*Electronic Supplementary Informations (ESI):*

# Facile synthesis of covalent organic frameworks COF-1 and COF-5 by sonochemical method

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## Experiemntal details

### *1. Synthesis of COF-1*

COF-1 was prepared using 250 mg of 1,4-benzene diboronic acid (BDBA, Aldrich) in 40 mL of a 1:1 *v:v* solution mixture of mesitylene (Aldrich) and 1,4-dioxane (Aldrich). The substrate mixture was introduced to a custom-made horn type Pyrex reactor, which was then fitted to a sonicator unit (VCX500, SONICS, USA) with an adjustable power output (maximum 500 W at 20 kHz). The reaction mixture was subjected to an ultrasonic treatment for 1 h. A white solid at bottom of the tube was isolated by centrifugation, washed with acetone (2×50 mL×10) and dried overnight at 353 K.

### *2. Synthesis of COF-5*

As a standard synthesis, COF-5 was prepared using 177 mg of BDBA and 242 mg of 2,3,6,7,10,11-hexahydroxytriphenylene (HHTP, TCI) in 40 mL of a 1:1 *v:v* solution mixture of mesitylene and 1,4-dioxane. The synthesis mixture was subjected to an ultrasonic treatment for 0.5 to 2 h at various power levels (40, 50, and 100%). A gray-purple solid powder at the bottom of the reactor was isolated by centrifugation, washed with acetone (2×50 mL×10) and dried at 353 K overnight.

### *3. COF-5 film on an alumina disc*

A COF-5 film was prepared using the same synthesis procedure for COF-5 powder but in the presence of an alumina disc positioned at the bottom of a horn type Pyrex reactor. The reaction mixture was subjected to an ultrasonic treatment for 1 h at a 50% power level. After sonication, the COF-5-coated alumina disc was placed in acetone (500 mL) for 2 days, and dried at 353 K for 12 h.

### *4. Characterization*

XRD patterns of the prepared samples were obtained on a Rigaku diffractometer (D/MAX 2200V/PC) using CuKα (λ=1.54 Å) radiation. $N_2$ adsorption-desorption isotherms were measured on a BELsorp-mini (BEL, Japan) at 77 K. Prior to the adsorption measurments, the prepared COF samples were activated at 423 K for 12 h under vacuum ($3\times10^{-3}$ torr). The specific surface areas of the samples were calculated by the BET method ($P/P_0$ = 0.05-0.1). The pore size distributions of the prepard COF samples were measured on a ASAP-2020 (Micromeritics, USA) at 77 K, and they were calculated by the HK method (COF-1) using Ar gas, and the DFT method (COF-5) using $N_2$ gas. The morphologies of the synthesized COF samples were examined by scanning electron microscopy (SEM) using a Hitachi S-4300 electron microscope. The thermal stability of the samples was tested using a thermogravimetric analyzer (TGA, SCINCO thermal gravimeter S-1000): 10 mg of the sample was heated at 10 K $min^{-1}$ to 1123 K under an $N_2$ flow (30 mL $min^{-1}$).

