Supporting Information

Recycle of Silicate Waste into Mesoporous Materials

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1. Experimental:

Synthesis of recycled mesoporous SBA-15 and SBA-16 silica

Recycled mesoporous SBA type silica samples were also synthesized using P123 ($EO_{20}PO_{70}EO_{20}$, BASF) and F127 ($EO_{106}PO_{70}EO_{106}$, BASF) by the procedure similar to that described in MCM-41. The 1.0 g of triblock surfactants was dissolved in 10 g deionized water, and 30 g silicate waste solution was added into the surfactant solution under vigorous stirring. Finally, 2.0 M HCl solution was slowly added until the pH of the solution decreased to 3.0. The composition of the final mixture was P123: HCl: H_2O: Na_2O: SiO_2 = 0.0008: 1.53: 48.80: 0.42: 1.00 for the synthesis of SBA-15 mesoporous silica, while F127: HCl: H_2O: Na_2O: SiO_2 = 0.0005: 1.53: 48.8: 0.42: 1.00 for the synthesis of SBA-16 mesoporous silica. The solution was stirred for about 1 h at ambient temperature to get a homogeneous solution, then charged into the stainless autoclave, and heated to 393 K for 2 days. The white product was filtered, washed with water/ethanol and calcined at 873 K for 6 h to remove the triblock copolymer.

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Characterization

Transmission electron microscopy (TEM) images were obtained with an EM912 Omega electron microscope with an acceleration voltage of 120 kV. The scanning electron microscopy (SEM) images were obtained using a Hitachi S-4700 microscope operated at an acceleration voltage of 10 kV. HR-TEM images were obtained by using transmission electron microscope (JEM 2200-FS) operated at 200 kV and HR-SEM images by using ultra high resolution-scanning electron microscope (Hitachi S-5500) operated at 30 kV. Powder X-ray diffraction (XRD) patterns of the samples were recorded by using a Rigaku Miniflex diffractometer with Cu Ka radiation using a Ni β-filter at a scan rate of 0.2 °/min and

operating at 30 kV and 15 mA.

The nitrogen adsorption-desorption isotherms were measured at 77 K using a KICT SPA-3000S system. Specific surface areas of the samples were determined by nitrogen adsorption branch in the relative pressure range from 0.05 to 0.2 using the Brunauer-Emmett-Teller (BET) equation. Total pore volumes were determined from the amount of gas adsorbed at the relative pressure of 0.99. Pore size distribution (PSD) was calculated from the adsorption branch by the Barrett-Joyner-Halenda (BJH) method.

The chemical composition of the samples was measured using an inductively coupled plasma mass spectrometer (ICP-MS). The ICP-MS used was performed using X-series system in which the liquid samples were introduced using a cross-flow nebulizer and a spray chamber. The aerosol was carried by 0.9 L/min argon. The Ar gas flow for the plasma was 15 L/min with an auxiliary gas flow of 0.8 L/min. The rf power was set to 1000 W. Viscosity of sample solutions was measured using a capillary viscometer (Newtonian fluids) at ambient conditions.

High resolution NMR spectra were collected using a Varian UNITY INOVA 500 NMR Spectrometer with a 11.8 T (500 MHz) wide-bore Oxford magnet. The MAS experiments were performed with a probe with 5 mm ZrO_2 rotors at a spinning rate of 6 KHz. The ^{29} Si MAS experiments were run with a recycle time of 400 s, 90 ^{o} pulse lengths, a 100 kHz bandwidth and 200 scans in each experiment.

