Fig. S1. Simplified net of 1 with (a) top view and (b) side view.
Fig. S2. (a) TG curves and (b) PXRD patterns of 1.
Fig. S3. Simplified net of 2 with (a) top view and (b) side view.
Fig. S4. (a) TG curves and (b) PXRD patterns of 2.
Fig. S5. (a) TG curves of as-prepared (3), MeOH-exchanged (3a), and activated (3b) samples. (b) Time-dependent TG curve of 3. One H$_2$O molecule (3.7%) per Zn was adsorbed from air when temperature was decreased to 30 °C.
Fig. S6. Nujol IR data of as-synthesized, MeOH-exchanged, and activated phases of 3. The dotted line indicates the C=O stretching vibration of DMA.
Fig. S7. (a) N$_2$ isotherms at 77 K and (b) CO$_2$ isotherms at 195, 273, and 298 K for 3b.
**Fig. S8.** TG curves of as-prepared (4) and MeOH-exchanged phases (4a).
Fig. S9. PXRD profiles of as-synthesized, MeOH-exchanged, activated, and resolvated samples of 4. Schematic diagram shows phase transformations during the solvation-desolvation process.
Fig. S10. Nujol IR data of as-synthesized, MeOH-exchanged, and activated phases of 4. The dotted line indicates the C=O stretching vibration of DMF.
Fig. S11. (a) N\textsubscript{2} isotherms at 77 K and (b) CO\textsubscript{2} isotherms at 195, 273, and 298 K for 4b.
Fig. S12. Photoluminescence spectra of dim, H₄dobdc and 3 in DMF. The inset shows the enlargement of the spectra of dim and H₄dobdc ($\lambda_{ex} = 295$ nm).

Fig. S13. Photoluminescence spectra of dim, H₄dobpdc and 4 in DMF.
**Fig. S14.** Photoluminescence spectra of 4 in several solvents ($\lambda_{ex} = 295$ nm).

**Fig. S15.** Concentration-dependent photoluminescence of 4 by the addition of different amounts of NB in DMF.
Fig. S16. Plot of intensity versus concentration of NB of 4.