Electronic Supplementary Information

Facile self-assembly synthesis of titanate/ Fe_3O_4 nanocomposites for the efficient removal of Pb^{2+} from aqueous systems

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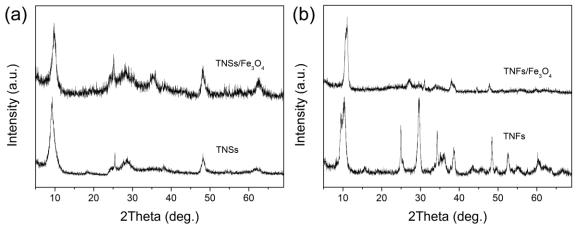


Fig. S1. XRD patterns of (a) TNSs and TNSs/Fe₃O₄; (b) TNFs and TNFs/Fe₃O₄.

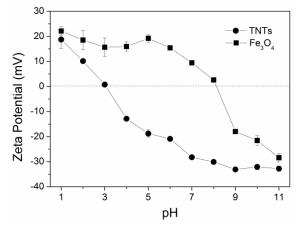


Fig. S2. Zeta potentials of TNTs and Fe₃O₄ NPs in aqueous solutions at different pH values.

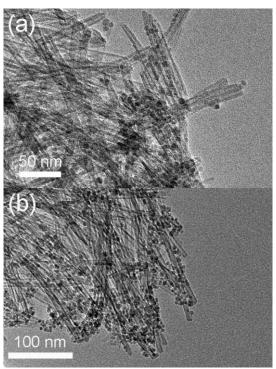


Fig. S3. TEM images of TNTs/Fe₃O₄ nanocomposites with different content of Fe₃O₄ NPs. (a) 10 μ L and (b) 30 μ L of Fe₃O₄ NPs solutions were separately added into different vials containing 12 mL deionized water and 5 mg TNTs.

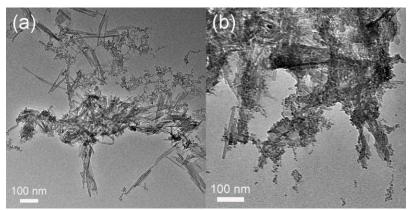


Fig. S4. TEM images of the original TNTs/Fe₃O₄ nanocomposites (a) before and (b) after Na₂CO₃ treatment.

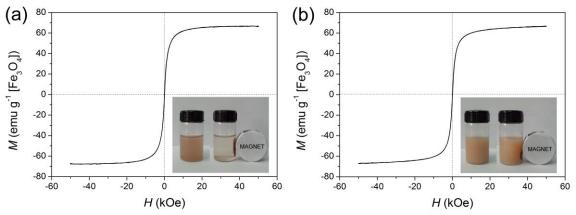


Fig. S5. Room temperature hysteresis loop of (a) TNSs/Fe₃O₄ and (b) TNFs/Fe₃O₄. The insets respectively show the water suspensions of the magnetic nanocomposites before and after magnetic separation.

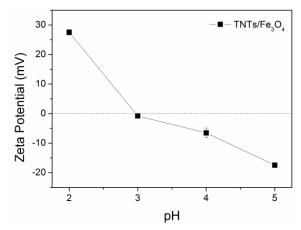


Fig. S6. Zeta potential of TNTs/Fe₃O₄ in aqueous solutions at different pH values.

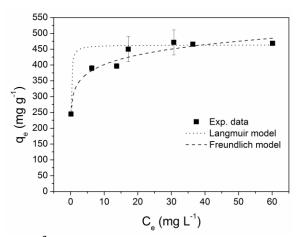


Fig. S7. Adsorption isotherm of Pb^{2+} on TNTs without coating Fe_3O_4 nanoparticles. (Materials dosage: 0.1 g L^{-1} , solution volume: 20 mL, pH: 5.0 and temperature: 25°C.)

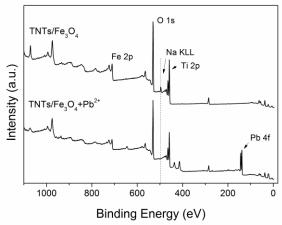


Fig. S8. XPS survey spectra of the TNTs/Fe $_3$ O $_4$ nanocomposites before and after Pb $^{2+}$ removal.

