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The K-edge of rare earth elements (REE): An EXAFS study of La and Ce sorption on clay minerals

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Ion adsorption deposits, involving clay minerals, are highly sought-after sources of rare earth elements (REE), owing to their relative ease of extraction and enrichment in heavy REE. A critical consideration when investigating these supergene deposits is how the REE are held by the clay minerals. Are the REE held in nanocrystalline phosphates, incorporated into the clay mineral structure, or adsorbed onto clay surfaces as readily extractable outer-sphere complexes? Extended X-ray absorption fine structure (EXAFS) spectroscopy provides a powerful approach to resolving these questions. Due to intensity limitations at high energies, REE are typically investigated at the L-edges. However, the close spacing of lanthanide L-edges complicates structural analysis in natural, compositionally complex samples. Recent advances have demonstrated the utility of the Y K-edge as a proxy for the other REE; however, Y is more similar to the heavy lanthanides (Gd-Lu), thus information on the structural state of light REE (La-Eu) remains less well constrained.

Following recent upgrades to the XMAS beamline at the ESRF, we successfully measured K-edges on the light lanthanides La and Ce, to determine the structural state of the light REE on ion adsorption deposits at multiple localities. Unlike the L-edge, the K-edge offers a broader energy range that is free from interference by neighbouring absorption edges, enabling more robust structural interpretation. Cerium K-edge EXAFS resolves both the oxidation state, as in previous L-edge studies, as well as two atomic shells corresponding to the presence of nanocrystalline cerianite (CeO_2). Preliminary analysis of La K-edge spectra indicates that most samples exhibit a first coordination shell consisting of 11±1.5 oxygen at 2.56±0.01 Å, consistent with REE being adsorbed to the clays as outer-sphere complexes. In contrast, a vermiculite-rich sample from Sokli, Finland, shows a clear second shell, and its first shell is better described by 9±2 oxygen at 2.52±0.01 Å. This difference may suggest the REE are structurally bound within the clays, sorbed as inner-sphere complexes, or associated with other nanocrystalline phases such as phosphate minerals. Ongoing EXAFS fitting is underway to determine the nature of this second shell and further refine first-shell parameters across all samples.