



# One-step synthesis of high-performance porous carbon from corn starch for supercapacitor



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## ABSTRACT

The novel route for preparation of high-performance porous carbon from corn starch by a one-step process of chemical activation with  $\text{H}_3\text{PO}_4$  was studied in this paper. The resulting porous carbon showed a high specific surface area of  $1167 \text{ m}^2 \text{ g}^{-1}$  and a large pore volume of  $1.80 \text{ cm}^3 \text{ g}^{-1}$ . The maximum specific capacitance was up to  $162 \text{ F g}^{-1}$  at a current density of  $0.625 \text{ A g}^{-1}$ , which was higher than commercial carbon ( $100 \text{ F g}^{-1}$ ). Meanwhile, the electrode exhibited larger energy density ( $22.5 \text{ Wh kg}^{-1}$  at a power density of  $313 \text{ W kg}^{-1}$ ) and longer cycle stability with above 93% capacitance retention after 5000 cycles. The better capacitive behavior was ascribed to unique pore structure, which indicated the porous carbon will be an ideal electrode material for supercapacitor.

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## 1. Introduction

Carbon-based supercapacitor is recognized as an significant type of energy storage device duo to the higher energy density, faster charge/discharge rate and longer cycle lifetime [1,2]. Until now, the porous carbon is the most commonly used as electrode material in terms of its attractive features such as high surface area, tunable pore structure and good electrical conductivity [3,4]. However, the electrochemical performance of the porous carbon strongly depends on activation method and precursor. Compared with physical activation, chemical activation is a desirable method to obtain high-performance porous carbon from various raw materials. Generally speaking,  $\text{H}_3\text{PO}_4$  is the preferred chemical activator owing to the activation condition is milder,  $\text{H}_3\text{PO}_4$  can be recovered and the corresponding porous carbon with high yield and well-developed pore structure [5,6]. Furthermore, starch is a promising renewable resource being rich in carbon and can be obtained from lots of renewable plant resources. It is also the cheapest natural biopolymer, which can be completely biodegradable [7,8].

However, as a green, inexpensive and renewable raw material, corn starch is rarely used as carbon source to synthesize the porous carbon through directly activated by  $\text{H}_3\text{PO}_4$ . In this work, we report a novel route for preparing corn starch-based porous carbon for supercapacitor electrode material via a simple one-step

method by chemical activation with  $\text{H}_3\text{PO}_4$ . Moreover, the electrochemical performance was also investigated in detail.

## 2. Experimental

Corn starch (CS) was obtained from local supermarket (Changchun, China). Phosphoric acid (85 wt%) was purchased from Beijing Chemicals Co. Ltd., which was of analytical grade. Commercial carbon (CC) was supplied by Kuraray Chemical Company (Japan, YP-80F). A one-step process of  $\text{H}_3\text{PO}_4$  activation was adopted for preparation of the porous carbon. CS was directly impregnated with 85 wt%  $\text{H}_3\text{PO}_4$ . The impregnation mass ratios of  $\text{H}_3\text{PO}_4$  to CS were varied from 2:1 to 6:1. Then the mixtures were heated up to the activation temperature 400–600 °C for 0.5–2.5 h. Finally, the solid products were washed to neutral and dried in an oven to obtain the porous carbons. The porous carbons will be denoted according to the expression: PC/Temperature (°C)/Time (h)/ $\text{H}_3\text{PO}_4$ :CS. All the prepared porous carbons were heated to 800 °C for 1 h under  $\text{N}_2$  atmosphere.

The morphology and size were observed by a JSM-6700F scanning electron microscope (SEM). The crystal structures were examined by powder X-ray diffraction (XRD). The Brunauer-Emmett-Teller (BET) surface area and the pore structure were measured by  $\text{N}_2$  adsorption at a Micromeritics ASAP 2420 surface analyzer. The electrochemical performance measurements were carried out on a CHI 660D electrochemical workstation in  $6 \text{ mol L}^{-1}$  KOH aqueous electrolyte with a conventional three-

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electrode configuration. The working electrodes were prepared by mixing the sample, acetylene black and polytetrafluoroethylene at a mass ratio of 8:1:1. Saturated calomel electrode (SCE) and Pt foil were used as the reference electrode and the counter electrode, respectively. The cyclic voltammetry (CV) measurement was performed at the scan rates from 10 to 50 mV s<sup>-1</sup>, the galvanostatic charge/discharge (GCD) measurement was conducted at the current densities from 0.625 to 3.125 A g<sup>-1</sup>. Both CV and GCD measurements were in a potential range of -1.1 to -0.1 V (vs. SCE). The specific capacitance (C, F g<sup>-1</sup>), energy density (E, W h kg<sup>-1</sup>) and power density (P, W kg<sup>-1</sup>) were calculated from GCD measurement using equations:

$$C = \frac{i\Delta t}{m\Delta V} \quad (1)$$

$$E = \frac{1}{2}C\Delta V^2 \quad (2)$$

$$P = \frac{E}{\Delta t} \quad (3)$$

where  $i$  (A) is the discharge current,  $\Delta t$  (s) is the discharge time,  $m$  (g) is the mass of the active material of individual electrode, and  $\Delta V$  (V) is the voltage range.

