Part S1. Chemicals and Reagents

Copper (II) acetate monohydrate, purchased from Aladdin. 1,3,5-benzenetricarboxylic acid (H₃BTC, 95wt%), and 1,4-benzendicarboxylic acid was purchased from Sigma-Aldrich (China). The deionized water used in our experiments was obtained from the Milli-Q System. Other analytical grade solvents including DMF, Ethanol, Methanol were supplied by Beijing Chemical Reagent Company (China). All the chemicals were used without further purification.

Part S2. General Characterization

Scanning electron microscopy (SEM) measurement was performed on a Hitachi SU8200 scanning electron microscope at 6.0 kV. Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) imaging and High angle annular dark field scanning transmission electron microscopy (HAADF-STEM) imaging was carried out using Tecnai G2 F20 S-TWIN at 200 kV. Powder X-ray diffraction (XRD) patterns were recorded on D/MAX-TTRIII (CBO) with Cu Ka radiation ($\lambda = 1.542$ Å) operating at 40 kV. X-ray photoelectron spectroscopy (XPS) spectra were performed by an ESCALAB 20 Xi XPS system, where the analysis chamber was 1.5 × 10 –9 mbar and the size of X-ray spot was 500 µm. The chemical structure of different MOFs was investigated by FT-IR spectroscopy (Nicolet Nexus 670, USA).

General synthesis method

Metal acetate (0.5 mmol) were dissolved in distilled water and filters through (0.22 μ m filter membrane). To this filtrate, the organic linker 1 mmol of (BDC or BTC dissolved in DMF) was added via a controlled dissolution by rapid solvent mixing method to get instant MOFs as shown in the Fig 1. Then products were centrifuged and washed several times with DMF and MeOH, dried at room temperature.



Fig S1 Minute-MOF Concept and Realization

Gram scale synthesis method

Cu-BDC MOF

Cu acetate 1g dissolved in 30ml of distilled water and filters through (0.22 μ m filter membrane). To this filtrate, the organic linker 1.6g of (1,4-benzendicarboxylic acid) dissolved in 10 ml of DMF was added via a controlled dissolution mixing method to get instant MOFs as shown in the Fig 1. Then products were centrifuged and washed several times with DMF and MeOH, dried at room temperature. Yield: 2.07g

Cu-BTC MOF

Cu acetate 1g dissolved in 30ml of distilled water and filters through (0.22 µm filter membrane). To this filtrate, the organic linker 2.1g of (1,4-benzendicarboxylic acid) dissolved in 10 ml of DMF was added via a controlled dissolution mixing method to get instant MOFs as shown in the Fig 1. Then products were centrifuged and washed several times with DMF and MeOH, dried at room temperature. Yield: 2.3g



Fig. S2 Gram scale synthesis (a) Cu-BDC (b) Cu-BTC.