

Electronic Supporting Informations

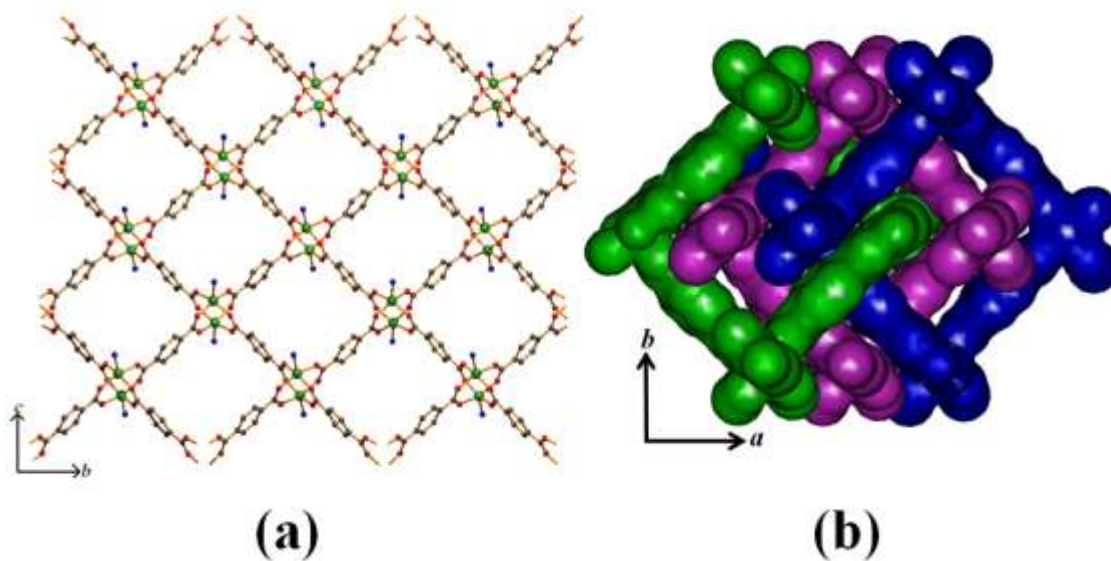


Fig. S1 (a) 2D brick-wall arrangement in **4** constructed by metal-terephthalate paddle-wheel moiety (*Meazpy* has been omitted for clarity); (b) View of the threefold interpenetrated of the 3D networks.

