Supplementary Information

Synthesis of Yolk-Shell Mesoporous Silica Nanoparticles via Facile One-pot Approach

Jing-Chuan Song, a Fei-Fei Xue, b Xing-Xing Zhang, a Zhong-Yuan Lu, c and Zhao-Yan Sun* a

a State Key Laboratory of Polymer Physics and Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun, 130022, China. Fax: +86 431 85262969, E-mail: zysun@ciac.ac.cn.

b School of Construction and Environment Engineering, Shenzhen Polytechnic, Shenzhen 518055, China.

c State Key Laboratory of Supramolecular Structure and Materials, Institute of Theoretical Chemistry, Jilin University, Changchun 130023, China.

Experimental Section:

Chemicals. Formaldehyde (37 wt %) was purchased from Aladdin Industrial Corporation (Shanghai, China). 3-Aminophenol was purchased from Xiya Reagent (Chengdu, China). Tetraethylorthosilicate was purchased from Xilong Chemical Co., Ltd. Ammonia aqueous solution (25%) and ethanol were purchased from Beijing Chemical works (Guangdong, China). All chemicals were used as received. Distilled water was used throughout the work.

Preparation of yolk-shell mesoporous silica nanoparticles. The yolk-shell mesoporous silica particles were prepared via one-pot approach in the presence of phenolic resin and tetraethylorthosilicate (TEOS). Typically, 0.20 g of 3-aminophenol was dissolved in the solution containing water (15-21 ml) and ethanol (6-12 ml). Then some amount of TEOS (0.36-1.44 g) was added under stirring. After stirring for 15 min at 30 °C, 0.28 ml of formaldehyde was injected and the resulting mixture was stirred continually at 30 °C for 4 h. The particle size can be tuned by varying the concentration of TEOS and the volume ratio of ethanol/water. The detailed synthesis parameters are given in Table S1. The product was washed with ethanol for three times and was collected by centrifugation. To obtain yolk-shell mesoporous silica particles, the as-made resin/silica nanocomposite particles were calcinated in air from room temperature to 550 °C for 6 h at a rate of 1 °C/min.

Characterization. Transmission electron microscopy (TEM) measurements were conducted on a JEM-2010 microscope (JEOL, Japan) operated at 100 kV. Field emission scanning electron microscopy (FE-SEM) images were recorded on a FEI XL30 ESEM FEG electron microscopy operating at 25 kV. Diffuse reflectance Fourier-transform infrared (FT-IR) spectra were recorded on a Bruker Vertex 70 spectrometer. The nitrogen adsorption measurement was conducted on a Micromeritics Tristar 3000 system with micropore analysis at 77 K. Surface area was calculated using the Brunauer–Emmett–Teller (BET) method from nitrogen adsorption–desorption isotherms. The total pore volume was estimated from the adsorbed amounts at a relative pressure (P/P0) of 0.99. Before measurements, samples were degassed in a vacuum for 6 h at 180°C.
Table S1. Synthesis parameters of yolk-shell mesoporous silica particles.

<table>
<thead>
<tr>
<th>Sample</th>
<th>3-Aminophenol (g)</th>
<th>Formaldehyde (ml)</th>
<th>TEOS (ml)</th>
<th>Ethanol (ml)</th>
<th>NH$_3$·H$_2$O (mL)</th>
<th>Water (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>YSNPs-1</td>
<td>0.20</td>
<td>0.28</td>
<td>1.08</td>
<td>8</td>
<td>0.1</td>
<td>19</td>
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<tr>
<td>YSNPs-2</td>
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<td>8</td>
<td>0.1</td>
<td>19</td>
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<tr>
<td>YSNPs-3</td>
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<td>8</td>
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<td>19</td>
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<td>YSNPs-4</td>
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<td>12</td>
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<tr>
<td>YSNPs-5</td>
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<td>17</td>
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<td>YSNPs-6</td>
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<td>1.08</td>
<td>6</td>
<td>0.1</td>
<td>21</td>
</tr>
</tbody>
</table>

Fig. S1 FE-SEM image of as-made yolk-shell mesoporous silica nanoparticles. The arrows show a crushed particle which presents the shell and yolk clearly.
Fig. S2 FT-IR spectra of a) the obtained yolk-shell mesoporous silica nanoparticles after calcinating and b) as-synthesized resin/silica nanocomposites particles. The bands at 1508 cm⁻¹, 1445 cm⁻¹ are characteristic peaks of resin and most of the peaks disappear after calcinating (a).

Fig. S3 TEM image of yolk-shell mesoporous silica nanoparticles prepared at 15 ml of water (YSNPs-4).
**Fig. S4** The comparison of specific surface area for the mesoporous silica yolk/mesoporous silica shell nanoparticles prepared in our work and other yolk-shell silica nanoparticles reported in the literatures. 

**References**