Facile synthesis of mesoporous MOF/silica composites

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Experimental

Materials

$Cu(NO_3)_2 \cdot 3H_2O$, cetyltrimethylammonium bromide (CTAB), tetraethoxysilane (TEOS), 1,3,5-Benzenetricarboxylic acid (H_3BTC) and ethanol were supplied by Sinoreagent. All solvents and chemicals are of analytical reagents and were used without further purification.

Characterization

The powder XRD patterns of all the samples were recorded on a Bruker D8 ADVANCE diffractometer with Cu Kα radiation at 40 KV and 30 mA. The N_2 adsorption-desorption isotherms were collected at 77 K using Micromeritics ASAP 2460 porosimeter. Prior to the measurement, the samples were degassed at 423 K overnight. The total pore volume (Vt) was determined as the volume of liquid nitrogen adsorbed at a relative pressure of 0.99. The pore size distributions were obtained using BJH methods from the desorption branch. The micropore volume (V_{micro}) was estimated using t-plot method and mesopore volumes were obtained by subtracting V_{micro} from the total pore volume. Scanning Electron Microscope (SEM) pictures were taken by a FEI Quanta 250 FEG device and elemental mapping was performed using an EDX Bruker Quantax 400. Transmission Electron Microscope (TEM) pictures were obtained using a FEI Tecnai G2 F20 device operated at 200 KV. XPS were recorded performed using a Thermo Scientific Escalab 250Xi instrument equipped with Al Kα radiation (hv=1486.6eV). Binding energies for the high-resolution spectra were calibrated by setting C 1s at 284.8 eV. Thermal gravimetric

analysis (TGA) measurements were performed on a DSC 8000 Series Perkin Elmer thermal analysis system. Samples were heated in a continuous-flow of N_2 gas with a ramp rate of 10 ^oC/min from 30 up to 800 ^oC.

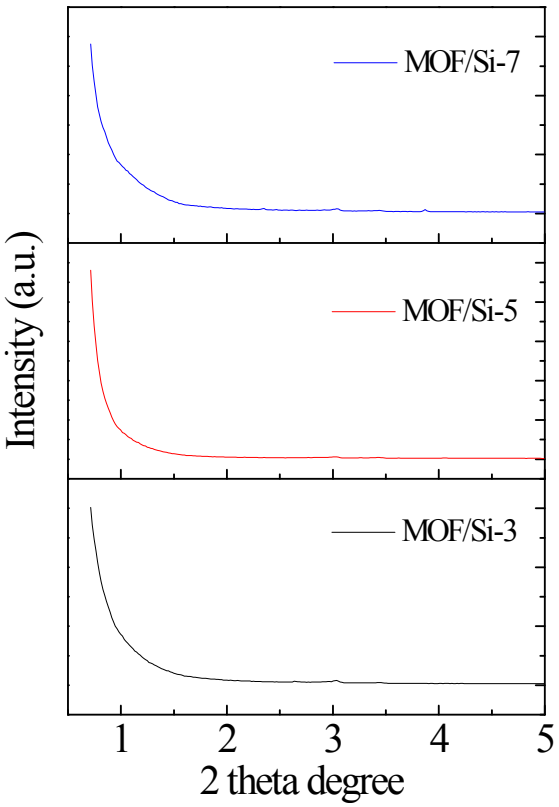


Figure S1. Small-angle XRD patterns of samples (a) MOF/Si-3; (b) MOF/Si-5; (c) MOF/Si-7

