Electronic Supplementary Information (ESI)

A thin-layered chromatography plate prepared from naphthalimide-based receptor immobilized SiO$_2$ nanoparticles as a portable chemosensor and adsorbent for Pb$^{2+}$

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Experimental Section

**General**: $^1$H and $^{13}$C NMR spectra were measured on a Bruker ARX 300 apparatus. IR spectra were obtained for KBr pellets, in the range 400 - 4000 cm$^{-1}$, with a shimadzu FT-IR 8400S instrument, and Mass spectra were obtained by a JEOL JMS-700 mass spectrometer. The optical absorption spectra of the samples were obtained at 278-77 K using a UV–vis spectrophotometer (Hitachi U-2900). All fluorescence spectra were recorded in RF-5301PC spectrophotometer.

**SEM observations**: For transmission electron microscopy (TEM), a piece of the gel was placed on a carbon-coated copper grid (400 mesh) and removed after one min, leaving some small patches the sample on the grid. The specimens were examined with a JEOL JEM-2010 transmission electron microscope operating at 200 kV using an accelerating voltage of 100 kV and a 16 mm working distance. Scanning electron micrographs of the samples were taken with a field emission scanning electron microscope (FE-SEM, Philips XL30 S FEG). The accelerating voltage of SEM was 5–15 kV and the emission current was 10 μA.

**Photophysical studies**: Fluorescence spectra of 1 (10.0 mg) were observed upon addition of various metal ions (0-10.0 equivalents) in aqueous solution at pH 7.

**Preparation of compound 3**: Under nitrogen, a solution of 4-bromo-1,8-naphthalic anhydride (0.6 g, 2.18 mmol) compound 4 (0.7 g, 3.26 mmol) in 2-methoxyethanol (40 mL) was refluxed for 48 h, the mixture was evaporated by rotary evaporation and dissolved in CH$_2$Cl$_2$ (150 mL). The organic layer was washed with water (3 x 100 mL), dried over anhydrous Na$_2$SO$_4$, and filtered. After the solution was evaporated under reduced pressure, the crude product was purified by column chromatography (silica, CHCl$_3$/EtOH 99:1) to give 0.52 g of 3 (6.7 % yield). $^1$H NMR (300 MHz, CDCl$_3$, δ): 1.24 (t, 6H, J = 25 Hz), 2.77 (t, 2H, J = 25 Hz), 2.86 (t, 2H, J = 20 Hz), 4.16 (q, 2H, J = 24 Hz), 6.75 (d, 1H, J = 28), 7.65 (t, 1H, J = 25 Hz), 8.15 (d, 1H, J = 28), 8.50 (d, 1H, J = 28 Hz), 8.62 (d, 2H, J = 24). ESI-MS (m/z) Calcd. for C$_{22}$H$_{23}$N$_1$O$_7$: 413.1. Found: 440.1 [Na(3)]$^+$. FT-IR: 3004.32, 1712.97, 1362.10, 1420.66, 1221.97, 1092.20 and 901.51 cm$^{-1}$. Element analysis: calculated for C$_{22}$H$_{23}$N$_1$O$_7$: C 63.92 H 5.61 N 3.39. Found C 64.12 H 5.87 N 3.19

**Preparation of compound 2**: Compound 3 (0.6 g, 1.4 mmol), was dissolved in ethanol (20 mL). To this was added NaOH (0.87 g, 21.7 mmol) and the resulting solution refluxed for 6 h. Progress of this hydrolysis reaction was periodically monitored by TLC. Upon complete consumption of the ester, the reaction mixture was acidified with conc. H$_2$O and extracted with ethyl acetate. The organic layer was
dried over Na$_2$SO$_4$, filtered and concentrated to dry ness to afford pure compound 2 (84.2% yield). $^1$H NMR (300 MHz, CDCl$_3$, $\delta$): 2.55 (t, 2H, J = 26 Hz), 2.71 (t, 2H, J = 23 Hz), 3.61 (t, 2H, J = 21 Hz), 4.23 (t, 2H, J = 25 Hz), 6.8 (d, 1H, J = 29 Hz), 7.68 (t, 1H, J = 26 Hz), 8.2 (d, 1H, J = 29 Hz), 8.4 (d, 1H, J = 25 Hz), 8.7 (d, 1H, J = 28 Hz), 12.3 (s, 2H). $^{13}$C NMR (75.4 MHz, DMSO-d$_6$): $\delta$ 173.5 173.2, 164.0 163.1 150.8 134.6 130.9 129.7 129.0 124.5 122.0 120.5 108.1 104.1 49.0 35.9 33.5 33.0. FT-IR : 3404, 2924, 2584, 1723, 1682, 1639, 1576, 1549, 1357, 1261, 1233 and 1022 cm$^{-1}$. ESI-MS (m/z) Calcd. for C$_{18}$H$_{15}$N$_7$O$_7$: 357.0. Found: 356.1 (M$^+$. Element analysis: calculated for C$_{18}$H$_{15}$N$_7$O$_7$: C 60.51 H 4.23 N 3.92 O 31.34 Found C 60.87 H 4.51 N 4.23.

**Preparation of compound 1:** Compound 2 (35 mg) was dissolved in anhydrous toluene (20 mL). The silica nanoparticle (27 mg) was added as a solid. The suspension of silica was stirred under reflux condiions for 24 h in toluene. Then, the collected solid was washed copiously with toluene (50 mL) to rinse away any surplus 2 and dried under vacuum. FT-IR : 3404, 2924, 2584, 1723, 1682, 1639, 1576, 1549, 1357, 1261, 1233 and 1022 cm$^{-1}$.
Scheme S1. Preparation Method of 2-immobilized SiO₂ nanoparticle (1).
Fig. S1 FT-IR spectra of (a) 2, (b) mesoporous silica and (c) 1.
Fig. S2 TOF-SIMS spectrum of 1.
Fig. S3 Time course of the fluorescence intensity of 1 (10.0 mg) upon addition of Pb$^{2+}$ (10.0 equiv) in aqueous solution at pH 7; a) 0 min, (b) 0.5 min, (c) 1 min, (d) 2 min, (e) 3 min, (f) 4 min, (g) 5 min, (h) 6 min, (i) 7 min and (j) 8 min.

Fig. S4 Fluorescence intensity of 1 (10.0 mg) with Pb$^{2+}$ (10.0 equiv) in the presence of other metal ions such as Cu$^{2+}$, Ca$^{2+}$, Cd$^{2+}$, Hg$^{2+}$, Fe$^{2+}$, Na$^{2+}$, Ni$^{2+}$, Zn$^{2+}$ and Mg$^{2+}$ ions in water at pH 7.
Fig. S5 Fluorescence intensity of 1 (10.0 mg) before and after addition of metal ions (5.0 equivalents) at different pH values.