

Probing the Rapid Formation Kinetics of Ni-Al LDH Precipitates on γ -Alumina Using QEXAFS

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Formation of mixed metal-Al surface precipitates as layered double hydroxide (LDH) phases is an important pathway of natural attenuation of toxic metals such as Co, Ni, and Zn. However, due to the inability of existing kinetic methods (i.e., *ex situ* batch, stirred-flow systems, and pressure-jump relaxation), the kinetics and mechanisms of precipitate formation are not well understood. In this research, we developed a novel *in-situ* quick scanning extended X-ray absorption fine structure spectroscopy (QEXAFS) technique coupled with a conventional flow-through cell to monitor the real-time adsorption/precipitation of Ni on Al oxide surfaces. With a rapid data collection rate, for the first time we show a rapid transition from surface adsorbed Ni to Ni-Al LDH precipitate formation as short as < 5 minutes. This suggests that surface precipitation could occur as rapidly as surface adsorption. Additionally, time-resolved synchrotron-based X-ray diffraction analysis further confirmed the formation of Ni-Al LDH phases and indicated that mineral surfaces promoted crystallization of LDH precipitates. Based on the *in-situ* real-time QEXAFS study, we excluded Ni hydroxides as an intermediate phase for LDH precipitate formation. This new method developed in this study will help advance the understanding of mineral-water interfacial geochemistry.