# **Supporting Information**

Novel Mesoporous Silica Spheres with Ultra-Large Pore Sizes and Their Application in Protein Separation

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#### Synthetic details of the Carbon replicas:

The carbon replicas of the products were prepared by using different ultra-large pore mesoporous silica spheres as replicas and sucrose as the carbon source. Typically, 1 g of the B<sub>56</sub>-E-20 products was added to a solution containing 1.25 g of sucrose and 0.14 g of sulphuric acid in 5 g of water. The obtained mixture was kept in an oven for 6 h at 100 °C followed by temperature increase to 160 °C for another 6 h. After addition of 0.8 g of sucrose and 0.09 g of sulphuric acid and 5 g of water, the mixture was again subjected to the similar thermal treatment. Thereafter, the composites were pyrolyzed in a nitrogen flow at 900 °C for 4 h. The carbon replicas were recovered after dissolution of the silica framework in a 5 wt% solution of hydrofluoric acid by filtration, washed several times with ethanol and dried at 120 °C.

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## Figure S1



Figure S1. The SEM images of the products. a and b, c and d, e and f are the low and high magnification images of  $B_{56}$ -E-20,  $B_{56}$ -D-33 and  $B_{97}$ -D-40, respectively.



## Figure S2:

Figure S2, SEM (a) and TEM (b) images of the carbon replicas from  $B_{56}$ -E-20.

#### Thermogravimetric Analysis (TGA)

The thermogravimetric analysis (TGA) curve of the mesoporous silica spheres ( $B_{56}$ -E-20) measured in air (Fig. S4) indicates that there are two significant weight-change steps. During the first step, which takes place from 25 to 170 °C, a small weight loss of 1.2 % occurs, due to the loss of water adsorbed on the surface. The second large weight-loss step of 21 %, which occurs between 175 and 750 °C, can be ascribed to the loss of the template Brij 56 and the additive APS.



**Figure S3.** The representative thermogravimetric analysis (TGA) curve of the mesoporous silica spheres (B<sub>56</sub>-E-20).

### **FTIR results:**

The postulation that esters work as swelling agent has been supported by FTIR spectroscopy. The IR spectrum of the as-synthesized  $B_{56}$ -D-33 shows bands at 1640 cm<sup>-1</sup> and 1740 cm<sup>-1</sup>, which are characteristic of the amino group (-NH<sub>2</sub>) and the ester group (-COOCH<sub>2</sub>-), respectively. The disappearance of the C-H groups (2850

cm<sup>-1</sup>-3000 cm<sup>-1</sup>, 1350 cm<sup>-1</sup>-1500 cm<sup>-1</sup>) and the ester group (1740 cm<sup>-1</sup>) from the IR spectra of the extracted sample can attributed to that the surfactant and ester have been removed by the extraction. These results provide evidence that the ester molecules penetrated into the core of micelles. At the same time, esters groups reacted with APS produce amides (-CONH-), which resulted in the appearance of amide bands in the IR (1540 cm<sup>-1</sup>) of the extracted products.



Figure S4. a, b and c are the as-synthesized, extracted and calcined B<sub>56</sub>-D-33 samples, respectively.

Figure S5, SEM images of the siliceous hollow microspheres



Figure S5. SEM images of the siliceous hollow microspheres.