### HIGHLIGHTS

- PCNs were bridged by CNFs through carbonization of BC and potassium citrate.
- The composite exhibits large specific surface area.
- The composite exhibits excellent electrochemical performances.
- The symmetric device shows an ultrahigh energy density in an aqueous electrolyte.

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### ABSTRACT

A facile and one-step method is demonstrated to prepare carbon nanofibers (CNFs)-bridged porous carbon nanosheets (PCNs) through carbonization of the mixture of bacterial cellulose and potassium citrate. The CNFs bridge PCNs to form integrated porous carbon architecture with high specific surface area of  $1037 \text{ m}^2 \text{ g}^{-1}$ , much higher than those of pure PCNs ( $381 \text{ m}^2 \text{ g}^{-1}$ ) and CNFs ( $510 \text{ m}^2 \text{ g}^{-1}$ ). As a consequence, the PCN/CNF composite displays high specific capacitance of  $261 \text{ F g}^{-1}$ , excellent rate capability and outstanding cycling stability (97.6% of capacitance retention after 10000 cycles). Moreover, the assembled symmetric supercapacitor with PCN/CNF electrodes delivers an ultrahigh energy density of  $20.4 \text{ Wh kg}^{-1}$  and outstanding cycling life (94.8% capacitance retention after 10000 cycles) in an aqueous electrolyte.

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### 1. Introduction

The rapid development of portable electronic devices and hybrid electric vehicles, ever-increasing demand for clean and renewable energy sources as well as growing concerns for the environment have stimulated intensive efforts to explore advanced energy-storage and conversion devices [1–4]. Specifically, supercapacitors bridging the storage gap between batteries and conventional capacitors have been widely regarded as promising candidates for energy-storage devices due to their high power density, fast charging/discharging rates, long cycle life, low environmental impact and low maintenance cost [5,6]. Among various

materials, porous carbon materials have attracted tremendous attention as the electrode materials for supercapacitors in recent years owing to their large surface area, controllable pore size, excellent electrical conductivity, high chemical stability and low cost [7]. Porous carbon is commonly produced through carbonization of carbonaceous organic precursors under an inert atmosphere followed by physical (air,  $\text{CO}_2$  and steam) or chemical ( $\text{KOH}$ ,  $\text{H}_3\text{PO}_4$  and  $\text{ZnCl}_2$ ) activation treatment. Although residues may remain in the final product, chemical activation is preferred owing to the lower temperature, shorter time needed, higher yield and higher development of porosity for the resultant carbon materials compared with physical activation [8]. Although chemical activation process can generate a well-developed microporous/mesoporous network, the operation process is somewhat complex and time-consuming and it usually requires to use a large excess of strong corrosive chemical agents, special equipments and rigorous washing conditions, which are unfavorable for large-scale

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production and industrial applications. For these reasons, exploring a facile, economic and sustainable approach to prepare highly porous carbon materials is highly desirable but still a big challenge needed to be overcome. In recent years, direct carbonization of organic salts, such as citrates [7], potassium tartrate [9], tetrasodium salt of ethylenediamine tetraacetic acid [10] and gluconates [11], has turned out to be a facile strategy to synthesize novel porous carbon materials.

Herein, we present a facile and activation-free one-step method to prepare carbon nanofibers (CNFs)-bridged porous carbon nanosheets (PCNs) for high-performance supercapacitors through the carbonization of the mixture of bacterial cellulose (BC) and potassium citrate (Fig. 1). The potassium citrate not only serves as carbon precursor for the formation of PCNs, but also provides potassium as chemical activating agent during heat-treatment. In the resultant carbon material, the CNFs derived from BC bridge the PCNs to form three-dimensional (3D) integrated porous carbon. The as-prepared carbon material exhibits high specific surface area and excellent electrochemical performances in aqueous electrolytes.

## 2. Experimental

### 2.1. Synthesis of PCN/CNF composite

All the chemicals were of analytical grade and were used without further purification. The BC pellicles (Hainan Yide Foods Co., Ltd., China) were first cut into small pieces with a sharp blade and washed with distilled water, and then frozen in liquid nitrogen ( $-196\text{ }^{\circ}\text{C}$ ), followed by freeze-drying in a bulk tray dryer at a sublimating temperature of  $-50\text{ }^{\circ}\text{C}$  and a pressure of 0.035 mbar. Subsequently, the obtained freeze-dried BC was mixed with potassium citrate with a weight ratio of 1:5 and heat-treated in a horizontal tube furnace under a flowing  $\text{N}_2$  atmosphere up to  $850\text{ }^{\circ}\text{C}$  at a heating rate of  $3\text{ }^{\circ}\text{C min}^{-1}$  and held at this temperature for 1 h. After cooling down to room temperature, the resulting black solid was washed with HCl (10%) and distilled water for several times, respectively. Finally, the obtained PCN/CNF solid was dried in an oven at  $100\text{ }^{\circ}\text{C}$  overnight. For comparison, the CNF and PCN were also prepared through direct pyrolysis of pristine BC and potassium citrate according to the above procedure, respectively.

