Supporting Information

Synthesis of dithioglycol-functionalized periodic mesoporous organosilicas for the simultaneous removal of mercury ions and organic dyes from water

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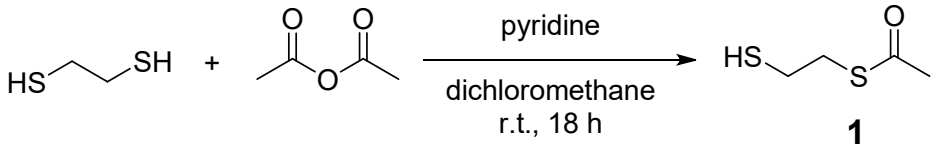
1. Synthesis of materials

2-acetylthioethanethiol (ref. 1): To a solution of 1,2-ethanedithiol (4.71 g, 50 mmol) in pyridine (13 mL) and dichloromethane (13 mL) was added acetic anhydride (5.105 g, 50 mmol). The resulting mixture was stirred at room temperature for 18 h. the solvent was removed under reduced pressure and the product (**1**) was purified by chromatography on silica gel using dichloromethane/hexanes (1:1, v/v) to give a light yellow liquid.



2. Characterization data

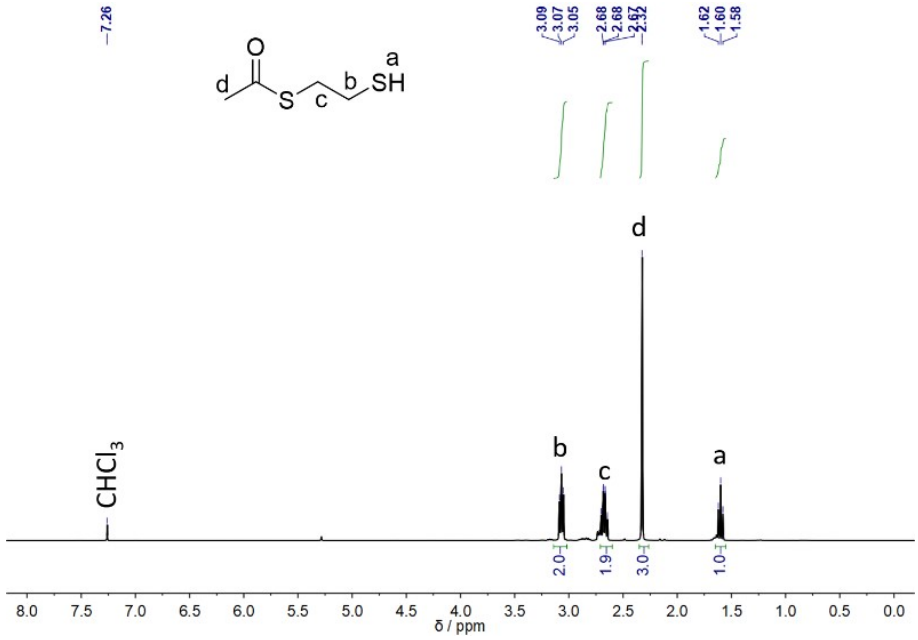


Figure S1. ^{1}H NMR (400 MHz, $CDCl_3$) of 2-acetylthioethanethiol (**1**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.09 (t, J = 7.4 Hz, 2H), 2.70 (dt, J = 8.6, 7.5 Hz, 2H), 2.35 (s, 3H), 1.62 (t, J = 8.4 Hz, 1H).

