Evaluation of Fluorapatite as a Waste-Form Material: Third Quarter Report, March 1- May 31, 2004

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Third Quarter Report 2004

Evaluation of Fluorapatite as a Waste-Form Material

03/01/04 – 05/31/04

Personnel

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Project Description

Fluorapatite, fluorinated calcium phosphate, has been identified as a potential matrix for the entombment of the zirconium fluoride fission product waste stream from the proposed FLEX process. If the efficacy of fluorapatite-based waste-storage can be demonstrated, then new and potentially more-efficient options for handling and separating high-level wastes, based on fluoride-salt extraction, will become feasible. This proposal will develop a dual-path research project to develop a process to fabricate a synthetic fluorapatite waste form for the ZrF₄ FP waste stream, characterize the waste form, examine its performance under environmental conditions, and correlate the behavior of the waste form with natural analogs. Characterization of the material will be accomplished through probing the molecular-scale electronic and geometric structure of the materials in order to relate them to macroscopic properties, with the goal of developing techniques to evaluate and predict the performance of different waste-form materials. Time and funding permitting, other waste forms for the zirconium fluoride, fission product salt waste stream will be examined and benchmarked against the fluorapatite matrix baseline.

Highlights of Accomplishments

1. Synthesized Sr-containing hydroxyapatite using the reflux method and acquired SEM, EDS and XPS data for these samples.

2. More samples of Sr-containing hydroxyapatite synthesized using various lengths of refluxing time and stirring to check the thermodynamic effects on the crystalline character/shape of the samples.

3. First samples of fluorapatite and Sr-containing fluorapatite synthesized and analyzed with IR spectroscopy.

4. Synthesized Zn-containing hydroxyapatite using the reflux method and obtained IR data.

Technical progress

In order to load surrogates successfully into the apatite structure it is critical to determine the best method and the right amounts of surrogates necessary. During the last quarter multiple samples of hydroxyapatite containing Sr and Zn have been synthesized as well as the first samples of Sr-containing fluorapatite. Two samples of Sr-containing hydroxyapatite were synthesized using the reflux method. The first sample was refluxed with the ratio 1:2 (hydroxyapatite to strontium chloride) while stirring for 24 hours in aqueous medium. The second sample was refluxed with the ratio 1:2 (hydroxyapatite to strontium nitrate) while stirring for 27 hours in aqueous medium and kept at room temperature for another 20 hours. Then both samples were dried in the vacuum and in a deccicator and then analyzed using SEM and IR spectroscopy. Only the second sample showed changes in both SEM data and in IR spectra which shows two phases one being hydroxyapatite and another phase different from other phases that have been observed before (see circle in figure 1). It looks like an amorphous phase with a chemical composition containing Ca, Sr, P and oxygen but no Cl and no nitrate.

![SEM image of hydroxyapatite synthesized containing strontium nitrate using the reflux method showing two different phases.](image-url)
With the recently purchased chemicals we were able to synthesize fluorapatite powder that was dried in the oven. IR spectra have been taken to compare with natural and commercial available fluorapatite samples. Synthesized Sr-containing fluorapatite with Sr to Ca ratios of 1:4, 2:3 and 3:2 which were heated to 2000 °C in air and then analyzed using SEM micrographs.

**Management Issues**

1. Are you spending according to your proposed schedule? Yes.
2. How are your completion goals tracking with your proposed timeline? Reasonably well.
3. What problems have you encountered? Do you need assistance from the UNLV program management on any of these issues? From the national program? No problems.
4. Has the proposed schedule/timeline changed? No changes.
5. What do you expect to accomplish in the next quarter? Chemical and physical modifications of the baseline materials to include surrogate wastes, followed by spectroscopic and microscopic studies to compare with the pure apatite samples. More measurements using modern x-ray spectroscopy at the ALS.