Supplementary Material (ESI) for Chemical Communications

Supporting Information

Rapid Synthesis of Zeolitic Imidazolate Framework-8 (ZIF-8) Nanocrystals in Aqueous System

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Materials

$Zn(NO_3)_2 \bullet 6H_2O$ (98%) and 2-methylimidazole (99%) were purchased from Sigma-Aldrich, and used as received without further purification.

Synthesis of ZIF-8 nanocrystals

Nanometer-sized ZIF-8 crystals with hexagonal shape were synthesized via rapid pouring an aqueous solution of $Zn(NO_3)_2 \bullet 6H_2O$ into an aqueous solution of 2-methylimidazole and the mixture was stirred at room temperature for 5 min. In addition, the two solutions were all filtered by filter paper before mixing. In a typical synthesis, 1.17 g of zinc nitrate (3.95 mmol) in 8 DI water was added into a solution of 2-methylimidazole (22.70 g, 276.50 mmol) in 80 g DI water, and the molar ratio of 2-methylimidazole to zinc was 70:1. In order to examine the effect of molar ratio of 2-methylimidazole to zinc on the particle size, we fixed the quantity of zinc added, but increased the amount of 2-methylimidazole to 32.4 g and 64.8 g when the molar ratio was increased to 100:1 and 200: 1, respectively. The product was collected by repeated centrifugation (6500 rpm, 30 min) and washed by DI water for three times. A small part of products was re-dispersed in methanol to form a stable colloid suspension, used for TEM and DLS characterizations, and another part of the product was dried at 65 ^{o}C overnight in a drying oven.

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Characterization of ZIF-8 nanocrystals

Powder x-ray diffraction (XRD) patterns were recorded at room temperature on a Bruker D8 ADVANCE diffractometer in transmission geometry using CuKα radiation (λ = 1.54059 Å) at 40 kV and 40 mA. In order to examine the thermal stability of product in vacuum, samples were measured from 30 to 450 ^{o}C on an in-situ non-ambient Bruker A8 ADVANCE diffractometer at 40 kV, 40 mA with CuKα radiation. Fourier-transform infrared (FTIR) spectra were obtained in a Nicolet iS10 system. Samples were mixed and ground with KBr for FTIR measurements in the wavenumber range of 2000-500 cm^{-1}. Dynamic light scattering (DLS) measurements were performed on a Zetasizer Nano ZS from Malvern instrument equipped with a He-Ne laser (λ = 632.8 nm) in the backscattering detection mode. Field-emission scanning electron microscope (SEM) pictures were taken by a FEI Quanta 600 FEG, and the acceleration voltage was 30 kV. Transmission electron microscope (TEM) pictures were obtained by evaporating a drop of methanol dispersion of ZIF-8 nanocrystals on carbon-coated copper grids followed by the measurement on Titan ST TEM operating at 300 kV. The mean particle size of the product was determined by manual measurement of about 300 crystals in SEM and TEM pictures. Thermal gravimetric analysis (TGA) measurements were performed on a Netzsch STA 449 F1 Jupiter thermoanalyzer. For this purpose, ca. 10 mg samples were filled into an alumina crucible and heated in a continuous-flow of helium gas with a ramp rate of 5 °C/min from 40 up to 800 °C. Nitrogen physisorption isotherms were measured at 77 K on an automatic volumetric adsorption apparatus (Micromertics ASAP 2420). The samples were filled into glass ampoules and outgassed in high vacuum at 473 K for 24 h before the start of the sorption measurements.

