

## Supporting information for

# Fabrication of FeOOH Hollow Microboxes for Purification of Heavy Metal-Contaminated Water

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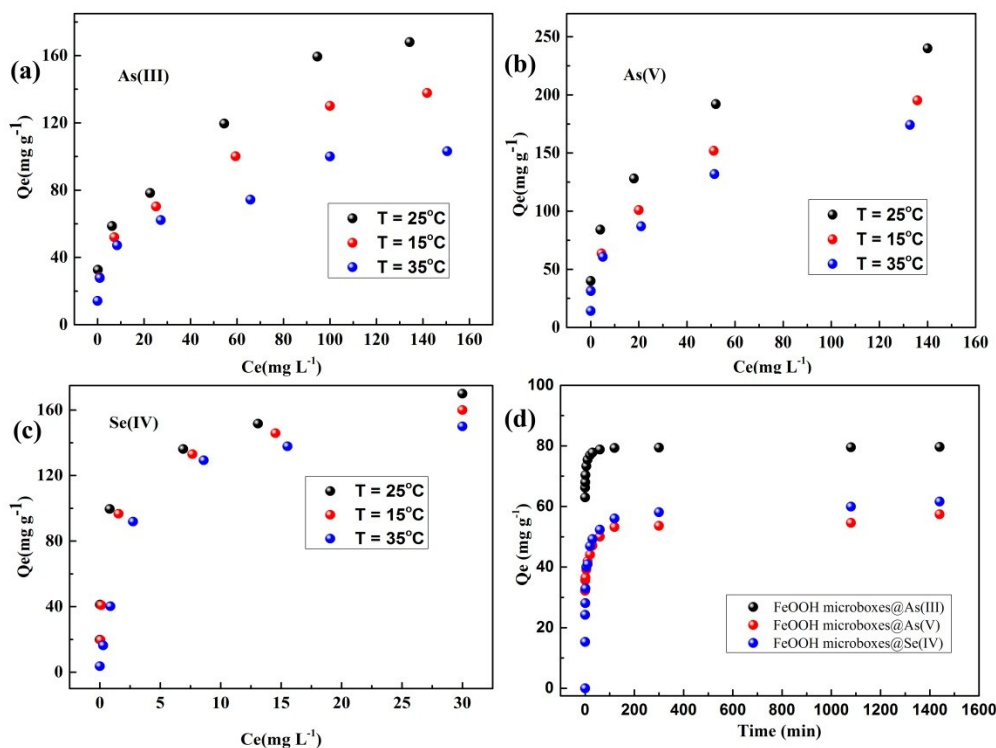
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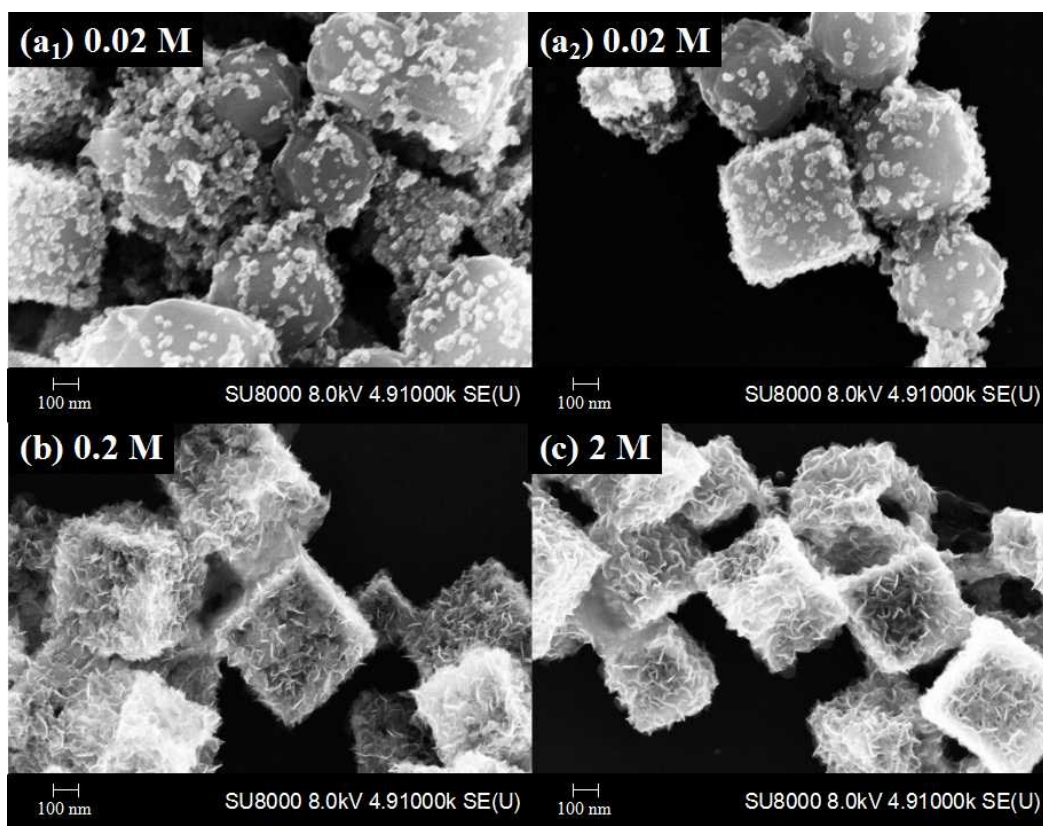
## **Experimental Section**

