Electronic Supplementary Material (ESI) for CrystEngComm.

Supporting Information

In Situ Reconstruction of ZIF-8 Loaded on Fibrous Supports

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Materials and Characterization Methods

Materials

All other chemicals were obtained from commercial sources and were used without further purification.

Characterization

High-resolution scanning electron microscopy (HRSEM) images were obtained using a JEOL JSM-IT-500HR SEM and JEOL JSM-7800F Prime SEM. Samples were coated with Au for 20 seconds using an SBC-12 sputter coater. Energy dispersive X-ray spectroscopy (EDS) elemental mapping results were obtained on a JSM-IT-500HR SEM (15 kV). Powder X-ray diffraction (PXRD) patterns were collected on an X-ray diffractometer (Bruker D2 PHASER or D8 ADVANCE). N_2 sorption data at 77 K were collected on a BELSORP Max II. Before gas adsorption-desorption measurement, all samples were activated under vacuum for 10 h at 120 °C.

Experimental Details

Synthesis of 900 ± 5 nm ZIF-8: 219.5 mg (1 mmol) $Zn(OAc)_2$ and 656.8 mg (8 mmol) 2-methylimidazole (2-MIM) was separately dissolved into 20.0 mL methanol (MeOH) as stock solutions. Then, them were mixed and stirred for 5 min. The mixture was aged at room temperature for 12 h. The as-synthesized product was washed by methanol three times. The resulting material was suspended in methanol for further use.

Synthesis of 15 ± 5 µm ZIF-8: 588 mg (2.0 mmol) $Zn(NO_3)_2 \cdot 6H_2O$ was dissolved in 40 ml methanol. 324 mg (4.0 mmol) 2-MIM and 538 mg (7.9 mmol) sodium formate ($NaHCO_2$) were dissolved in 40 ml methanol. The molar ratio of Zn/2-MIM

/$NaHCO_2$/MeOH was set at 1:2:4:1000. The ligand solution was poured into the metal solution under stirring. After mixing, stirring was immediately stopped. The solution was heated at 90 ℃ for 24 hours in a Teflon-lined autoclave. The product was rinsed by methanol for three times. The resulting material was suspended in methanol for further use.

Preparation of ZIF-8⊂fibrous support: a prescribed amount of 120 mg/ml ZIF-8 suspension in methanol was drop-casted onto a piece of fibrous support (2×2 cm cotton fabric, filter paper or rockwool) and was allowed to evaporate naturally under room temperature. Then this process was repeated 2-3 times to afford three composites denoted as ZIF-8⊂cotton, ZIF-8⊂filter, and ZIF-8⊂rockwool, respectively.

Degradation of ZIF-8⊂fibrous support: ZIF-8⊂cotton, ZIF-8⊂filter, and ZIF-8⊂rockwool, was placed on a glass slide and then loaded into a container containing an acetic acid/ethylene glycol mixture ($V_{acetic\ acid}$: $V_{ethylene\ glycol}$ = 3:1) at room temperature for 1 h to give ZIF-8⊂cotton-DE, ZIF-8⊂filter-DE, and ZIF-8⊂rockwool-DE, respectively.

In situ vapor-assisted reconstruction (VAR) process: ZIF-8⊂cotton-DE, ZIF-8⊂filter-DE, and ZIF-8⊂rockwool-DE were placed on a glass slide and loaded into a sealed stainless steel chamber (Figure S4) along with a small amount of methanol. The glass slide was suspended above the solvent layer. The chamber was placed in a 70 ℃ oven for 24 h. After cooling to room temperature, the sample was taken out, collected, and denoted as ZIF-8⊂cotton-RE, ZIF-8⊂filter-RE, and ZIF-8⊂rockwool-RE, respectively.

Estimate the porosity of fibrous materials: Three fibrous materials were cut into square-shaped samples. The weight of each sample and the weight of the sample filled with water were measured by electronic balance. The porosity of these samples was calculated based on the retained water volume divided by the fibre mass which was 4.4, 2.3 and 12.5 cc/g for ZIF-8⊂cotton, ZIF-8⊂filter and ZIF-8⊂rockwool, respectively.

Supplementary table

Table S1. ZIF-8 loading capacity of ZIF-8⊂cotton and ZIF-8⊂filter calculated from the N_2 uptake capacity.