2. Results

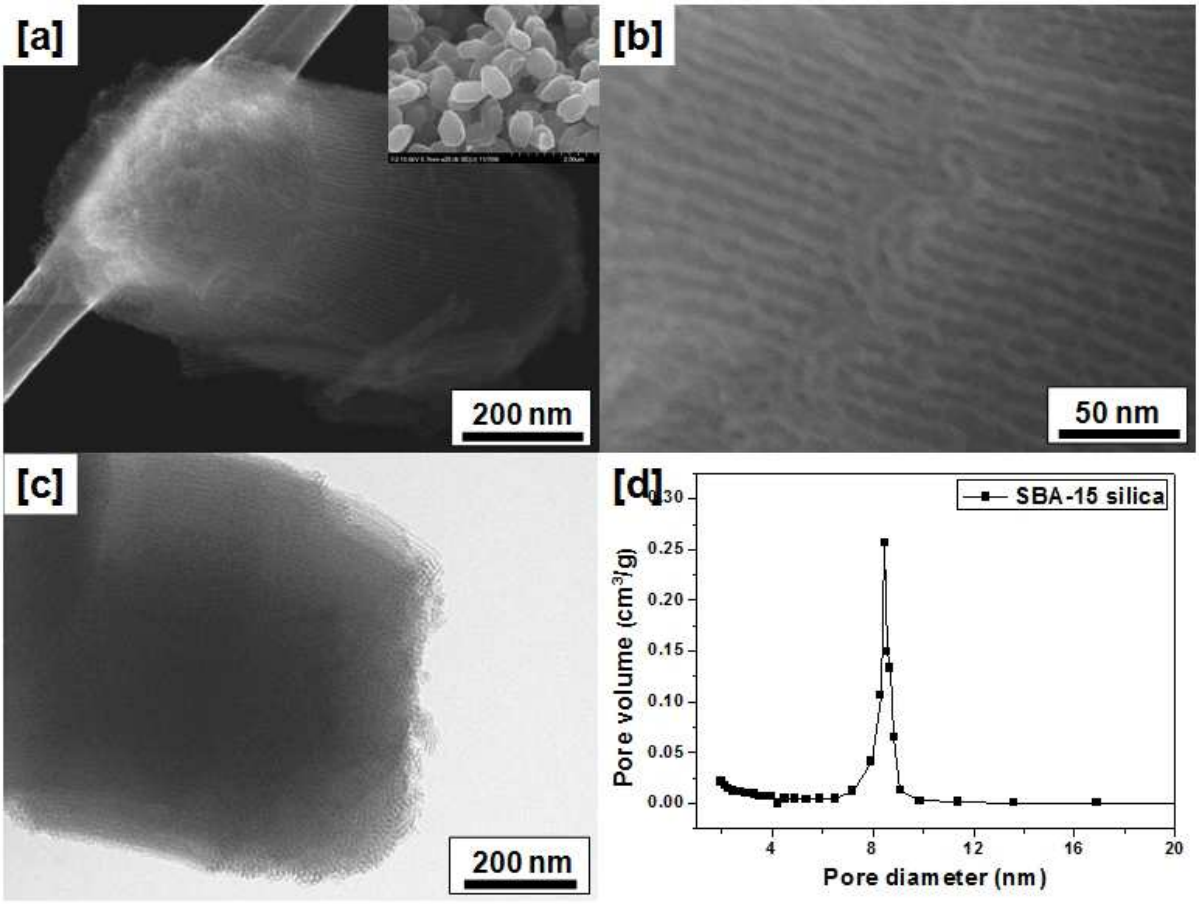

Figure S1. Representative SEM (a), HR-SEM (b) and TEM (c) images and pore-size distribution curve (d) of SBA-15 silica host.