*Surface Hydroxyl groups analysis:* Boehm titration method was used to measure the surface hydroxyl groups of both traditional FeOOH and the as-prepared FeOOH hollow microboxes. Firstly, both these two adsorbents were dispersed in deionized water respectively to make a turbid liquid, and the initial pH were kept at 10 using 0.1 M NaOH standard solution. Then, 0.1 M HCl standard solution was used to regulate the pH value, and the final pH value was changed to 4 in this experiment. The volume of HCl standard solution was the indication of the surface hydroxyl groups in the adsorbents.

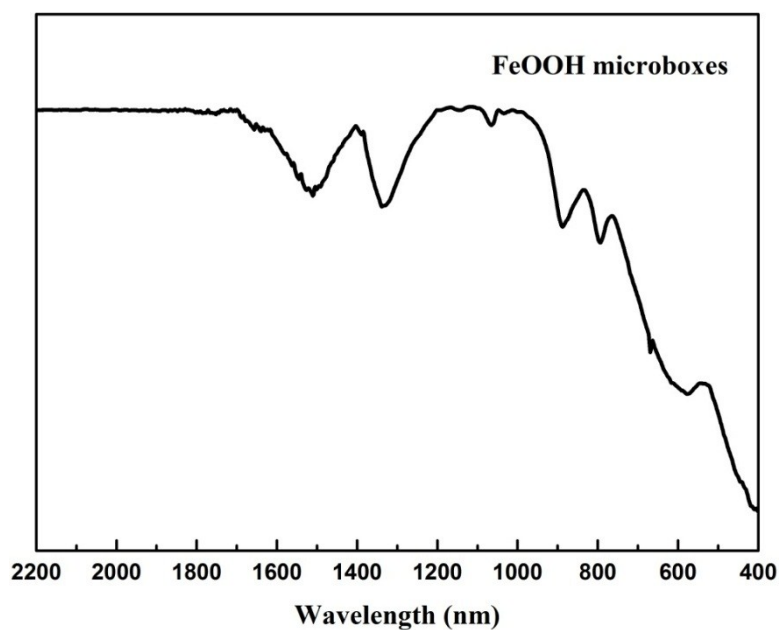
*Adsorption/Desorption experiments:* The adsorption/desorption experiments were conducted as follows. 40 mL As (III), As (V) and Se (IV) containing solutions with a concentration of 20 mg L<sup>-1</sup> and 10 mg of FeOOH microboxes were stirred for 24 h at pH=7, 4, 5, respectively. The heavy metal-loaded adsorbents were separated from the solution and washed by using DI water. It was subsequently immersed into 40 mL of 0.1 mol L<sup>-1</sup> NaOH to neutralize hydrogen ions adhered on the adsorbent surface and mechanically stirred at 80 rpm for 24 h. The regenerated adsorbents were used in the subsequent five-cycle adsorption/desorption experiments.



