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**Article** in *Journal of Radioanalytical and Nuclear Chemistry* · November 1995

DOI: 10.1007/BF02164050

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## URANIUM LEACHING FROM PHOSPHATE ROCK

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Received 21 July 1995  
Accepted 4 August 1995

Uranium in phosphate rock was removed by means of alkaline leach solutions. Ammonium carbonate/bicarbonate solution produced a very stable uranyl carbonate compound which was separated by centrifugation. Radiometric analysis showed that about 40% of uranium was solubilized and it can be recuperated. This process could be used before the manufacture of phosphatic fertilizers and the final products would contain smaller uranium quantities.

## INTRODUCTION

Concentrated phosphate rock used in fertilizer plants contains about 130 ppm uranium. When rock is treated with sulfuric acid to produce phosphoric acid, most of the uranium goes into solution. Further manufacture of

fertilizers brings out all the uranium and the products are then contaminated.<sup>1</sup>

During the 1960-70's, considerable research effort was directed towards uranium extraction from phosphoric acid by means of solvent extraction methods, but they were very expensive and produced contamination problems in some steps of the wet-process phosphoric acid. Furthermore, large volumes of phosphate acid produced, disabled its treatment<sup>2</sup>. At present, uranium remains in phosphate fertilizers, its intensive application redistributes the uranium in farming soils<sup>3</sup>. Therefore, the goal of this work was to remove uranium from fertilizers considering environmental protection.

It is well known that hydrometallurgical mining techniques using carbonate solutions are useful to recover uranium from its ores<sup>4,5</sup>. This leaching technique is employed with success in in-situ or in-pile extraction process in uranium rich minerals<sup>6,7</sup>. It was established that carbonate solutions produce a soluble uranyl tri-carbonate complex. It was expected that uranium in phosphate rock was suitable for following the same extraction process, with the advantage of leaching uranium before the wet-process phosphoric acid without further contamination problems during the fertilizers production process.

## EXPERIMENTAL

### Reagents

All reagents were of analytical grade. 1M sodium and potassium carbonate solutions were used as leaching solutions and ammonium carbonate/bicarbonate at different

concentrations. Nuclear grade uranium dioxide was used for standard solutions. Calcium and phosphorus commercial standard solutions were used to prepare stock solutions for atomic absorption and UV/Vis technique.

### Equipment

The phosphate rock minerals were characterized by X-ray diffraction with a Siemens 500 using the  $K\alpha$  line of copper. Calcium in the leaching solution was determined with an atomic absorption spectrometer Perkin-Elmer 2380. UV/Vis spectrophotometry was used together with 1-cm absorption cells to measure the intensity of the phospho-molybdo-vanadate complex formed, as described elsewhere<sup>6</sup>. Neutron activation analysis to determine uranium was performed as follows:

- a - Weighed sample were irradiated for 2 min in a Triga Mark III nuclear reactor at ININ in a neutron flux of  $10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$ .
- b - Hyperpure germanium detector connected to a multi-channel analyzer board for  $\gamma$ -counting was used to detect the radioactivity in the samples.

### Procedure

Leaching experiments were performed as follows. A weighed phosphoric rock sample was placed in a 250 ml glass balloon, then a given volume of leaching solution was added at the desired concentration. After some shaking, the phases were centrifuged at 30000 rpm for 5 min, then uranium, calcium and phosphorus were analyzed in the solid and liquid phases. XRD spectra were performed before and after leaching process.

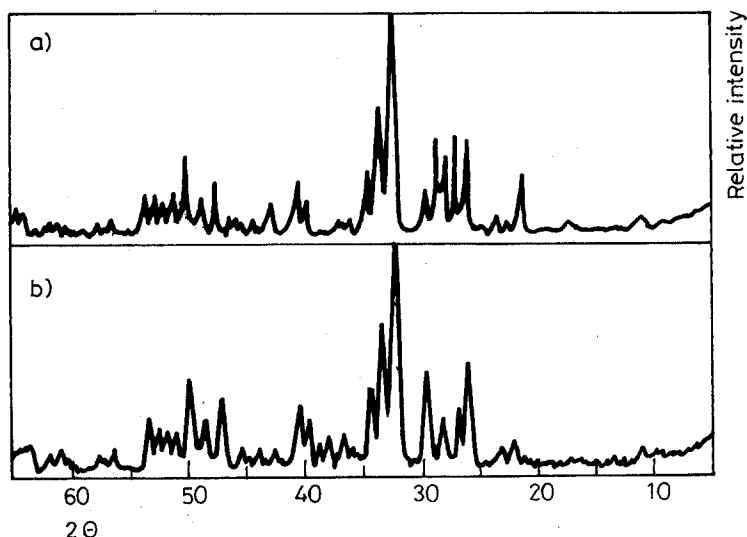


Fig. 1. XRD patterns of concentrated phosphate rock.  
a - Natural sample and b - treated sample

## RESULTS AND DISCUSSION

Figure 1a shows the XRD pattern obtained from direct analysis of concentrated phosphoric rock. The main component of the bulk was francolite (JCPDS card 31-267), and silica was the minor component (JCPDS card 33-1161)<sup>8</sup>.

Leaching experiments showed that, when sodium or potassium carbonate leach solutions were employed to recover uranium from concentrated phosphate rock, the soluble uranyl carbonate complex precipitated because hydroxide ions were formed, increasing the pH of the solution. When an ammonium carbonate/bicarbonate solution was employed, the pH did not increase due to the presence of bicarbonate ions in the solution.

