AN INTEGRATED APPROACH TO THE CHARACTERIZATION AND DECONTAMINATION OF URANIUM CONTAMINATED SOILS

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ABSTRACT

An Integrated Demonstration (ID) Program, hosted by the Fernald Environmental Restoration Management Company, has been established for investigating technologies applicable to the characterization and remediation of soils contaminated with uranium. Chemical and physical characterization of Fernald soils and the uranium wastes contained therein is being accomplished by means of standard analytical techniques as well as a suite of non-standard microscopy and spectroscopy techniques. Likewise, a suite of physical and chemical extraction technologies are being designed and tested for accomplishing soil decontamination. However, the main theme of this paper is not the technologies being tested but the approach taken to integrate characterization, decontamination, and risk assessment efforts. It is our intent to outline the critical components of an integrated approach for characterizing and remediating uranium contaminated soils as well as provide a real-world example based on the lessons learned in the ID program.

INTRODUCTION

One of the major problems facing the U.S. Department of Energy's (DOE) Environmental Restoration Program is the remediation of uranium-contaminated soils. In response to this problem the Office of Technology Development has initiated an Integrated Demonstration program to evaluate and compare the versatility, efficiency, and economics of various technologies that may be combined into systems for the characterization and remediation of uranium contaminated soils. Fernald, the former DOE production complex for processing uranium from natural uranium ore concentrates, was selected as the host site for this program based on its past operating history and known environmental problems.

The primary objective of the Uranium in Soils Integrated Demonstration (USID) is to develop treatment technologies capable of achieving volume reduction in uranium contaminated soils. The purpose of this paper is to outline the approach being taken to meet this objective. The adopted approach calls for close interaction between soil characterization, soil decontamination, and risk assessment efforts. In this paper, specific examples are given, based on the experiences of the USID, concerning:

- utilization of soil characterization information in the screening of alternative soil decontamination technologies,
- remedial design via bench-scale treatability studies, and
- role of risk assessment in soil treatment design and decontamination efforts.

The first step in this approach, soils characterization, follows very closely the *Characterization Protocol for Radioactive Contaminated Soils* published by the Environmental Protection Agency.[1] Because of this similarity, results from the USID represent an important example of the implementation of this protocol.

The paper begins with a brief description of the soils characterization program along with a description of the standard and state-of-the-art analytical techniques being employed. This is followed by the results of initial site characterization efforts. Attention is then turned to the soil decontamination program where a basic outline of the program is given as well as a

description of the technologies being tested. Results of preliminary bench-scale treatability studies are also provided. Finally, an overview of the risk assessment program is given.

GEOCHEMICAL CHARACTERIZATION OF SOILS

The USID program has established a soil geochemical characterization program[2] to acquire information necessary for selecting and designing soil decontamination technologies and for performing risk assessment calculations. As such, the general approach and methods employed by the characterization program are tailored to the needs of the data users. A suite of standard analytical techniques coupled with state-of-the-art molecular spectroscopy and microscopy techniques have been assembled to address the following data needs:

- bulk physical and chemical soil characteristics;
- nature of uranium partitioning within the soil;
- physical characteristics of uranium wastes (particulate, sorbed, coating);
- uranium chemistry (oxidation state, speciation); and
- migration potential and toxicity of the uranium.

A two phase-approach has been adopted for the soil characterization program. In the first phase, soils from across the site are collected and analyzed. The acquired data are used to screen alternative soil treatment technologies and for baseline design and risk assessment calculations. Phase II of the program focuses on optimization of selected soil treatment technologies. This involves the characterization of soils both before and after bench scale treatability studies. Comparison of the results will help identify those uranium phases for which the treatment is effective as well as those phases unaffected by the treatment. Other important information includes the effects of treatment on the soil matrix, characteristics of the secondary wastes, and whether repartitioning of the uranium occurs.