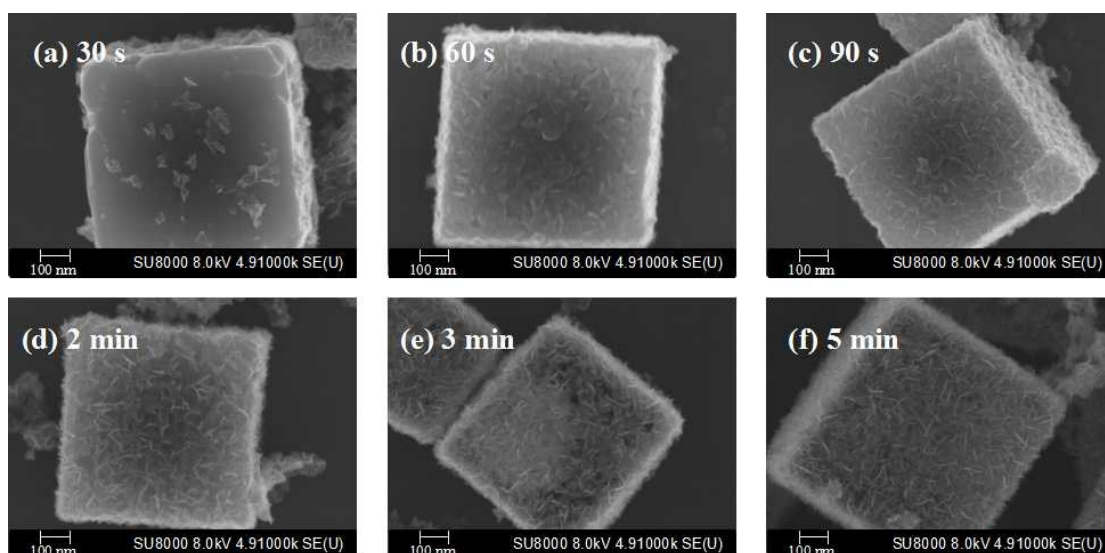
**Figure S1.** Adsorption isotherm and kinetics of As (III), As (V) and Se (IV) adsorbed on FeOOH microboxes with a dosage of 250 mg L<sup>-1</sup>: (a) adsorption isotherm of As (III) with the initial concentration ranges from 5 to 200 mg L<sup>-1</sup>; (b) adsorption isotherm of As (V) with the initial concentration ranges from 10 to 200 mg L<sup>-1</sup>; (c) adsorption isotherm of Se (IV) with the initial concentration ranges from 1 to 100 mg L<sup>-1</sup> at different temperature and (d) removal efficiencies of As (III), As (V) and Se (IV) with initial concentration of 20 mg L<sup>-1</sup> at optimal condition. These data clearly showed that FeOOH microboxes performed better at room temperature (25±1°C), which can be set as the optimal condition. And after reacting for about 24 h, all these three heavy metals were reached to the adsorption equilibrium, with the initial concentration of 20 mg L<sup>-1</sup>, respectively. Moreover, the adsorption performance of FeOOH microboxes to As (III), As (V) and Se (IV) were better than that of the traditional ones for the lower equilibrium concentration, respectively.



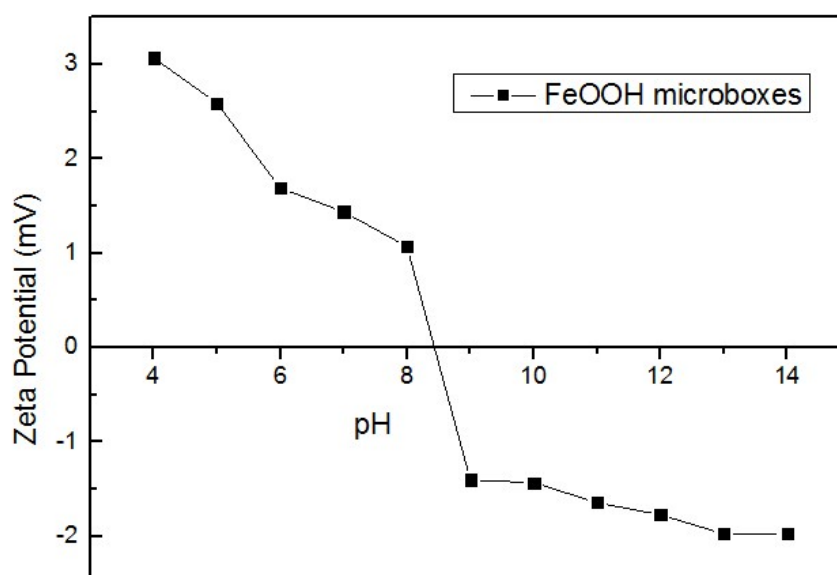
**Figure S2.** SEM images of the changes in morphology of FeOOH microboxes with different reactant concentrations. According to the images above, the PB cannot be etched totally with a low NaOH concentration. The final product, FeOOH microboxes, can be synthesized under a higher NaOH concentration of 0.2 M.



