Supplementary Material (ESI) for Chemical Communications

Hydrothermal Synthesis of a Novel Thermally Stable Three-Dimensional Ytterbium-Organic Framework

Supporting Information

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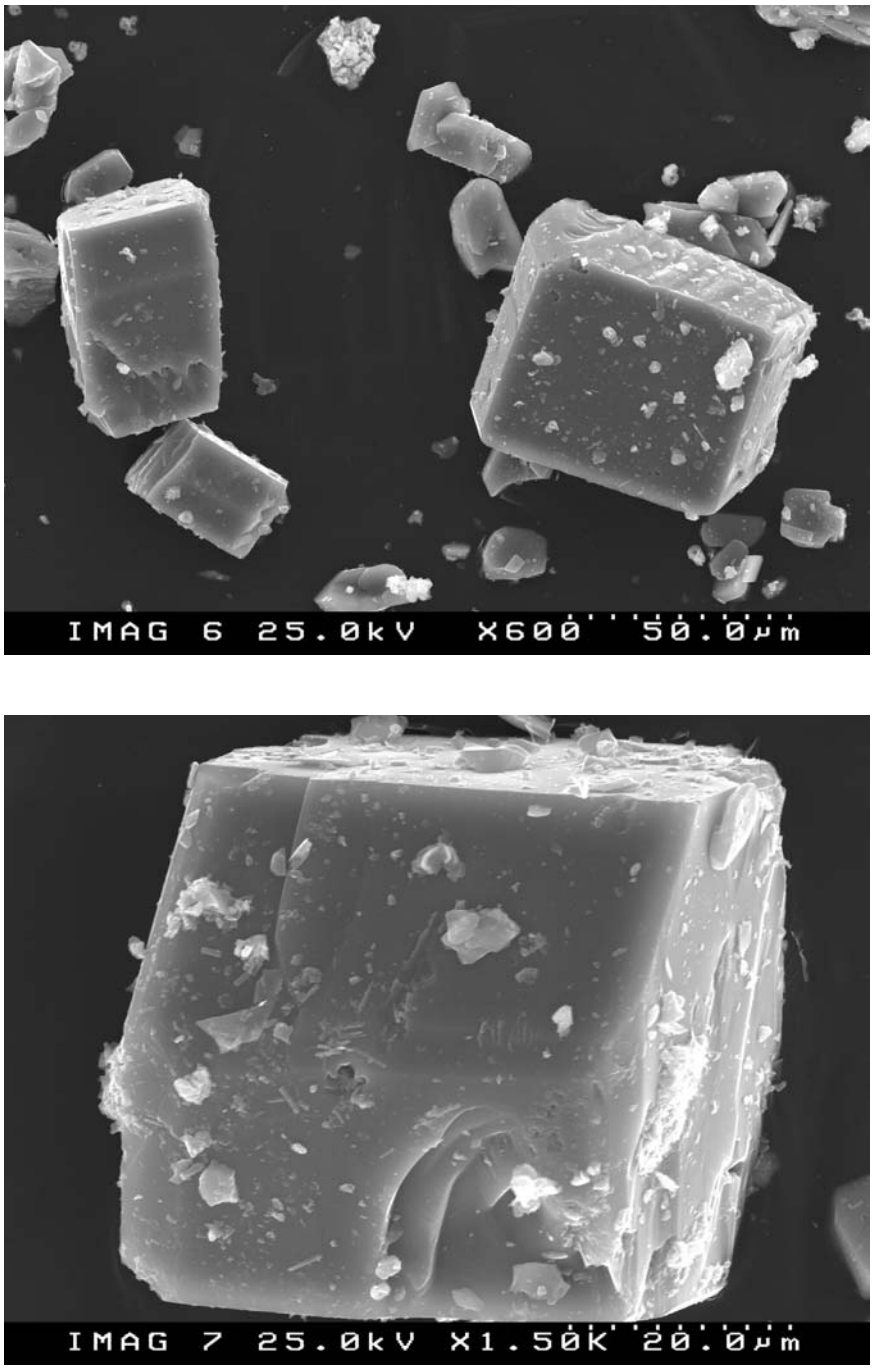

Fig. S1 Scanning Electron Microscopy (SEM) images of CUmof-9 obtained using a FEG-SEM Hitachi S4100 microscope operating at 25 kV. Samples were prepared by depositing CUmof-9 on aluminium foil and carbon coating.