Methods

One of the critical aspects of the characterization program is the strong integration and synergism of the various analytical techniques employed. It is clear from the list of data needs that no single technique is capable of providing the depth and breadth of information required. To meet these needs we have brought together conventional and advanced methods capable of microscopic to macroscopic scale investigation. Based on the findings of the USID, this integrated approach is crucial for any characterization effort focused on soils with uranium contamination.

Soils are first subjected to a suite of standard analytical techniques to determine the bulk characteristics of the soil and the nature of the uranium partitioning within the soil.[3] In particular pH, organic content, and cation exchange capacity are measured. Uranium content of the bulk soils is then determined using either gamma spectroscopy, chemical dissolution and inductively coupled plasma-mass spectroscopy, or neutron activation analysis. Next, particle size and particle density separations are performed. These size and density fractions are then analyzed for their uranium content (by the same methods as listed above) to discern the partitioning of uranium among these fractions. Mineralogy of the soils is also determined by means of x-ray diffraction and scanning electron microscopy (SEM).

Techniques capable of probing the molecular structure of the soils are available to better predict the chemical behavior of uranium in soils. The bulk chemical characteristics of the uranium (i.e., oxidation state and speciation) in soils are analyzed using a combination of spectroscopic techniques.[4] Four basic methods are employed, x-ray absorption spectroscopy, optical luminescence spectroscopy, photoacoustic spectroscopy, and Raman vibrational spectroscopy. These methods are complementary in the information they provide, and span an analyte concentration range from ~ 1 % to ~100 parts-per-trillion and below. Of the four methods, x-ray absorption spectroscopy is the most incisive. It provides oxidation state information [e.g., U(IV) vs U(VI)] and information on the local coordination environment

about the target (uranium) ion. Optical luminescence spectroscopy is a very valuable probe for the speciation of hexavalent uranium moieties (i.e., UO_2^{2+} species) and other emissive species. Photoacoustic spectroscopy is a very useful complement to optical luminescence because species that are not emissive tend to yield excellent photoacoustic spectra. The final method, Raman vibrational spectroscopy, provides molecular structure information explicitly via the number, energy, and intensity of the observed vibrational transitions.

Soils bearing multiple uranium phases (species), as is the case at Fernald, require characterization on a particle by particle basis. Such information is acquired by means of an integrated suite of microscopy technologies;[5] SEM, selected area electron diffraction (SAED), convergent beam electron diffraction (CBED), and x-ray energy dispersive spectroscopy (EDS). SEM with back-scattered electron detection and EDS are used in conjunction to locate uranium particles and establish their compositional character. The resolution of these techniques range from 150μm to 50 nm. The SEM images are also used to determine the nature of the relation between the uranium and the host soil (i.e., sorbed, coating, particulate). By combining compositional information with structural identification, as achieved using SAED to a resolution of 1 μm and CBED down to 20 nm, direct determination of the uranium species present in the soil is possible. Integrating the bulk uranium characteristics as measured by molecular spectroscopy with detailed particle information acquired with the microscopy techniques (which provide positive identification of the uranium phases involved), fine scale uranium phase data is translated to the macroscopic scale to facilitate use in the design of soil treatment strategies.

Results and Discussion

The characterization methods and approach detailed above have been applied as part of the USID. Most of the work to date has been in support of the Phase I objectives, soil characterization. Although Phase II activities have been initiated, results are preliminary and thus are not discussed in detail here. While characterizing the geochemistry of Fernald soils, it was recognized that the wide variety of uranium compounds and chemical processing utilized in the past may give rise to a range of unique uranium waste forms in the environment. To address this potential variability, sampling sites were selected according to the mode of release (i.e., air borne, aqueous, or product spill), which can be closely correlated to an associated source term. Five areas, representative of each release type, as well as mixtures of different release types, were selected for sampling. Two soil samples were collected from each area according to a strategy biased by activity readings obtained from a portable NaI detector. To establish a baseline, two samples of representative soil classes were also collected from off-site and subsequently characterized.

