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A Complete Remediation Process for a Uranium-Contaminated Site and Application to Other Sites

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Abstract

During the summer of 1996 we were able to test, at the pilot scale, the concept of leaching uranium (U) from contaminated soils. The results of this pilot scale operation showed that the system we previously had developed at the laboratory scale is applicable at the pilot scale. The paper discusses these results, together with laboratory scale results using soil from the Fernald Environmental Management Project (FEMP), Ohio. These FEMP results show how, with suitable adaptations, the process is widely applicable to other sites.

Introduction

The purpose of this paper is to describe results that demonstrate remediation of uranium-contaminated soils may be accomplished through a leach scheme using sodium bicarbonate. The authors present data from a pilot scale system implemented at Los Alamos. Additionally laboratory data is presented using soils from the FEMP site to show how the process can be adapted to different soils.

The concept of remediating uranium-contaminated sites using a leaching mechanism has its roots in the mining industry (Merritt, 1971). Alkaline leach is an established method for mining uranium in areas of high carbonate rocks where acid leach is inappropriate. More recently in-situ mining techniques are currently operating using an alkaline leach scheme (Vogt et al., 1982). The adaptation to in-situ mining reduces the overall costs so U.S. uranium mines become competitive on the world market (HRI, 1997). Heap leaching (unsaturated flow) has successfully produced economic amounts of precious metal from tailing piles heretofore considered uneconomic (Homestake Mining Co., 1991).

There are significant differences between mining and remediation. In mining, economic considerations are paramount whereas for remediation, regulatory drivers of the permissible levels of uranium left in the soil, determined by risk calculations, dominate the procedures. Never the less, the two procedures have much in common as they are both governed by the same chemical process. This reaction is the formation of the soluble uranyl tricarbonate complex when insoluble uranium species in the VI oxidation state are contacted by soluble carbonate:

\[ \text{UO}_2^{2+} + 3 \text{HCO}_3^- \rightarrow \text{UO}_2(\text{CO}_3)_3^{4+} + 3 \text{H}^+ \]
Since uranium is one of the few commonly occurring metals which forms soluble carbonate species, this reaction is specific for uranium and therefore does not result in large-scale soil degradation.

The efficiency of the leaching process is influenced by the mineralogy and oxidation state of uranium as well as the soil properties. An additional significant factor is the degree of homogeneity of the contamination. Cocontaminants are also a major concern but are not addressed here.

In mining operations most uranium is found in the IV oxidation state usually as uraninite (uranium dioxide), \( \text{UO}_2 \), or as coffinite (uranium silicate), \( \text{USiO}_4 \). Natural occurring oxidized forms will, for the most part, have migrated away from uranium bearing minerals due to the solubility of the \( \text{UO}_2^{2+} \) in alkaline ground waters. Thus oxidative pretreatment of the U(IV) species is essential in mining and may be necessary in remediation. This is accomplished using oxygen (HRI, 1997), peroxide (Vogt et al., 1982, Mason et al., 1997) or other strong oxidizers. Whether minor forms of IV need to be oxidized depends on the clean-up criteria determined by risk calculations. Soil structure varies significantly from site to site. The FEMP site consists of about 80% clays and silts whereas Los Alamos has typical sandy soil with 80% sands and gravel. For soils high in clays and silts, in order to prevent swelling of clays and retarded hydrologic flow, sand additions are found to be effective. A method often used in the mining industry is agglomeration with water or cement to improve its structural integrity and so prevent retarded flow. Results on these two methods for enhancing flow are given here.

Finally, remedial actions may be assisted by pretreatment before a leach step. In general physical treatments are less expensive than chemical treatment. For solid contamination, as at firing sites, a size separation will be effective at reducing the volume of contaminated soil. At firing sites, such as at Yuma Proving Ground (Mason et al., 1996) and Los Alamos, large pieces of penetrators are found to be still be intact. Additionally a radioactive separation (segregating the soil by radioactivity) will further reduce the volume of soil to be leached. Two commercial systems are available for such separations: The Segmented Gate System (SGS) (ThermoNutech, 1996) which uses gamma detectors and the Alpha Separator (MacArthur et al., 1998) using alpha
detectors. Separations based on size and radioactivity will be ineffective for liquid spills due to the more uniform uranium distribution.