Samples	ZIF-8⊂cotton	ZIF-8⊂filter	ZIF-8
N_2 uptake capacity at 0.95 P/P_0	118	164	410
ZIF-8 loading capacity	29 wt%	40 wt%	N/A

Supplementary Figure

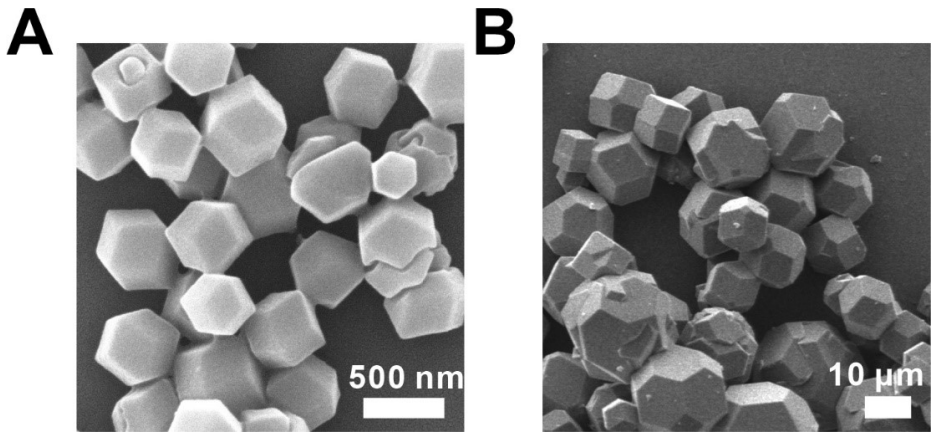

Fig. S1 SEM images of (A) small and (B) large ZIF-8 particles

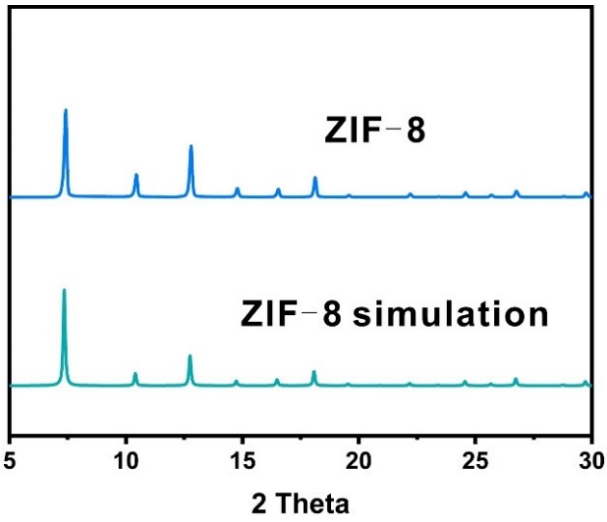

Fig. S2 The PXRD pattern of as synthesized ZIF-8 and ZIF-8 simulated from its crystal structure

