



# Synthesis and supercapacitive performance of three-dimensional cubic-ordered mesoporous carbons



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## ABSTRACT

3D cubic-ordered mesoporous carbons (OMCs) are prepared by nanocasting approach using silica KIT-6 as the template, furfuryl alcohol as the carbon precursor and subsequently carbonized at the temperature of 500 °C, 600 °C and 700 °C. The physicochemical and electrochemical properties of the as-prepared OMCs are investigated by transmission electron microscopy (TEM), nitrogen adsorption/desorption isotherm, Fourier infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), cyclic voltammetry, galvanostatic charge/discharge, electrochemical impedance spectroscopy (EIS) and cycle life measurement in 6 mol L<sup>-1</sup> KOH electrolyte. The results demonstrate that the synthesized technology is a crucial factor impacting the characteristics of OMCs. With the change of carbonization temperature, the OMC-600 exhibits the honeycomb-like morphology with high specific surface area (1257.8 m<sup>2</sup> g<sup>-1</sup>), large pore volume (1.56 cm<sup>3</sup> g<sup>-1</sup>), and high capacitance of 221.8 F g<sup>-1</sup> at the current density of 1 A g<sup>-1</sup>. In addition, the supercapacitor using OMC-600 as the active material shows high specific capacitance and excellent cycle stability, which exhibits a specific capacitance of 52.8 F g<sup>-1</sup> at the charge/discharge current density of 0.5 A g<sup>-1</sup>. Moreover, the OMC-600 supercapacitor delivers high energy density of 6.53 Wh kg<sup>-1</sup> at the power density of 5000 W kg<sup>-1</sup>, indicating a promising application for the high performance supercapacitors.

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## 1. Introduction

In recent years, the quick development of new energy vehicles and hybrid electric devices is promoted by the crisis of energy, and thus accelerate the development of the energy storage devices such as fuel cells, lithium-ion batteries, and supercapacitors. In comparison with batteries and conventional capacitors, supercapacitors have the advantages of high power density, fast charge/discharge processes, and long cycle life, and have been considered as a good choice for high-power energy to meet the need for fast growth in power required by devices [1–4].

According to the mechanism of electrical energy storage, the capacitive behavior of supercapacitors can be divided into two types: (1) using ion absorption (electrochemical double layer capacitors (EDLCs)), such as carbons; (2) by surface redox reaction (pseudocapacitors), such as metal-oxides and conducting polymers. Compared with the pseudocapacitors using metal-oxides and conducting polymers as electrode materials, the carbon-based EDLCs exhibit longer life, higher specific surface area, lower

cost, and higher power density but lower specific capacitance. Thus, more and more attention has been paid on the enhancement of the specific capacitance of EDLCs. The high capacitance of EDLC is produced by the quick formation of electrical double layer at electrode/electrolyte interface, which accumulates electrical charges on the electrode surface. On the basis of the energy storage mechanism of EDLCs, the porous electrode materials with high surface area and capable of high charge accumulation are demanding for high capacitance [5,6]. In addition, the pore structure of carbon materials (micro-, meso- and macropore) may critically affect their physicochemical properties, specially serving as an electrode material involving the electrolyte accessibility, ion transportation, electron conductivity, etc. [7–9]. The types of pores as defined by IUPAC are micropores, pores with width not exceeding about 2.0 nm, mesopores, pores with width between 2.0 nm and 50 nm and macropores, pores with width exceeding about 50 nm [9]. Compared with the other carbon electrode materials, such as activated carbon materials [10,11], carbon aerogels [12,13], carbon nanotubes [14,15], carbide-derived carbons [16,17], carbon fibers [18,19], the mesoporous carbons with pores size between 2.0 nm and 50 nm possess high specific pore volume and high specific surface area, which can facilitate the ions diffusion. Therefore, the

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mesoporous carbons seem to be more attractive candidates as electrode materials for EDLCs.

The pore size distribution and topology of pores in materials of similar compositions can be very different, depending on the method of synthesis. Ordered mesoporous carbons (OMCs) possess tunable pore structure, uniform and adjustable pore size, and are expected to show much higher specific capacitance and longer cyclic life for the application of supercapacitors [20]. Since the first synthesis of ordered mesoporous carbon, denoted as CMK-1, was reported by Ryoo et al. using the MCM-48 silica with cubic  $Ia3d$  symmetry as the template [21], many kinds of OMCs materials with different structures have been prepared using various types of silica template, such as SBA-16 [22], MCM-48 [23], MSU-H [24], KIT-6 [3], SBA-15 [25], etc. In particular, mesoporous silica KIT-6 consists of two interwoven mesoporous subnetworks quite similar as in the case of MCM-48 [26], but it may be prepared with much larger mean pore diameters. Consequently, the mesoporous silica templates with 3-dimensional bicontinuous mesoporous structure are more attractive for supercapacitors.

The preparation of ordered mesoporous carbons involves filling the pores of the silicate template with a suitable carbon precursor, the following carbonization and the final removal of the silica template. There are several critical parameters in the preparation of OMCs that would affect its structure, one of which is the carbonization temperature. The carbonization temperature will affect the collapse degree of the pore and hole structure, the porosity and the internal ordering of the carbons. Although the synthesis of 3D cubic-ordered mesoporous carbons using KIT-6 as the template has been previously reported, little attention has been paid on the effects of carbonization temperature on the textural parameters of mesoporous carbons as well as the electrochemical performance for the application of supercapacitor. Thus, in this work, 3D cubic-ordered mesoporous carbons have been successfully synthesized employing KIT-6 silica as the template, furfuryl alcohol as the carbon source at the temperature ranging from 500 °C to 700 °C, and the effects of carbonization temperatures on structure, electrochemical performance and supercapacitive behaviors of OMCs are further investigated.

