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Spectroscopic Study on Heavy Metals Stabilization Using *In Situ* Iron oxide Synthesis: Effect of Repeated Synthesis on Stabilization

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In a spatially limited contaminated site, *in situ* synthesis of iron oxide would be an option for soil heavy metals stabilization. In this study, ferrihydrite was synthesized for heavy metals stabilization, and the behavior of immobilization of Cd, Zn, and As was investigated. Cd, Zn, and As were surface adsorbed on the first synthesized ferrihydrite 69±0.97%, 38±1.6%, and 23±0.96%, respectively. The remaining metals were adsorbed into the ferrihydrite structure (i.e., incorporation). Repeating an application of the synthesizing solution resulted in reduced surface adsorption of Cd and Zn, with rates of 54±2.6% and 23±1.5%, respectively, while As adsorption remained at 23±0.54%. Encapsulation of the surface adsorbed metals occurred due to particle growth pre-synthesized ferrihydrite by agglomeration on the surface. However, encapsulation of surface adsorbed As was not observed, probably due to its inhibitory effect on ferrihydrite agglomeration. The incorporation of heavy metals was determined by measuring the difference in ferrihydrite interplaner lattice space (*d*-spacing) using X-ray diffraction (XRD). The differences were 0.002~0.035 nm for Cd, -0.031~-0.017 nm for Zn, and 0.017~0.029 nm for As compared to pure ferrihydrite. Spherical aberration-corrected transmission electron microscopy (Cs-TEM) integrated with energy dispersive X-ray spectroscopy (EDS) line scanning technique, and Cd and Zn K-edge extended X-ray absorption fine structure spectroscopy (EXAFS) were used to determine the encapsulation of surface adsorbed Cd and Zn. The Fourier Transform of the EXAFS spectra showed that surface adsorbed Zn on the first synthesized ferrihydrite exhibited octahedral coordination, while tetrahedral coordination similar to that of franklinite ($ZnFe_2O_4$) appeared in the repeatedly synthesized ferrihydrite. In addition, an increased magnitude of a peak in the Fourier Transform in the range of 1.7 to 2 Å, which relates to Cd-Fe, was observed in the repeatedly synthesized ferrihydrite. EDS line-scanning visually observed that Cd and Zn were evenly distributed on the first ferrihydrite, while both fractions were lower at the outer side of the repeatedly synthesized ferrihydrite. The findings suggest that repeated synthesis of ferrihydrite can enhance the stabilization of heavy metals by encapsulating the Cd and Zn previously adsorbed on the ferrihydrite surface synthesized by single application.