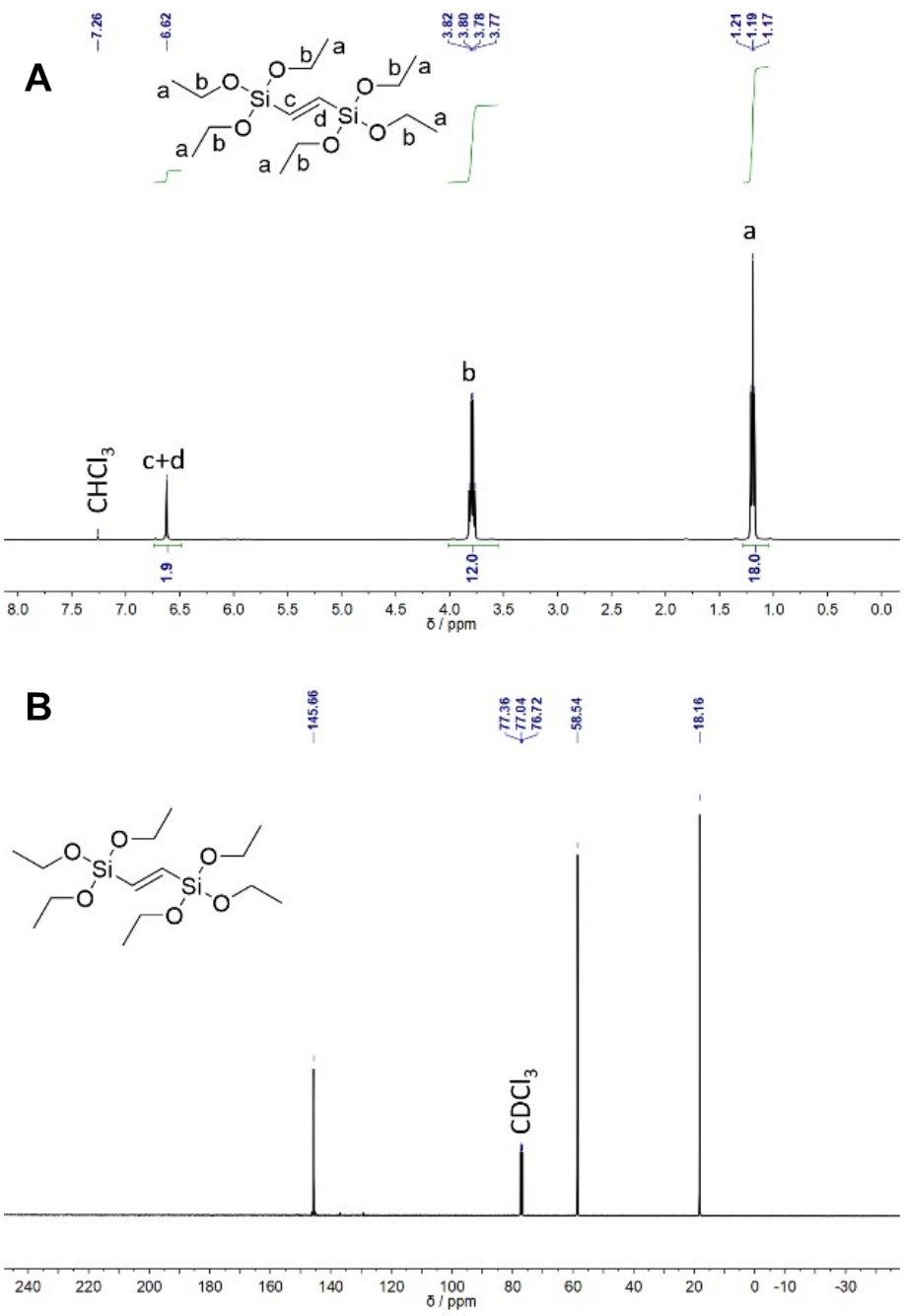


Figure S2. (A) ^{1}H NMR (400 MHz, $CDCl_3$) and (B) ^{13}C NMR (101 MHz, $CDCl_3$) spectra of 1,2-(*E*)-bis(triethoxysilyl)ethene (BTEE). ^{1}H NMR: δ 6.62 (s, 2H), 3.79 (q, J = 7.0 Hz, 12H), 1.19 (t, J = 7.0 Hz, 18H); ^{13}C NMR: δ 145.66 (s), 58.54 (s), 18.16 (s).

