Electronic Supporting Information

Periodic Mesoporous Organosilicas for Ultrahigh Selective Copper(II) Detection and The Sensing Mechanism

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1. \(^1\text{H} \) NMR Spectra of BRh and BRh-Si\(_4\).

Figure S1. \(^1\text{H} \) NMR spectrum of BRh.

Figure S2. \(^1\text{H} \) NMR spectrum of BRh-Si\(_4\).
2. Structural Characterization of BRhPMOs.

**Figure S3.** Small angle X-ray scattering (SAXS) patterns for solvent-extracted BRhPMO-10 and BRhPMO-15.
Figure S4. Nitrogen adsorption/desorption isotherms and pore size distribution of solvent-extracted BRhPMOs.

The BET surface areas, pore volume, and BJH pore diameter for BRhPMO-1 were calculated to be 643 m$^2$ g$^{-1}$, 0.45 cm$^3$ g$^{-1}$, and 2.78 nm, respectively. For BRhPMO-5, these values were 579 m$^2$ g$^{-1}$, 0.41 cm$^3$ g$^{-1}$, and 2.75 nm, while for BRhPMO-15, these values were 413 m$^2$ g$^{-1}$, 0.39 cm$^3$ g$^{-1}$, and 4.41 nm respectively.
Figure S5. FT-IR spectra of BRh-Si₄, solvent-extracted BRhPMO-0 and BRhPMO-10.

Figure S6. $^{29}$Si MAS NMR spectrum of solvent-extracted BRhPMO-10.
**Figure S7.** Thermal analysis for BRh and solvent-extracted BRhPMO-10: TG curves (solid line) and DTA curves (dash line).

**3. Optical Characterization of BRhPMOs.**

**Figure S8.** Fluorescence response of BRhPMOs with different BRh-Si₄/TEOS molar ratios upon the addition of Cu²⁺ (10⁻⁴ M) in C₂H₅OH/HEPES (8:2 v/v, pH 6.8).
Figure S9. The mechanism for Cu$^{2+}$-induced ring-opening of BRh units in BRhPMOs.

Figure S10. Job’s plot for BRh-Si$_4$ and Cu$^{2+}$ in THF. The total concentration of BRh-Si$_4$ and copper ion was 1.0×10$^{-4}$ M.
**Figure S11.** Fluorescence response of BRhPMOs (0.1 mg mL\(^{-1}\)) upon the addition of Cu\(^{2+}\) in C\(_2\)H\(_5\)OH/HEPES (8:2 v/v, pH 6.8). Inset: emission intensity at 548 nm of BRhPMOs (0.1 mg mL\(^{-1}\)) as a function of Cu\(^{2+}\) concentration in 10\(^{-5}\) M range (1.0×10\(^{-5}\) to 10×10\(^{-5}\) M). Excitation at 500 nm.