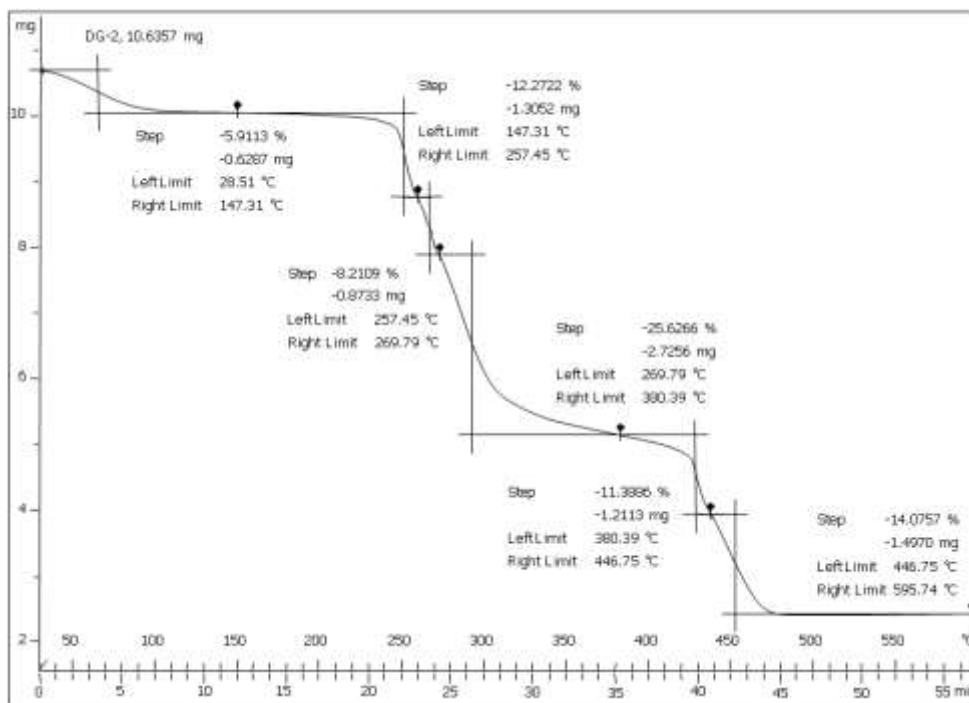


Fig. S2 TGA of the compound **2** under N₂ atmosphere.