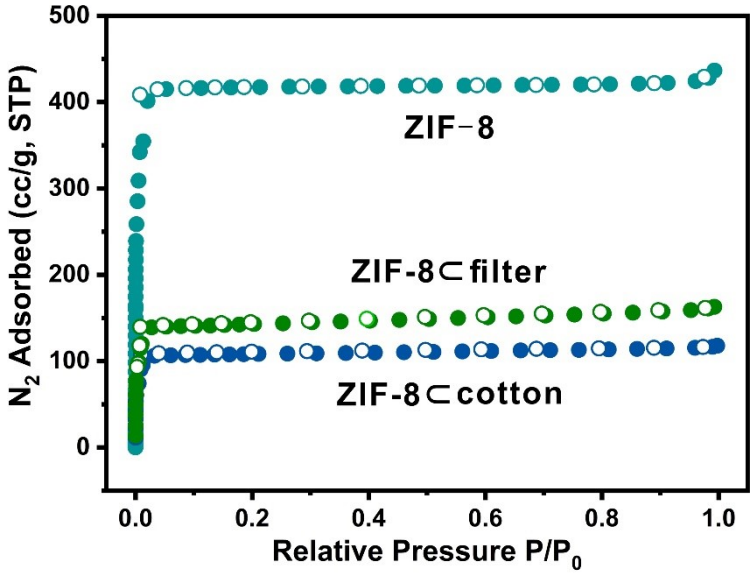

Fig. S3 77 K N_2 sorption isotherms of neat ZIF-8, ZIF-8⊂cotton and ZIF-8⊂filter

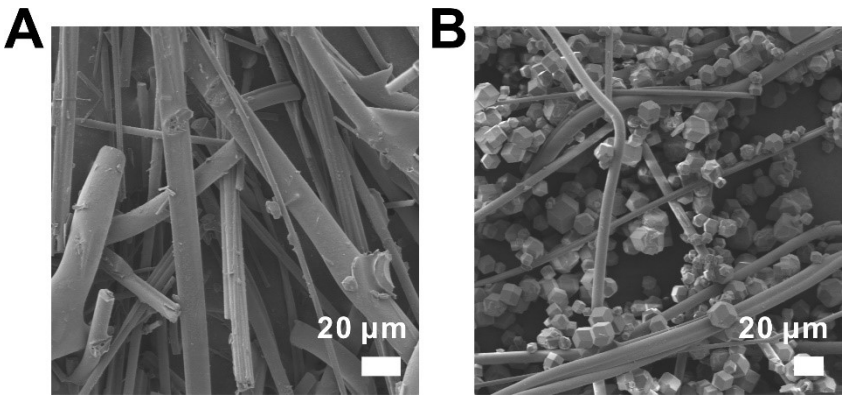

Fig. S4 SEM images of (A) rockwool and (B) ZIF-8⊂rockwool

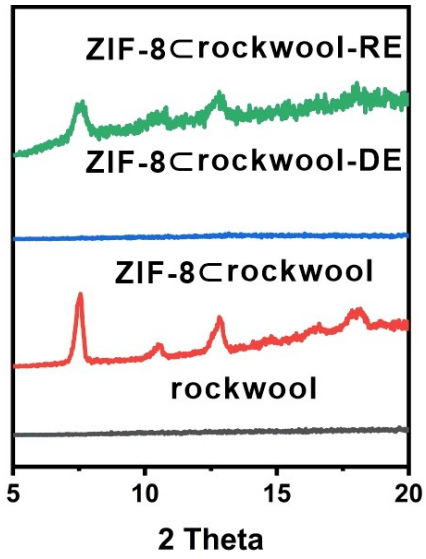

Fig. S5 PXRD patterns of ZIF-8⊂rockwool before degradation, after degradation, and after reconstruction.

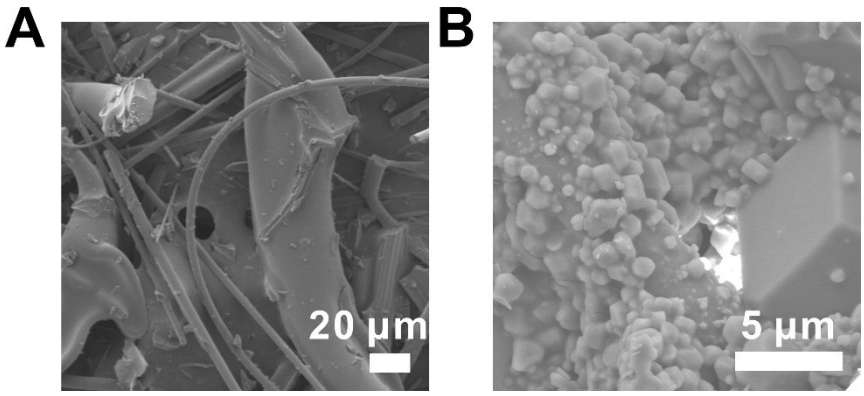

Fig. S6 SEM images of (A) ZIF-8⊂rockwool-DE and (B) ZIF-8⊂rockwool-RE

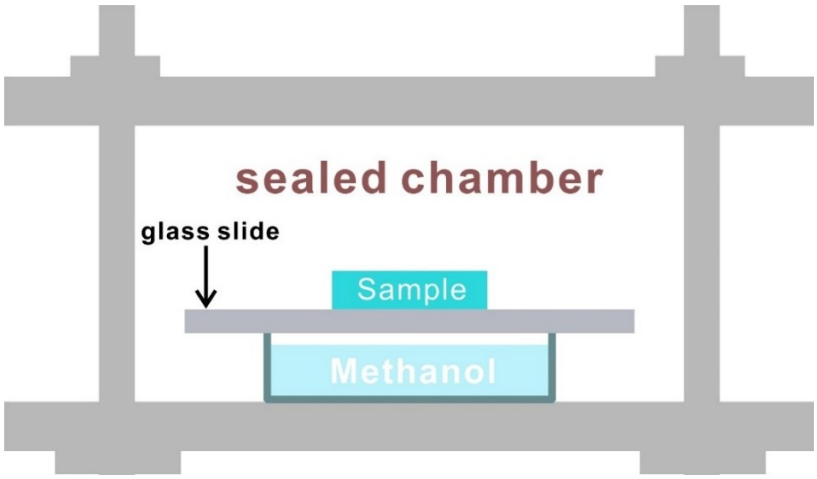

Fig. S7 Homemade stainless-steel sealed chamber for in situ vapor-assisted reconstruction.