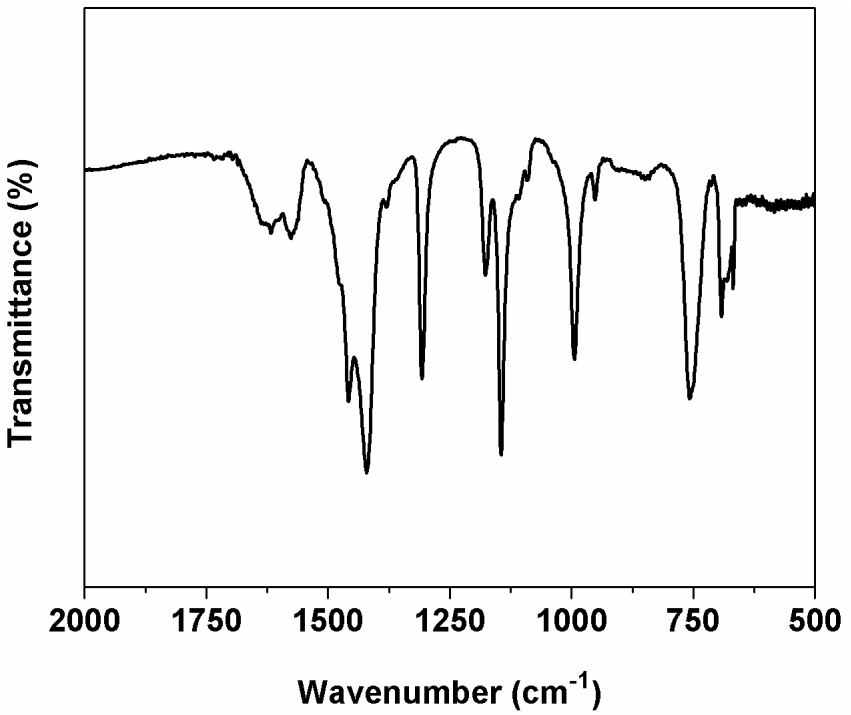

Fig. S1 FTIR spectrum of as-synthesized ZIF-8 nanocrystals

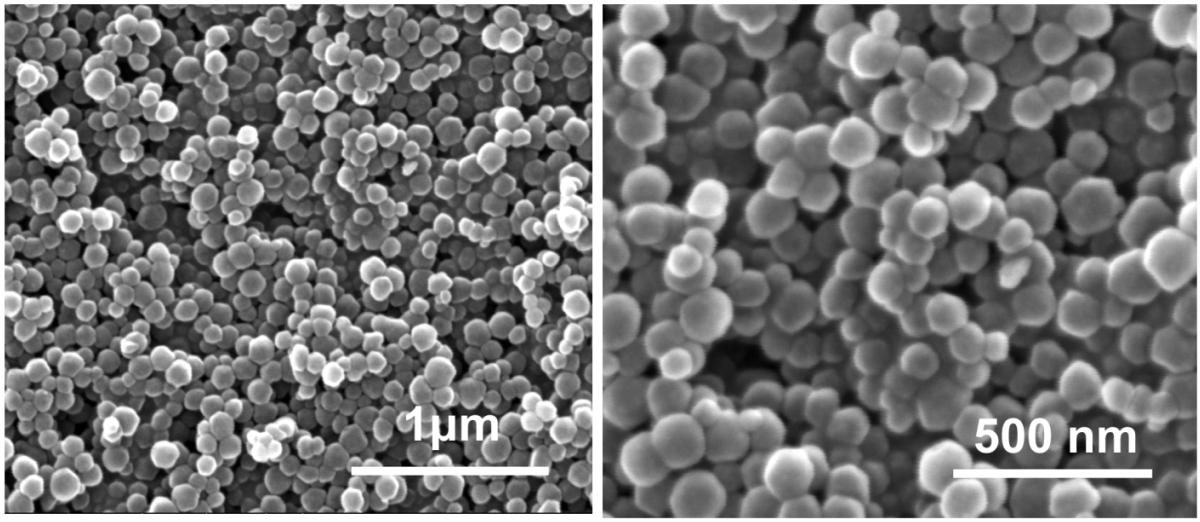

Fig. S2 SEM pictures of as-synthesized ZIF-8 nanoparticles

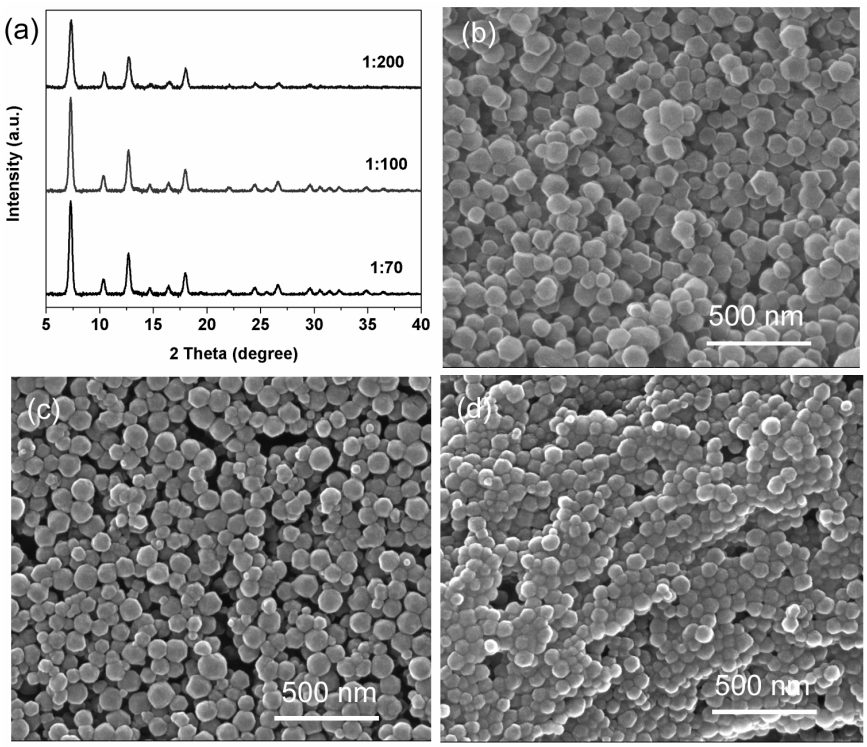

Fig. S3 (a) XRD and (b-d) SEM pictures of as-synthesized ZIF-8 nanoparticles with various molar ratio of 