Syntheses

SBA-15 powder was prepared using the triblock copolymer, $EO_{20}PO_{70}EO_{20}$ (Pluronic P123, BASF) and tetraethoxysilane (TEOS, 98%, Acros). In a typical synthesis, 1 g of P123 was dissolved in 30 g of 2 M HCl and 7.5 g of H_2O, then 2.08 g of TEOS or 1.96 g of sodium silicate was added. The slurry was hydrothermally treated at 100 ºC for 48 h after stirring at 40 ºC for 16 h . The product was filtered off, dried at 80 ºC for 10 h.

The synthesis procedures for SBA-16 and FDU-1 powder were similar to that of SBA-15 powder, except for SBA-16, $EO_{106}PO_{70}EO_{106}$ (Pluronic F127, BASF) was used as the structure directing agent (SDA) and 1.1g of KCl was added to the synthetic mixture, and for FDU-1, $EO_{39}BO_{47}EO_{39}$ (Polyglycol B50-6600, Dow) was the SDA and 0.5 M instead of 2 M HCl was used.

MCM-41 was prepared by vigorously stirring the gel mixture at ambient temperature. The typical gel composition is, expressed as mass ratios, 0.4 cetyltrimethylammonium (CTAB); 18 H_2O; 10 concentrated $NH_3 \cdot H_2O$ (28 %); 2.08 TEOS. The product was filtered off, dried at 80 ºC for 10 h.

Mesoporous films were synthesized by using dip-coating rapid solvent evaporation method. P123 and F127 were used as the SDAs for MF-1 and MF-2 respectively. In typical, 1.0 g of P123 or 0.8 g of F127 were dissolved in 10 g of ethanol, and 2.08 g of TEOS was added to another solution containing 5 g of ethanol and 0.3 g of 2M of HCl. The above two solutions were mixed after separately stirring for 2 h and the mixture was further stirred for another 2 h. Finally the films (MF-1 and MF-2) were deposited on glass wafers by dip coating and dried at 40 ºC for 48 h.

Macro- mesoporous membranes were prepared by using PS spheres (the radii were 270 nm, 300 nm and 130 nm for MMM-1, MMM-2 and MMM-3, respectively) and tri-block copolymer P123 as co-templates. Monodisperse PS spheres were patterned on silicon wafer by self-assembly. Then the voids were filled with a drop of siliceous sol (the same as the mother solution of MF-1) by capillary flow. The samples were dried at 40 ºC for 48 h.

Ethanol extraction was conducted by refluxing 0.2 g of sample in 150 ml of absolute ethanol at 78 ºC for 24 h. Thermocalcination for samples was carried out in an oven at 550 ºC for 5 h.

Microwave assisted functionalization was done by suspending 0.4 g of digested sample in 20 mL of 10 % v/v solution of (3-thiolpropyl)-trimethoxysilane (MPTS) in tetrahydrofuran or ethanol within the microwave sample preparation system model MK-Ⅱ and was given microwave irradiation for 5 min. The microwave oven was operated at approximately 1200 W and the working frequency is 2450 MHz.

N_2 measurements were performed at 77K using a Micrometities Tri star-3000 analyzer. TEM photographs were obtained with a TEM-1200EX microscope operated at 80 KV. PXRD were conducted with a Regaku D/MAX-IIA using Cu-K α radiation. Solid-state ^{29}Si NMR experiments were performed on a DSX300 spectrometer produced by Bruker Company with a frequency of 59.63 MHz, a recycling delay of 600s and a radiation frequency intensity of 62.5 KHz. TG curves were obtained with Thermogravimetric Analyzer TGA 7. The temperature range is 25—1000 ºC. Chemical elemental analyses (C, H, N) were done with Elemental Analyzer Carlo Erba 1106. Sulfur contents were measured by Oxygen Burning Method. FT-IR curves were obtained with AUATAR 360 FT-IR (Nicolet) analyzer. UV-vis curves were obtained with JASCO V-550 UV-VIS Spectrometer.

Figure 1 XRD patterns of a) as-made, b) microwave digested and c) calcined SBA-15. Inset, N_2 sorption curves of calcined and digested SBA-15.