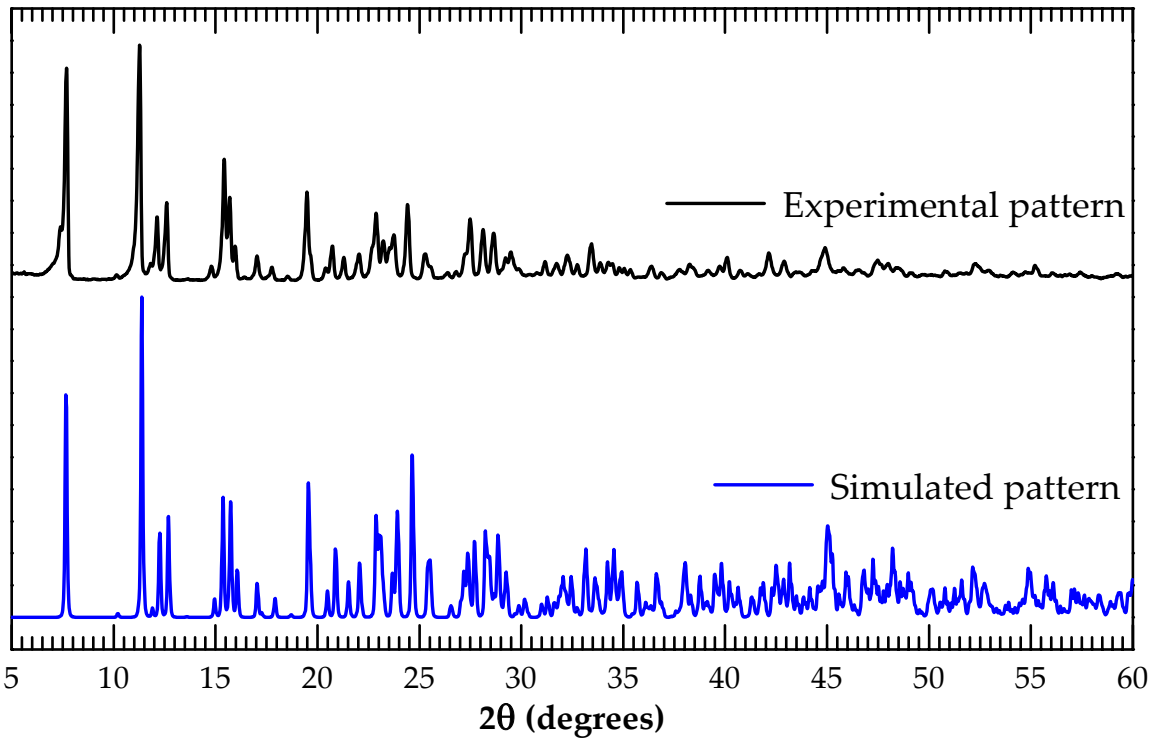

Fig. S2 Comparison of the experimental (top) and simulated (bottom) powder X-ray patterns for CUmof-9. Data were collected at ambient temperature using the step counting method (step 0.5˚, time 460s) on a STOE STADI-P high-resolution transmission diffractometer with Ge(111)-monochromated Cu Kα radiation (λ=1.5406 Å), and a position-sensitive detector covering a 6˚ 2θ angle (40 kV, 40 mA). The simulated powder pattern was based on single-crystal data and calculated using the STOE Win XPOW software package.