2-methylimidazole to zinc: (b) 70, (c) 100, (d) 200.

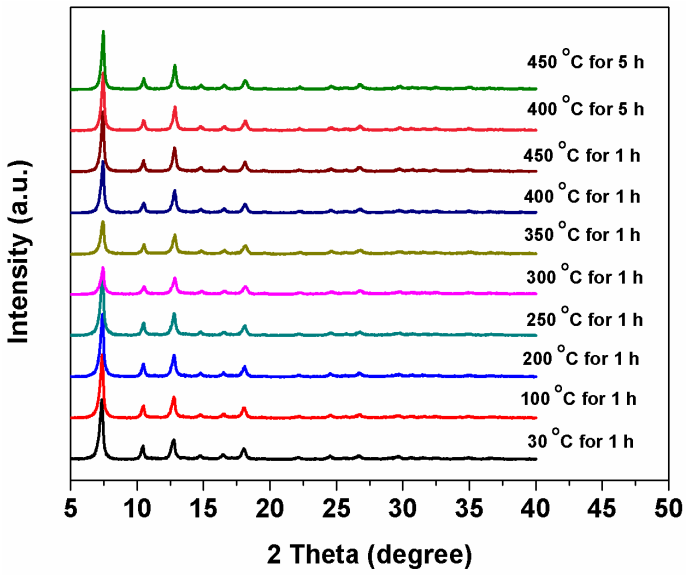

Fig. S4 XRD patterns of as-synthesized samples measured on an in-situ non-ambient XRD diffractometer

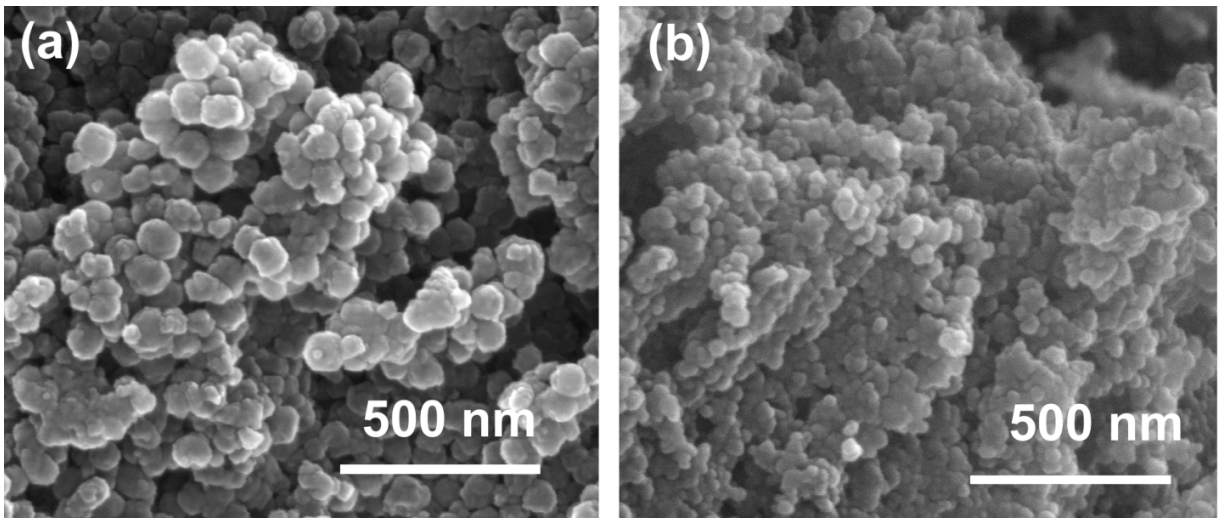

Fig. S5 SEM images of ZIF-8 nanocrystals calcinated in air: (a) 300 ^{o}C for 24 h, and (b) 400 ^{o}C for 5 h