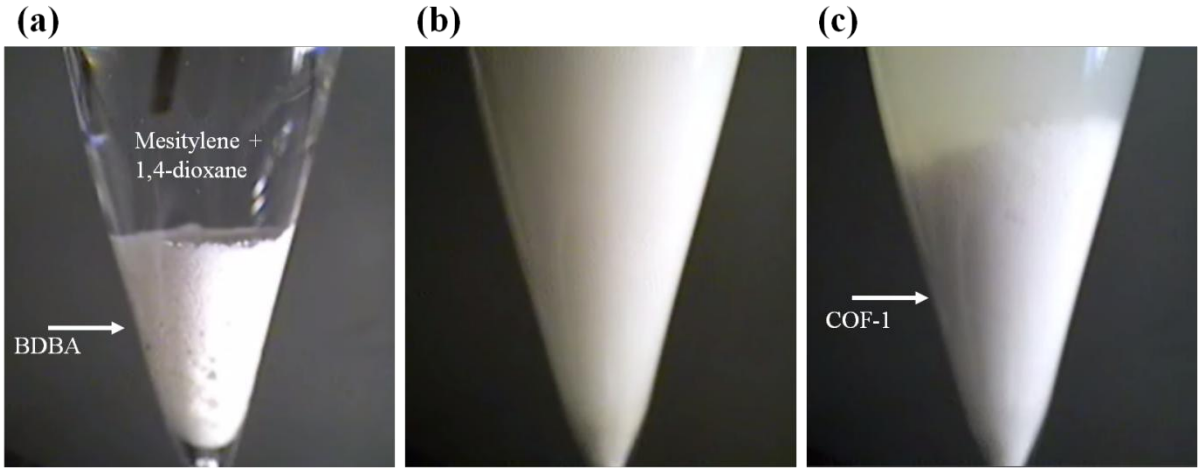


**Fig. S1.** Video Capture images of COF-1 sonochemical synthesis; (a) setup, (b) applying sonication, and (c) after the reaction.