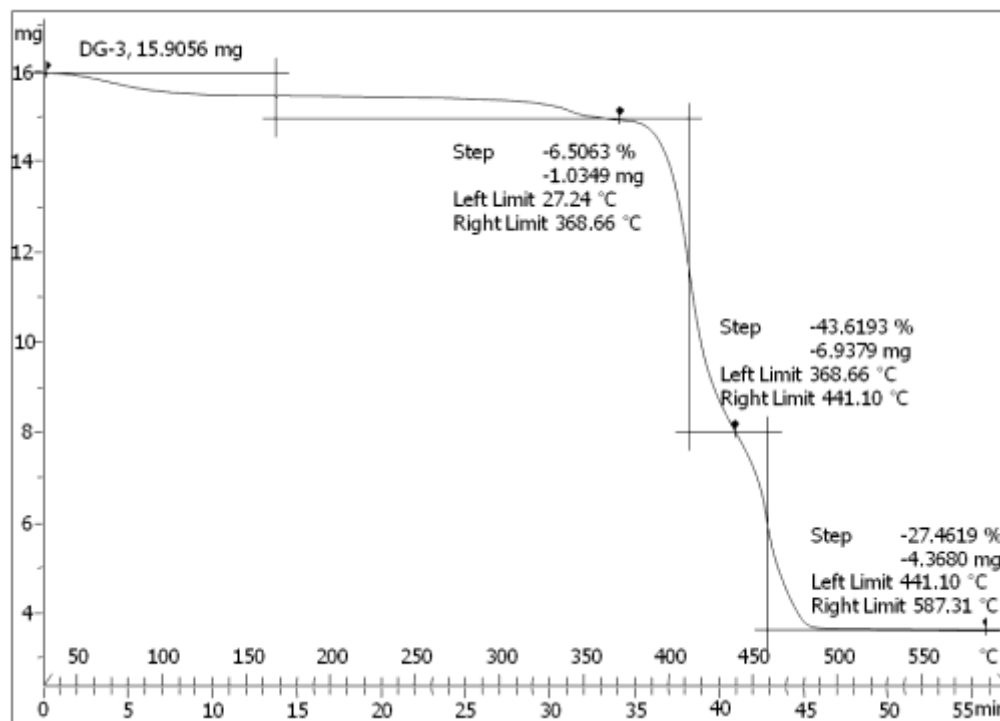


Fig. S3 TGA of the compound 3 under N₂ atmosphere

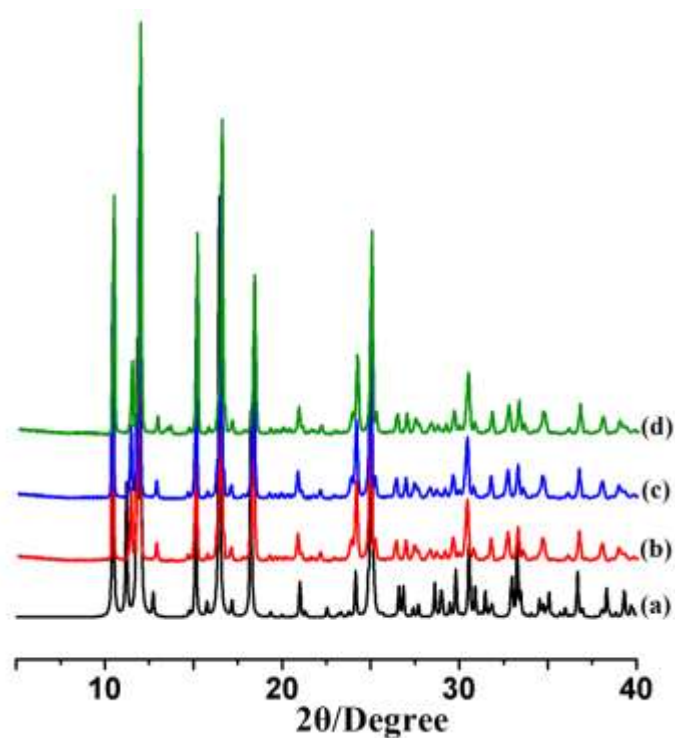


Fig. S4 PXRD patterns of **3** in different states. (a) Simulated from X-ray single crystal data; (b) bulk as-synthesized compound; (c) at 150 °C; (d) exposed to the water vapour for seven days.

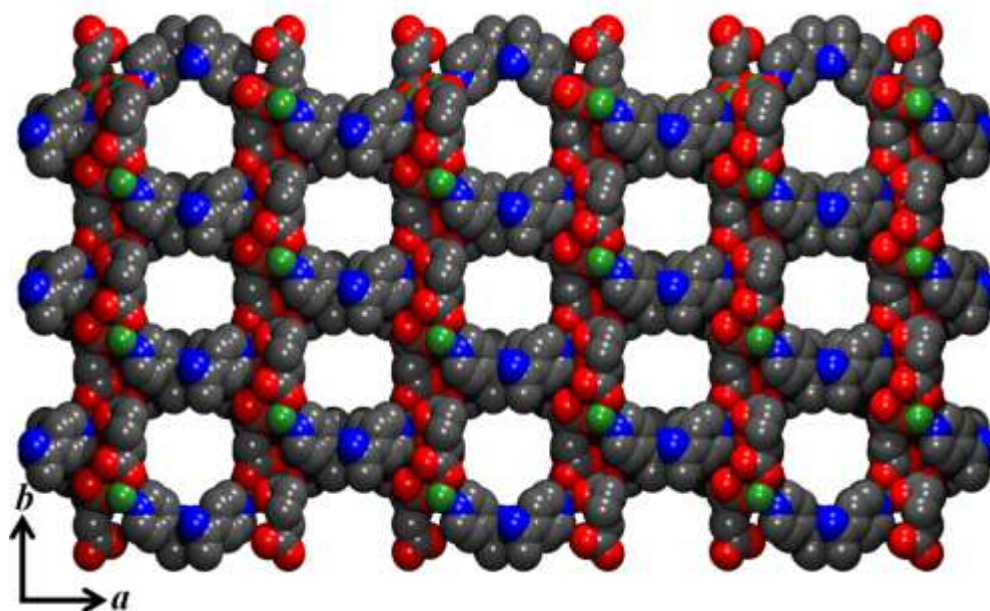


Fig. S5 The space filling model of the dehydrated species of **2** showing the 3D honeycomb-like empty channel along *c*-axis.