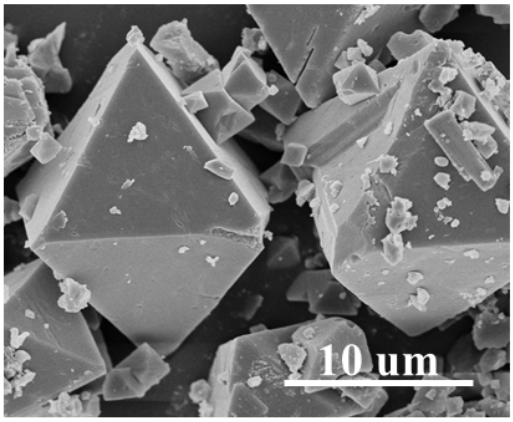


Figure S2. SEM image of pure HKUST-1

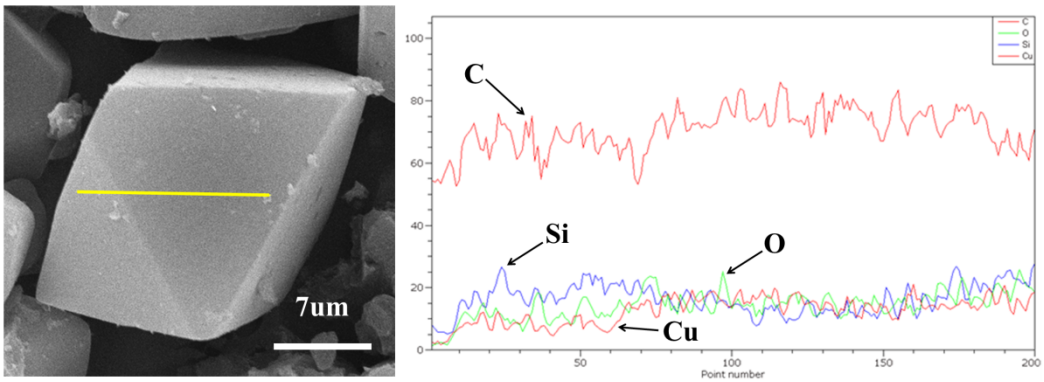


Figure S3. SEM image and corresponding line scans of MOF/Si-5 sample.

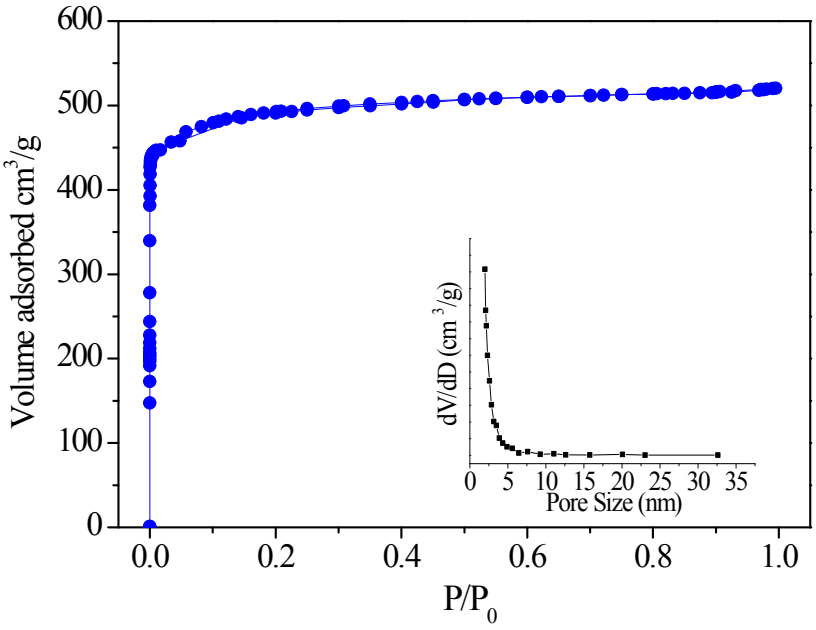


Figure S4. Nitrogen adsorption-desorption isotherms and mesopore size distributions (inserted) of pure HKUST-1