Finally, once the uranium is in solution, it may be concentrated by evaporation, precipitation and by immobilization on an ion exchange resin. Resins have been used both in the mining industry (Vogt et al., 1982) and also by us (Lu et al., 1997). Whether immobilization is followed by resin stripping or by long term storage of the resin depends on a cost comparison between stripping and storage.

This paper is divided into two sections:

Pilot scale study at a Los Alamos National Laboratory (LANL) Site, and

Laboratory scale studies using high clay soils from FEMP.

Pilot Scale Study

The site chosen, containing of 218 cubic yards of contaminated soil, consisted of two catch boxes filled with earth which had received natural uranium slugs fired into them from 1948 to 1955 as part of the nuclear weapons experimental program. Although other contaminants were at one time present in these catch boxes, they were not found during initial characterization. The uranium slugs were at various stages of oxidation and disintegration - those closest to the earth surface had few recognizable characteristics whereas towards the back of the boxes, intact and partially intact slugs were found. The predominant form of weathered contamination consisted of yellow fragments varying in size from about 5cm diameter downwards and identified by x-ray diffraction analysis as schoepite, UO$_3$·2H$_2$O.

A three-step process was chosen for volume reduction for this site:

1. physical separation based on levels of radioactive contamination and incorporating an initial size separation, followed by
2. chemical separation using a leach scheme employing sodium bicarbonate (0.5M) as the leaching agent in an unsaturated flow scheme, and
3. ion exchange process for removing soluble uranium from solution.
Risk calculations revealed that for this site, acceptable risk was obtained for soil containing < 4218 Bq/kg (114 pCi/g) of radioactivity assuming a recreational scenario for a trail hiker (Turney et al., 1997).

Results

Physical Separation for radioactive segregation and accompanying size segregation.

Figure 1. shows a schematic of the system deployed at the pilot site. The system used, the Segmented Gate System (SGS), is commercially available (ThermoNUtech, 1996). The soil was moved by a front end loader and placed onto a moving belt after pretreatment, both by a hammermill to break up large pieces of rock and by a jig to remove large pieces of debris (> 1.9 cm). The jig removed several partially intact penetrators and functioned as an initial size separator. The moving belt passed under a bank of sodium iodide detectors which detects gamma emissions > 3700 Bq/Kg (100 pCi/g), scanning in the range of 30 to 200 KeV including gamma emissions from the daughter of U-238, and thorium-234. These readings were fed to a computer that automatically diverted soil contaminated above a certain preset level (>100 pCi/g) by opening gates. The contaminated soil is passed through screens for a secondary size separation and fed into bags for the second stage leach scheme. Clean soil was returned to the site.

Figure 2. presents the results of the soil separation after radioactive segregation.

Chemical Separation

Radioactive soils were contained in one cubic yard bags (total of nine) hung from a metal frame and suspended over a catch basin. A drip irrigation system placed on the top surface distributed a 0.5M sodium bicarbonate solution (previous bench scale results had shown that 0.5M sodium bicarbonate gave optimal leach results (Mason et al., 1997)). The leach solution passed through the soil, dissolving uranium to form the uranyltricarbonate species, \( \text{UO}_2(\text{CO}_3)_{3}^{4-} \). A schematic of the process is shown in Figure 3.

No other metal ions are dissolved to any great extent by sodium bicarbonate (Mason, 1997). A pump transferred the leach solution to the top of the vat after passing through three filters: a filter (10µm) at the exit tube from the leach bags, a 10µm filter and finally a 1 µm filter. An ion exchange resin system was added to remove the soluble uranium prior to recirculation.
The filters and sediment (that collected at the base of the catch basin) were found to contain significant quantities of uranium-238 and also, only in the case of the exit filter, some natural thorium (thorium-232). The results for Bag 3 are shown in Table I.

The post-leached soil was analyzed for residual U and these were compared to the preleached soil analyses (Mason et al., 1998). The results are summarized in Figure 4. After leaching was terminated, the soil in one bag (bag 3) was leached with water to remove any soluble uranium species still with the soil. We found that ~ 37% of the residual uranium was removed. Also for bag 3, nine cores were taken (center, corners and center of sides). The core (7.6 cm diameter) was divided into five sections. These were composited and analyzed for total U (U(IV) + U(VI)) and U (VI). The residual uranium (IV) was found to vary between 100-800Bq/kg (2.7- 21.6pCi/g). The total residual uranium (IV and VI) in bag 3 is 3.33 x 10^3 Bq/kg (90pCi/g) thus the contribution from uranium IV varies between 3% and 24%. This emphasizes the extreme heterogeneity of the distribution of uranium and the importance of sieving the soil prior to leaching and the small contribution to total uranium the leached soil after processing.