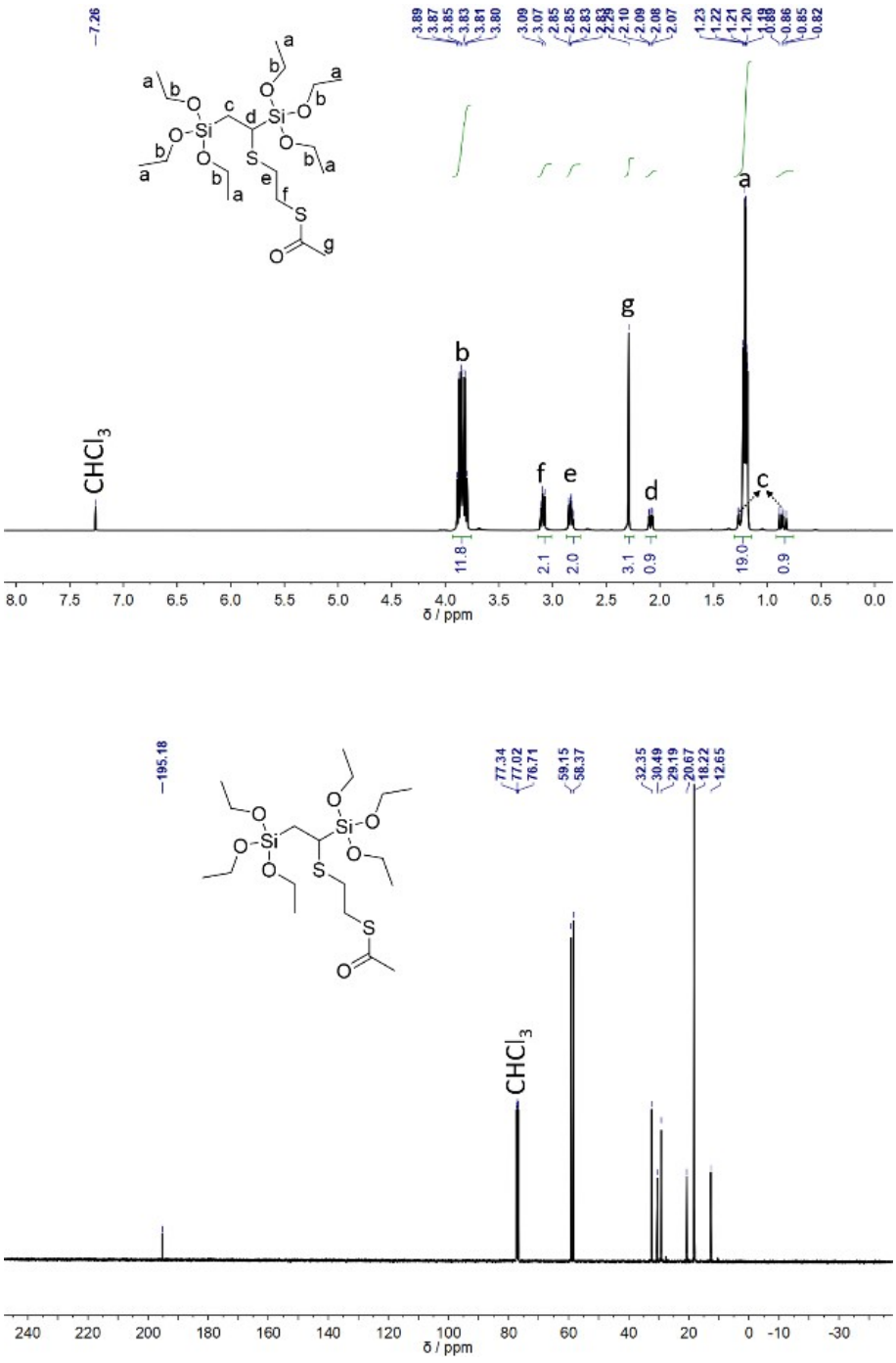


Figure S3. (A) ^{1}H NMR (400 MHz, $CDCl_3$) and (B) ^{13}C NMR (101 MHz, $CDCl_3$) spectra of 1-(2-thioacetylethylthio)-1,2-bis(triethoxysilyl)ethane (**2**). ^{1}H NMR δ 3.96–3.75 (m, 12H), 3.17–3.02 (m, 2H), 2.91–2.76 (m, 2H), 2.31 (s, 3H), 2.11 (dd, J = 10.7, 3.8 Hz, 1H), 1.37–1.11 (m, 19H), 0.96–0.80 (m, 1H). ^{13}C NMR δ 195.18 (s), 59.15 (s), 58.37 (s), 32.35 (s), 30.49 (s), 29.19 (s), 20.67 (s), 18.22 (s), 12.65 (s).