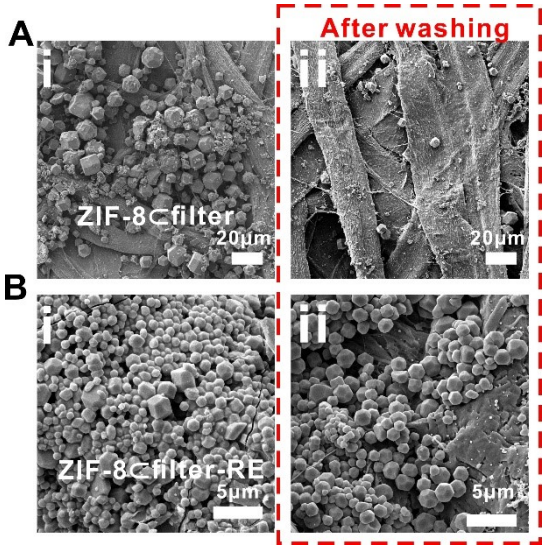

Fig. S8 SEM images of (A) ZIF-8⊂filter and after washing (20 mins), (B) ZIF-8⊂filter-RE and after washing (20 mins).

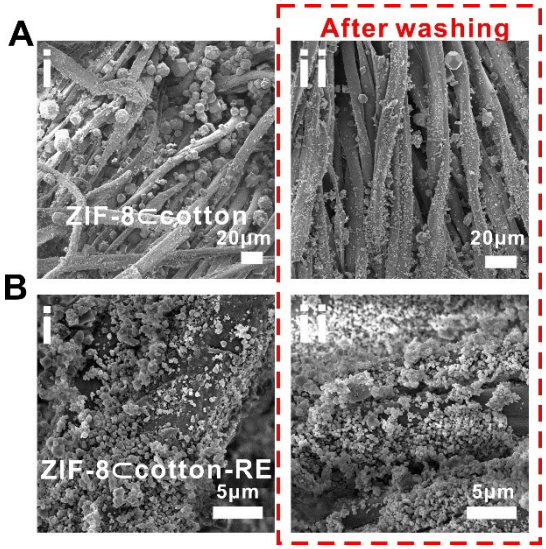

Fig. S9 SEM images of (A) ZIF-8⊂cotton and after washing (20 mins), (B) ZIF-8⊂cotton-RE and after washing (20 mins).

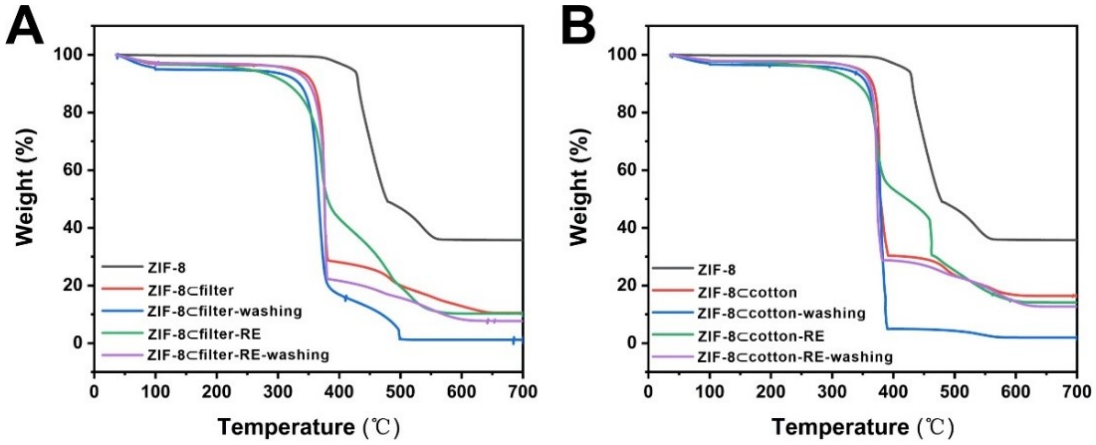

Fig. S10 TGA images of (A) ZIF-8, ZIF-8⊂filter, ZIF-8⊂filter-washing, ZIF-8⊂filter-RE and ZIF-8⊂filter-RE-washing, (B) ZIF-8, ZIF-8⊂cotton, ZIF-8⊂cotton -washing, ZIF-8⊂cotton-RE and ZIF-8⊂cotton-RE-washing.