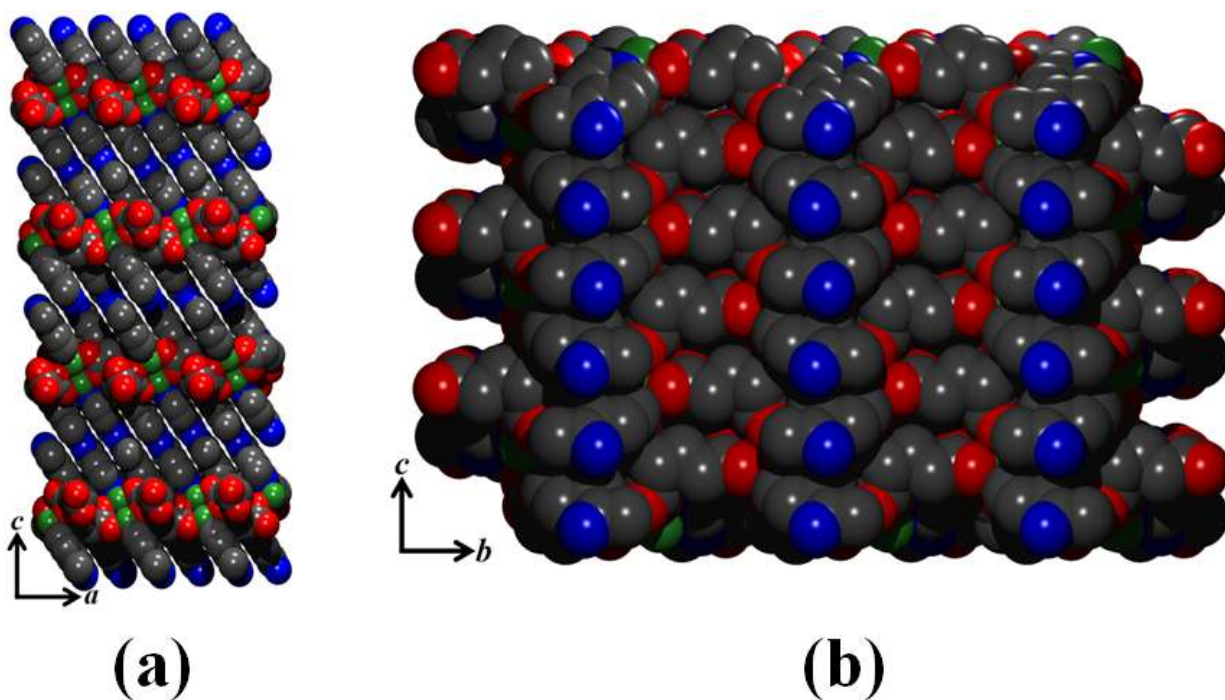


Fig. S6 The space filling model of the dehydrated species of **2** viewed along *b*-axis and *a*-axis.

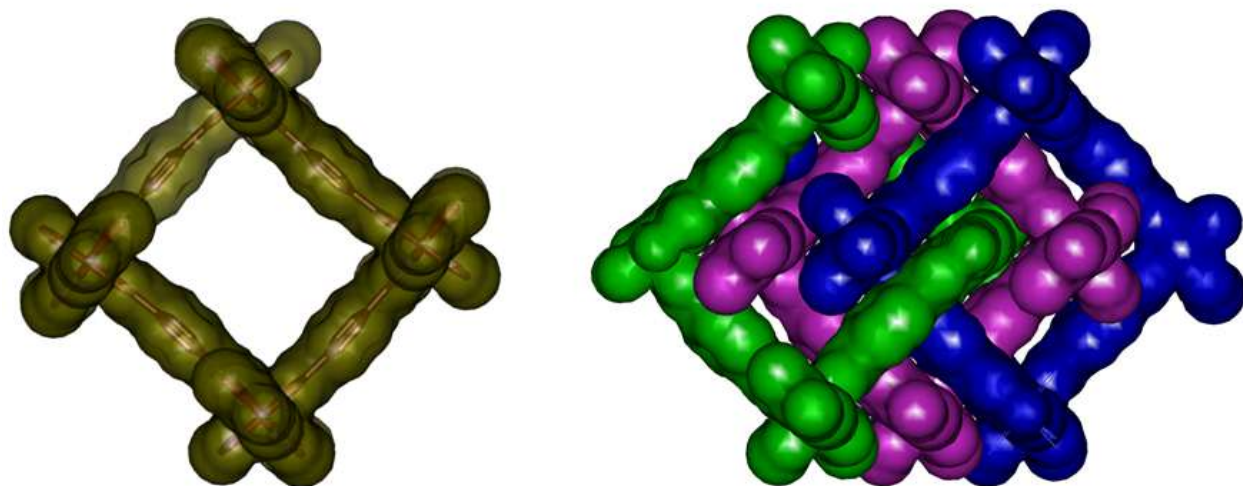


Fig. S7 Blockage of pore due to three-fold interpenetration in **3** and **4**.