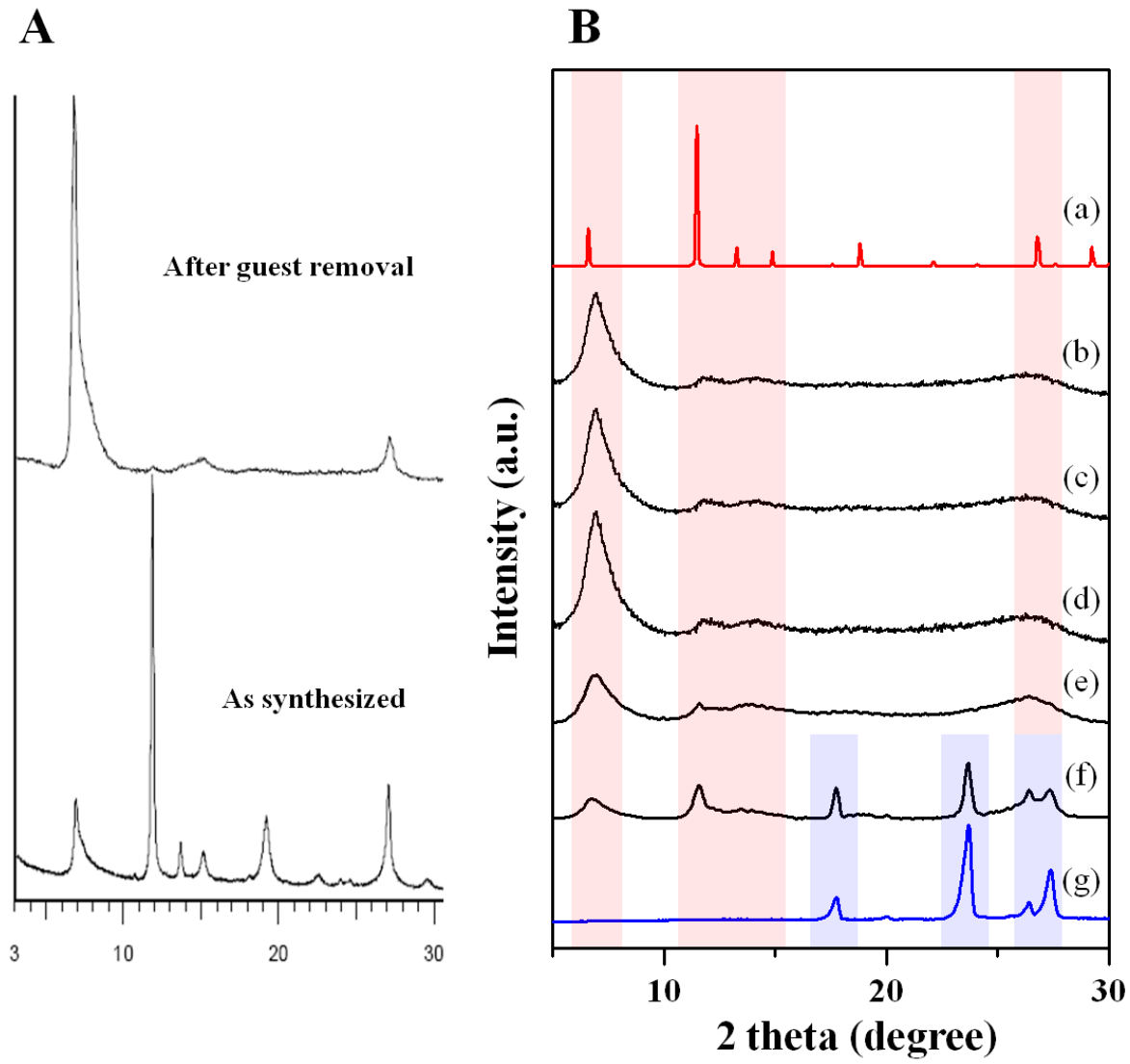


**Fig. S2.** XRD patterns; (A) experimental data of COF-1 XRD patterns before and after activation in the literature,[§] and (B) COF-1 simulated (a), COF-1 samples in Table 1 of (b) entry 5, (c) entry 4, (d) entry 3, (e) entry 2, and (f) entry 1, and (g) BDBA.

[§] XRD patterns of COF-1 after the evacuation of guest molecules and gas adsorption measurements (top) and as-synthesized (bottom), illustrating the shifting layers upon guest removal. The principal [100] and [002] diffraction peaks of COF-1 were retained: A. P. Côté, A. I. Benin, N. W. Ockwig, M. O'Keeffe, A. J. Matzger, O. M. Yaghi, *Science*, 2005, **310**, 1166; B. Lukose, Al. Kuc, T. Heine, *Chem. Eur. J.*, 2011, **17**, 2388.

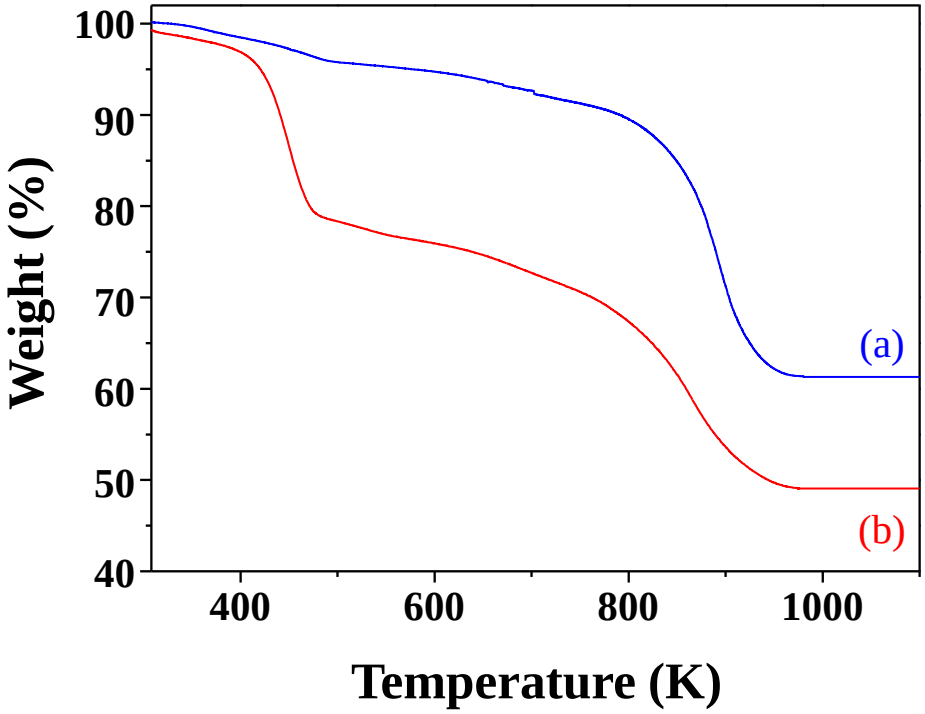


**Fig. S3.** Thermogravimetric analysis of the COF-1 sample: (a) washed with acetone (2×50 mL×10) and dried at 353 K overnight and (b) as-synthesized (acetone washed once and dried).

