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Design and synthesis of novel mesostructured metal-organic frameworks templated by cationic surfactants via cooperative self-organization

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Supporting Information

Experiment Section

Materials

$Cu(NO_3)_2 \cdot 3H_2O$ was purchased from Sinopharm Chemical Reagent Co.,Ltd. 5-Hydroxyisophthalic acid was purchased from Alfa Aesar. Methanol was purchased from Tiantai Fine Chemical Co.,Ltd. Of Tianjin. Nitric acid was purchased from Xilong Chemical Co.,Ltd. Supramolecular templating agents were all purchased from HuiShi biochemical reagent Co.,Ltd of Shanghai. All materials were used without further purification.

Measurements

Powder X-ray diffraction (XRD) data were collected on a Rigaku 2550 diffractometer with Cu Kα radiation (λ = 1.5418 Å). The step size was 0.02^o and the count time was 2s.

Transmission electron microscope (TEM) was carried out by using a FEI Tecnai G2 F20 s-twin D573 transmission electron microscope operated at 200 kV, respectively. The sample for TEM was mounted on a carbon polymer supported on a copper grid.

The infrared (IR) spectra were recorded within the 400-4000cm^{-1} region on a Nicolet Impact 410 FTIR spectrometer using KBr pellets.

The elemental analyses were performed on a Perkin-Elmer 2400 LSII CHN analyzer.

The thermal gravimetric analyses (TGA) were performed on a TGA Q500 V20.10 Build 36 thermogravimetric analyzer in air atmospheric environment with a heating rate of 10 $^oC \cdot min^{-1}$.

The elemental analyses were performed on a Perkin-Elmer 2400 LSII CHN analyzer.

Electron paramagnetic resonance spectra (EPR) were recorded on a JES-FA200 electronic spin resonance spectrometer with powder samples at 298 K.

UV/vis spectra were recorded using a Shimadzu UV2450 spectrometer. The sample was dispersed in methanol solution and then filtered using a membrane filter with 0.45 μm pore size for Uv/vis measurement.

High-performance liquid chromatograms(HPLC) were performed using the

UltiMate^{TM} 3000 system, including autosampler ASI-100, degasser DG-1210, pump P680 and Detector VWD-3400. The column was Acclaim TM 120, C18(4.6× 100mm), 5μm. The mobile phase was the mixed solution of methanol and water with the volume ratio of 9:1 and the flow-rate was 0.8ml/min. The sample was injected with 15μl every time detected by UV absorbance at 312 nm.

Sample preparation for HPLC

Methanol and concentrated HNO_3 at 100:3.5 (volume ratio) were mixed to prepare the standard solution. Then, 0.01g of Cu-(5-OH-BDC)-C_{16} was added into 70ml of standard solution to decompose. After 20 minutes slowly stirring, 20ml of the disassembled solution were filtered using a membrane filter with 0.22 μm pore size for HPLC measurement.

Experimental results Figures

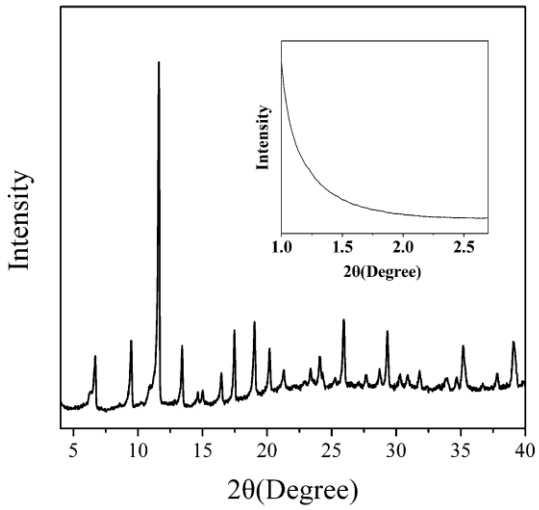

Figure S1 X-ray diffraction (XRD) patterns of Cu-BTC-C_{16}

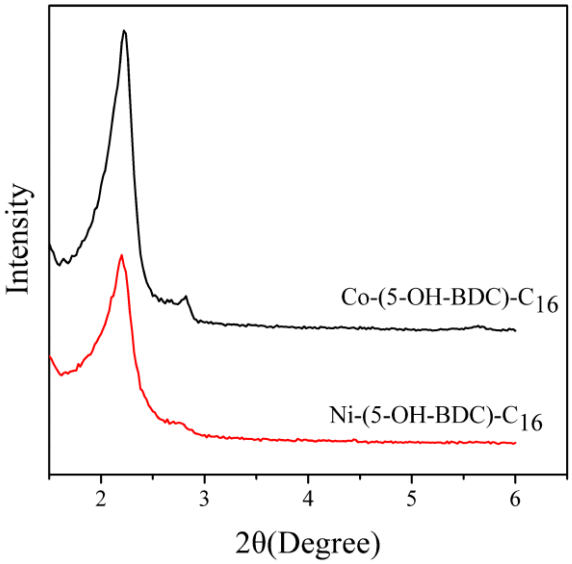

Figure S2 X-ray diffraction (XRD) patterns of other metal ions Co-(5-OH-BDC)-C_{16}, Ni-(5-OH-BDC)-C_{16}