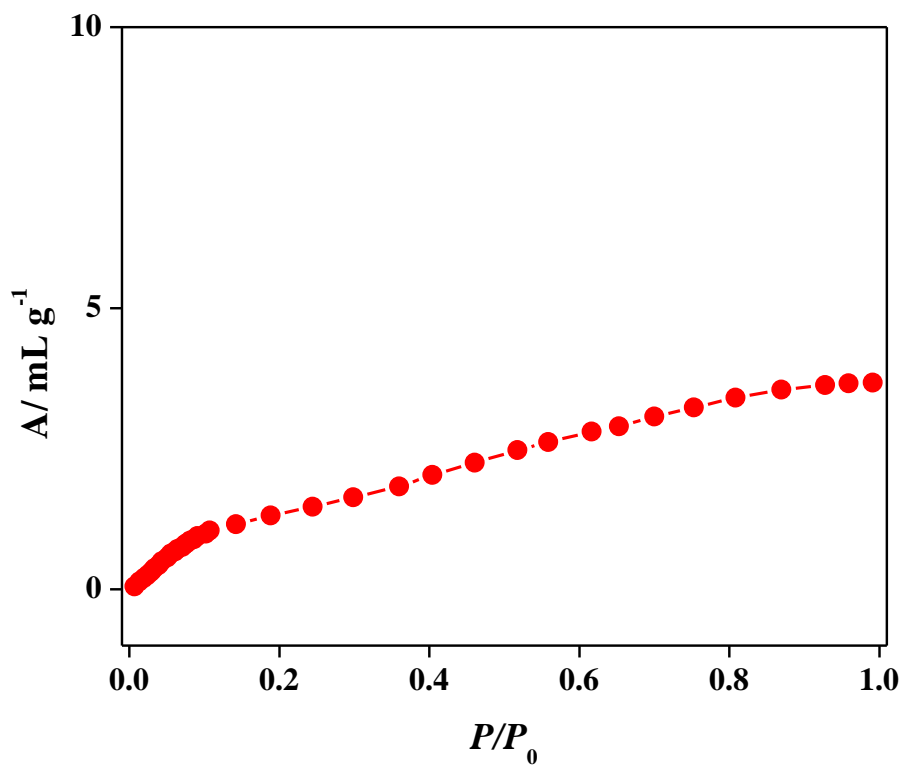


Fig. S8 H_2 adsorption isotherms of **3** below 1.0 bar pressure at 77 K.