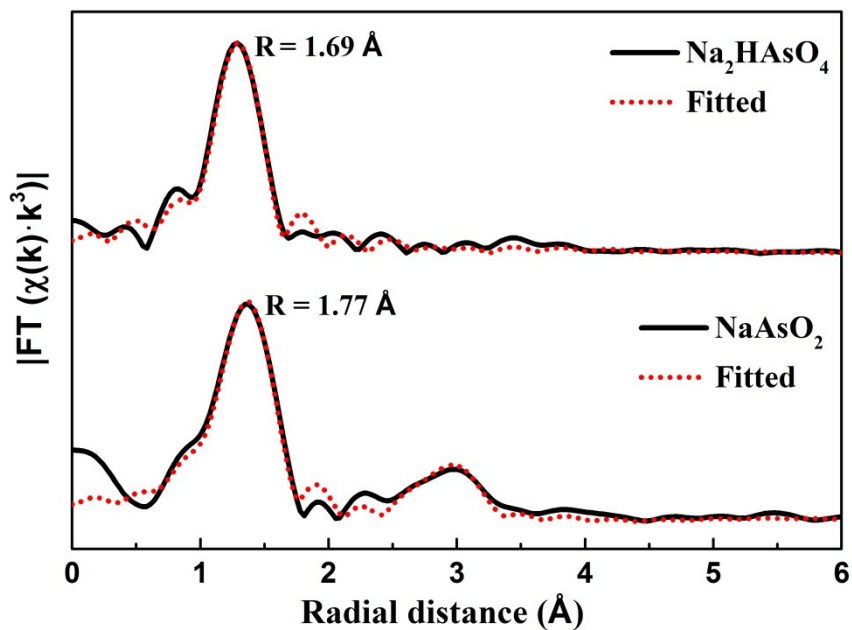
**Figure S3.** Infrared spectroscopy results of FeOOH microboxes.



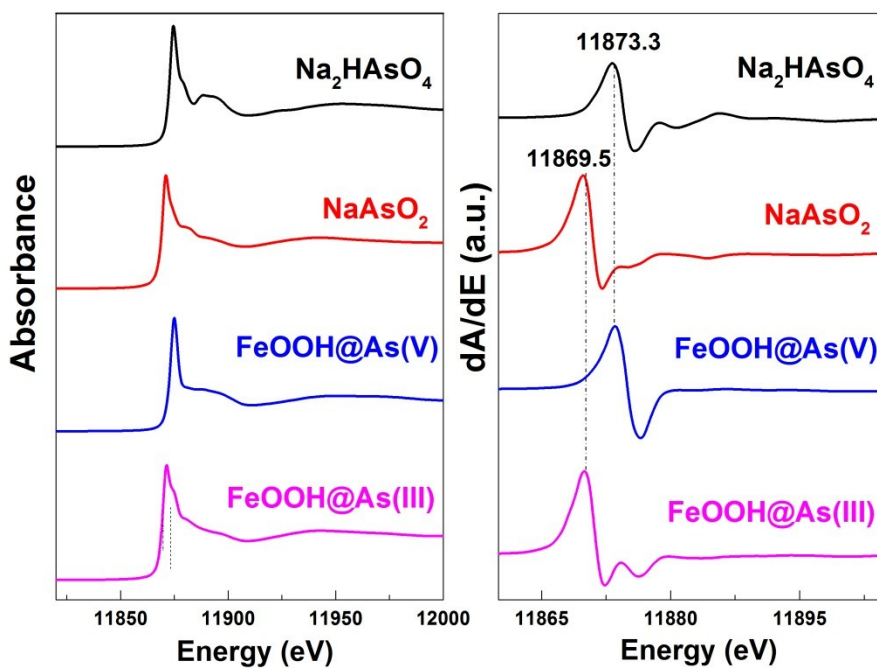
**Figure S4.** SEM images of the changes in morphology of FeOOH microboxes with time variation. Firstly, the outer surface of PB was reacted with NaOH and the interior retained the original shape to be as the template. As the reacting time extended, the solid interior was decreasing and more twisted FeOOH nanosheets were accumulating. Finally, FeOOH microboxes contained by numerous twisted nanosheets were synthesized.



