Facile Synthesis of MOF-5 Confined in SBA-15 Hybrid Material for Enhanced Hydrostability

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Supporting Information

Content

Section 1 Detailed synthesis of MOF hybrid materials

Chemicals for synthesis: 1, 4-Benzenedicarboxylic acid (1,4-BDC), *N*, *N*-Diethylformamide (DEF), $Zn(NO_3)_2 \cdot 6H_2O$, amphiphilic triblock copolymer, poly(ethylene glycol)-*block*-poly(propylene glycol)- *block*-poly(ethylene glycol) with average molecular weight 5800 (P123), and tetraethyl orthosilicate (TEOS). All starting materials were received from commercial sources and used without further purification.

Synthesis of MOF-5: The synthesis of MOF-5 was based on the procedure reported by Yaghi and co-workers.[1] Typically, 360 mg (1.21 mmol) of $Zn(NO_3)_2 \cdot 6H_2O$ and 66 mg (0.40mmol) of 1,4-BDC were dissolved in 10 mL DEF solvent in a 20-mL scintillation vial. The mixture was then kept at 100 ^oC for 12 hours for the formation of MOF-5 crystals. Upon cooling, the crystals were obtained by filtration and washed several times with *N*, *N*-Diethylformamide (DEF). To activate the MOF-5 pores, the MOF-5 crystals were washed three times with chloroform and then soaked in chloroform for additional 24 hours. Before the surface area measurement and gas/vapor adsorption study, the MOF-5 crystals were degassed under 105 ^oC under ultra-high vacuum (< 6 μmHg) for at least 24 hours.

Synthesis of SBA-15 materials: The preparation of SBA-15 porous material followed previous report.[2] Briefly, 3.2 g of P123 polymer was dispersed in to a mixture solution of water (24g) and 2M HCl (96g). 6.8 g of TEOS was then added into the resulting solution under stirring. The TEOS solution was stirred at 40 ^oC for 24 hours and then underwent gelation at 100 ^oC for 48 hours. After the gelation, the white solid powder was obtained by filtration, washed by DI water and dried at 80 ^oC for overnight. The as-synthesized SBA-15 material was then calcinated in air at 550 ^oC for 12 hours to remove the P123 polymer.