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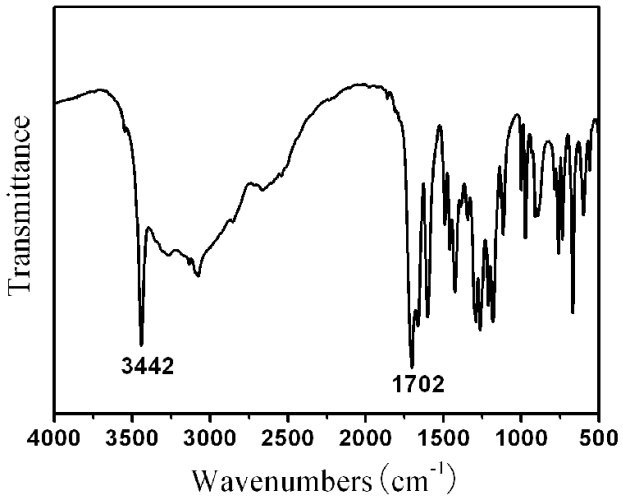
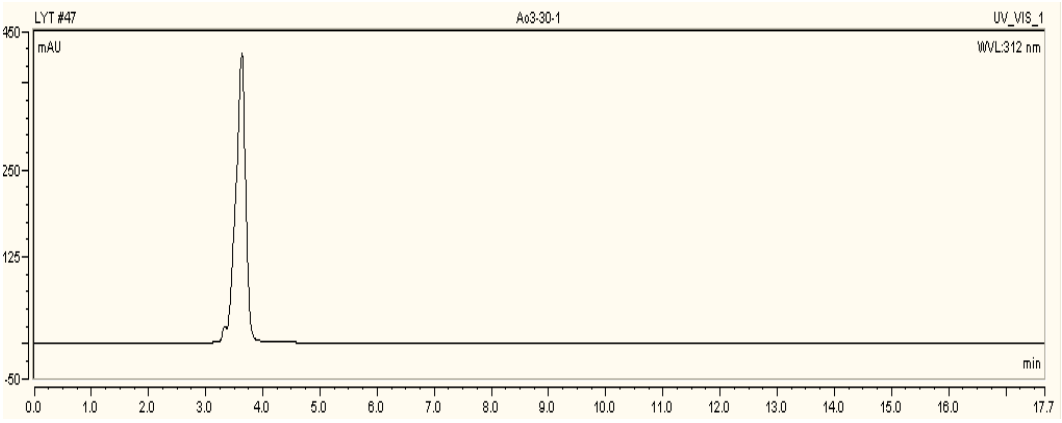
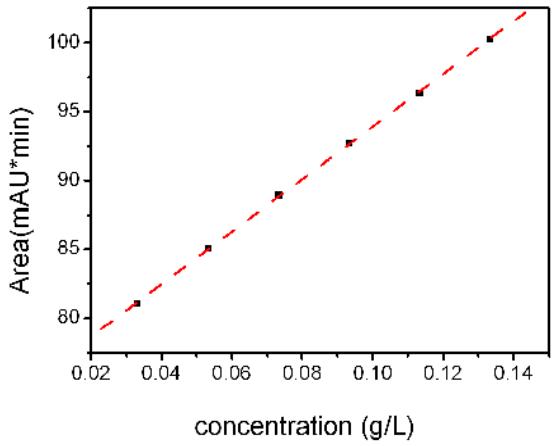


Figure S3 IR spectra of 5-OH-BDC



a) HPLC of 5-OH-BDC in decomposed solution of Cu-(5-OH-BDC)-C_{16}



b) the standard concentration curve of 5-OH-BDC

Figure S4 a) The integral area value of 5-OH-BDC in decomposed solution of Cu-(5-OH-BDC)-C_{16} is 84.13 mAU*min. b) The correlation co-efficient of the standard curve could reaches 0.999 when the concentration of 5-OH-BDC is from 0.02 g/L to 0.14 g/L in the standard solution.