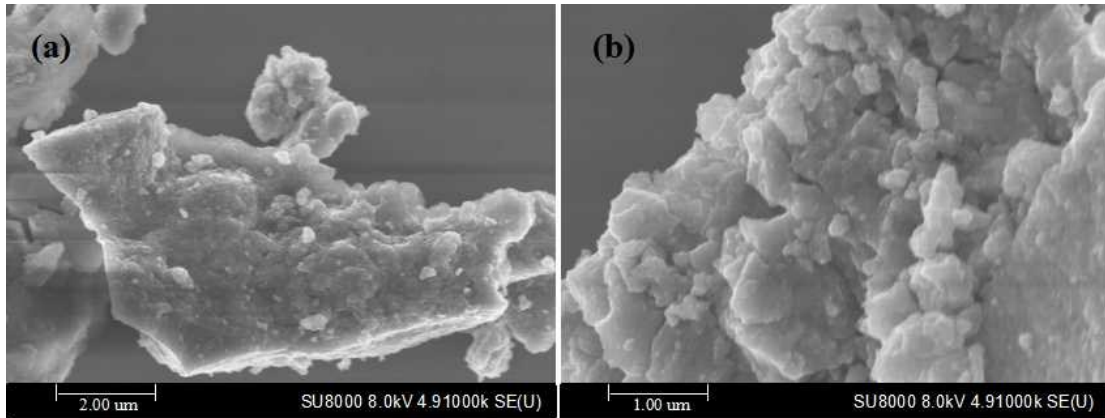
**Figure S5.** Zeta potential of the synthesized FeOOH microboxes, where the FeOOH microboxes are positive when the pH



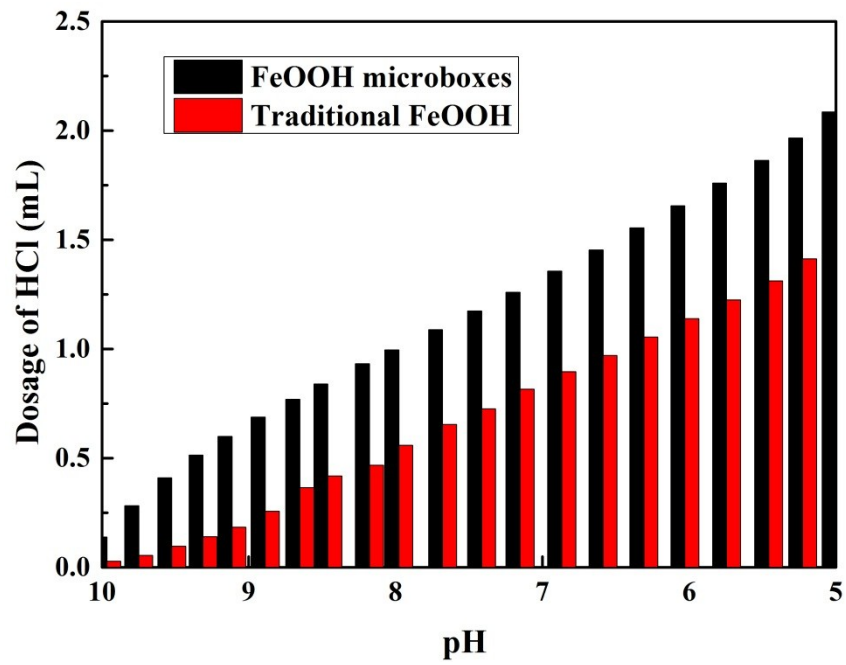
**Figure S6.** EXAFS spectra of As K edge in As-containing reference samples of Fourier transforms of  $k^3\chi(k)$  into R space, where the red dotted lines correspond to the curve-fitting results.



**Figure S7.** Normalized XANES and first-order derivatives of XANES of As K edge in As-containing and reference samples



**Figure S8.** SEM images of traditional FeOOH.



**Figure S9.** Boehm titration results for both FeOOH microboxes and traditional FeOOH.