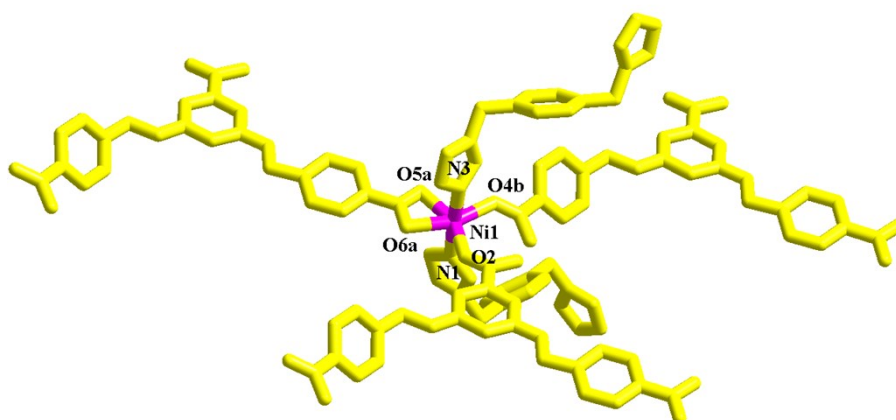
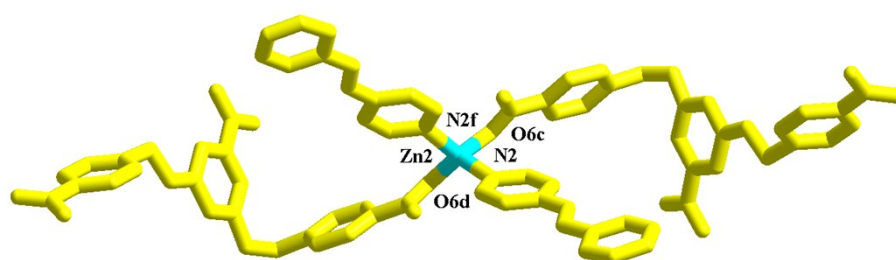
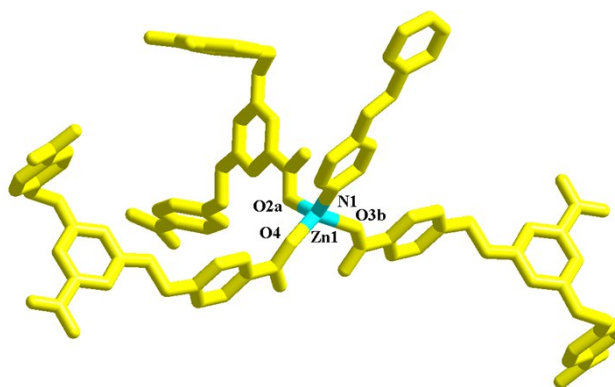