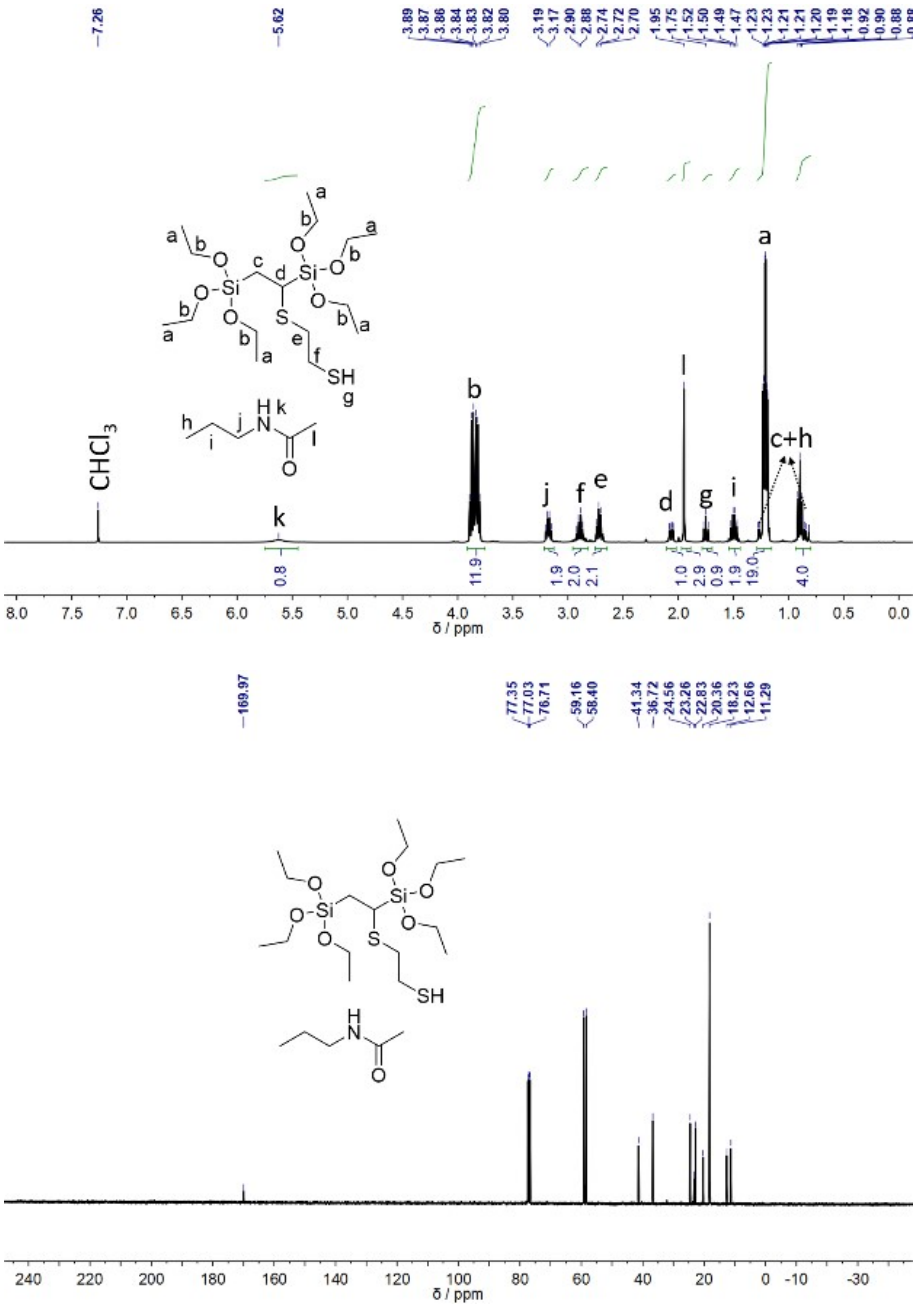


Figure S4. ^{1}H NMR and ^{13}C NMR of a mixture containing 1-(2-mercaptoethylthio)-1,2-bis(triethoxysilyl)ethane (DT-BTEE) and propylacetamide. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.62 (s, 1H), 3.84 (dq, J = 10.5, 7.0 Hz, 12H), 3.18 (dd, J = 13.6, 6.6 Hz, 2H), 2.93–2.85 (m, 2H), 2.75–2.69 (m, 2H), 2.06 (dd, J = 10.7, 3.7 Hz, 1H), 1.95 (s, 3H), 1.75 (t, J = 8.1 Hz, 1H), 