The uranyl tricarbonat complex is very stable and it could be separated easily by centrifugation, uranium,

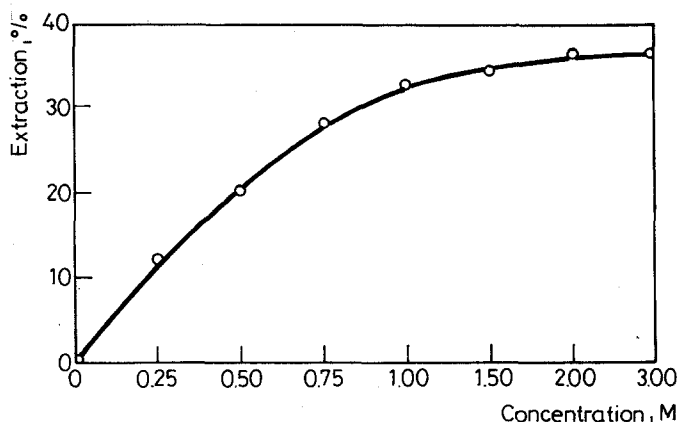


Fig. 2. Uranium extraction behavior with 1M ammonium carbonate/bicarbonate solution

calcium and phosphorus were then analyzed in the liquid phase, and uranium in the solid phase.

To determine the susceptibility of phosphate rock to alkaline leach solutions for the extraction of uranium, the concentration, volume and time of leaching were considered. We have determined the highest limit of uranium extraction with 1M ammonium carbonate/bicarbonate leach solution. Higher concentrations did not improve the rate of extraction of uranium, as shown in Fig. 2.

To evaluate the effect of leaching time, tests were run each hour during 24 h, the optimum leaching time was found to be 6 h. After this time the precipitation of ammonium uranate began to be important and the soluble uranium complex decreased rapidly (Fig. 3).

At last, we established the ratio of leaching solution volume vs. the quantity of phosphate rock. Tests were run with 10 g of concentrated phosphate rock, varying the volume of ammonium carbonate/bicarbonate solution. The maximum yield of extraction was reached when 50 ml of leaching solution was employed. Figure 4 shows that

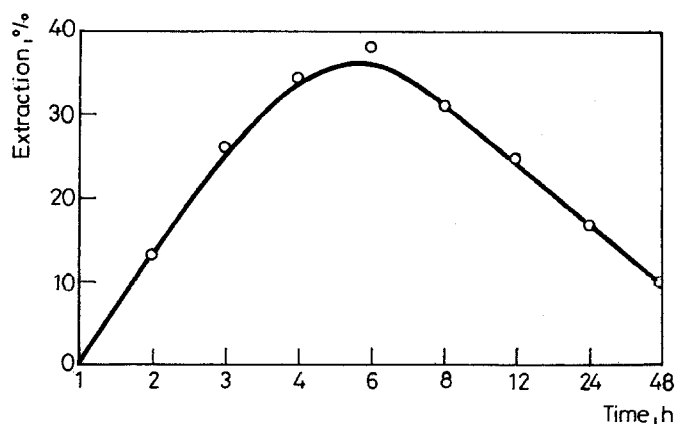


Fig. 3. Uranium extraction behavior at different stirring times

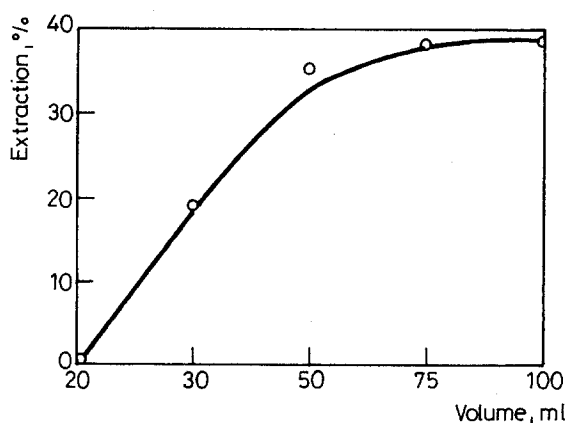


Fig. 4. Uranium extraction behavior at different volumes of ammonium carbonate/bicarbonate solution

despite the addition of leaching solution to the system, the rate of dissolution of uranium was only improved slightly. These data showed that to get maximum extraction of uranium in the phosphate rock, 10 g of phosphate rock need to be mixed with 50 ml of 1M ammo-

nium carbonate/bicarbonate solution and strongly stirred for 6 h. Chemical and spectrographic analysis for calcium and phosphorus in leached solutions showed negligible dissolved quantities (under 0.1%). In the X-ray spectra it was possible to find a very small modification of the basic diffraction peaks of francolite in the treated samples. In these conditions the best extraction rate of uranium was 40% of total uranium in phosphate rock.

## CONCLUSIONS

The data obtained from this study have demonstrated that the ammonium carbonate/bicarbonate leaching process is effective to extract about 40% of uranium from the concentrated phosphate rock.

There are insignificant changes in the chemical composition and crystallographic lattice of francolitic mineral.

This process may prevent corrosion during the phosphate fertilizer production and contamination problems. It is an attractive, simple and inexpensive process to ensure the reduction of radioactive contamination in the final commercial products.

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