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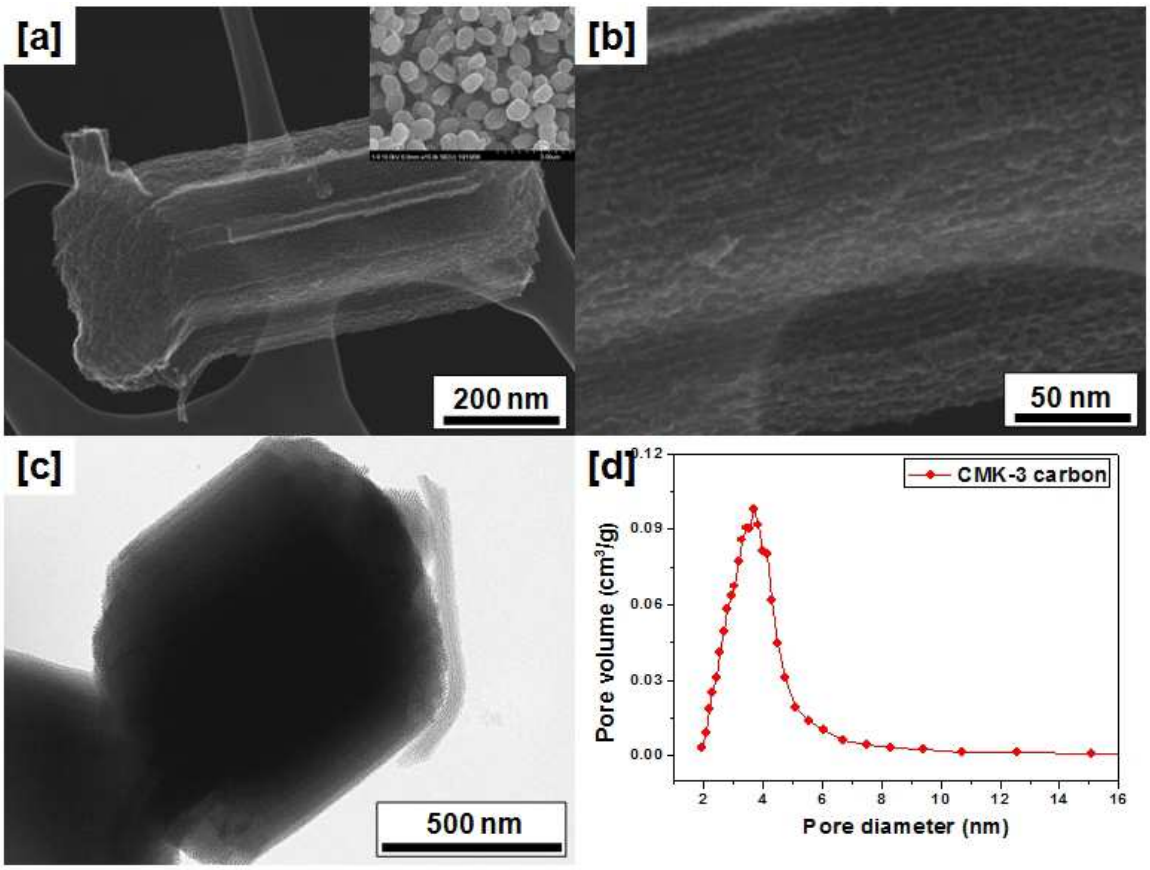

Figure S2. Representative SEM (a), HR-SEM (b) and TEM (c) images and pore-size distribution curve (d) of CMK-3 carbon replica.