**Ion Exchange Resins for Uptake of Soluble Uranium.**

Ion exchange resins are used in the mining industry to concentrate uranium. (Vogt et al., 1982) Using leach liquors from Crownpoint, New Mexico and the anion exchange resin Dowex 21K not only was uranyl (UO$_2$(CO$_3$)$_4$) anion exchanged onto the resin but also the molybdate (MoO$_4^{2-}$) anion.

In our studies (Lu et al., 1997) we used Ionac™ A641 which is a macroporous strong-base anion exchange resin with the trimethylamine, [R-N-((CH$_3$)$_3$)Cl], as the functional group attached to a styrene divinyl benzene copolymer. During the pretreatment the chloride form becomes the bicarbonate form: [R-N-((CH$_3$)$_3$)HCO$_3$]. When U-bicarbonate liquors, (UO$_2$(CO$_3$)$_4$), were introduced into the columns, the exchange reaction occurs resulting in the formation of resin-uranyl carbonate complexes [(R-N-((CH$_3$)$_3$)$_4$UO$_2$(CO$_3$)]. Each uranyl carbonate requires 4 resin exchange sites. In addition, significant competition between anionic SiO$_4^{2-}$ and uranyl carbonate anions results in loss of capacity for exchanging with uranium anions, in a similar way that loss in capacity resulted from the presence of molybdate in the mining study. No molybdenum was
Remediation of a Uranium-Contaminated Site

present in our leach solutions. This competition with silicate was also seen in the bench scale tests. Improvements are expected to remove 99% or better of the U from solution up from the 95% achieved in the initial scale-up.

**Laboratory scale studies using high clay soils from FEMP.**

**Results**

Small-scale laboratory column experiments on the leachability of uranium from contaminated soils from FEMP, Ohio have previously been reported (Mason et al., 1997). Column lengths of up to 12 ft were used. Proof-of principle was established.

Differences between FEMP soil and LANL soil are summarized in Table II. We have previously reported results (Mason et al., 1997) where in-situ oxidation of U(IV), using sodium peroxide, in the FEMP soils results in a 10-15% increase in leachable uranium. This is roughly equivalent to the amount of U(IV) uranium present in FEMP soils.

One difference between the two soils is the organic content. This phenomenon was illustrated during a pretreatment of FEMP soil consisting of a size segregation that resulted in significant decrease in uranium. The results are shown in Table III. Two different samples of the same soil were received from FEMP. Although they were from the same area, one had undergone sieving through a 4-mm sieve prior to shipment in order to remove organic debris (such as twigs). These results show that significant fraction of the uranium is associated with the organic matter.

The difference in clay content and resultant difference in porosity make concerns about hydrologic conductivity a major issue. For the FEMP soils, representative of soils high in clays, our investigations centered on treatments to make the soil more amenable to leaching. These treatments include agglomeration with water and with cement and also sand addition to large-scale columns.

Agglomeration is a process used in the mining industry whereby the structural integrity of the medium is enhanced by tumbling with an agglomerating agent, in this case water or cement. The tumbling results in small soil pellets that are then placed in columns. Figure 4. shows the results of column studies using soil agglomerated with water and agglomerated with cement, otherwise under identical conditions (9kg soil, 150 cm length, 10 cm diameter columns),
application rate of leach solution = 0.012mL/min/cm². The leach solution first exits from the base of the column after ~ 20 h. The exit time from the column base was found to be linear with column length suggesting flow is not inhibited within the column ranges studied.

Figure 5. shows the results using soil from FEMP amended with sand (50% and 25%) in large column experiments (210 kg soil + sand, 365 cm (12 ft) length, 25 cm diameter). It is clear that, even for columns at this height, sand is effective at preventing retarded flow although it also did not accelerate the initial flow through the column. Over the 15 days duration of the experiment, no impedance was seen.

Analyses

The concentrations of U in the effluent were analyzed using a Varian Liberty 200 inductively coupled plasma atomic emission mass spectrophotometer (ICP-AES) or a kinetic phosphorescence analyzer (KPA). U in soil samples were analyzed in the same way following pretreatment either with sodium bicarbonate (dissolve U(VI)), nitric acid (0.5N) (dissolve total U) or deionized water (to remove dissolved U).