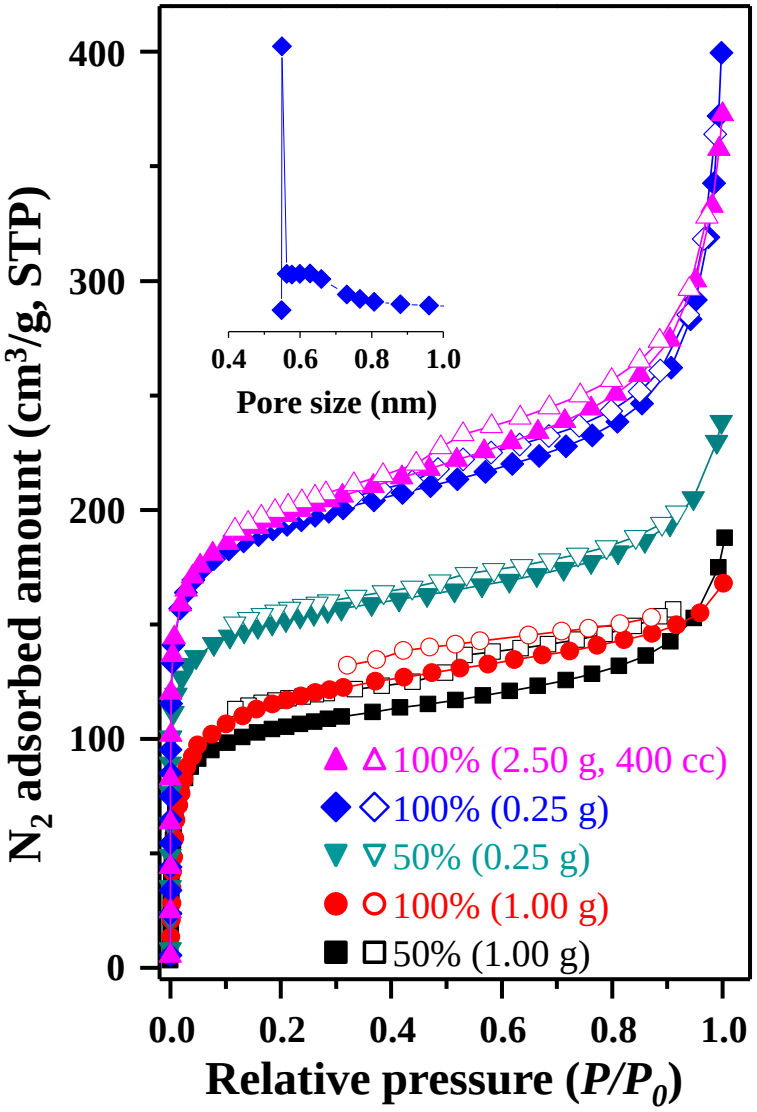


**Fig. S4.** $N_2$ adsorption-desorption isotherms of the COF-1 samples (inset is the pore size distribution of COF-1 sample of entry 4 in Table 1): ■□ (entry 1 in Table 1); ●○ (entry 2); ▼▽ (entry 3); ◆◇ (entry 4); and ▲△ (entry 5).

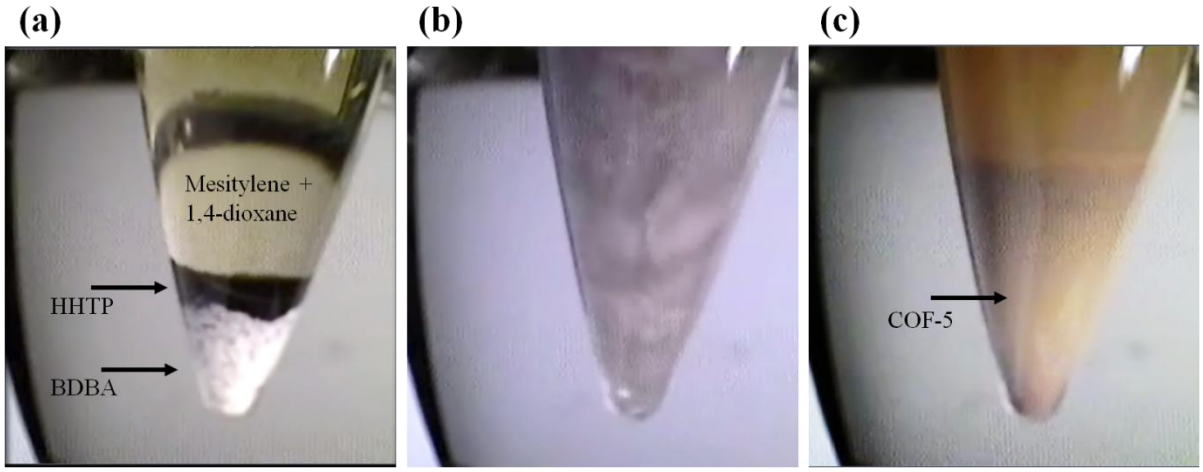


**Fig. S5.** Video Capture images of COF-5 sonochemical synthesis; (a) setup, (b) applying sonication, and (c) after reaction (top portion in (a) is the reflected image of HHTP).