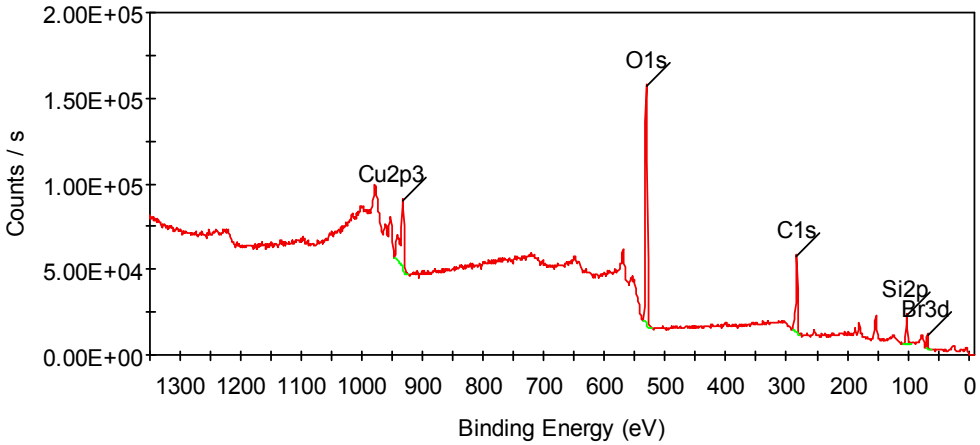
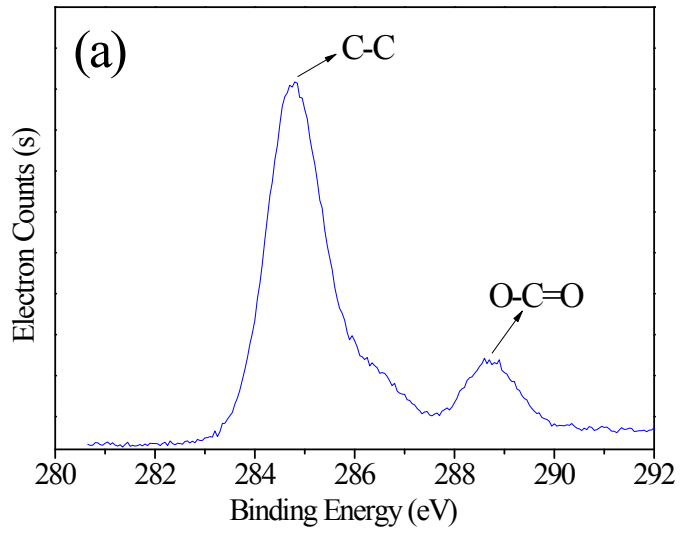
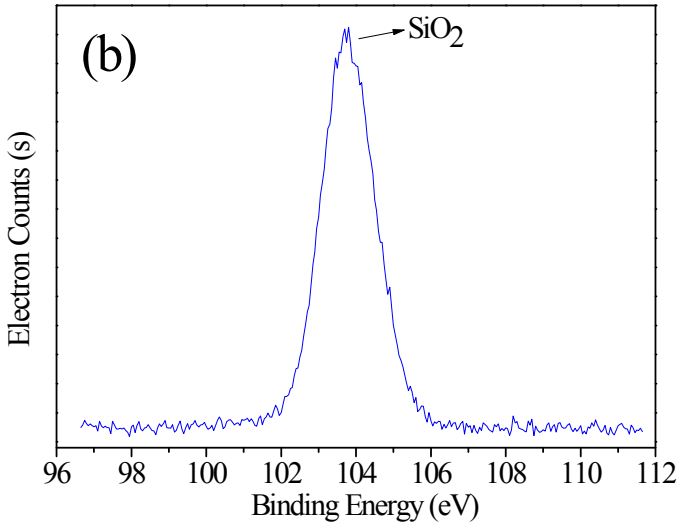


Figure S5. Survey XPS spectra of MOF/Si-5 sample





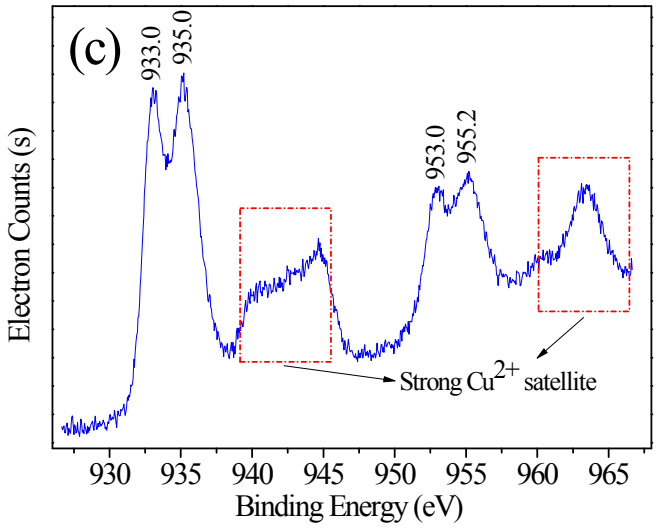


Figure S6. XPS spectra in the C1s region(a), Si 2p region(b) and Cu 2p region(c) for MOF/Si-5 sample

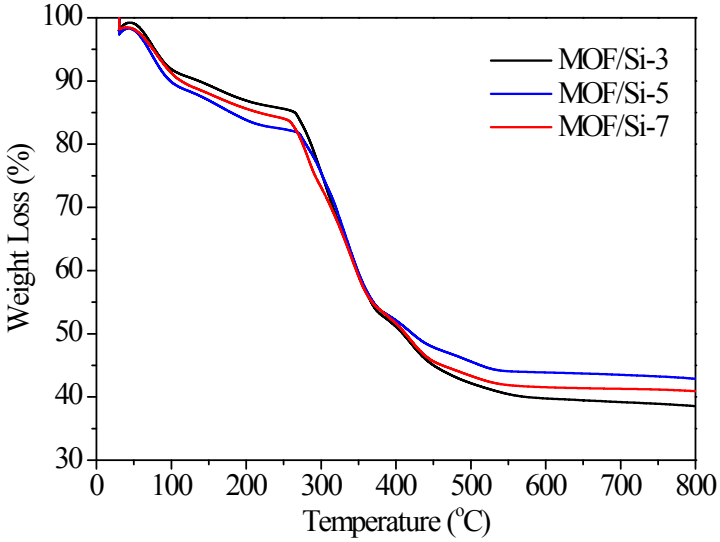


Figure S7. TGA curves of the as-synthesized samples.