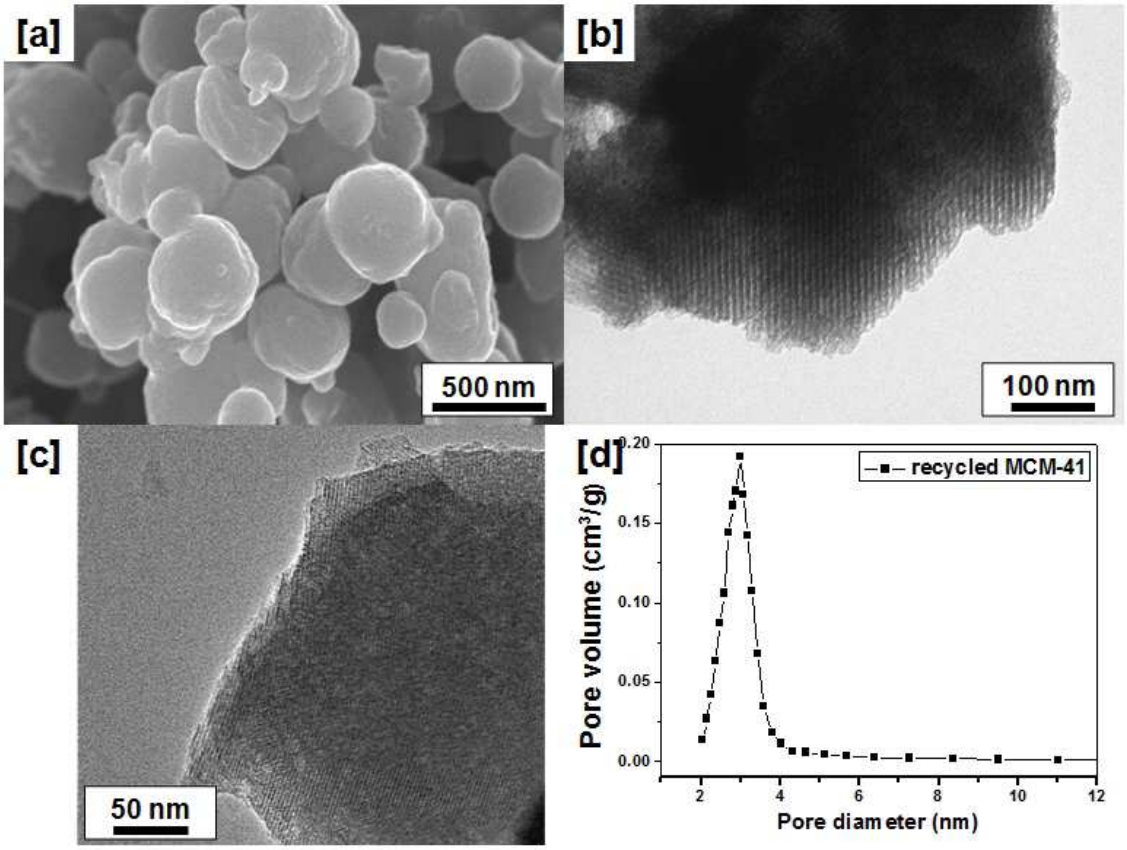

Figure S3. Representative SEM (a), TEM (b) and HR-TEM (c) images and pore-size distribution curve (d) of recycled MCM-41 silica.

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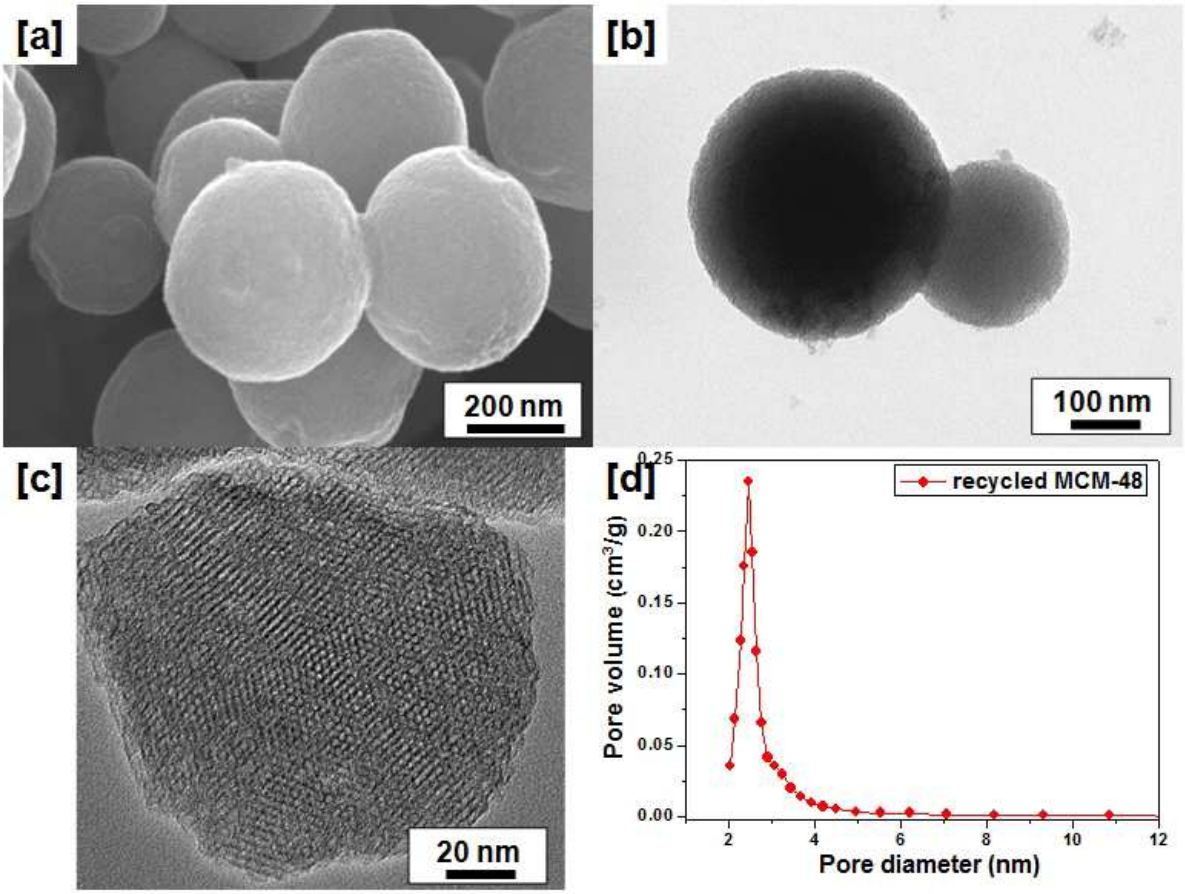