1.49 (dt, J = 14.6, 7.3 Hz, 2H), 1.28–1.18 (m, 19H), 0.94–0.77 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.97 (s), 59.16 (s), 58.40 (s), 41.34 (s), 36.72 (s), 24.56 (s), 23.26 (s), 22.83 (s), 20.36 (s), 18.23 (s), 12.66 (s), 11.29 (s).

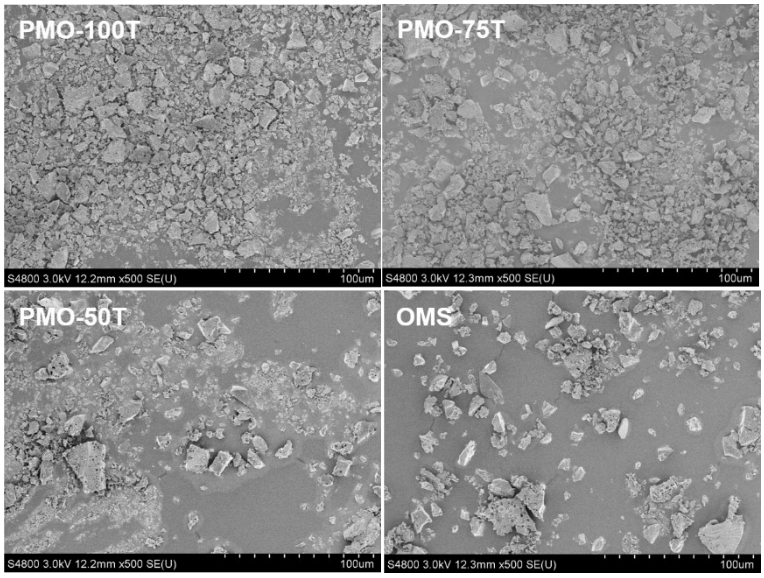


Figure S5. SEM images of DT-PMO and OMS particles.

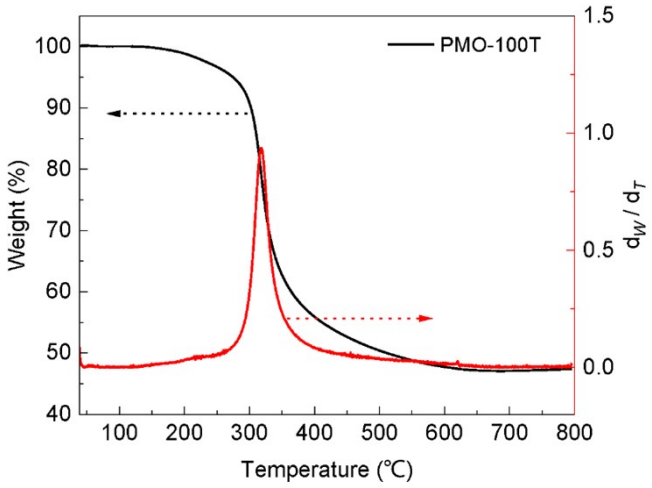


Figure S6. TGA and DTG curves of PMO-100T.