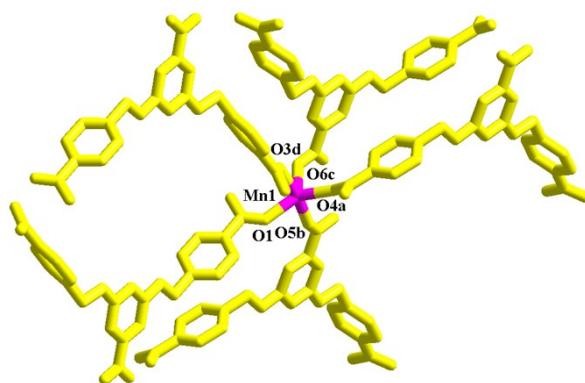
## Supporting materials



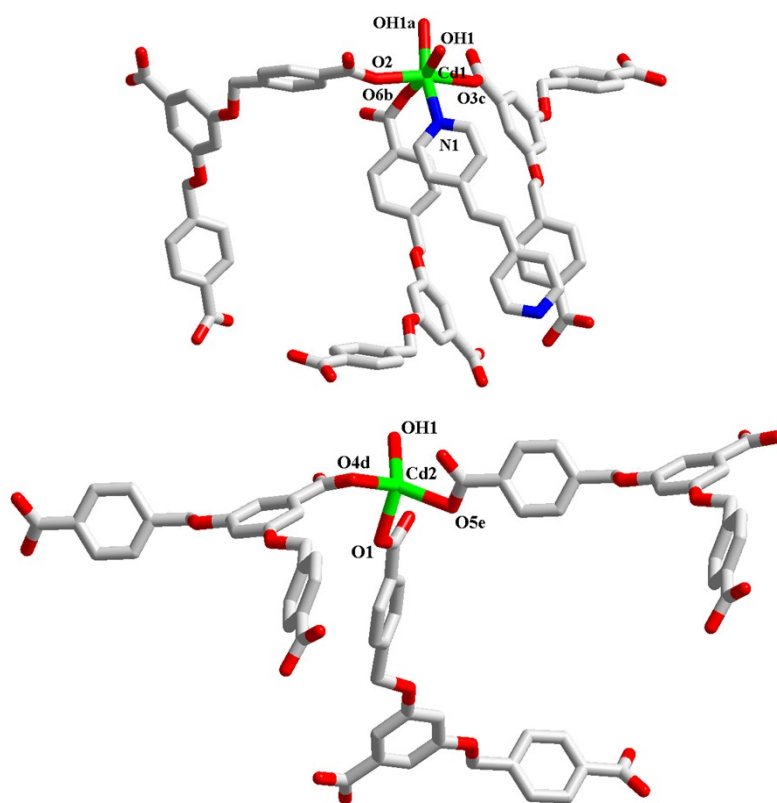
**Fig. S1** Coordination environment around Ni1 in **1** (a:  $x-1, y-1, z$ ; b:  $x, y, z-1$ ).



**Fig. S2** Coordination environments around Zn1 and Zn2 in **2** (f:  $-x, y, -z+3/2$ ).



**Fig. S3** Coordination environment around Mn1 in 3 (a:  $x+1, -y+1, z-1/2$ ; b:  $x+1/2, -y+3/2, z-1/2$ ; c:  $-x+5/2, y-1/2, -z-1/2$ ; d:  $-x+2, -y+1, -z$ ).



**Fig. S4** Coordination environments around Cd1 and Cd2 in 4 (a:  $x, y+1, z$ ; b:  $-x+1/2, y-1/2, -z+3/2$ ; c:  $-x+1, -y, -z+1$ ; d:  $-x, -y+2, -z+1$ ; e:  $x-1/2, -y-1/2, z+1/2$ ; f:  $-x, -y+1, -z+1$ ).

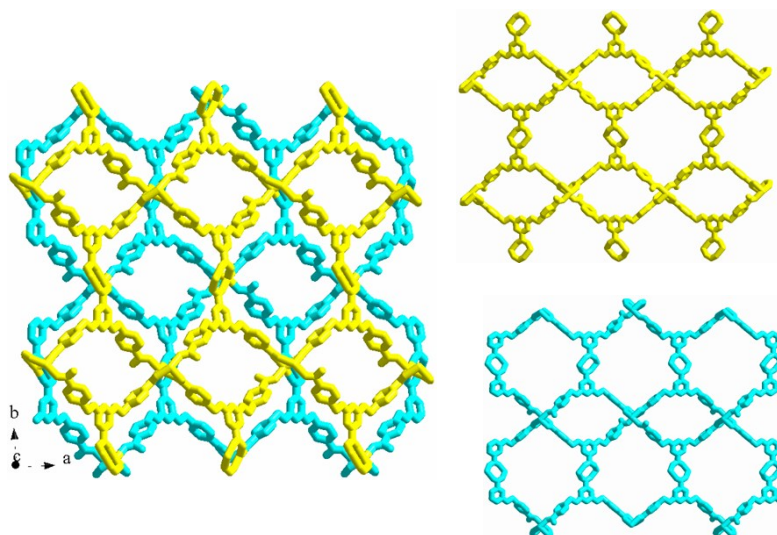


Fig. S5 A 3-D network constructed from two types of layers in **3**.