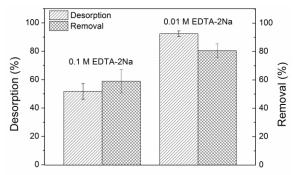


Fig. S9. Desorption of Pb²⁺ by EDTA-2Na with different concentration (0.01 M and 0.1M) and Pb²⁺ adsorption by regenerated adsorbents.

Table S1. pH values before and after adsorption. (Materials dosage: 0.1 g L^{-1} , solution volume: 20 mL, initial Pb²⁺ concentration: 40 mg L^{-1} , temperature: 25°C and contact time: 240 min.)

| | pH values | | | |
|-------------------|-----------|------|------|------|
| Before adsorption | 2.03 | 3.04 | 4.03 | 4.93 |
| After adsorption | 2.04 | 3.04 | 3.74 | 4.31 |

Table S2. Theoretical and Calculated q_e Values, Pseudo-Second-Order Rate Constants, k_2 , and Correlation Coefficient Values (\mathbb{R}^2). (Materials dosage: 0.1 g \mathbb{L}^{-1} , solution volume: 50 mL, initial metal ion concentration: 20, 60 and 100 mg \mathbb{L}^{-1} , pH: 5.0 and temperature: 25°C.)

| Theoretical q_e (mg g ⁻¹) | Calculated q_e (mg g ⁻¹) | $k_2 (\mathrm{g \ mg}^{-1} \mathrm{min}^{-1})$ | R^2 |
|---|--|--|--------|
| 200 | 200.00 | 1.48 | 1 |
| 600 | 303.95 | 0.0026 | 0.9995 |
| 1000 | 316.46 | 0.0052 | 0.9998 |

Table S3. Isotherm constants for the adsorption of Pb²⁺ by TNTs and TNTs/Fe₃O₄. The equilibrium metal ion concentration, C_e , the amount of metal ion adsorbed at equilibrium, q_e , the maximum adsorption capacity of the adsorbent, Q, the Langmuir constant related to the free energy of adsorption, b, the Freundlich constant related to the adsorption capacity of the adsorbent, K_F , the heterogeneity factor indicating the adsorption intensity of the adsorbent, n, and Correlation Coefficient Values (R²).

| Adsorption isotherm models | Isotherm constants | Adsorbents | |
|----------------------------|--------------------------|------------|-------------------------------------|
| Adsorption isotherm moders | Isotherm constants | TNTs | TNTs/Fe ₃ O ₄ |
| Langmuir isotherm model | $Q (\text{mg g}^{-1})$ | 463.66 | 382.30 |
| $a = QbC_e$ | $b (\mathrm{Lmg}^{-1})$ | 12.261 | 0.647 |
| $q_e = \frac{1}{1 + bC_e}$ | \mathbb{R}^2 | 0.99305 | 0.98539 |
| Freundlich isotherm model | $K_F(\text{mg g}^{-1})$ | 315.11 | 275.80 |
| $q_e = K_F C_e^{1/n}$ | n | 9.52 | 13.44 |
| $q_e - K_F C_e$ | \mathbb{R}^2 | 0.99731 | 0.97041 |

Preparation of titanate nanostructures and Fe₃O₄ nanoparticles. For the synthesis of titanate nanotubes, 3 g titanium dioxide were mixed with 10 M NaOH solution and placed into a Teflon-lined autoclave. After vigorously stirring for 1h, the mixture was sealed and hydrothermally treated at 130°C for 24h. The precipitation was separated by filtration and washed thoroughly with deionized water until a pH value was reached about 8.0. After filtration, the products were oven-dried at vacuum at 70°C overnight. Titanate nanofibers and nanosheets were obtained by simply adjust the hydrothermal conditions. For titanate nanofibers, the temperature was changed to 180°C, while for titanate nanosheets, 5 M NaOH was used for synthesis.

Water-soluble Fe_3O_4 NPs were prepared by a thermal decomposition method. In a typical synthesis, 2 mmol $Fe(acac)_3$ and 30ml TREG (triethylene glycol) were mixed and slowly heated to 278°C at a rate of 3°C/min under N_2 protection. The mixture was kept at reflux for 30min before it was cooled down to room temperature. After ethyl acetate was added to the black colloid suspensions, the products were collected by centrifugation at 20,000 rpm. The precipitations were transformed into ethyl acetate to form homogeneous suspensions after ultrasonication and separated by a magnet. After washing with ethyl acetate for three times, the precipitations were re-dispersed into water and stored at 4°C for further use.