Section 2 Powder XRD patterns of MOF-5 and SBA-15

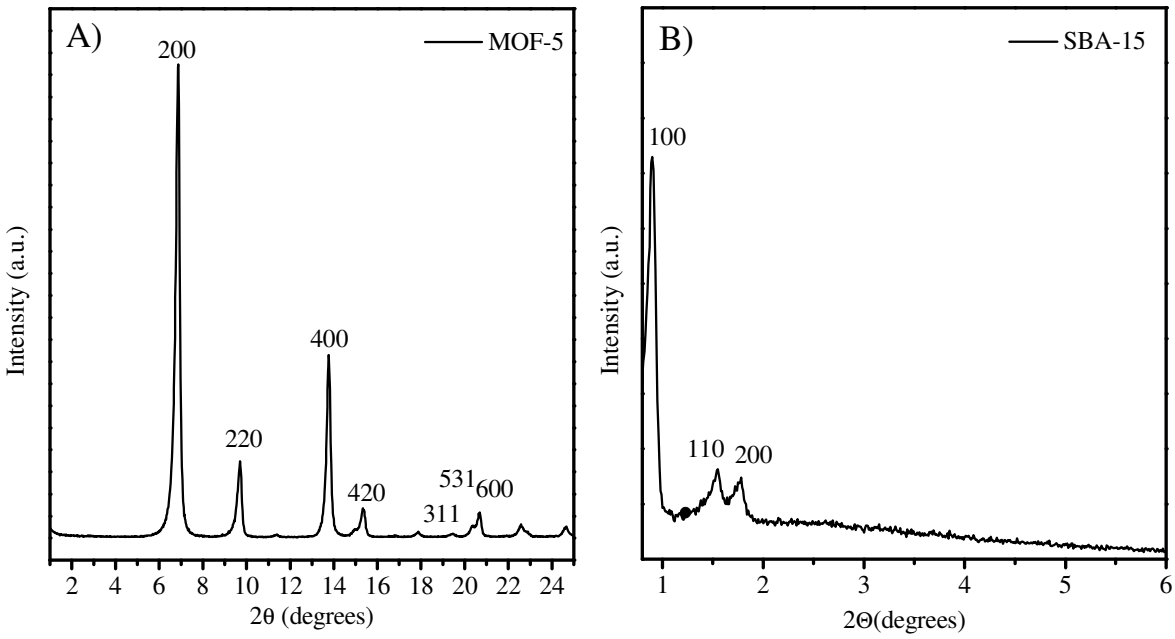

Figure S1. Powder-XRD patterns of (A) activated MOF-5 and (B) SBA-15.

Section 3 TGA measurements of MOF-5, SBA-15, and MOF-5-SBA-15

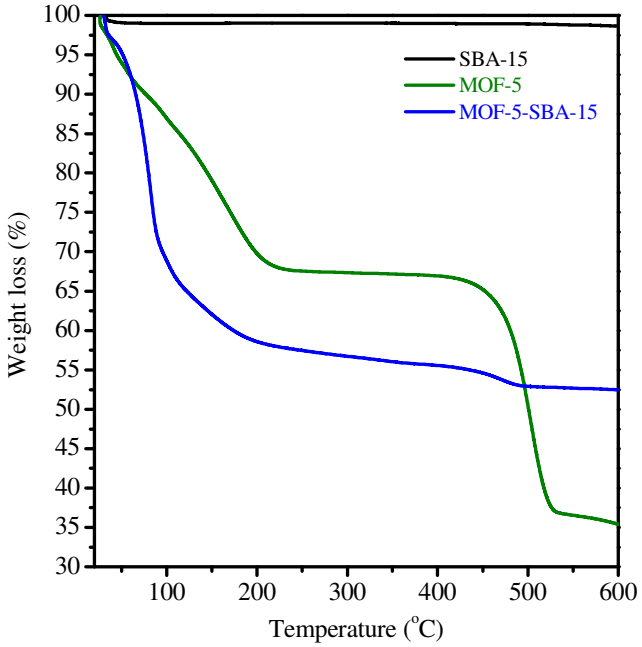

Figure S2. TGA patterns of SBA-15(black), as-synthesized MOF-5(green), and MOF-5-SBA-15(blue).

Section 4 FTIR spectra of MOF-5-SBA-15, SBA-15, and MOF-5

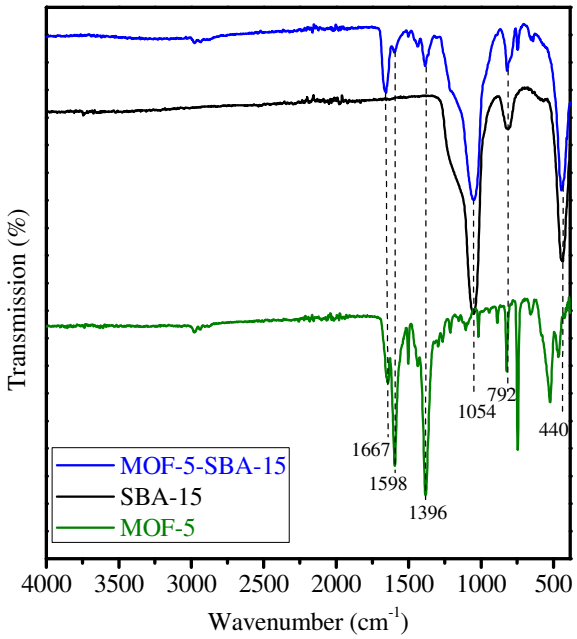

Figure S3. FTIR patterns of MOF-5-SBA-15(blue), SBA-15(black), and MOF-5(green) materials.