Figure S4. Representative SEM (a), TEM (b) and HR-TEM (c) images and pore-size distribution curve (d) of recycled MCM-48 silica.

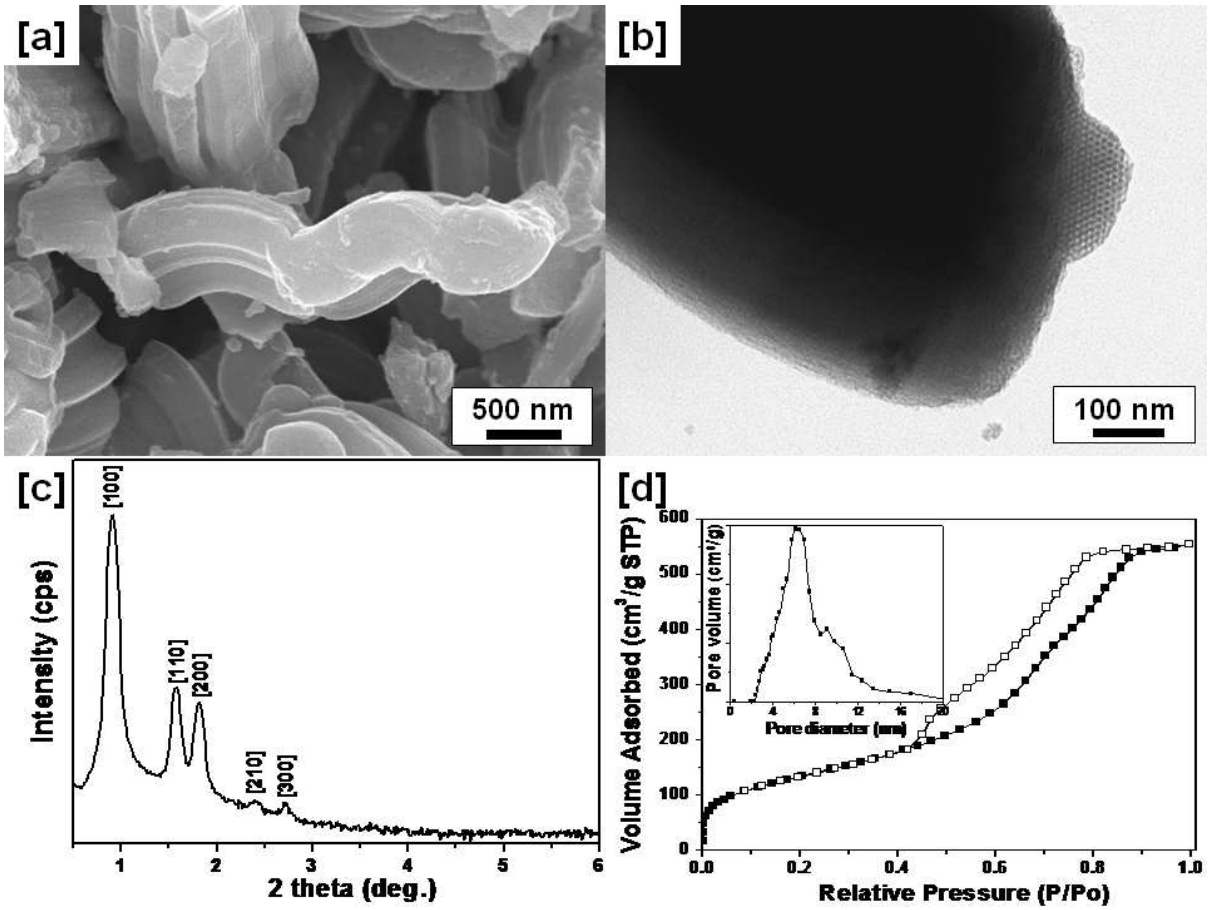

Figure S5. Typical SEM (a) and TEM (b) images of recycled mesoporous silica (SBA-15-like) synthesized using P123 surfactant, and corresponding XRD (c) and N_2 adsorption isotherm (d).

