

# Blowing and in-situ activation of carbonaceous “lather” from starch: Preparation and potential application



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## ABSTRACT

Activated carbon foam with nano-thickness cell walls (ACF-NCW) is prepared via a simple method. Starch, a natural polysaccharide, is selectively hydrolyzed by mixed-acid firstly. Then, the hydrolyzed starch is dried and mixed with zinc chloride. Finally, ACF-NCW can be prepared with the assistance of microwave irradiation under the synergistic effect of foaming, carbonization and in situ activation. Different from the traditional carbon foams, the ACF-NCW possesses the characteristic of three-dimensional (3D) graphene and porous carbon nanosheet. Its porous cell walls are in the thickness of ~50 nm, which shorten the transmission distance of ions during its application. The laminated polycrystalline walls and 3D interconnected structure are propitious to electron transfer. Due to its large specific surface area (BET: 926 m<sup>2</sup>/g, DFT: 730 m<sup>2</sup>/g), the ACF-NCW electrode exhibits high specific gravimetric capacitance of 172 F/g at 0.5 A/g in 6 mol/L KOH and good electrochemical stability with gravimetric capacitance retention up to 92% after 1000 cycles at 2.5 A/g.

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## 1. Introduction

Carbon foam is a new carbonaceous material with 3D spongy like structure. It has many novel features, such as exceptionally high void volume, low specific weight, very high resistance to temperatures in non-oxidizing environments, good energy absorption and high thermal conductivity or insulation as desired [1]. It has attracted much attention as its wide potential applications in the areas like electrode for energy storage [2,3], electromagnetic shielding material [4], catalyst support [5], scaffold for bone growth [6], thermal conducting/insulating material [7], etc. It is also immensely appealing to apply carbon foam by combining its different features. For instance, it may be applied as both filler and electrode in the walls of buildings for thermal insulation and solar energy storage by constructing a super large supercapacitor and solar panels circuit. Lately, the preparation and application of carbon foam were reviewed in detail by Inagaki et al. [8].

As a potential application area of carbon foam, supercapacitor has received growing attention because of its high power density, long cycle life and safe operation. Electrode material is a key component that determines a supercapacitor's performance. Carbonaceous materials are valuable candidates for the electrodes of supercapacitor as they have excellent properties such as high surface area, good corrosion resistance, excellent temperature stability and relatively low cost. Recently, activated carbon, hierarchical porous carbon, three-dimensional (3D)

graphene, porous carbon nanosheet, etc., have been extensively studied. Activated carbon is a commonly used material for supercapacitor electrodes. Theoretically, a higher specific surface area would lead to a higher capacity of ions and therefore a higher energy density of the supercapacitor. In spite of the mature technology of preparing activated carbon with various specific surface areas, getting a higher specific surface area by increasing the amount of micropores would inevitably sacrifice the conductivity of the material. The random connected micropores also hinder the access and transfer of electrolyte ions in the activated carbon particles. Carbonaceous material with hierarchical porous structure then provides an option to solve this problem from the perspective of mass transfer [9,10]. Encouraging results have been obtained. Three-dimensional graphene foam is another attractive electrode material for supercapacitor [11]. It overcomes the restacking issue of normally used two-dimensional graphene sheets, and has large accessible surface area because of its 3D porous/hollow structure. But low yield and long preparation cycle are still challenges for the large-scale application of graphene. Inspired by the excellent electrochemical capacitance of graphene nanomesh [12], Fan et al. developed a pillared-porous carbon nanosheet [13]. This architecture also shows high performance for supercapacitor.

Although the application of carbon foam in supercapacitor has been reported, the two traditional structures of carbon foam (i.e., the one having a reticulated structure and the one having a cellular structure, usually made from non renewable materials such as thermosetting resins, coal, mesophase pitch) are not good enough for its electrode performance unfortunately. The former with large pores is propitious

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to mass transfer, but not conductive to the storage of ions for its small specific surface area. The latter after activation, however, will face the same mass transfer problem of activated carbon because of its thick cell walls (usually in micron scale). In the recent attempts, carbon foam with hierarchical pores and honeycomb-like structure were developed [14,15]. These two carbon foams showed excellent performance as supercapacitor electrodes.

In the present work, carbon foam with novel structure was prepared by a simple method. Different from the reported structures of carbon foam, it has ultra-thin porous cell walls. The obtained structure possesses the characteristic of 3D graphene foam and porous carbon nanosheet. Ions can easily transmit in the carbonaceous walls with short transmission distance. The layered polycrystalline structure of the material is propitious to the electron transfer during application. This work shows a chance to obtain a material possessing both large porosity and appropriate channels for fast mass transfer without losing its conductivity.

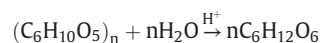
## 2. Materials and methods

### 2.1. Materials

Cornstarch was obtained from Xi'an Xiadian Corn Development Co., Ltd. (Shanxi, China). Starch is a carbohydrate consisting of a large number of glucose units joined by 1,4- $\alpha$ -glycosidic and 1,6- $\alpha$ -glycosidic linkages. It usually consists of two types of molecules: the linear and helical amylose and the branched amylopectin. Depending on the plant, starch generally contains 20 to 25% amylose and 75 to 80% amylopectin by weight. The starch used in our work contains ~25% amylose. Concentrated sulfuric acid (98%), phosphoric acid (85%) and concentrated hydrochloric acid (37.5%) were purchased from Chengdu KeLong Chemical Co., Ltd. (Sichuan, China). Iodine (AR), sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) (AR), potassium iodide (AR), potassium bichromate (AR) and soluble starch used for the determination of iodine number were also purchased from Chengdu KeLong Chemical Co., Ltd. (Sichuan, China). Zinc chloride (AR), KOH (AR), polytetrafluoroethylene (PTFE) and nickel foam were bought from Chengdu United Institute of Chemical & Reagent (Sichuan, China). The microwave absorber  $\text{Fe}_3\text{O}_4$  (CP) was supplied by Shanghai Shanhai Gongxuetuan No. 2 Experiment Factory (Shanghai, China).

