

Abstract

Metal oxalates are salts of very low solubility allowing an easy precipitation of metal ions from the acidic aqueous solutions into crystalline material. Due to this property, oxalates have an important role in the technology of lanthanides and actinides. It is namely the separation of actinides from the spent nuclear fuel that is its industrial application of interest. *4f*-lanthanides are often used as *5f*-actinides surrogates when optimizing or modelling the precipitation process.

A synthetic route for homogeneous precipitation of oxalates based on the thermal decomposition of oxamic acid was developed by our research team. The present work deals with further investigation into the structure and morphology of simple lanthanide oxalates. The synthesis products, developed microcrystals, were studied by means of solid-state analysis (optical microscopy, single crystal X-ray diffraction, X-ray powder diffraction, TGA, SEM, AFM). Among the known, the previously not-specified structures of $\text{Er}_2(\text{C}_2\text{O}_4)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Yb}_2(\text{C}_2\text{O}_4)_3 \cdot 6\text{H}_2\text{O}$ were obtained and refined. According to our observations, the synthesized oxalates form two isostructural series: decahydrates and hexahydrates. Structurally, both types of Ln oxalates form 2D frameworks that are stacked together in one direction and connected by hydrogen bonds to form a 3D supramolecular structure. Each lanthanide atom within decahydrate (resp. hexahydrate) oxalate is coordinated by three bidentate oxalates, three (resp. two) water molecules and weakly by two (resp. one) water molecules residing in a cavity. The cavities observed in the structure increase in size with decreasing atomic number of lanthanoid and are of great interest for its possible applications.