EGU22-7905
https://doi.org/10.5194/egusphere-egu22-7905
EGU General Assembly 2022

The influence of the synthesis procedure on the morphology of REE-enriched Pb-apatite (pyromorphite)

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Synthetic REE-enriched Pb-apatites are potentially important materials in the industry. The size and morphology of the crystals can influence the physical properties, and therefore affect technological processes. The conditions of the synthesis can determine the size and morphology of the crystals. Lead apatite $Pb_5(PO_4)_3Cl$ (analogue of mineral pyromorphite) was chosen for this study because the morphology of its crystals shows particular sensitivity to changes in synthesis conditions or solution composition. The addition of REE elements was used because there are reports that the morphology of synthetic Ca-apatite crystals depends on the presence of REE elements. Therefore, in the present study, synthesis by solution mixing at room temperature was carried out and the change in morphology of the precipitated pyromorphite crystals was observed as a function of solution chemistry (presence or absence of La or Sm) and concentration, pH, and mixing parameters. Powder X-ray diffraction (XRPD) was used to identify the phase composition of precipitates, scanning electron microscopy (SEM) to examine the morphology of the crystals, and energy-dispersive X-ray spectroscopy (EDS) for analysis of the elemental composition of analyzed crystals.

XRPD results showed that pyromorphite was identified in all samples. No changes in the crystalline structure were observed (hexagonal system, $P6_3/m$ space group, typical for apatites). Also, EDS analyses showed that the chemical composition remained unchanged despite the morphological differences and the studied REEs (La or Sm) were incorporated into the structure in similar amounts in all precipitates. SEM images indicated that both the size and morphology of the pyromorphite crystals were sensitive to small modifications of the synthesis conditions. The size ranged from 2 μm up to 500 μm. Stirring resulted in smaller crystals than precipitation in the still water column. Crystals appeared in the form of long hexagonal needles (both single and cross-twinned), or slightly rounded, elongated and spear-like rods, or flower-like forms and intergrowths. The presence of REE caused elongation parallel to crystallographic c axis and formation of long needles compare to stubby hexagonal rods in the control sample.

The variation in size and morphology of Pb-apatites synthesized by the precipitation in aqueous solutions in different conditions were reported for the first time. Further research is needed to explain the contributing factors.

Slight changes in the synthesis protocol strongly affect the size and shape of Pb-apatite crystals. Therefore, determining the optimal conditions for the synthesis of homogeneous and well-formed crystals could be of great importance in the potential future applications of these materials.

This research was funded by NCN research grant no. 2019/35/B/ST10/03379.