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Supporting Information for

Fine control over the morphology and structure of mesoporous silica nanomaterials by a dual-templating approach

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Materials. Cetyltrimethylammonium bromide (CTAB) and sodium hydroxide (NaOH) were obtained from Beijing Chemical Reagent Company. Tetraethoxysilane (TEOS) was purchased from Alfa Aesar. Sodium bis (2-ethylhexyl) sulfosuccinate (AOT) was purchased from Acros. All chemicals were of analytic grade and used without further purification. Ultrapure water used in all experiments had a resistivity of 18.2 M\(\Omega\) cm, and was obtained from a Milli-Q system (Millipore).

Preparation of Mesoporous Silica Nanomaterials. In a typical procedure, 0.2 g of CTAB was dissolved in 96 mL of H\(_2\)O. After 0.28 mL of NaOH (5 M) was added, the mixture was vigorously stirred in a closed round-bottomed flask at room temperature for 30 min. A given amount of AOT was added to the solution. After 1.34 mL of TEOS was dripped to the mixture at 80 °C, the resulting mixture was vigorously stirred at 80 °C for 2 h. A white precipitate was obtained,
filtered, washed with pure water, and dried at 80 °C in air. The organic components of the product were removed by calcination in air at 550 °C for 5 h. The calcined mesoporous silica samples were named as S0, S1, S2 and S3 when the weight ratio (R) of AOT/CTAB was 0, 0.1, 0.2 and 0.5, respectively.

**Characterization.** X-ray diffraction (XRD) patterns of the calcined silica samples were recorded on a Holand PANalytical X'Pert PRO MPD X-ray diffractometer using Cu Kα radiation (λ = 0.1542 nm) operated at 40 kV and 40 mA. Scanning electron microscopy (SEM) observations were carried out on a Hitachi S-4300 field emission scanning electron microscope (FESEM). For transmission electron microscopy (TEM) observations, powder samples were added on carbon-coated copper grids and observed on a JEOL JEM-200CX transmission electron microscope at an acceleration voltage of 150 kV. Nitrogen adsorption-desorption measurements were performed on a Micromeritics ASAP 2010 accelerated surface area analyzer at -196 °C using the volumetric method. The calcined silica samples were dried at 200 °C before analysis. The Brunauer-Emmett-Teller (BET) specific surface areas were calculated by using adsorption data in $P/P_0=0.05-0.20$ (eight points collected). Pore size distributions were estimated from adsorption and desorption branches of the isotherms by using the Barrett, Joyner, and Halenda (BJH) method. Pore volumes were determined from the amounts of N2 adsorbed at the single point of $P/P_0=0.99$. 
Fig. S1 SEM images of calcined silica samples: a (S1), and b (S2), respectively.
Fig. S2 Nitrogen adsorption-desorption isotherms (a) and corresponding pore size distributions obtained from adsorption (b), and desorption (c) of calcined silica samples, respectively.
Table S1 Morphology and physicochemical properties of calcined silica samples synthesized at different AOT/CTAB weight ratios ($R$)

<table>
<thead>
<tr>
<th>Sample</th>
<th>$R$</th>
<th>Morphology</th>
<th>$d$ spacing$^a$ (nm)</th>
<th>Pore size distribution$^b$ (nm)</th>
<th>Surface area$^c$ (m$^2$/g)</th>
<th>Pore volume$^d$ (cm$^3$/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S0</td>
<td>0</td>
<td>nanospheres</td>
<td>4.6</td>
<td>2.61(2.6)</td>
<td>610.6</td>
<td>1.19</td>
</tr>
<tr>
<td>S1</td>
<td>0.1</td>
<td>nanoellipsoids</td>
<td>3.7</td>
<td>2.50(2.6)</td>
<td>662.6</td>
<td>0.565</td>
</tr>
<tr>
<td>S2</td>
<td>0.2</td>
<td>helical nanorods</td>
<td>4.0</td>
<td>2.49(2.5)</td>
<td>640.2</td>
<td>0.528</td>
</tr>
<tr>
<td>S3</td>
<td>0.5</td>
<td>multi-lamellar</td>
<td>—</td>
<td>2.93(2.5,3.6)</td>
<td>733.4</td>
<td>1.14</td>
</tr>
</tbody>
</table>

$^a$ Calculated from the XRD peak of (10) diffraction.

$^b$ Calculated from the N$_2$ adsorption (desorption) branch using the BJH method.

$^c$ Calculated from the N$_2$ adsorption branch using the BET method.

$^d$ Estimated from the single point amount adsorbed at $P/P_0=0.99$.

According to previous reports, the classical BJH method underestimates the pore width for MCM-41. Thus the pore width in the current results might also have been underestimated. It may be estimated more accurately on the basis of condensation pressure.$^{14b}$ For example, according to Table 1 in reference 14b, we estimated the pore width of S1 to be ca. 3.73 nm.