248-8 An Investigation On the Formation Mechanism of Zn/Al Layer Double Hydroxide Surface Precipitates On Aluminum Oxide.

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Surface precipitation of transition metals such as nickel (Ni), zinc (Zn), and cobalt (Co) as layered double hydroxide (LDH) phases is an important pathway for toxic metal sequestration and decontamination in soil environment. While the metal surface adsorption/complexation mechanism is well established, the mechanism for precipitate formation is not well understood. Accordingly, we investigated the mechanisms of Zn sorption/precipitation on γ-Al_2O_3 using extended X-ray absorption fine structure (EXAFS) and ^{27}Al solid state nuclear magnetic resonance (NMR) spectroscopies as well as high resolution transmission electron microscopy (HRTEM). Results from EXAFS show LDH surface precipitates form only when Zn concentration larger than 0.4 mM. The LDH surface precipitates were directly observed by HRTEM as separated phases. The identified precipitate area is about 250 nm in length and 8 nm in width, whereas the γ-Al_2O_3 particles show spherical shape and 20 nm in size. Energy dispersed spectroscopy (EDS) clearly show the presence of Al in the LDH precipitates and a Zn:Al mole ratio of around 2:1. More interestingly, at the high resolution (HR) mode, we observed some precipitates are crystalline as a sheet shape and some are amorphous. The ^{27}Al solid state NMR spectroscopy sheds light on the formation of Zn-Al LDH, showing an additional shoulder at $\delta_{Al\text{-}27}$ = + 12.5 ppm in addition to those peaks denoting bulk γ-Al_2O_3. This chemical shift is consistent with octahedral Al environment in Zn-Al LDH material. Also a signal intensity reduction of the peak at $\delta_{Al\text{-}27}$ = + 64 ppm was observed, indicating a transformation of some tetrahedral Al in the bulk γ-Al_2O_3 to octahedral Al in the Zn-Al LDH surface precipitates. These results augment previous findings that mineral dissolution appears to be a key factor in the formation of LDH and phyllosilicate-like precipitates during transition metal sorption to Al oxides and Al-rich clays.

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