XRD analysis of Doped Monoclinic LaPO4 (Monazite)
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Monazite has unique properties when doped with divalent or rare-earth elements; rare-earth doped LaPO4 is luminescent\(^1\), Sr\(^{2+}\) doped LaPO4 is an intermediate temperature proton conductor\(^2\). LaPO4 can also accommodate a wide range of nuclear fission products. Solution routes to form monazite usually result in the hexagonal hydrate, rhabdophane (LaPO4•0.5H2O), but we have developed a direct precipitation route to monoclinic LaPO4\(^3\).

Monoclinic LaPO4 was synthesized via the direct precipitation route at different temperatures. Change in particle morphology and size are observed as a function of lowering temperature. The phase transition to rhabdophane was observed at 90-100 °C. This is due to incorporation of H2O in the system since the synthesis is adding La and Sr nitride aqueous solution drop wise into heated phosphoric acid. In order to eliminate H2O from the system, the acid temperature needs to be above 100 °C. X-ray diffraction peaks of rhabdophane are observed among monazite peaks at 90-100 °C (Fig. 1).

The direct precipitation route was found to be successful for incorporation of dopants of rare-earth or Sr\(^{2+}\). Sr\(^{3+}\) or Eu\(^{3+}\) single doped LaPO4 were prepared with nominal dopant ratio of 10, 20 mol% for Sr\(^{2+}\) and 5 mol% for Eu\(^{3+}\). The dopant ratio were determined by Rietveld refinement. The result of refinement for Sr\(^{2+}\) 10 mol% corresponded to the nominal dopant concentration of 10 mol%, whereas refinement was not at all consistent for Sr\(^{2+}\) 20 mol%, possible due to (OH)\(^-\) in the crystal. The 5 mol% for Eu\(^{3+}\) refinement indicated the dopant ratio of Eu\(^{3+}\) is 2%, which implies Eu\(^{3+}\) ions in the solution were only partially incorporated. The complete solid solubility of Sr in LaPO4 via this precipitation route was also investigated. Ideally, were Sr to have complete solid solubility, SrHPO4 might be expected to form, however, Sr was found to precipitate as Sr(H2PO4)\(_2\) (Fig. 2). The solid solubility limit is currently under investigation.

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