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## Evaluation of Fluorapatite as a Waste-Form Material: First Quarter Report (2004-05), October 1 - December 31, 2004

Dennis W. Lindle

*University of Nevada, Las Vegas, lindle@unlv.nevada.edu*

Oliver Hemmers

*University of Nevada, Las Vegas, Oliver.Hemmers@unlv.edu*

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# First Quarter Report 2004-5

## Evaluation of Fluorapatite as a Waste-Form Material

10/01/04 – 12/31/04

### Personnel

The current project participants are listed below.

Principal Investigator (PI): Dennis W. Lindle  
Chemistry, UNLV  
4505 Maryland Parkway, Las Vegas, NV 89154-4003  
Phone: (702) 895-4426 Email: lindle@unlv.nevada.edu

Co-Principal Investigator: Oliver A. Hemmers  
Chemistry, UNLV  
4505 Maryland Parkway, Las Vegas, NV 89154-4003  
Phone: (702) 895-2691 Email: hemmers@unlv.nevada.edu

International Collaborators: V.G. Khlopin  
Radium Institute St. Petersburg, Russia

Boris E. Burakov  
KRI Principle Investigator, Head of Mineralogical Group  
Email: burakov@riand.spb.su

Evgeniy B. Anderson  
KRI Principal Co-Investigator

Alexander Rimsky-Korsakov  
KRI Director General

Collaborators (UNLV): Gary S. Cerefice (Research Scientist, Harry Reid Center)  
Phone: (702) 895-2612 Email: Cerefice@unlv.nevada.edu

Chirantha Rodrigo  
Master Student in Chemistry, UNLV  
Email: chirantharod@yahoo.com

Chinthaka Silva  
Master Student in Chemistry, UNLV  
Email: silvag2@unlv.nevada.edu

Collaborators (DOE): Jim Laidler  
Argonne National Laboratory  
Phone: (630) 252-4479 Email: laidler@cmt.anl.gov

## 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  $ZrF_4$ , 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

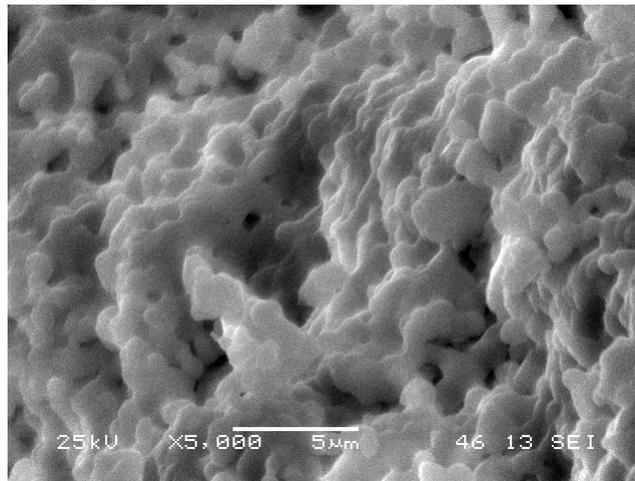
- 1) Multiple samples of hydroxyapatite prepared some with surrogates (varying Ca/Zn weight ratios) and some pure samples which have then been analyzed with IR, TGA/DSC and XRD techniques.
- 2) Fluorapatite samples synthesized using the reflux method and analyzed with SEM, XPS, XRD and single point IR.
- 3) Experiments conducted with natural fluorapatite and hydroxyl apatite using the precipitation method to determine if procedure to synthesize fluorapatite differs based on samples used. XRD was used to analyze the synthesized samples.
- 4) Synthesized yttrium and cesium containing FAP samples using the same method that was used for Strontium (explained in last report) and obtained XRD, SEM and DSC data for samples.

## Technical progress

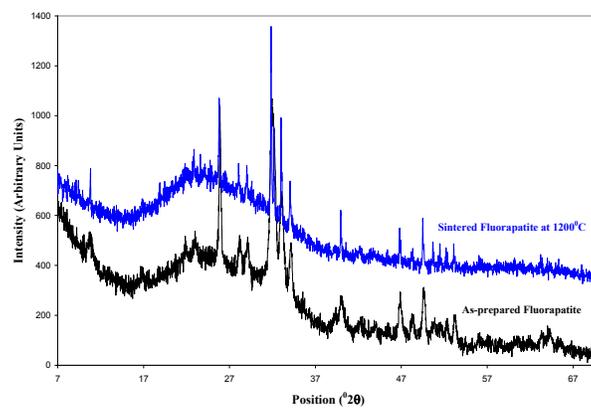
The XANES spectra that were obtained for samples including hydroxyl apatite and natural apatite near the Ca, Sr and oxygen edges at the Advanced Light Source of LBNL at beam line 6.3.1 do not indicate significant changes in the absorption edges. This is an indication that the electronic structure of apatite does not change very much after the insertion of foreign element like Sr. Further investigation is needed based on some more significant changes that were observed in the oxygen K-edge spectra.

Fluorapatite is a naturally occurring mineral with a strong crystal structure and is an abundant mineral around the world. It is desirable to find a way to use natural apatite to synthesize nuclear waste bearing apatite rather than using the more expensive pure chemical reactants to begin with. We therefore tried to find a procedure to re-synthesizing apatite starting with natural apatite. This re-synthesis of apatite using natural apatite as a starting material was partially successful. Even though, our intention was to synthesize fluorapatite we obtained hydroxyl apatite as final product. Further experiments are necessary to refine the procedures and optimize the results.

An important property of fluorapatite is its crystallinity and the resulting tensile strength. Nuclear waste bearing apatite should have a crystalline structure to be more stable. It is therefore of interest to find methods to improve the crystallinity of waste bearing apatites. The figures below show the characteristics of fluorapatite samples that were prepared using wet methods (also called precipitation methods). Figure 1 is a SEM micrograph of a fluorapatite sample after sintering at  $1200^{\circ}C$ . This figure clearly shows that sintering of fluorapatite is creating particles of a more definite size and shape. A not sintered sample of fluorapatite was compared to the sintered sample using XRD data taken from both samples. It turns out that sintering makes the sample more crystalline based on the fact that the peaks of the XRD data shown in figure 2 are narrower for the sintered sample. Similar experiments have been conducted with Zn-bearing fluorapatite with similar results but an overall lower crystallinity.



**Figure 1:** SEM micrograph of sintered fluorapatite at 1200<sup>0</sup>C



**Figure 2.** XRD patterns of fluorapatite before and after sintered at 1200<sup>0</sup>C