### 2.2. Experimental procedure

The procedure of preparing ACF-NCW is illustrated in Fig. 1. In a typical preparation, 5 g of cornstarch was dispersed in 50 mL of mixed-acid solution containing 0.1 mol/L of  $\text{H}_2\text{SO}_4$  and 0.5 mol/L of  $\text{H}_3\text{PO}_4$ . The starch was selectively hydrolyzed for 3 h, and then collected by filtration and oven dried at 105 °C for 1 h. During the hydrolysis process, the amorphous part of the starch particles was hydrolyzed and the crystallized part was remained. The hydrolysis process can be depicted as the following reaction. The hydrolysis produces some aqueous soluble dextrin and finally glucose after completely hydrolyzation.



After hydrolysis, filtration and oven drying, two grams of the dried starch was mixed with 2.5 g of zinc chloride. The mixture was put in a "Fe<sub>3</sub>O<sub>4</sub> bath" composed of two nested quartz crucibles (25 mL and 160 mL) with Fe<sub>3</sub>O<sub>4</sub> powder between them, where the Fe<sub>3</sub>O<sub>4</sub> powder is a recyclable microwave absorber. Subsequently, the crucibles were filled with nitrogen. Immediately after the crucibles were covered with lids, they were put in a household microwave oven (Galanz, WG700TL2011-K6, 2.45 GHz, 700 W) with quartz insulation cotton padding in the chamber for microwave irradiation. Combination of two irradiation programs (power I and II) was adopted in this research. For power I, the bath and its contents were treated in cycles with 6 s of full power irradiation (700 W) and 23 s of interval. For power II, the treatment was conducted with 18 s of full power irradiation (700 W) and 11 s of interval. The temperature after 5 min of treatment was measured with thermocouple. It was about 150 °C for power I and 400 °C for power II. Various irradiation times (5, 7.5, 10, 12.5 and 15 min) were investigated. Foaming, carbonization and in situ activation happened in the same procedure. We believe that dehydration plays an important role in transforming starch ((C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>)<sub>n</sub>) to carbonaceous product (C<sub>x</sub>H<sub>y</sub>O<sub>z</sub>), as sketched in the left of Fig. 1. The reaction may also be depicted not strictly as the following reaction.

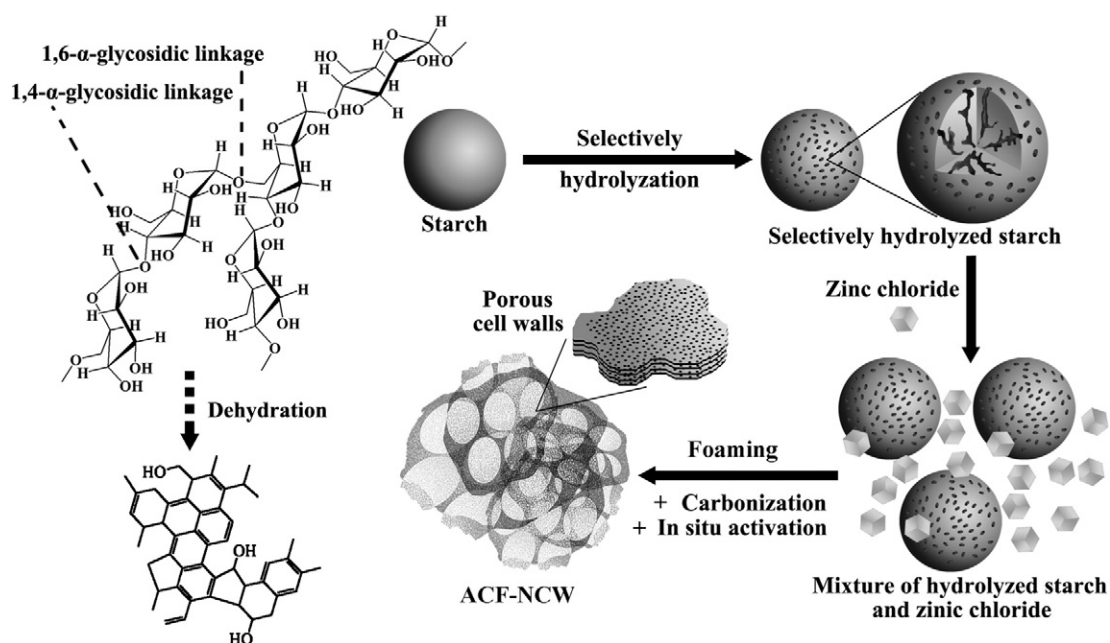
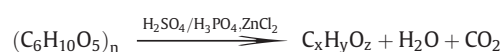


Fig. 1. Schematic illustration of the procedure of preparing ACF-NCW.

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