Based on the mineralogical analyses and scanning electron microscopy of ten different samples from five sites, we have determined that the uranium exists predominately in particulate form. The uranium is associated primarily with the sand and silt fractions of the soil, while samples associated with aqueous releases have a significant amount of uranium absorbed to the clay fraction (no appreciable quantities of uranium as a coating were noted). In many cases, the uranium is found to be associated principally with the more dense fractions of the soil components. However, significant variations were also noted in the density fractions of the samples with which the uranium is associated. Because of this variability in particle-size and particle-density distribution, the effectiveness of soil-washing using water as the eluent is expected to be minimal.

Based on x-ray absorption analyses, the uranium examined thus far exists principally (80 - 90 %) in the hexavalent oxidation state. In general, hexavalent uranium has greater solubility than uranium in other oxidation states, thus strong oxidizing agents may not be necessary as part of a chemical remediation scheme. Note, however, that this result implies that the uranium is less soluble than would be a tetravalent uranium species. Therefore, the extent of subsurface migration is expected to be greater for the hexavalent uranium species. In addition, because hexavalent uranium (as uranyl species) has a very small magnetic

moment, remediation based on magnetic separation technologies will be of limited value unless the uranium is associated with some other paramagnetic phase (e.g., iron oxides).

Both microscopic and spectroscopic (luminescence) techniques indicate that much of the particulate uranium exists in discrete, crystalline mineralogical phases, and these different techniques are in agreement with respect to the classes of minerals that are present. In contrast to the dominance of a single oxidation state, there is a wide variability in the speciation of the hexavalent uranium from site to site and in some cases with depth at a given site. Microscopy results further demonstrate that there is considerable mineralogical heterogeneity. Uranium-bearing phases identified to date include; uranium silicates, uranium phosphates (autunites), uranium oxides (UO_{2+x}), calcium uranium oxide, uranium contained within a calcium fluorite phase, and uranium adsorbed onto iron oxides. While the full implications of these macro-scale and micro-scale heterogeneities are not clear as to their influences on remediation strategies, they indicate that a range of chemical treatment technologies or a combination of chemical and physical separation technologies may be required to effect complete remediation.

For additional information on each of the characterization technologies as well as discussion of recent results, see Elles,[6] Allen,[7] and Buck [8] in these proceedings.

SOIL DECONTAMINATION

Approach

The fundamental goal in soil decontamination is the selective extraction/leaching of uranium from soil. The objective is to selectively remove uranium from soil without seriously degrading the soil's physicochemical characteristics or generating waste forms that are difficult to manage and/or dispose. Emphasis in research has been placed on chemical extraction techniques rather than physical extraction techniques. This strategy is being followed because most of the highly contaminated uranium soils are located in the eastern United States (predominately at Fernald and the Oak Ridge Y-12 Plant) and, consequently, these soils characteristically contain high levels of fine-textured materials (i.e., silt and clay) that are not directly amenable to physical separation techniques such as soil washing with water. Chemical extraction techniques being evaluated include traditional uranium extractions that use sulfuric acid and carbonate, as well as some nontraditional extraction techniques that use citric acid and complex organic chelating agents such as naturally occurring microbial siderophores.

Sulfuric acid and carbonate extractions have been used to commercially remove uranium from ore deposits in the uranium mining and milling industry; thus, their effectiveness in the decontamination of uranium from soils is a logical extension. Citrate also forms strong complexes with uranium over a wide pH range; thus, its effectiveness in removing uranium from soils is being investigated. Some naturally occurring microbial metal chelators (siderophores such as enterobactin and desferrioxamine B) have very high binding constants for uranium(IV), a form of uranium characteristically found in many uranium bearing minerals. To further assist in the dissolution of insoluble forms of uranium in soil, soils have been amended with cultures of sulfur and ferrous oxidizing microbes [to enhance dissolution of uranium(IV) forms] or cultures of fungi whose role is to generate mycorrhiza that excrete strong complexers for uranium that can solubilize insoluble uranium(VI) secondary minerals. One physical separation technology (aqueous biphasic extraction) is also being investigated because of its ability to segregate fine particulates, a fundamental requirement of soils containing high levels of silt and clay (i.e., Fernald soils contain >70% by weight <50 μm diameter particles). Aqueous biphasic extraction involves the selective partitioning of either solutes or colloid-size particles between two immiscible aqueous phases. Aqueous mixtures of unlike polymers (e.g., a straight-chain polymer such as polyethylene glycol and highly branched polymer such as dextran) can be used because they are immiscible and form upper and lower phases based on their densities. Mixtures of polyethylene glycol and aqueous solutions of inorganic salts (e.g., sodium carbonate or

