

Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics
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Supporting Information

Facile synthesis and application of a carbon foam with large mesopores

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1. Chemicals

Triblock copolymer poly(ethylene oxide)-b-poly(propylene oxide)-b-poly(ethylene oxide) (Pluronic P123, $\text{EO}_{20}\text{PO}_{70}\text{EO}_{20}$, MW = 5800), polyethylenimine (PEI, $M_n = 423$), sodium silicate, acetic acid, ammonium fluoride, ethanol (90 wt%), sodium hydroxide, bis-(trifluoromethanesulfonyl)imide lithium (LiTFSI), and tetraglyme were purchased from Aldrich. Millipore water was used in all experiments.

2. Synthesis of Mesoporous Carbons

In a typical synthesis of the carbon with large mesopores, 3.0 g of neutral triblock co-polymer surfactant, Pluronic 123, was dissolved in a mixture of acetic acid (3.0 g), DI water (52 g), and ammonium fluoride (0.3 g) at 40 °C. After stirring for 2 h, a solution of sodium silicate (2.35 g) in water (40 g) at 40 °C was added and the resultant mixture was reacted for 5 min under vigorous stirring. Then the mixture was kept static for 24 h at 40 °C, followed by aging at 70 °C overnight. The solid products were collected by filtration, dried in the air at 25 °C, and then calcined at 800 °C for 2 h under nitrogen. The mesoporous carbon foam was obtained after etching the silica using a 2 M NaOH aqueous solution at 80 °C. The mesoporous CMK-3 carbon was synthesized following the reported method¹.

3. Characterization

TEM analysis was conducted using an FEI TECNAI T12 Twin transmission electron microscope, operated at 120 kV. TEM samples were prepared by placing a droplet of ethanol-diluted carbon foam dispersion on a formvar - carbon coated microscope grid and dried in air. Raman spectra were recorded from 500 to 2000 cm^{-1} at excitation wavelengths of 488 nm on a Renishaw InVia Confocal Raman Microscope with resolution of $\sim 1 \text{ cm}^{-1}$. Nitrogen adsorption-desorption isotherms were measured at 77 K on a Micromeritics ASAP 2020 surface area and porosity analyzer. The mesoporous samples were degassed at 423 K in a high vacuum for 24 h prior to the analysis. Specific surface areas were calculated by the Brunauer-Emmett-Teller (BET) method. Pore size distributions were obtained from the related adsorption isotherms by using the Barrett-Joyner-Halenda (BJH) model and non-local density functional theory (NLDFT) model. The stability of the carbon under harsh conditions was assessed by performing a steam treatment. A 4 mL glass vial containing 0.2 g of the foam was put into a 20 mL glass vial that was filled with 10 mL of DI water. The vials were then sealed in a 100 mL Teflon-lined laboratory autoclave from Parr Instruments. The autoclave was heated to 120 °C and kept at that

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temperature for 30 h. The obtained carbon sample was dried under 0.02 mbar at 25 °C for 24 h and stored in a desiccator.

4. Sorbent Preparation and CO₂ Capture

The sorbent was prepared by wet impregnation. In a typical preparation, 0.8 g of PEI in ethanol (10 wt%) was added into 20 mg of the mesoporous carbon foam. The resultant slurry was stirred at room temperature for 30 min and then dried under reduced pressure (-700 mmHg).

The CO₂ adsorption measurement was performed on a TA Instruments Q500 thermal gravimetric analyzer. Dried pure CO₂ (99.99%) at 1 atm was used for the adsorption run and ultra-high purity N₂ (99.995%) was used as a purging gas for CO₂ desorption. In a typical adsorption process, about 5 mg of the sorbent was placed in a platinum sample pan. After the sorbent was heated to 100 °C in a N₂ stream (40 mL min⁻¹) and held at that temperature for 45 min to remove the moisture and the adsorbed CO₂. The sorbent was cooled down to 75 °C at a rate of 5 °C min⁻¹ and equilibrated at that temperature for 30 min. The gas was then switched from N₂ to CO₂ (40 mL min⁻¹) at 75 °C for 60 min for the sorption measurement. The CO₂ capacity of the sorbent in mmol g⁻¹ was calculated based on the weight gain of the sorbent during the adsorption.