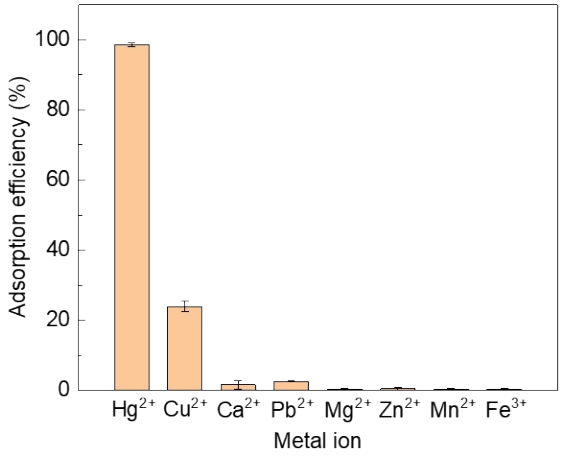


Figure S7. The adsorption selectivity of PMO-100T (the initial concentration of each coexisting ion was 10 ppm).

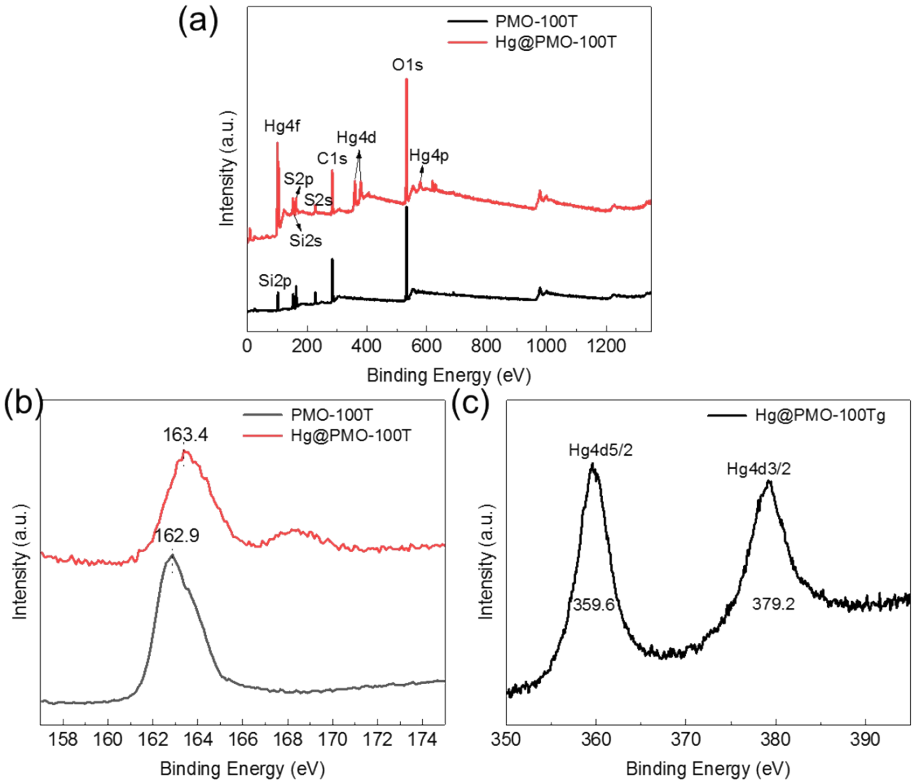


Figure S8. (a) Full survey XPS spectra of PMO-100T and Hg@PMO-100T samples. High resolution spectra of (b) S2p and (c) Hg4d.