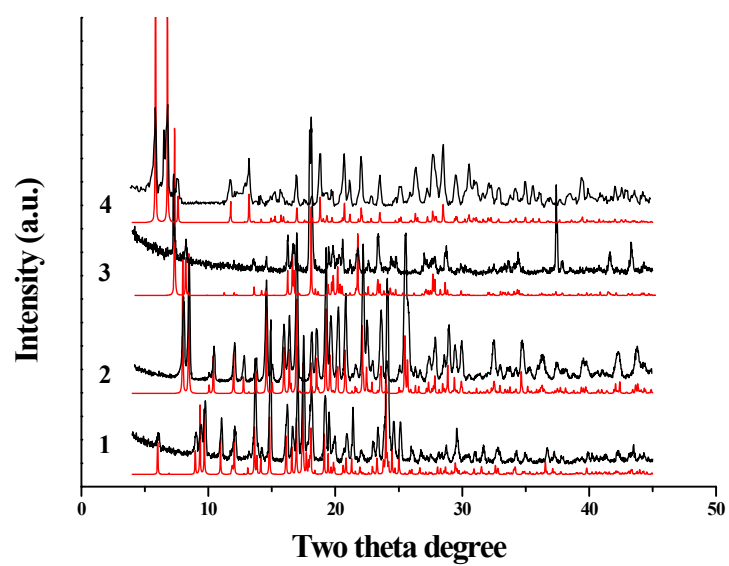


Fig. S6 Powder XRD patterns of **1-4**.

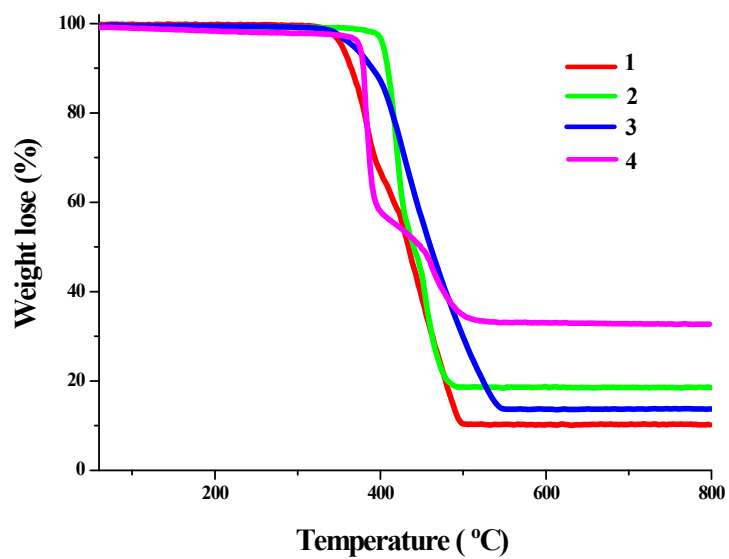


Fig. S7. TG curves of 1-4.

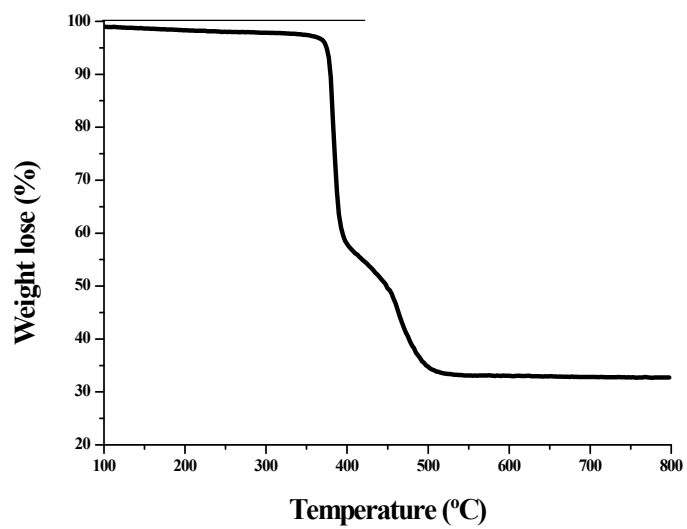


Fig. S8 TG curve of 4.

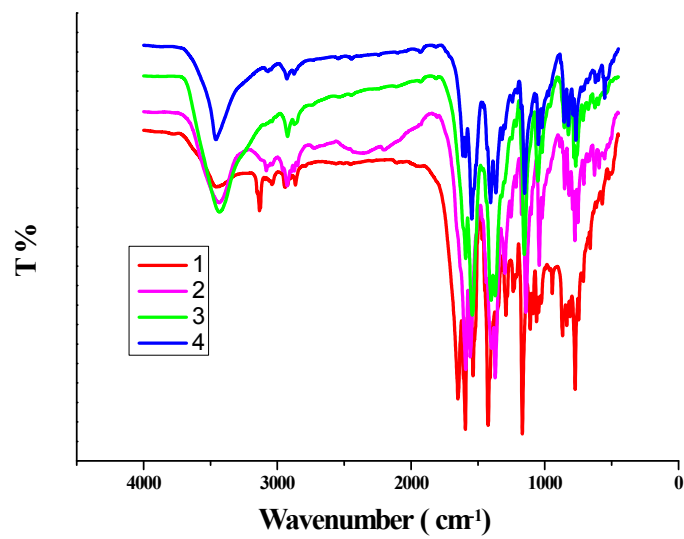


Fig. S9 IR spectra of 1-4.