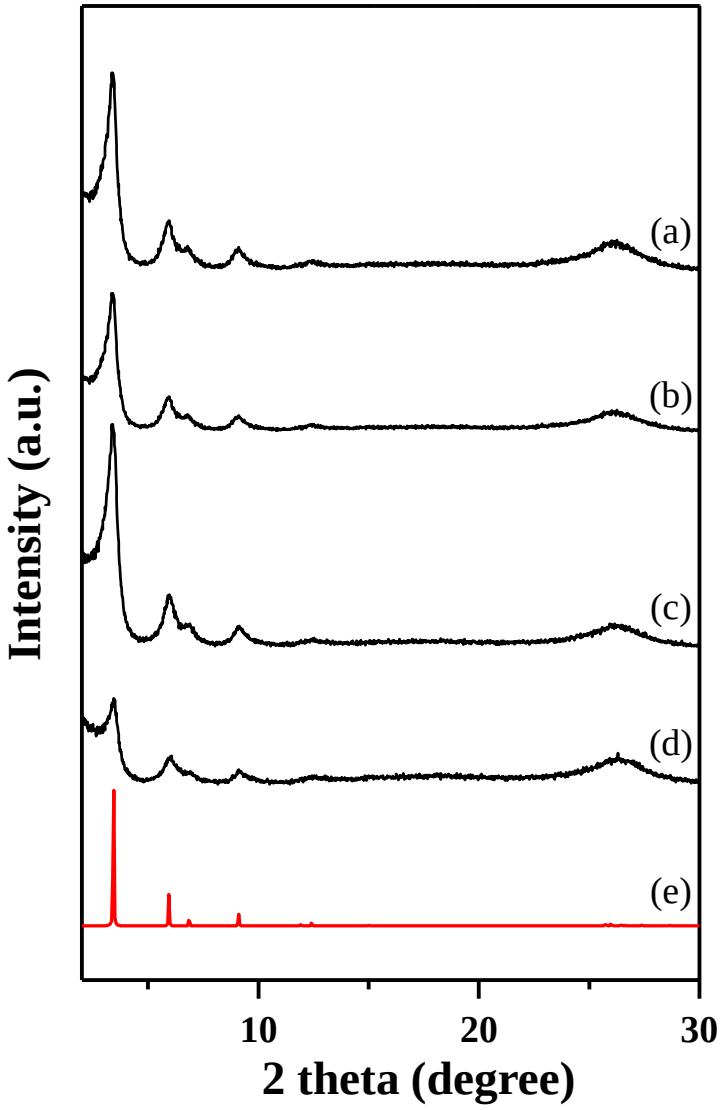


**Fig. S6.** XRD patterns after guest removal; (a) entry 14 in Table 1, (b) entry 12, (c) entry 8, and (d) entry 6 of COF-5 samples, and (e) simulated COF-5.