### 3. Results and discussion

#### 3.1. SEM and XRD

The morphology and size were observed by SEM. As shown in Fig. 1a, the corn starch particles were polygonal and exhibited a size distribution in the range of 5–25 μm. After H<sub>3</sub>PO<sub>4</sub> activation, all the porous carbons presented irregular shape, which appeared blocky structure and rough external surface. The obvious changes in the surface morphology of PC/500/1/4:1 can be seen from Fig. 1b. The diameter became smaller compare to the corn starch particles, which were around 1–5 μm in size. Notably, H<sub>3</sub>PO<sub>4</sub> played a crucial role in the activation process. When corn starch was directly impregnated with H<sub>3</sub>PO<sub>4</sub>, starch particles would assemble together and H<sub>3</sub>PO<sub>4</sub> interacted with them to form phosphate and polyphosphate bridges. The pore structures created with the insertion of phosphate groups during the dilation processes [9]. The crystal structures were examined by XRD. Fig. 1c showed the XRD pattern of PC/500/1/4:1. XRD pattern displayed two broad diffraction peaks at around 24° and 44°, corresponding to the (002) and (100) crystal planes of the graphitic carbon, respectively. The occurrence of broad peaks at these 2θ suggested an increasing regularity of crystal structure and resulting in better layer alignment [10].

#### 3.2. The pore structure properties

The N<sub>2</sub> adsorption/desorption isotherms (Fig. 2a) presented a combined I/II type isotherms with strong N<sub>2</sub> adsorption at low relative pressure and slight N<sub>2</sub> adsorption at middle and high relative pressure, indicated large amounts micropores and mesopores coexist in the porous carbons. The obvious H1 hysteresis loops were observed at a relative pressure P/P<sub>0</sub>=0.4, which were attributed to developed mesoporous structure and the appearance of macropores [11]. The DFT pore size distribution curves (Fig. 2b) also demonstrated the samples coexistence of micropores and mesopores/macropores, which were consistent with the results of the N<sub>2</sub> adsorption/desorption isotherms. Table 1 listed the texture

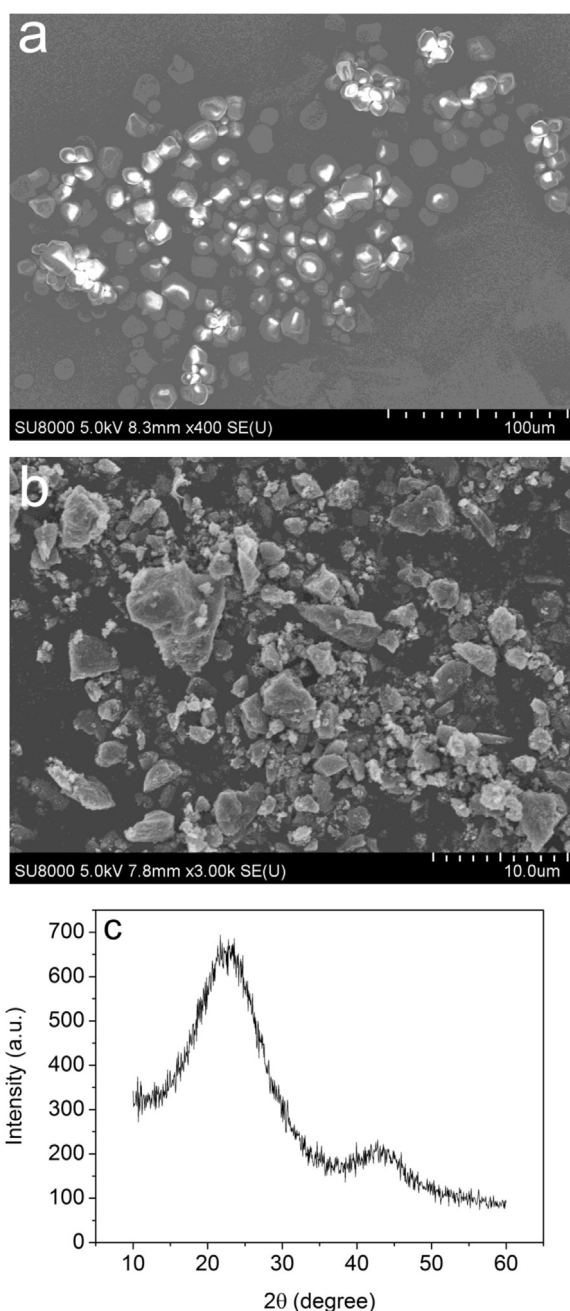


Fig. 1. SEM micrographs of (a) corn starch and (b) PC/500/1/4:1; (c) XRD pattern of PC/500/1/4:1.

properties of the porous carbons in different H<sub>3</sub>PO<sub>4</sub>/CS ratios. The H<sub>3</sub>PO<sub>4</sub>/CS ratio was significant for the development of porosity. The BET surface area, total pore volume, average pore diameter and external surface area presented an upward trend with increasing the H<sub>3</sub>PO<sub>4</sub>/CS ratio from 2 to 4. However, they dropped abruptly when the H<sub>3</sub>PO<sub>4</sub>/CS ratio was changed from 4 to 6. The micropore area and micropore volume presented an upward trend with increasing the H<sub>3</sub>PO<sub>4</sub>/CS ratio from 2 to 6. The pore size distribution also showed a majority of the pores fell into the range of mesopores. The results revealed the activation opening the closed pores, widening the pre-existent pores and forming the mesopores in quantities.

#### 3.3. Electrochemical performances

The electrochemical performances were estimated by CV and

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