5. Preparation of Carbon/sulfur cathode and Battery Test

The carbon/sulfur (C/S) composite was prepared by mixing the carbon foam and sulfur powders in an agate mortar and then heated at 159 °C for 20 h for the melt infusion of sulfur into the carbon (S content = 60 wt%). The C/S cathode was fabricated by making a slurry of the C/S composite with PVDF using NMP as the solvent. The slurry was then casted onto a 0.001" thick aluminum sheet and dried at 70 °C overnight. The electrode contained 92.5 wt% of the C/S composite and 7.5 wt% PVDF. The electrochemical performance of the cathode was tested galvanostatically in the MACCOR battery tester in 2032-type coin cells where the coin cells were cycled between 1.0 V and 3.0 V. Lithium metal was used as the anode and 1 M LiTFSI in tetraglyme was used as the electrolyte. The charge and discharge of the coin cells was performed at 1.0 C-rate.

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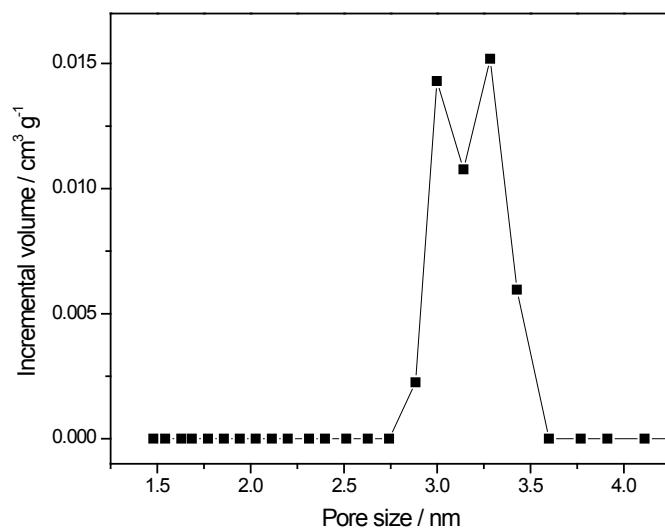


Fig. S1. The pore size distribution of the small mesopores of the mesoporous carbon foam calculated using the NLDFT method.

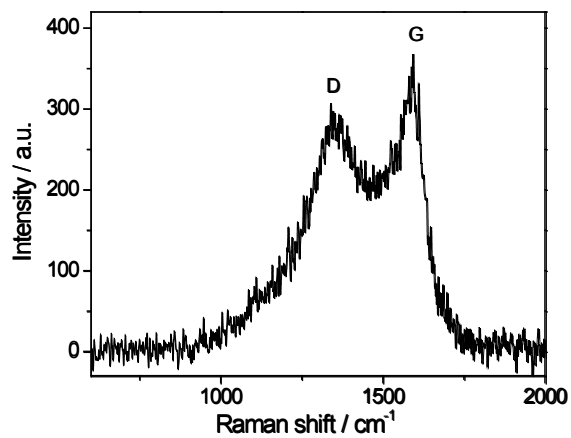


Fig. S2. Raman spectrum of the mesoporous carbon foam.

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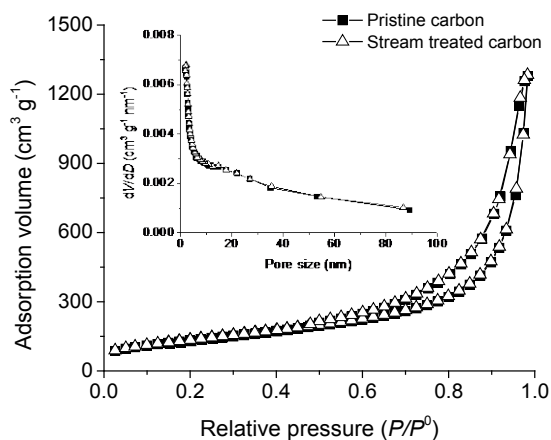


Fig. S3. The nitrogen sorption isotherms and the pore size distributions (the inset) of the pristine mesoporous carbon and the steam treated carbon. The nitrogen sorption isotherm of the pristine mesoporous carbon is slightly different from that in the main text because of batch-to-batch differences.

1. X. Ji, K. T. Lee and L. F. Nazar, *Nature materials*, 2009, **8**, 500-506.