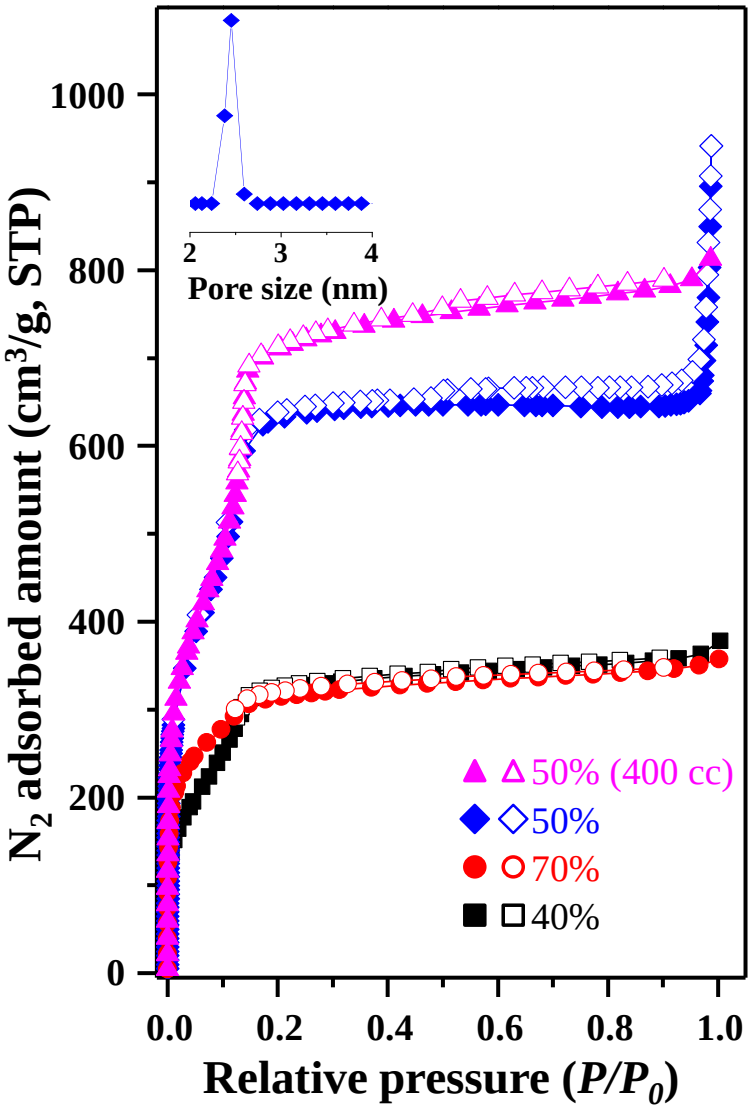


**Fig. S7.** $N_2$ adsorption-desorption isotherms of the COF-5 samples (inset is the pore size distribution of COF-5 sample of entry 8 in Table 1): ■□ (entry 6 in Table 1); ◆◇ (entry 8); ●○ (entry 12); ▲△ (entry 14).

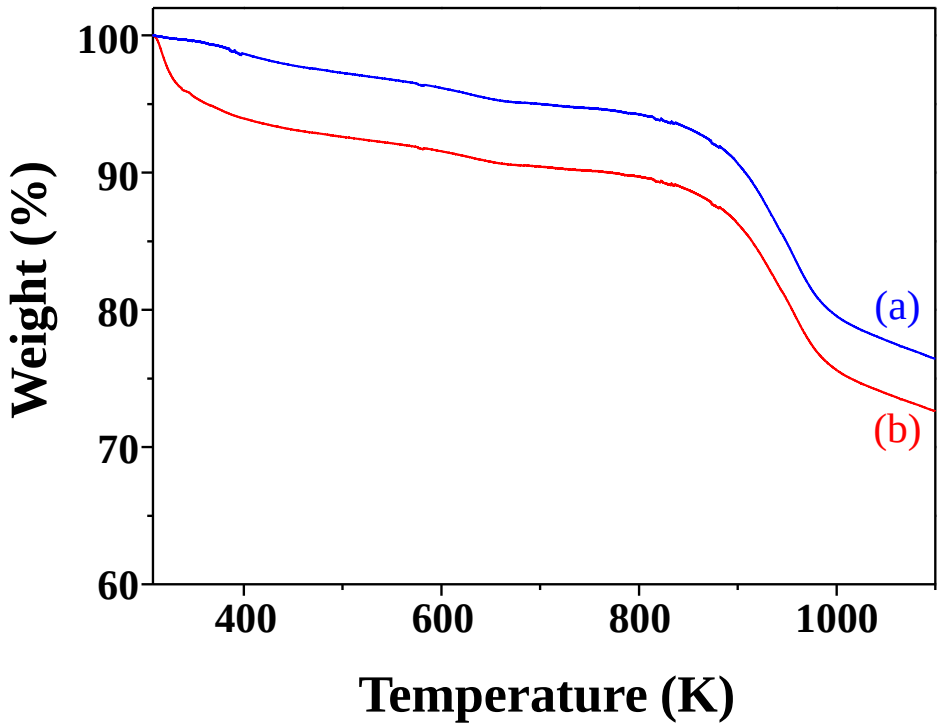


**Fig. S8.** Thermogravimetric analysis of the COF-5 sample: (a) washed with acetone (2×50 mL×10) and dried at 353 K overnight and (b) as-synthesized (acetone washed once and dried).