Gamma spectroscopy was used for determination of U in solid and liquid samples. The gamma counters utilized are generally high-purity germanium detectors with resolution of ~ 1-2 KeV FWHM at 1000 KeV. The counters are energy and efficiency calibrated using multi-line NIST traceable standards. Two computer analysis codes, RAYGUN and SPECANL analyze the counts generated. The data provided for the determination of U-235 was based on the 185.7 KeV emissions of U-235.

X-ray diffraction (XRD) analysis on the solid mineral phases was used to identify the mineralogical speciation of U using a computer controlled INEL CPSS-120X-Ray Powder Diffractometer

Discussion

Pilot Scale Results

The SGS for radioactive separation reduced the volume of contaminated soil from 218 cubic yards to 6.5 cubic yards—a 97% reduction in volume. The leach system further reduced the contaminant level in the nine bags of soil by an overall amount of 89%—six of the nine bags
having residual concentrations of less than the free release limits. Of the remaining three, the values were 2%, 17%, and 41% over the release limit. These were due to large pieces of schoepite in the bags and the rate determining surface reaction of the solution with the solid. It is also possible that some channeling took place but we have no direct evidence. Later in the leach sequence sieves were added so the soil was sieved again immediately before entering the leach bag removing any remaining large pieces of contaminant. In subsequent remedial actions such sieving would be an integral part of the system.

Both the filters and sludge on the bottom of the catch basin became very radioactive, mainly with uranium. The volumes are small so these filters and sludge can be placed in low-level radioactive storage sites. The uranium is sorbed so strongly to the smallest clays (<10μm) that uranium is not readily leached. We also found that there is a slow migration of the smallest size soil constituents to the base of the leach bags.

Resins are considered the preferred way to immobilize uranium from solution. Evaporation can only accomplish a limited increase in concentration and is dependent on external conditions. Precipitation reactions, although effective, are regulatory discouraged because of the very low levels of uranium allowed for free release of the residual solution. Resins have been shown to be effective at immobilizing uranium.

We have shown that, for the LANL site, sorting and leaching techniques are capable of remediating a contaminated site.

It can be argued that the Los Alamos site is ideal for a leach scheme (oxidized contaminant form, low organic content, high porosity, and heterogeneous contamination). Thus we have also worked on a site (FEMP) where the opposite is true (partially reduced form, higher organic content, low porosity, and more homogeneous distribution.)

Laboratory scale studies using high clay soils from FEMP.

Uranium (IV) has been shown, both in mining and remediation, to be oxidized in-situ readily. Our own pilot scale studies show that very little uranium (IV) is present when uranium metal is oxidized in an oxidizing rich environment. This suggests the rate determining step is oxidation to IV and followed by rapid oxidation to VI.
Uranium ions can bind to humus by surface complex formation through carboxylic acids, phenolic and alcoholic hydroxy groups and amino functional groups. For the FEMP soils, the organic fraction is removed before treatment with any aqueous agent so that the soluble fractions of humic and fulvic acid complexes will also be removed. The early FEMP soil showed dissolution of organic compounds as seen by the dark coloration of the leach solutions. In the FEMP soils, the organic fraction complexes with ~ 38% of the total uranium (Tables II and III).

Low porosity of the FEMP soils lead to the experimental work on agglomeration and tall columns. Both experiments were designed to stop impeded transport of leach solution through the soil because of clays that may lead to plugged columns. Figure 4. shows that there is little time difference in the establishment of equilibrium (leach solution in = leach solution out) with agglomeration with water and cement. Slightly better uranium removal is seen fiom the water agglomeration due to there being better surface contact in the absence of cement. In neither case did the columns plug.

Since it was expected that 356 cm (12 ft) columns with no soil additions would plug, the two experiments were done using 50% sand and 25% sand, reducing the 80 % clay concentration to 53% and 64%. As can be seen no plugging was found over 7 days. We can conclude that there is no interrupted flow for clay content > 53%. The limit may be significantly less than this.

Extension to Other Sites

We have performed some initial studies on several other uranium-contaminated sites (Kitten et al., 1995). We are confident that we can remediate them using techniques designed to address the specific requirements of the site. Thus a radioactive sort is suitable for sites with heterogeneous contamination and not appropriate for homogeneous contamination. The leach system will act as a final step in some cases. This latter is particularly important when dealing with highly contaminated sites, such as transuranic wastes.