## 2. Experimental

### 2.1. Synthesis of cubic mesoporous silica KIT-6

The silica KIT-6 template was synthesized according to literature procedures [9]. 4.0 g of triblock copolymer Pluronic P123 (poly (ethylene oxide)-*b*-poly (propylene oxide)-*b*-poly (ethylene oxide),  $EO_{20}PO_{70}EO_{20}$ ,  $M_w = 5800 \text{ g mol}^{-1}$ ), was dissolved in 144 ml deionized water and 7.9 g of 37 wt% HCl mixture aqueous solution with the aid of stirring at 35 °C. Then, 4.0 g of *n*-butanol was added in the above solution, and the mixture was kept on stirring for 1 h. Subsequently, 8.6 g of tetraethyl orthosilicate (TEOS) was added at once under vigorous stirring and the mixture was kept on stirring at 35 °C for 24 h. The mixture was put into a Telfon autoclave and kept at 100 °C for 24 h. The obtained white product was filtered with a mixture of ethanol-HCl, and dried at 80 °C overnight. Finally, the silica KIT-6 was obtained by the calcinations at 550 °C for 6 h in air with a heating rate of 1 °C  $\text{min}^{-1}$ .

### 2.2. Synthesis of 3D cubic-ordered mesoporous carbons

3D cubic-ordered mesoporous carbons were synthesized by nanocasting pathway which furfuryl alcohol was used as the carbon resource and mesoporous silica as the template, and the general preparation strategy of the OMCs can be briefly described by the following process as shown in Fig. 1. Briefly, 1.0 g furfuryl

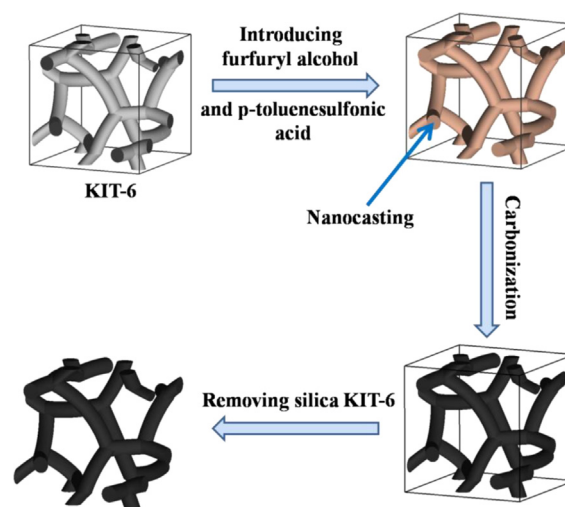


Fig. 1. A schematic mechanism of OMCs formation.

alcohol was impregnated into 1.0 g KIT-6 by nanocasting approach in the presence of 0.1537 g toluene-*p*-sulfonic acid for 1 h. In order to polymerize furfuryl alcohol completely, the mixture was cured in the oven at 80 °C and 160 °C for 2 h, 4 h respectively. Afterwards, the mixture was carbonized under Ar atmosphere for 3 h at a heating rate of 5 °C  $\text{min}^{-1}$ . Finally, the OMCs were obtained by the removing of silica template using 5% HF aqueous and drying at 100 °C in air. The resulted OMCs materials carbonized at different temperature were named as OMC-X (where X represents the carbonization temperature).

### 2.3. Characterization of structure and morphology

Transmission electron microscopy (TEM) measurements were conducted on a JEM-2100F microscope operated at 200 kV, to reveal the ordered structure of the OMCs. Small-angle X-ray diffraction (XRD, D/MAX-3C, Rigaku) patterns were evaluated using a diffractometer with Cu  $K\alpha$  radiation (30 kV, 30 mA,  $\lambda = 1.5406 \text{ \AA}$ ). The specific surface area and pore structure of the OMCs were determined by  $N_2$  adsorption/desorption isotherm at 77 K (JW-BK112) after the prepared samples were degassed at 110 °C overnight. The specific surface areas were calculated by the conventional Brunauer–Emmett–Teller (BET) method. Pore volumes ( $V_{\text{total}}$ ) and the pore size distribution (PSD) plots were derived from the adsorption branch of the isotherm based on the Barrett–Joyner–Halenda (BJH) model. The FTIR measurement of the sample was characterized via a Fourier transform infrared (FTIR) spectrometer (Perkin-Elmer Spectrum One). The surface characteristics of the samples were finished by X-ray photoelectron spectroscopy (XPS) (K-Alpha 1063, Thermo. Fisher Scientific, Britain).

### 2.4. Electrode preparation and electrochemical measurements

The supercapacitor electrode materials were made up of 80 wt% OMCs material, 10 wt% acetylene black, and 10 wt% polyvinylidene fluoride (PVDF). After well mixed, the obtained paste was pressed into the nickel foams uniformly which were used as the current collectors and dried in vacuum at 110 °C for 12 h. Finally, the nickel foams were pressed under a pressure of 15–16 MPa. The geometric surface area of the electrodes was kept to be 1.0  $\text{cm}^2$ , the electrodes typically had a thickness of about 0.1 mm and the loading active matter was 4–5 mg.

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