



# Synthesis, characterization and electrochemical properties of S-doped carbon aerogels

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## ABSTRACT

Heteroatom doping in carbon aerogels is critical for improving the surface areas and electronic properties of these aerogels. This paper demonstrates doping sulfur (S) atom into carbon aerogels. S-doped carbon aerogels (S-CAs) were prepared via a sol-gel process using phloroglucinol (P), resorcinol (R) and formaldehyde as the polymerization precursors with sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) as the basic catalyst and sodium 4-hydroxybenzenesulfonate (HBS) as the surfactant. The as-synthesized products were dried in normal temperature and carbonized with temperature-programmed. The effects of S-doping on the surface area and electronic properties of the S-CAs were studied by a surface area analyzer and electrochemical workstation. When the S loading was up to 1.38%, the corresponding S-CA exhibited the most optimal surface area and specific capacitance. The textural properties were characterized using scanning electron microscopy (SEM), transmission electron microscopy (TEM), a surface area analyzer, an X-ray diffraction spectrometer, a Raman spectrometer, the X-ray photoelectron spectroscopy (XPS) spectra, an elemental analyzer and an EDX detector, and the electrochemical properties were measured using cyclic voltammetry, galvanostatic charge–discharge measurements and electrochemical impedance spectroscopy (EIS). When the molar ratio of HBS/(P + R) was 0.05, the S loading was 1.38%, the specific surface area of the as-synthesized CA was  $831 \text{ m}^2/\text{g}$ , and its specific capacitance was  $105 \text{ F/g}$ .

## 1. Introduction

Porous carbon materials have attracted attention because of their interconnected three-dimensional (3D) porous structure and favorable chemical properties [1–12]. Carbon aerogels (CAs) have been widely studied and reported in recent years [13–15]. CAs have good potential for applications in water purification due to their plentiful porosity and various adsorption properties [16]. CAs are attractive candidates for supercapacitors and batteries because of their electrochemical double layer properties [17–19]. In the field of catalysis, CAs are attractive catalyst supports because of their porous texture [20,21]; CAs have been widely used in fireproof materials due to their low thermal conductivity and low density [22,23].

A CA preparation method was first reported by Pekala et al. in the 1990s [24,25]. The CAs were prepared via the polycondensation of traditional organic precursors, e.g., phenols and aldehydes [26]. Recently, other techniques for the preparation of CAs have been reported [27–30]. Heteroatom doping is a new technique used to improve the surface and electronic properties of CAs [27,28]. Song et al. reported a

wet impregnation method for a Mn-doped CA that was used in a supercapacitor, and the as-synthesized CA showed a higher specific capacitance than the other CAs [29]. A preparation method for copper-doped CAs has been reported by Baumann et al.  $\text{Cu}(\text{NO}_3)_2$  was used as the heteroatom source and was added to the polycondensation precursors [30]. The Cu-doped CAs exhibited high surface areas and micro- and meso-scale pore diameters. Sulfur is a lightweight element used for the heteroatom doping of CAs, and S is a promising doping material because of its advantages, such as its natural abundance, low cost, and environmental friendliness [31–33]. The reported S-doping methods include: (i) using organic sulfur compounds that are added into the polycondensation precursors as the sulfur source; and (ii) activating CAs with  $\text{H}_2\text{S}$ , which is a pyrolysis process sulfur source [34,35].

S-doping of CAs has been widely studied and reported, but the previous methods of producing CAs have some disadvantages [31,35]: (i) small pore diameters and low surface areas, and (ii) high resistances and poor electrochemical properties. Zhou et al. have reported a new S-doping method, they directly carbonized potassium salts of m-aminophenol-m-mecaptophenol co-resin to prepare S-doped carbon

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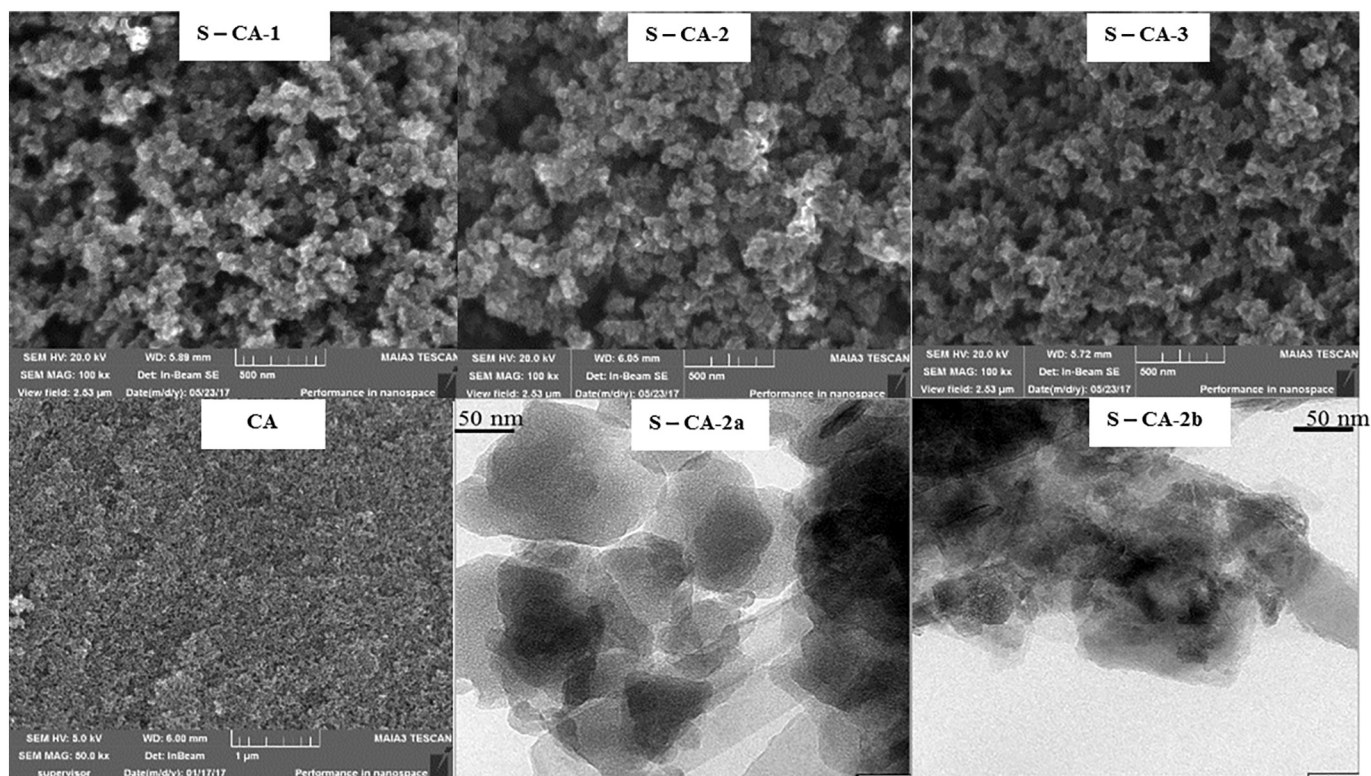


