Supplementary information

Strong Silica Monoliths with Large Mesopores using Agarose Gel Templates

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Experimental conditions

Materials. Tetraethyl orthosilicate (Sigma Aldrich), ethanol (Merck), agarose powder (Scientifix), 1 M tetrabutyl ammonium fluoride in tetrahydrofuran (Aldrich) were used as received.

Synthesis of silica pellets. Agarose gels of 3, 5, 7 and 8 wt % were used as templates. To make a 5 wt % gel, 5 g of agarose powder were dispersed in 95 g of distilled water. The dispersion was stirred and heated to 80 °C at which point, the powder dissolved. The clear solution was poured while hot into test tube molds and allowed to set over night. The gels were removed from the molds and cut into roughly 1 cm^3 cubes. Over a period of 36 hours, the solvent was exchanged from pure water to ethanol by first placing the pellets in 1:2 ethanol:water (vol/vol), then 2:1 ethanol:water, followed by two exchanges of absolute ethanol. Fifty gel pellets were soaked overnight in 100 mL of solution composed of 50 vol % TEOS and 50 vol % 1 M TBAF. The infused agarose gel pellets were transferred to distilled water to initiate hydrolysis and condensation reactions. After 6 h, the pellets were removed and dried at room temperature for a few days and then at 50 °C for 6 h. The template was removed through calcination under air flow using the following program: 30-600 °C at 1 °C min^{-1}, 600 °C for 5 h. Robust, crack free silica monoliths resulted when an excess of TEOS/TBAF solution was used in comparison to the volume of the agarose gel, when the pellets were completely dry at the time of calcination and when the calcination ramp was fairly gradual (1 ºC min^{-1}).

Characterization.

Scanning electron microscopy was performed on a FEI QUANTA 200F microscope operated at voltages between 15-20 kV. Samples were mounted on carbon SEM stubs and then sputter coated with a thin layer of gold using an Edwards S150B Gold Sputter Coater.

Transmission electron microscopy analyses were conducted using a Philips CM120 BioTWIN microscope operating at 120 kV. TEM samples were prepared by finely grinding the sample in ethanol using an agate mortar and pestle, sonicating for 20 minutes and then drop depositing the suspension on to holey carbon-coated copper grids.

The surface area and pore sizes of the synthesized materials were determined by nitrogen physisorption using a Micromeritics Tristar 3000 instrument. Samples were degassed at 150 °C and at a pressure below 100 mTorr for a minimum of 4 h prior to analysis using a Micromeritics VacPrep 061. The surface area was calculated using the Brunauer-Emmett-Teller (BET) method. The Barrett-Joyner-Halenda (BJH) method was used to calculate the pore size distribution using the adsorption branch of the isotherm.

Compression tests were performed on an Instron 5848 MicroTester, equipped with an Instron 5800 computer and Bluehill1 Version 2.22 software. A crushing plate with a maximum load capacity of 2000 N was used. The approach rate was 3 mm min^{-1} and the compression rate was 1 mm min^{-1}. Ten pellets were crushed individually and the results were averaged.