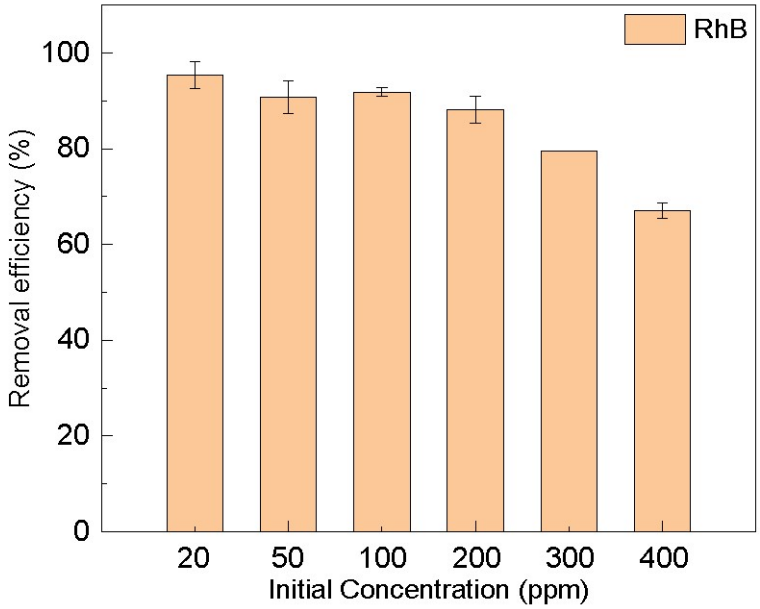


Figure S9. Histogram showing the % RhB removal from water by PMO-100T at the initial concentration of 20–400 ppm (adsorbent conc.= 1.5 mg/mL, pH = 5).

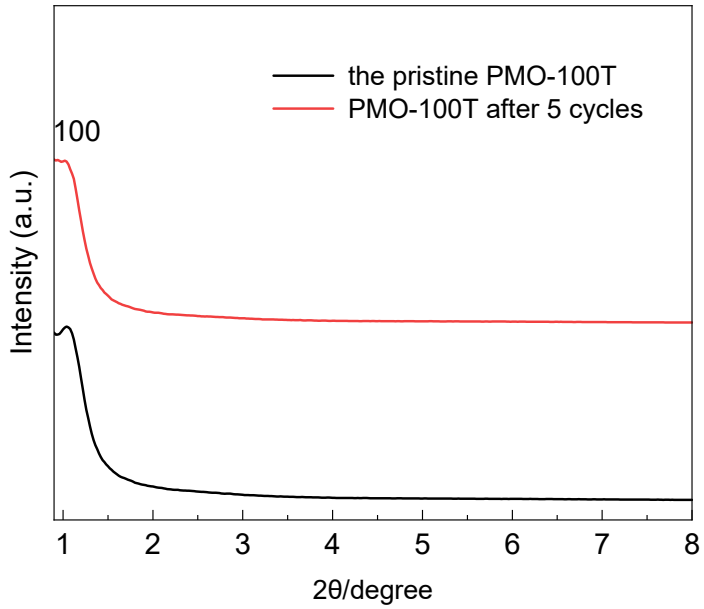


Figure S10. XRD patterns of PMO-100T before Hg(II) adsorption and after it uses for 5th cycle.

Table S1. The parameters of the Langmuir and Freundlich models

Pollutants	Langmuir model			Freundlich model		
	q_m (mg g^{-1})	K (L mg^{-1})	R^2	K_f (mg g^{-1})	n	R^2
Hg^{2+}	1253.0	0.04042	0.9861	49.7172	1.5650	0.9097
RhB	317.9	0.0491	0.9989	27.5328	1.9062	0.9982

Table S2. The parameters of pseudo-first-order and pseudo-second-order kinetics models

Pollutants	$q_{e,exp.}$ (mg/g)	pseudo-first			pseudo-second		
		q_e (mg/g)	k_1 (min^{-1})	R^2	q_e (mg/g)	k_2 (g mg^{-1} min^{-1})	R^2
Hg^{2+}	898.8	176.5	0.02657	0.8709	900.9	6.55×10^{-4}	0.9999
RhB	268.6	17.9	0.00541	0.5206	262.5	3.0×10^{-3}	0.9999

References:

[1] J. Xun, H. D. Huang, K. W. Vogel, D. G. Drueckhammer, The importance of the amide bond nearest the thiol group in enzymatic reactions of coenzyme A. *Bioorg. Chem.*, 2005, 33, 90–107.