### 2.2. Materials characterization

X-ray diffraction (XRD) patterns were recorded on a Rigaku D/MAX-TTR III X-ray diffractometer equipped with  $\text{Cu K}\alpha$  radiation ( $\lambda = 0.15406\text{ nm}$ ). The morphology and microstructure of the as-prepared samples were analyzed using a Hitachi S-4800 scanning electron microscope (SEM) and a FEI Tecnai G2 F30 transmission electron microscope (TEM) operating at 200 kV. Raman spectra were collected with a JY HR-800 Raman spectrometer (Jobin Yvon, France) with laser wavelength of 458 nm. Nitrogen adsorption/desorption isotherms were measured on an Autosorb iQ2 automated gas sorption analyzer (Quantachrome, USA) at 77 K. The specific surface area was calculated from the Brunauer–Emmett–Teller (BET) plot of the adsorption isotherm and the pore size distribution were obtained from the non-local density functional theory (NLDFT) and Barret–Joyner–Halenda (BJH) method. Thermogravimetric analysis (TGA) was conducted on a Perkin–Elmer Diamond thermal analyzer. The electrical conductivity of the sample was measured through a two-probe method with a home-made apparatus by pressing the carbon powders into cylinders under a pressure of 10 MPa. The packing density of the sample was measured under a pressure of 10 MPa. X-ray photoelectron spectra (XPS) were determined by a PHI 5700 ESCA X-ray photoelectron spectrometer with a monochromated  $\text{Al K}\alpha$  X-ray source (1486.6 eV).

### 2.3. Electrochemical measurement

Electroactive materials, carbon black and polytetrafluoroethylene (PTFE) were mixed together with the mass ratio of 75:20:5 by adding a small amount of ethanol, coated onto the nickel foam current collector with a coating area of  $1\text{ cm} \times 1\text{ cm}$ , pressed under a pressure of 10 MPa and then dried at  $100\text{ }^{\circ}\text{C}$  for 12 h in a vacuum oven. The total material loading on each electrode was approximately 3 mg. For the three-electrode tests in the 6 M KOH aqueous electrolyte, Ni foam coated with electroactive materials was used as the working electrode, platinum foil and Hg/HgO electrode were used as the counter and reference electrodes, respectively. For the two-electrode tests, two electrodes with exactly the same sizes were symmetrically assembled with a non-woven fabric as a separator with 1 M  $\text{Na}_2\text{SO}_4$  or 6 M KOH as the electrolytes. Cyclic

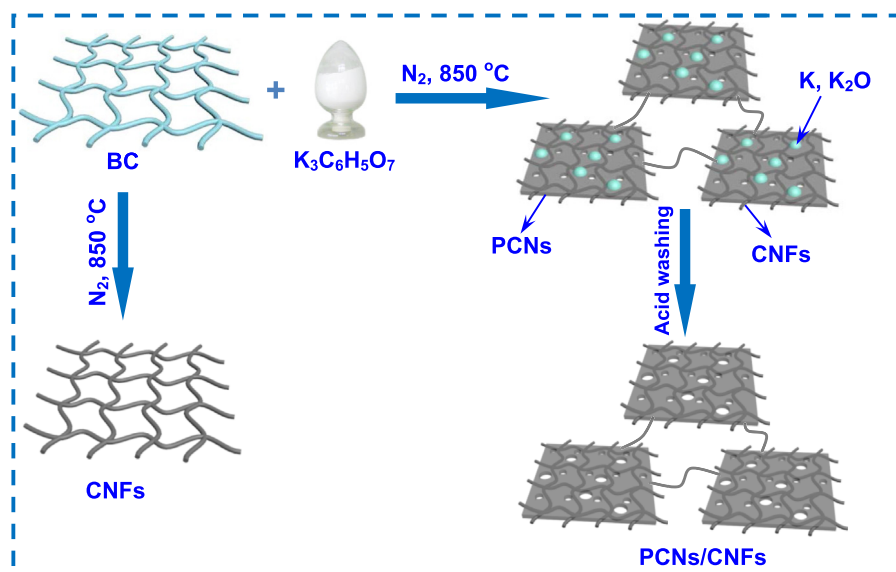


Fig. 1. Scheme illustration of the synthesis of the PCN/CNF composite.

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