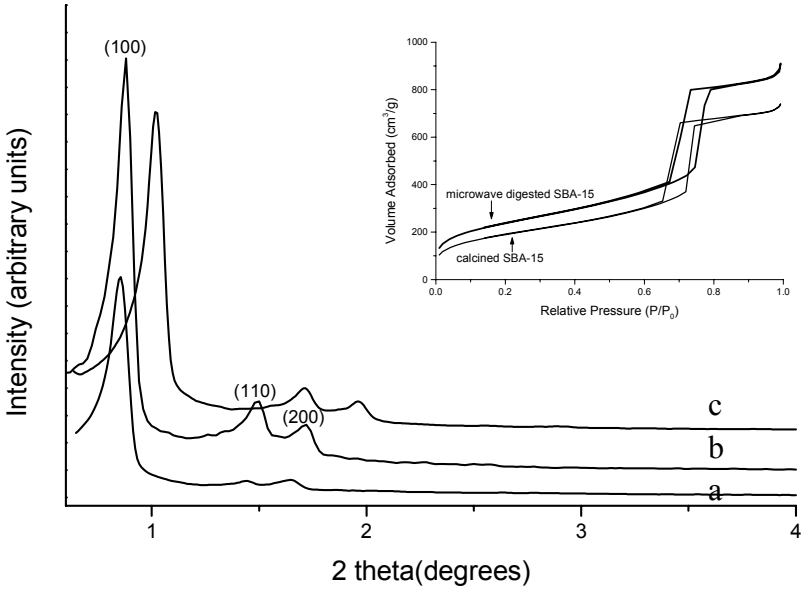

Figure 2 SEM image of microwave digested SBA-15 powders. The macro-morphology is wheat-like and is as same as that of typical as-made SBA-15.

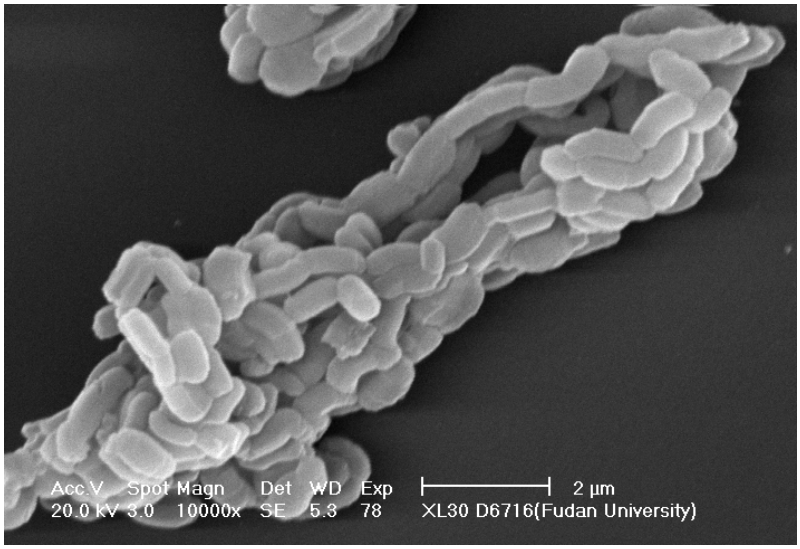

Figure 3 Photograph of Pb^{2+} adsorbed by thiol- functionalized digested (left), extracted (middle) and calcined (right) SBA-15 samples in ethanol solutions. The deep yellow left one indicates the most Pb^{2+} loading amount.

Figure 4 ^{29}Si MAS NMR of calcined, digested and as-made SBA-15.

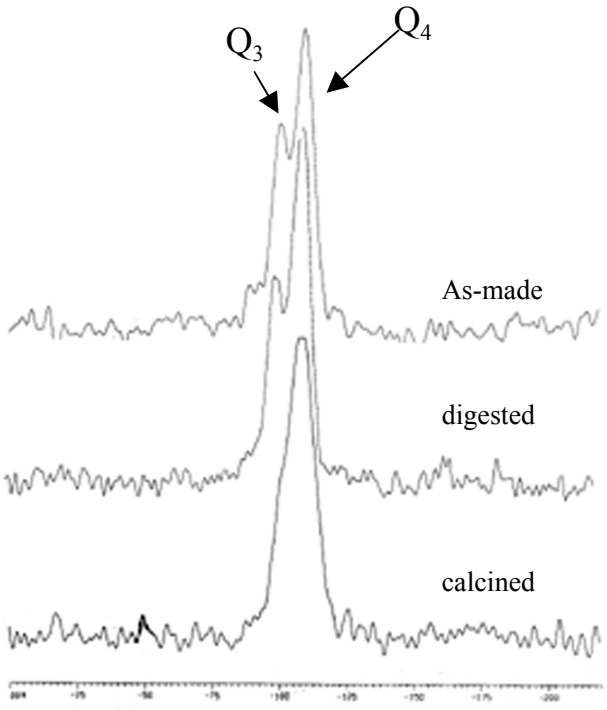

Figure 5 TG curves of calcined and microwave digested SBA-15.