Fig. 1. SEM images of the CAs and TEM images of the S-doped CA-1. (P, C, R and F were constant; S – CA-1, HBS = 10 mol%; S – CA-2, HBS = 5 mol%; S – CA-3, HBS = 2 mol%; CA: Blank).

aerogels [34]. In their report, S-doped carbon aerogels exhibited ideal surface areas and electrochemical properties. In this article, we report an original preparation method for S-doped CAs. Sodium 4-hydroxybenzenesulfonate was used as both the surfactant and sulfur source to improve the texture and electrochemical properties, respectively. The as-synthesized S-doped CAs exhibited higher surface areas and specific capacitances than the blank samples. Furthermore, the as-synthesized S-doped CAs were characterized using various textural spectroscopy instruments and electrochemical instruments.

## 2. Experimental

### 2.1. Synthesis of the CAs

Phloroglucinol (P), resorcinol (R), a formaldehyde solution (F), sodium carbonate ( $\text{Na}_2\text{CO}_3$ , C), and sodium 4-hydroxybenzenesulfonate (HBS) were purchased from Aladdin Reagent Co. All reagents were analytical reagent (AR) grade and were used as-received without further treatment.

The preparation method for the CAs was described in detail in our previous work [36]. Sodium 4-hydroxybenzenesulfonate (HBS) was added into the reaction solution as a surfactant before the reaction solution was sealed in test tubes. Briefly, the molar ratio of P/R was 0.14. The molar ratios of HBS/(P + R) were 0.02, 0.05 and 0.1. The molar ratio of C/(P + R) was 0.002. The mass concentration of the reactants (P + R + F + C + HBS) in the solution was 45%. A Tubular carbonizing furnace (GSL-1700 × -S60, Kejing, Hefei) was used to carbonize samples. The drying and carbonization method were similar to our previous method [37]. The synthesis and pyrolysis processes were similar to those used in our previous work [37].

### 2.2. Characterization

The surface areas were measured using an ASAP 2420 surface area

analyzer (Micromeritics, USA) with the Brunauer–Emmett–Teller (BET) method. The samples were degassed using a temperature-programmable process, and the temperature conditions for BET analysis was similar to our previous work [36]. The textural properties were obtained from the adsorption–desorption isotherms via the Barrett–Joyner–Halenda (BJH) model. The total pore volume ( $V_{\text{total}}$ ) was obtained from the quantity of adsorbed  $\text{N}_2$  at a relative pressure ( $P/P_0$ ) of 0.998. The as-synthesized S-doped CAs were characterized via X-ray diffraction (XRD, Ultima IV X-ray diffractometer, Rigaku, Japan) with CuK $\alpha$  radiation ( $\lambda = 1.54 \text{ \AA}$ ) and a Labram HR evolution Raman spectrometer (532 nm). The morphologies of the ternary CAs were observed via scanning electron microscopy (SEM, TESCAN MAIA3, USA) with an energy dispersive X-ray spectrometer (EDX) and transmission electron microscopy (TEM, JEM-2100F, JEOL, Japan). The surface compositions of the as-synthesized S-doped CAs were obtained via X-ray photoelectron spectroscopy (XPS, PHI5600 Physical Electronics). The elemental compositions of the as-synthesized S-doped CAs were obtained from an elemental analyzer (EA 3000).

### 2.3. Electrochemical property measurements

The preparation of the as-synthesized S-doped CA electrodes was described in detail in our previous work [36]. Briefly, the as-synthesized S-doped CAs were ground into a powder and filtered through a 200-mesh sieve. Polytetrafluoroethylene (PTFE) was used as the binder, and the weight ratio of the S-doped CAs/PTFE was 95:5. Nickel foam was used as the packing material.

The electrochemical properties of the as-synthesized S-doped CAs were obtained using cyclic voltammetry (CV), galvanostatic charge–discharge and electrochemical impedance spectroscopy (EIS) analysis methods. A GAMRY Interface 1000 electrochemical workstation (GAMRY, USA) was used to measure the electrochemical properties. The samples were dried on  $105^\circ\text{C}$  in a vacuum drying oven for 24 h, weighed, and soaked in 6 M KOH for 24 h before the sample electrodes

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