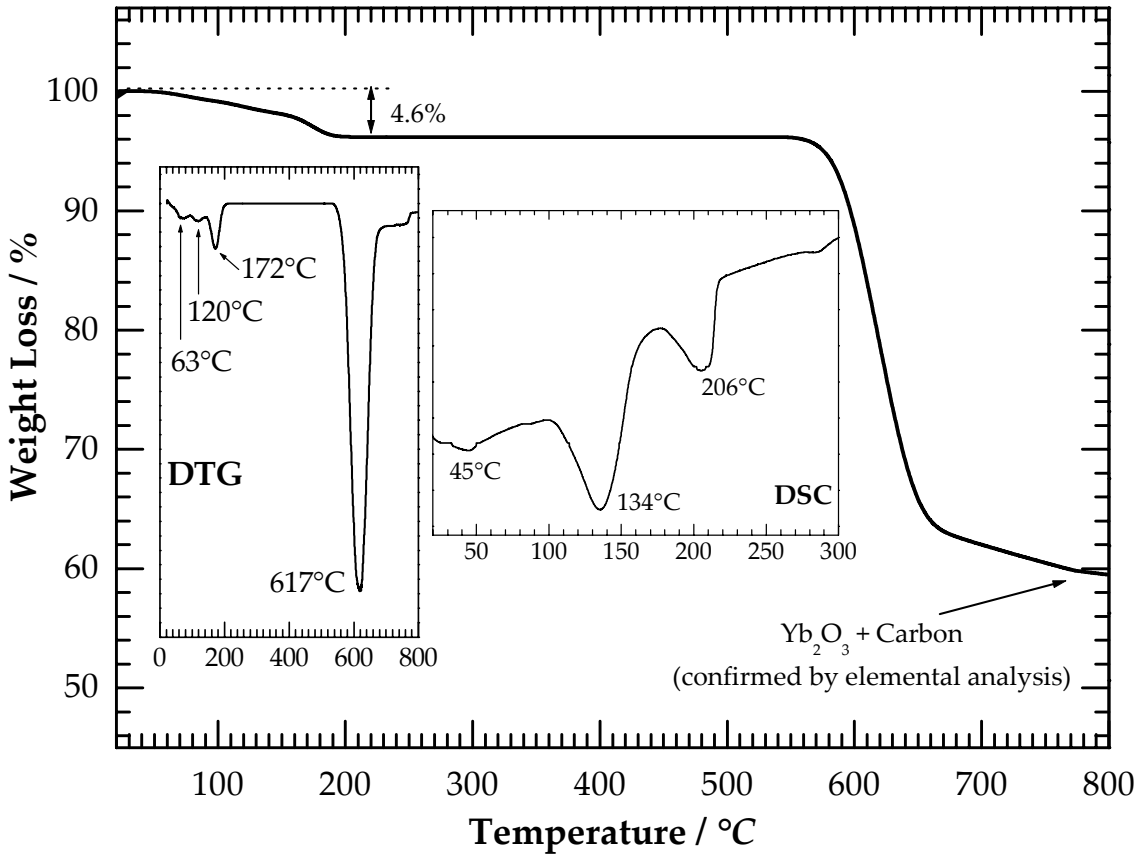

Fig. S3 Thermal analysis of CUmof-9: TG, DTG and DSC curves. The compound loses almost all the crystallisation water below *ca.* 200°C, with the oxidation of the framework starting only above *ca.* 550°C. TGA was carried out using a Shimadzu TGA-50 and DSC on a Shimadzu DSC-50 analysers. Measurements were performed at a heating rate of 5˚C/min under a nitrogen atmosphere with a flow rate of 20 cm^3/min.

