Supporting Information

Microwave-assisted preparation of sepiolite-supported magnetite nanoparticles and its removal ability to low concentration Cr(VI)

Sheng-Hui Yu¹⁾, Han-Li¹⁾, Qi-Zhi Yao²⁾, Sheng-Quan Fu³⁾, Gen-Tao Zhou¹⁾*

 ¹ CAS Key Laboratory of Crust-Mantle Materials and Environments, School of Earth and Space Sciences, University of Science and Technology of China, Hefei 230026, P. R. China.
² School of Chemistry and Materials Science, University of Science and Technology of China, Hefei 230026, P. R. China.

³ Hefei National Laboratory for Physical Sciences at Microscale, University of Science and Technology of China, Hefei 230026, P. R. China.

> Corresponding author: Prof. Dr. Gen-Tao Zhou Tel.: 86 551 63600533 Fax: 86 551 63600533 Email: gtzhou@ustc.edu.cn



Figure S1. XRD (a), Fe XPS spectrum (b), SEM (c), and TEM (d) of magnetite prepared by microwave-assisted method without sepiolite.

Figure S1a shows that all reflection peaks can be well assigned to a spinel structure with the characteristic reflections of magnetite (JCPDS: 79-0419) or maghemite (JCPDS: 39-1346). No diffraction peaks from hematite or other impurities can be detected in the XRD pattern. The diffraction peaks of our product are slightly broadened, implying that the prepared product may consist of nanocrystals. Estimated in terms of Scherrer equation,⁵¹ the average grain size is ca. 12 nm based on the measurement of the full width at half-maximum (fwhm) of the (311) peak ($2\theta = 35.5^{\circ}$). XPS analysis confirms that the black magnetic precipitate is magnetite (Figure S1b), as the photoelectron peaks at 710.5 and 724 eV match well with the characteristic doublet of Fe $2p_{3/2}$ and $2p_{1/2}$ core-level spectra of Fe₃O₄.⁵⁴ The magnetite is nanoscale, and obvious agglomerates could be observed from SEM image of the obtained magnetite product (Figure S1c). The aggregation phenomenon of the nanoparticles was also observed in the TEM image, and the average size of magnetite nanoparticles is ca. 10 nm, which is nearly consistent with the calculation according to the Scherrer equation (Figure S1d).



Figure S2. TEM images of sepiolite-supported magnetite nanoparticles prepared by oil method for 20 min (a) and 1 h (b).



Figure S3. Relationship between the Cr(VI) removal efficiency and final pH at initial Cr(VI) concentration 1.0 mg/L and adsorbent concentration 1.0 g/L.



Figure S4. Typical TEM image of SSM-3

Reproducibility study on the microwave-assisted synthesis processes

In order to testify the reliability of microwave-assisted method applied in the synthesis of unsupported and sepiolite-supported magnetite nanoparticles, another two sets of preparation experiments with the same conditions as the standard run was performed, and the results are shown below. Both SEM images (Figure S5 a and c) and XRD patterns (Figure S5 b and d) of the samples obtained without the support of sepiolite are similar with the result exhibited in Figure S1, indicating that the synthesis of magnetite nanoparticles by the microwave-assisted method can be easily repeated. After the introduction of sepiolite, the SEM images (Figure S5 e and f) also unequivocally reveal that the magnetite nanoparticles are dispersed on the surface of sepiolite with less aggregation, which is consistent with the result described in Figure 1. All the repeated experiments show good consistence, further confirming that the synthesis of the sepiolite-supported and unsupported magnetite nanoparticles by this microwave-assisted method is reliable and reproducible.





Figure S5. SEM images (a, c) and corresponding XRD pattern (b, d) for unsupported magnetite nanoparticles; SEM images (e, f) for sepiolite-supported magnetite nanoparticles.