**Supplementary Information**

**Experimental Section**

**Figure S1.** XRD patterns of Mn₃[Co(CN)₆]₂·nH₂O formed at different conditions a) no ethanol added to the reaction system with other experimental parameters kept constant; b) the ratio of ethanol/water in reaction system is 1:2 with other experimental parameters kept constant; c) no PVP added to the reaction system with other experimental parameters kept constant.

**Figure S2.** FT-IR spectra of Mn₃[Co(CN)₆]₂·nH₂O formed at different conditions a) no ethanol added to the reaction system with other experimental parameters kept constant; b) the ratio of ethanol/water in reaction system is 1:2 with other experimental parameters kept constant; c) no PVP added to the reaction system with other experimental parameters kept constant.

**Figure S3.** (a) Optical photograph of the mixture containing the Mn₃[Co(CN)₆]₂·nH₂O nanocubes and Pb²⁺. (b) Magnetic separation of Mn₃[Co(CN)₆]₂·nH₂O nanocubes. Because the magnetic field strength of the edge of the magnet (0.3 T) is much stronger than that of the center (≈0 T), the white precipitates were mainly located in the edge of centrifugal tube (the white box in the b).
Experimental Section:
The Removal of Pb\(^{2+}\) in the presence of Mn\(_3[Co(CN)\_6]\)\(_2\cdot nH\_2O\) nanocubes

Typically, the nanocubes suspension (1 g/L, 4 mL) dissolved in distilled water and aqueous solution (100mg/L, 1 mL) of Pb\(^{2+}\) were transferred to the centrifugal tube (10 mL). After uniform oscillated with 2h using a shaker ensure sufficient interaction between nanocubes and Pb\(^{2+}\), the nanocubes were separated from the solution with the help of a 0.3 T magnet. The amount of Pb\(^{2+}\) in solution was characterized by Plasma-atomic emission spectroscopy. The initial concentration of Pb\(^{2+}\) in the system was 20µg/mL, while the concentration of Pb\(^{2+}\) was 2.775µg/mL after adsorbing by Mn\(_3[Co(CN)\_6]\)\(_2\cdot nH\_2O\) nanocubes according to the Plasma-atomic emission spectroscopy. Therefore, the removal efficiency of Pb\(^{2+}\) can be calculated to 86%
Figure S1.
Figure S2.
Figure S3.