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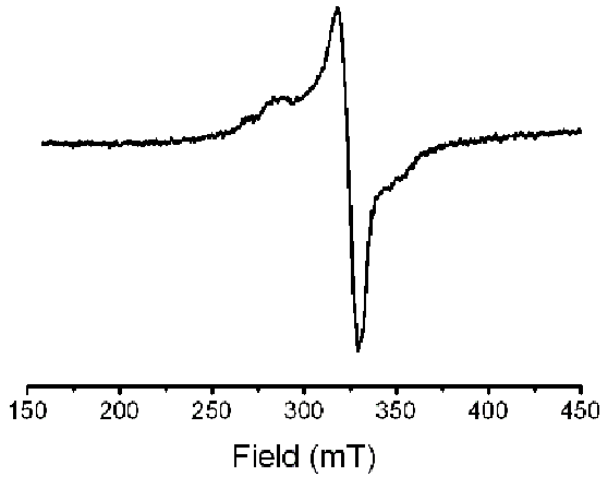

Figure S5 The EPR spectrum of Cu-(5-OH-BDC)-C_{16}

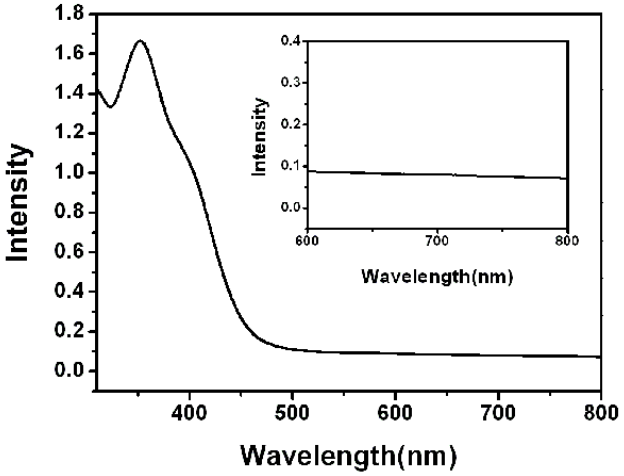

Figure S6 UV Vis spectrum of the solution of dissolved Cu-(5-OH-BDC)-C_{16} in methanol.

Because the dicopper paddlewheel cluster is very stable in methanol solution, even if the sample decomposes into small clusters and fragments in methanol, the dicopper paddlewheel cluster (if any) would still be detectable. The fact of no absorption band at 700nm proves the absence of dicopper paddlewheel cluster in our product Cu-(5-OH-BDC)-C_{16}.

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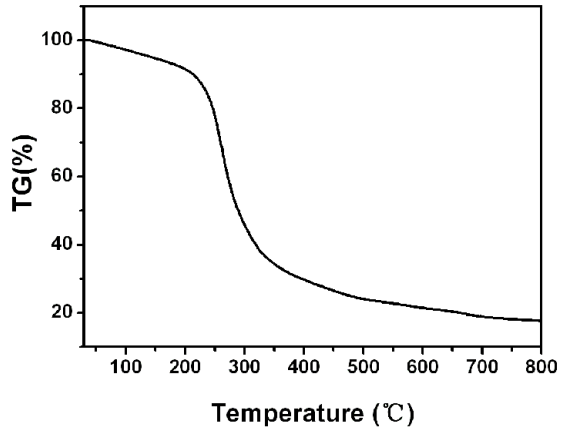

Figure S7 Thermogravimetric analysis of Cu-(5-OH-BDC)-C_{16}.
Weight loss of sample is about 83%.

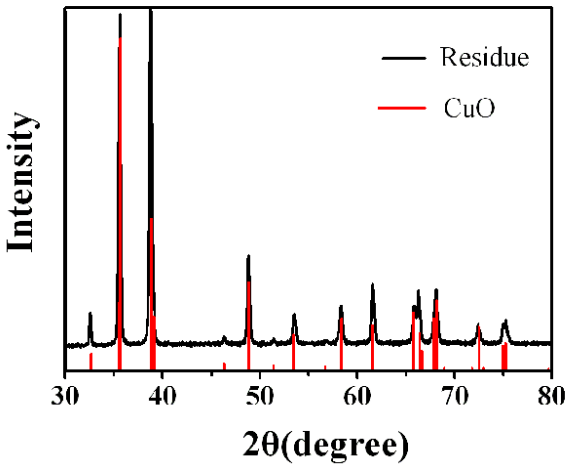

Figure S8 The XRD patterns of residue coinciding with that of CuO