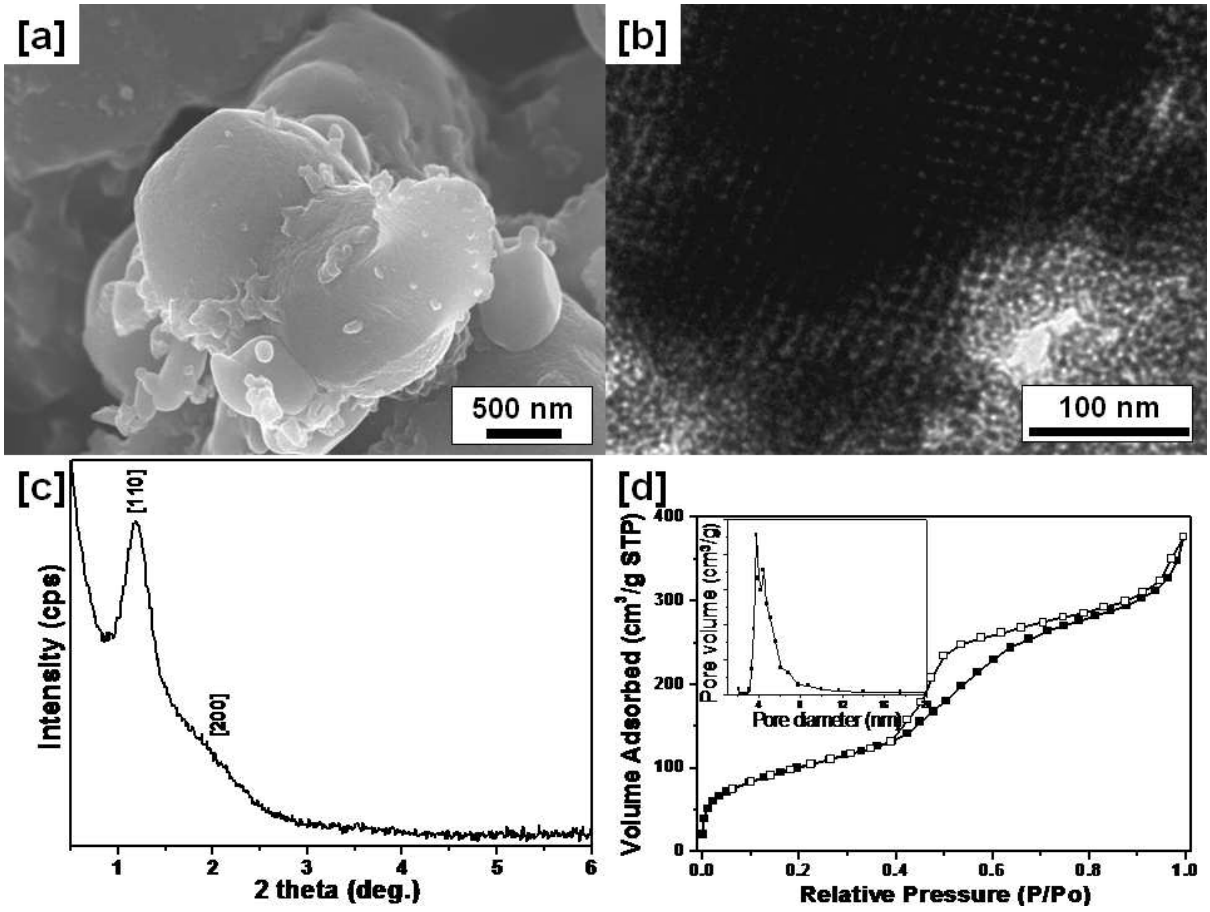

Figure S6. Typical SEM (a) and TEM (b) images of recycled mesoporous silica (SBA-16-like) synthesized using F127 surfactant, and corresponding XRD (c) and N_2 adsorption isotherm (d).