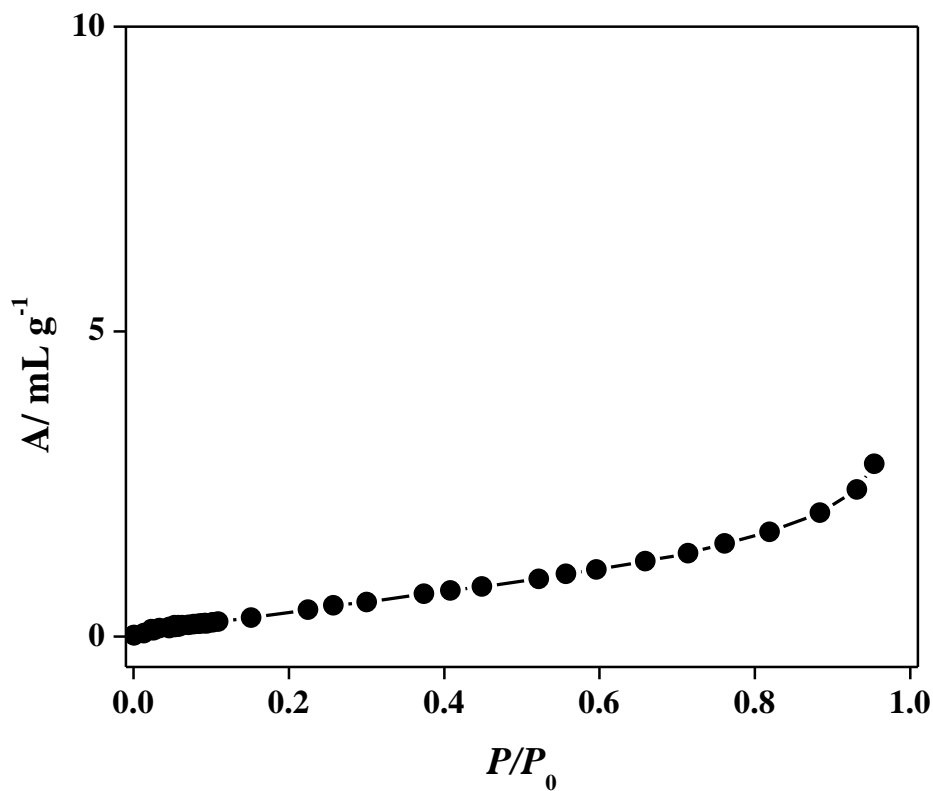


Fig. S9 N₂ adsorption isotherms of **3** below 1.0 bar pressure at 77 K.

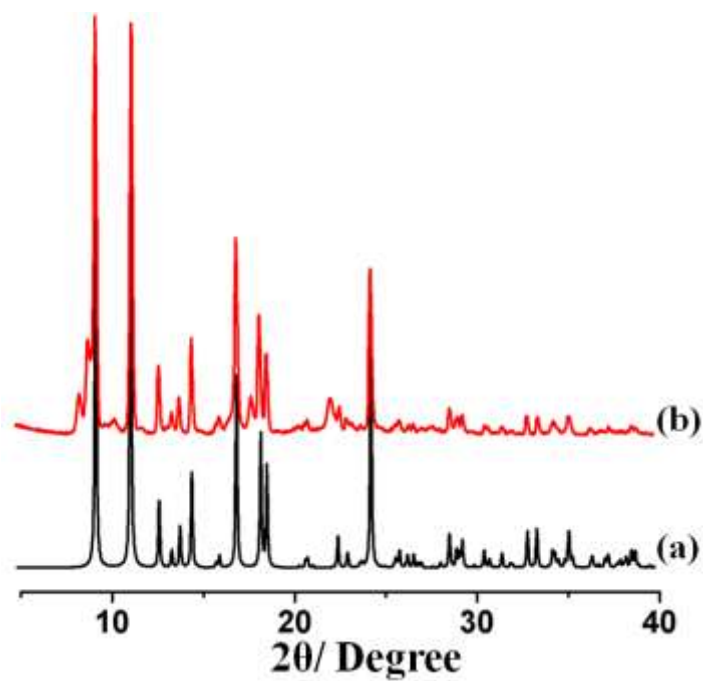


Fig. S10 PXRD patterns of **4** in different states (a) Simulated from X-ray single crystal data; (b) bulk as-synthesized compound.

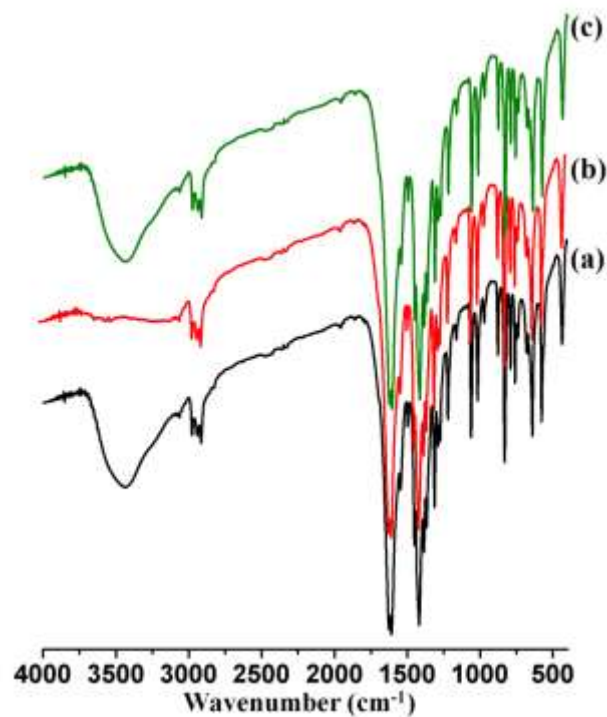


Fig. S11 FT-IR spectra of **2** in different states. (a) As-synthesized; (b) Dehydrated; (c) Rehydrated.