Section 5 Estimation of MOF-5 loading in MOF-5-SBA-15 hybrid materials based on TGA measurements

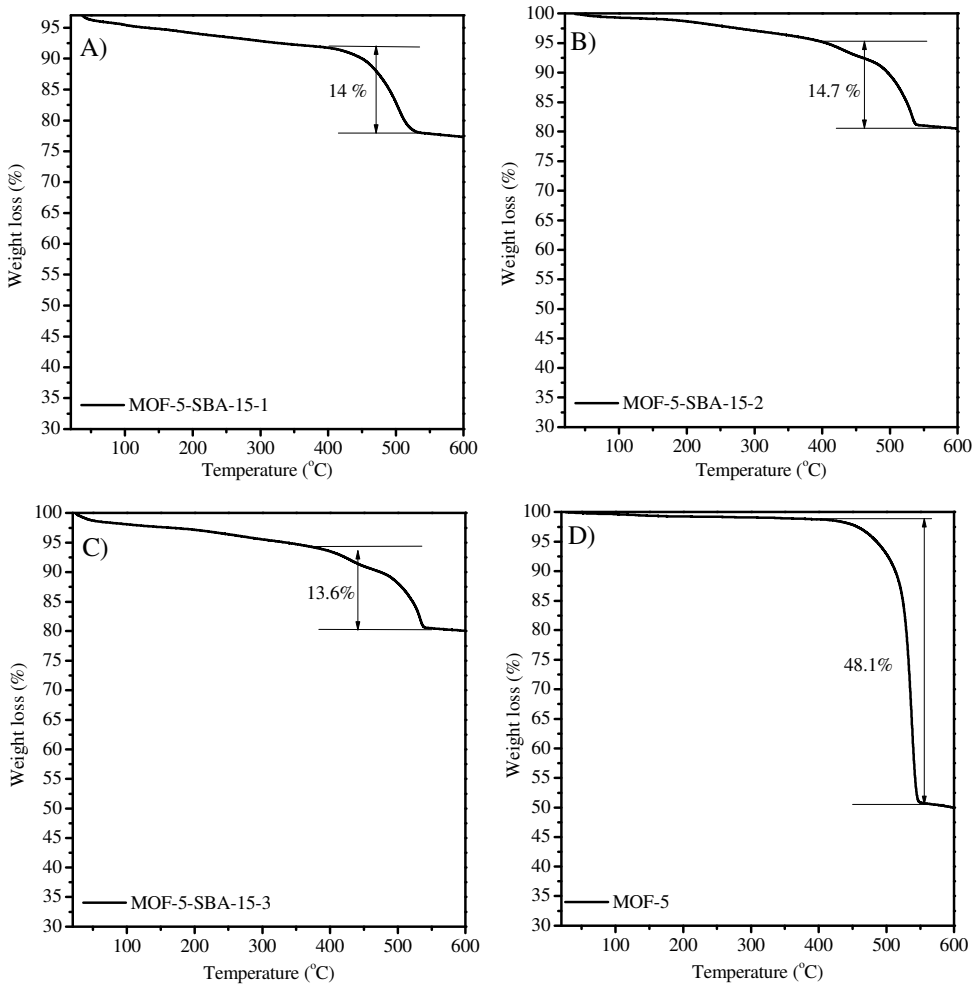

Figure S4. TGA measurements of dried MOF-5-SBA-15 samples in three independent trials. The loading amount of MOF-5 composition in the MOF-5-SBA-15 hybrid was estimated based on the ratio of the weight loss of dried MOF-5-SBA-15 to the weight loss of the dried MOF-5 at ~450 ^{o}C, since SBA-15 template itself shows no weight loss at this temperature range (see Figure S2). The weight loss of dried MOF-5 is 48.1 % at ~ 450 ^{o}C; thus, the loading of MOF-5 in dried MOF-5-SBA-15 from three different batches was estimated to be 29.1 wt% (14%/48.1%), 30.5 wt% (14.7%/48.1%), and 28.3 wt% (13.6%/48.1%), respectively, i.e., the average loading of MOF-5 is 29.3±1.1 wt%.

(1) Li, H.; Eddaoudi, M.; O'Keeffe, M.; Yaghi, O. M. *Nature* **1999**, *402*, 276.
(2) Murali, A.; Chang, Z.; Ranjit, K. T.; Krishna, R. M.; Kurshev, V.; Kevan, L. *The Journal of Physical Chemistry B* **2002**, *106*, 6913.