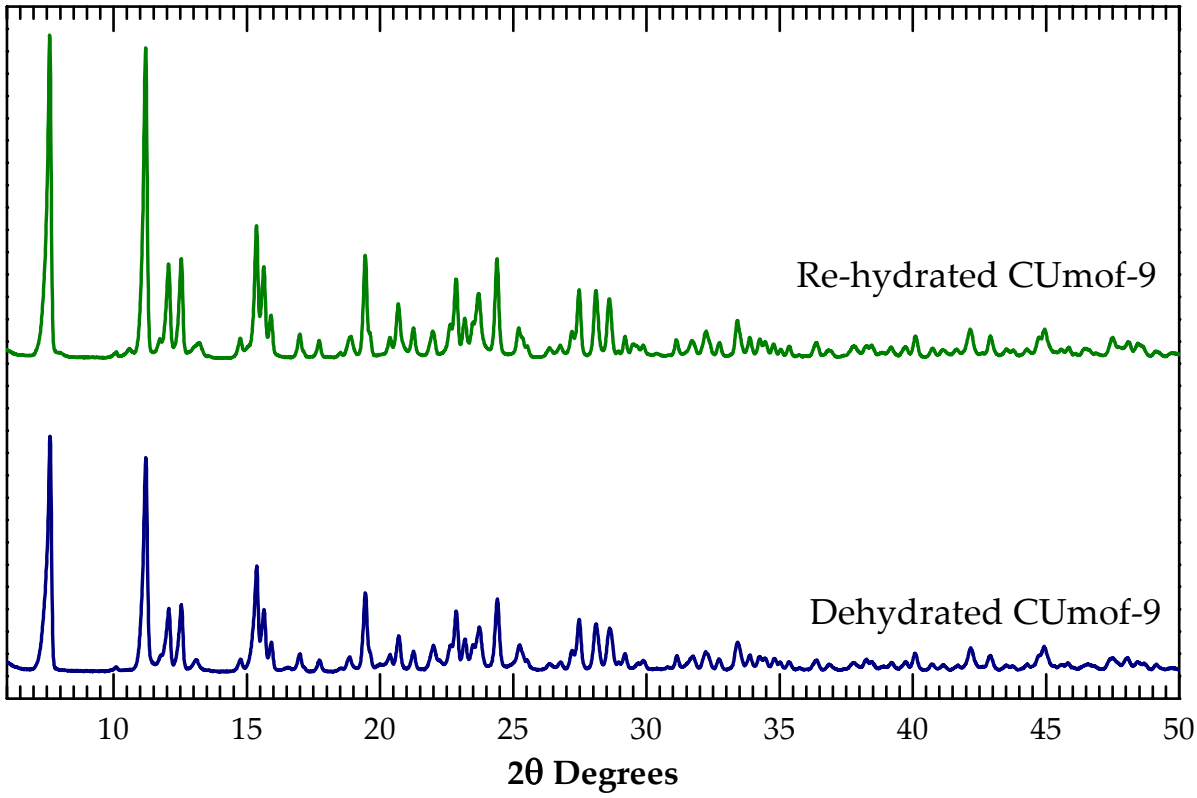

Fig. S4 Comparison of the experimental powder X-ray patterns for dehydrated (bottom) and re-hydrated (top) CUmof-9. Data were collected under the same experimental conditions as those in figure S2. Removal of water from CUmof-9 was achieved by slow heating in an oven to 350°C, after which the system was left undisturbed for 12 hours. Dehydrated CUmof-9 was then quickly transferred into a desiccator. Elemental analysis: C 43.07%, H 1.92%. Calculated for $[Yb_2(NDC)_3]_n$: C 43.70%, H 1.84%.
Absorption of water by the dehydrated CUmof-9 was performed under a humid atmosphere inside a desiccator containing a saturated solution of NH_4NO_3. Elemental analysis: C 41.96%, H 2.27%.

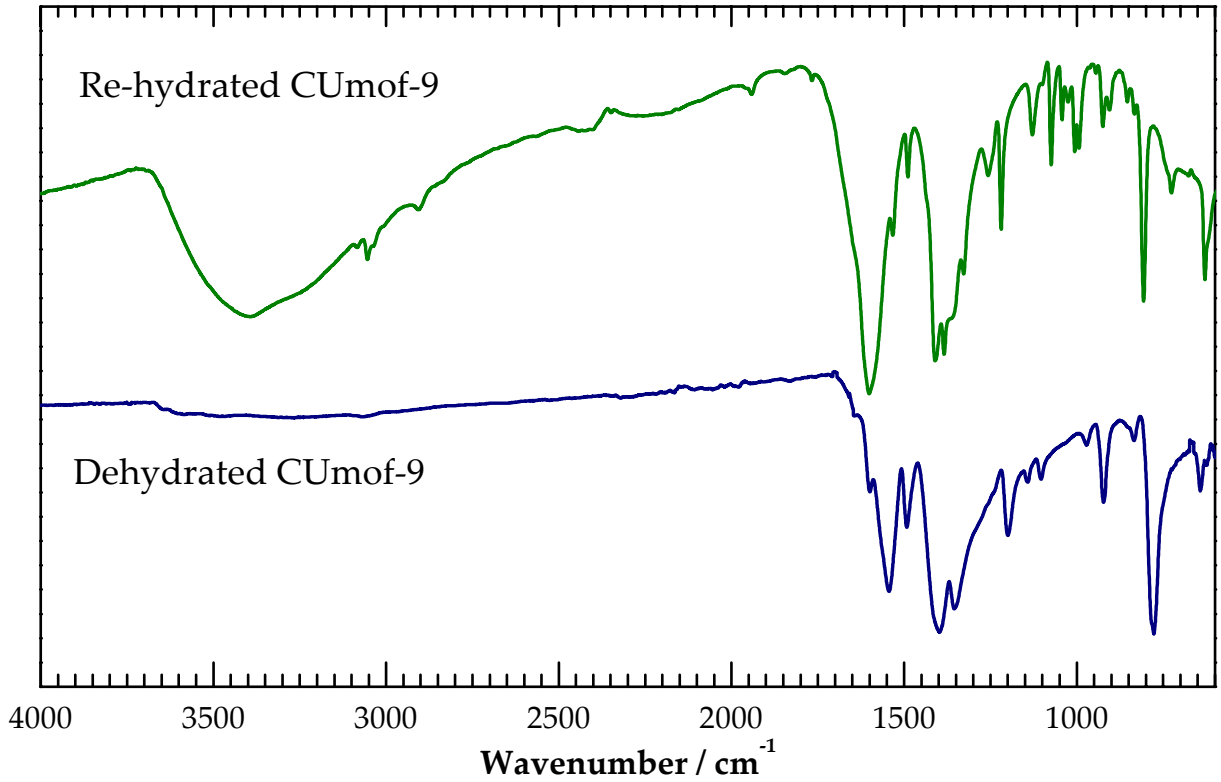

Fig. S5 Comparison of the FT-IR spectra for dehydrated (bottom) and re-hydrated (top) CUmof-9. FT-IR spectra were collected using a Thermo Nicolet spectrometer (NEXUS family) equipped with a Smart Golden Gate for ATR analyses.