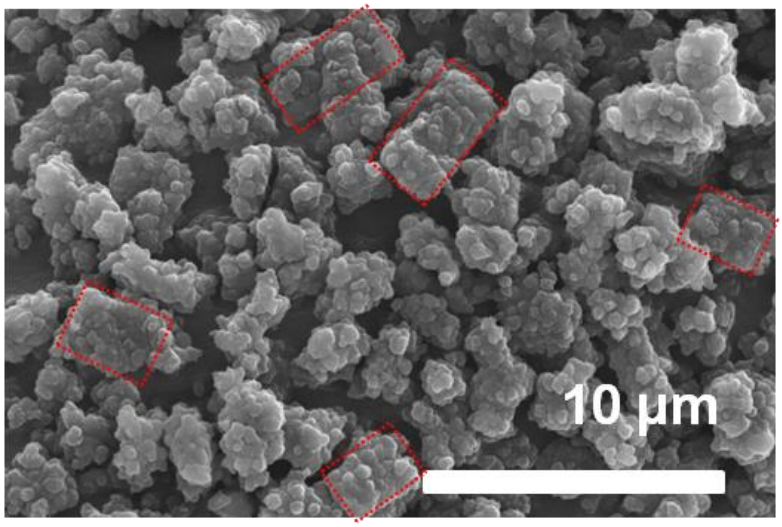


**Fig. S9.** SEM image of the COF-5 samples (entry 10 in Table 1, red rectangle: BDBA morphology).

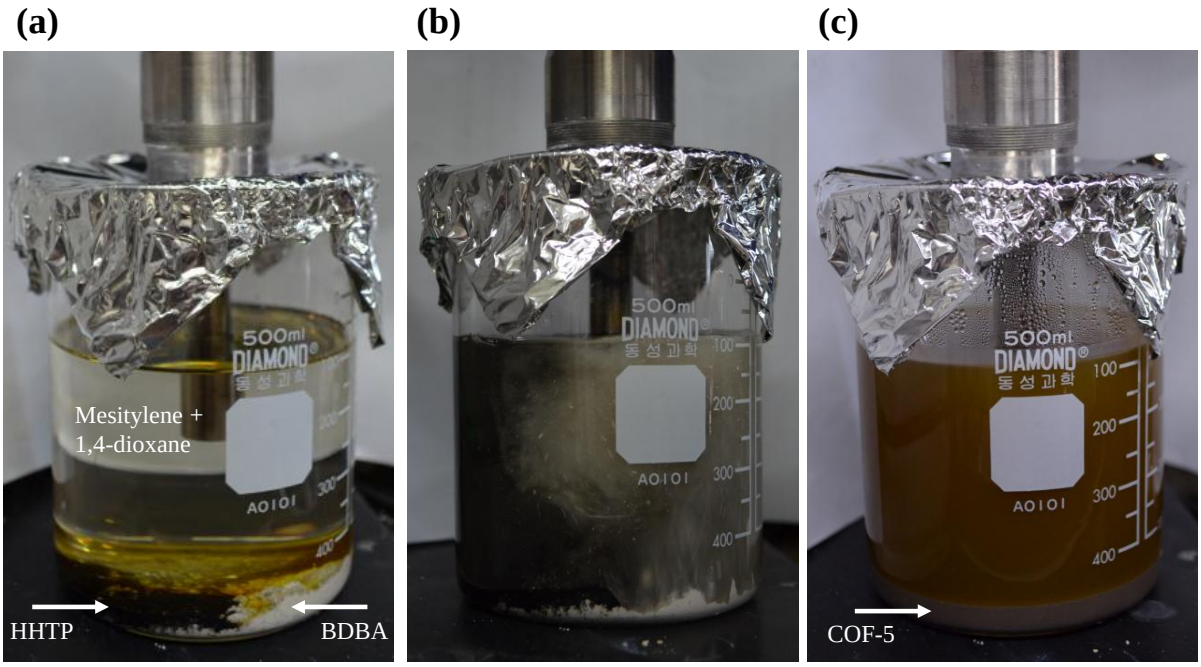


**Fig. S10.** COF-5 sonochemical synthesis procedures in a 0.5 L-batch (entry 14 in Table 1); (a) setup, (b) applying sonication, and (c) after reaction.