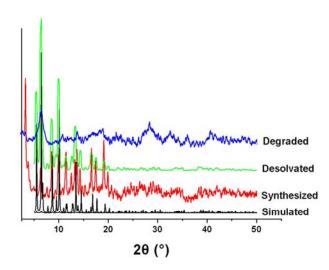
## **Supporting Information**

## Stepwise Assembly of Metal–Organic Framework Based on a Metal–Organic Polyhedron Precursor for Drug Delivery

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**Fig. S1** Experimental and simulated powder X-Ray diffraction patterns for **2**. The blue line represents the PXRD pattern of **2** after the drug release experiments.

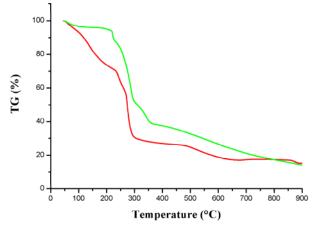


Fig. S2 TG profiles of 2 (red) and evacuated 2 (green).

**Drug Loading** Dissolving 5-FU (30 mg) and 2 (20 mg) in methanol (5 mL) three days yielded heterogeneous green solution. The precipitation was isolated by centrifuging and washed with methanol. The 5-FU content was calculated using UV method ( $\lambda = 266$  nm).

**Drug Release** 7 mg of drug-loaded **2** was dissolved into 2 mL of PBS buffer solution (pH 7.4), and loaded into a dialysis bag, which was dialyzed against 5 mL of deionized water at 37°C. During each time interval, about 1 mL of the solution was pulled out to test, and decanted back when the test was over. The content of 5-FU in the samples taken out was monitored by fluorometry, in which the detection wavelength was 453 nm.