We have also completed leaching studies using different leaching agents for the remediation of plutonium and americium contaminated soils (Lu et al., 1998).

Costs.
Remediation of a Uranium-Contaminated Site

A detailed cost analysis has been made and published elsewhere (Cummings and Booth, 1996). In summary, the larger the volume of contaminated soil, the better the cost benefit for a leach scheme coupled with a radioactive presort compared with conventional dig and haul. The system has the potential of saving in excess of $45 million for remediation of each 100,000 cubic yards of radioactively contaminated soil, a cost savings of approximately 75% over conventional dig and haul methods, providing on-site disposal is not an option. The greater the initial heterogeneity of the soil the greater the efficiency of the radionuclide sort.

Conclusions

An array of different techniques is available for remediating actinide-contaminated soils. These can be tailored to specific sites, each site having its own unique characteristics and clean-up requirements.

Acknowledgments

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References


Remediation of a Uranium-Contaminated Site


Table I.
Radioactivity on Filters and Sludge for Leach Bag 3.

<table>
<thead>
<tr>
<th>Filter type or sludge</th>
<th>Weight</th>
<th>Activity Bq/kg x 10^5</th>
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<tr>
<td>Bag exit filter</td>
<td>57 g</td>
<td>3.3</td>
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<tr>
<td>sludge</td>
<td>385 g</td>
<td>14.4</td>
</tr>
<tr>
<td>10(\mu)m filter</td>
<td>245 g</td>
<td>9.1</td>
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<tr>
<td>1(\mu)m filter</td>
<td>136 g</td>
<td>7.2</td>
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</table>
Table II.
Comparison of Pilot Site (LANL) Soil with FEMP Site Soil.

<table>
<thead>
<tr>
<th>Pilot Site</th>
<th>FEMP Site</th>
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</thead>
<tbody>
<tr>
<td>~80% sand and gravel</td>
<td>80% clays and silts</td>
</tr>
<tr>
<td>~100% U(VI)</td>
<td>~85% U(VI)</td>
</tr>
<tr>
<td>schoepite UO$_2$-2H$_2$O</td>
<td>~15% U(IV)</td>
</tr>
<tr>
<td>Traces uraninite UO$_2$</td>
<td>autunite Ca(UO$_2$)$_2$(PO$_4$)$_2$-2H$_2$O</td>
</tr>
<tr>
<td>Oxidation solid uranium metal</td>
<td>uranium UO$_2$</td>
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<tr>
<td>Total organic matter 1%</td>
<td>uranium phosphate U(PO$_4$)$_3$</td>
</tr>
<tr>
<td></td>
<td>Waste process stream, incineration</td>
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<tr>
<td></td>
<td>Total organic matter ~ 5%</td>
</tr>
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<td>(See Table III)</td>
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### Table III.

Effect of Screening FEMP Soil through 4cm Screen.

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<tr>
<th>Soil identifier</th>
<th>Soil pH</th>
<th>SOM %</th>
<th>Gravel %</th>
<th>Sand %</th>
<th>Silt %</th>
<th>Clay %</th>
<th>Total U mg/kg</th>
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<tr>
<td>SP-93 pretreated</td>
<td>7.4</td>
<td>2.3</td>
<td>0.2</td>
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<td>50.8</td>
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Figure 1.
Schematic of Segmented Gate System (SGS)
Remediation of a Uranium-Contaminated Site

Figure 2

Soil Separations Achieved by SGS

Soil Separation Achieved by SGS

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<th>Day</th>
<th>Kg so</th>
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</tbody>
</table>
Figure 3.

Schematic of Leach System
Remediation of a Uranium-Contaminated Site

Figure 4.

Pre- and Post- Leach Concentrations of Uranium

Figure 5
Remediation of a Uranium-Contaminated Site

Figure 5.

Effect of Pretreatment on Rate of U Removal

![Graph showing the effect of pretreatment on the rate of uranium removal over 7 days. The graph plots the percentage of uranium remaining in soil against days. Two lines are shown: one for samples agglomerated with cement and another for samples homogenized with water. The graph indicates a significant decrease in uranium content over time for both treatments.](image-url)
Figure 6. Scale-up Leach Experiment Using FEMP Soil, 12 ft columns

% Uranium Removal

Days after leach solution first exits

- 50% sand
- 25% sand