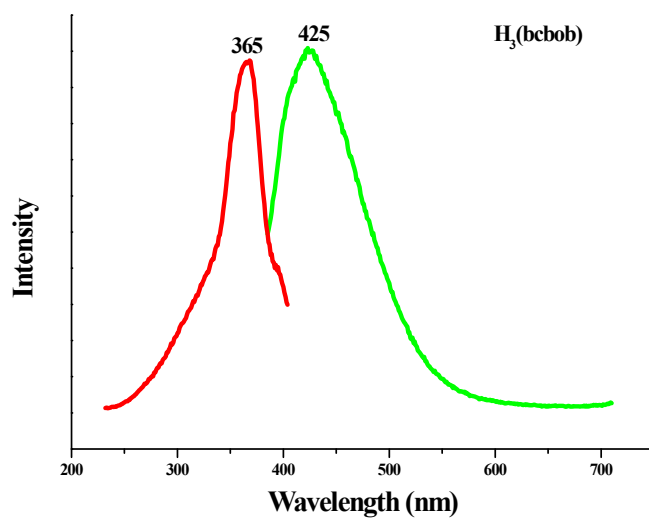
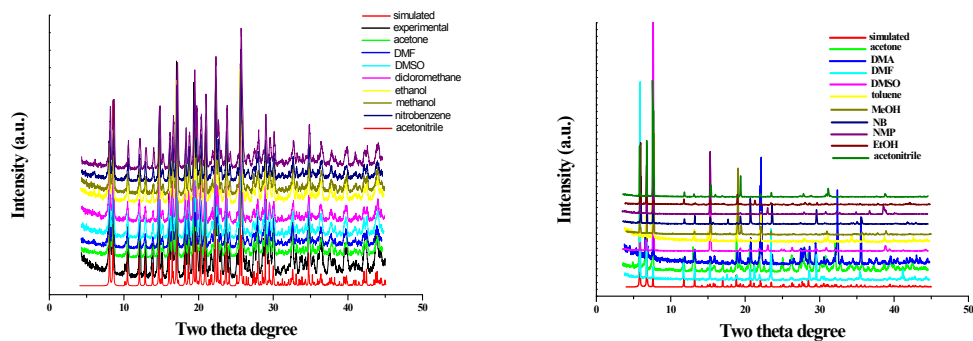


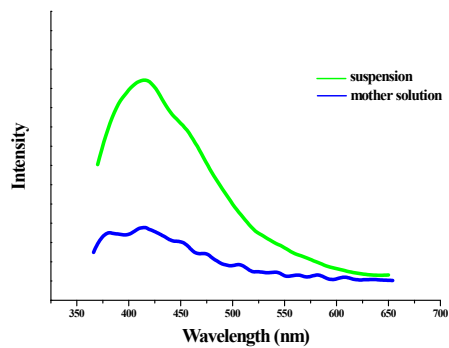
Fig. S10 Photoluminescence spectra of  $H_3(bcbob)$ .



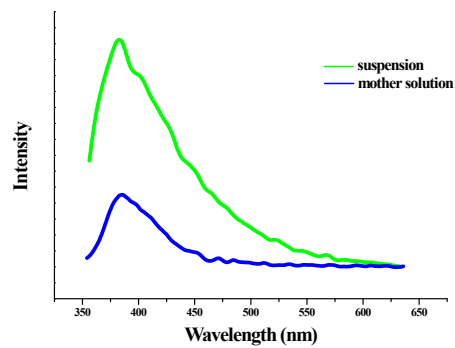
(a)

(b)

Fig. S11 Powder XRD patterns of 2 (a) and 4 (b) obtained after immersing in different solvents.



(a)



(b)

Fig. S12 Emission spectra of suspension and mother solution for 2 (a) and 4 (b).