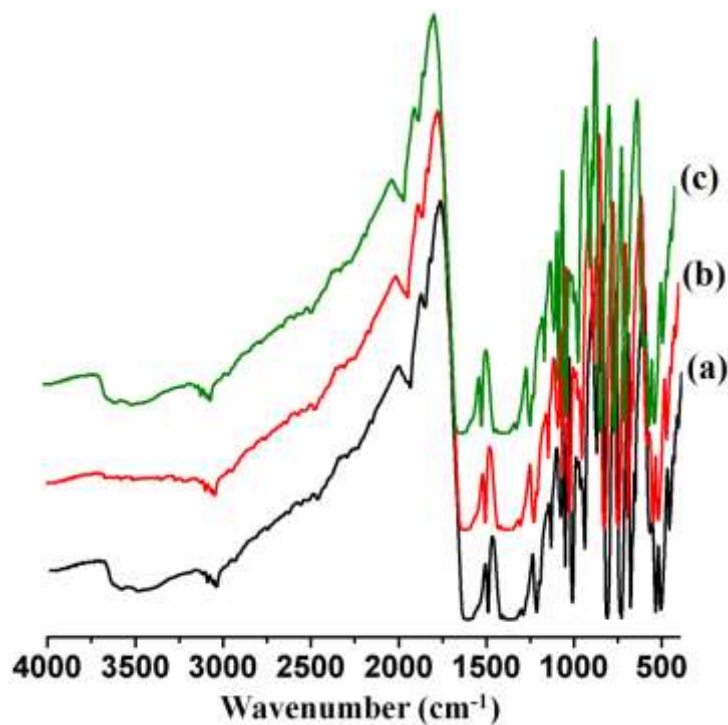


Fig. S12 FT-IR spectra of **3** in different states. (a) As-synthesized; (b) Dehydrated; (c) Rehydrated.

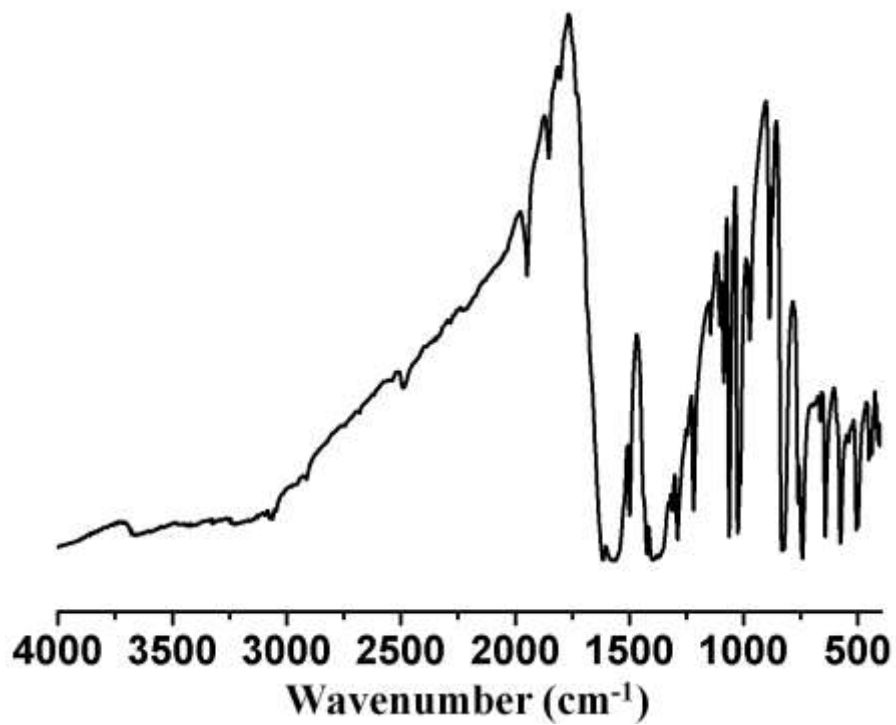


Fig. S13 FT-IR spectra of compound 4.