sulfate) can also be used and are likely candidates for extraction of uranium. Partitioning to selected phases is determined by the surface chemistry of the particle and preferential wetting by one of the liquid phases.

Decontamination/Characterization Linkages

Characterization of uranium in soils provides invaluable information as to the treatment technology most likely to succeed. The characterization strategy is centered around three major themes. The information obtained from investigations within each of these themes has helped define the course in which current decontamination efforts are directed. In the text that follows, each of these themes are discussed in detail.

Distribution of Uranium within Particle Size/Density Fractions of Soil

While this is the first and fundamental issue that needs to be addressed, in many remediation efforts it is not. Most remedial investigation/feasability studies only address the total concentration of a particular contaminant in soil, which is of little help with respect to identifying a useful remedial technology. The simple distribution of uranium among particle size/density fractions determines the likelihood of soil cleanup by conventional soil washing (beneficial classification with water) or mineral agitation approaches. Initial efforts by Lee et al.[3] indicated that such decontamination efforts would not be productive for the Fernald soils because of the relatively uniform distribution of uranium among the sand, silt, and clay fractions and the lack of significant enrichment of uranium in the heavy density fraction.

Oxidation State of Uranium in Soils

The oxidation state of uranium in soils determines the effectiveness of two important factors governing removal of uranium from soils: (1) the extent to which uranium is complexed into a water-soluble form, preferably an anion form which prevents hydrolysis and subsequent sorption onto soil surfaces, and (2) the potential for the dissolution of uranium from its solid mineralogical phase. The extraction of uranium using carbonate based media is a classic example. Carbonate forms soluble complexes with only the uranyl, uranium(VI) form; thus, uranium must be present in the soil as uranium(VI) or the reduced forms of uranium must be oxidized to make carbonate an effective extract media. Carbonate extraction of uranium in the mining industry is often coupled with amendments of an oxidant ($KMnO_4$, perchlorate, or H_2O_2) to assure the formation of the uranyl carbonate complex.

Direct determination of the oxidation state of uranium in soils is a difficult task. In fact, only recent advances in x-ray absorption techniques have made such determinations a reality. Uranium speciation studies at Los Alamos National Laboratory[4] on soils from Fernald have indicated that 80-90% of the uranium to be in the hexavalent form. Indirect leaching studies with carbonate/$KMnO_4$ also support these conclusions. In fact, statistically significant quantities of uranium could only be leached from the Fernald incinerator soil (approximately 10% more) in the presence of $KMnO_4$, indicating that most of the uranium was in the uranium(VI) form.[9] In the case of another Fernald soil, collected near a storage pad, use of an oxidant had no apparent effect. These studies motivated Brainard et al.[10] and Francis et al.[11] to investigate the role of reductive dissolution in the removal of uranium from these soils. Brainard et al.[10] observed that the addition of 0.1 M concentrations of dithionite to 0.1 M levels of Tiron (1,2-dihydroxy-3,5-benzenedisulfonic acid) increased removal of uranium from the Fernald incinerator soil from approximately 6% to > 83%. Francis et al.[11] observed that treatment of the Fernald incinerator soils with a citrate-carbonate-dithionite (CBD) extractant removed 92% of the uranium as compared to a carbonate and $KMnO_4$ extractant which removed only 48%. The role of dithionite in the extraction process for uranium is not entirely clear. Dithionite (as $Na_2S_2O_4$) in the CBD extractant (bufffered at pH 7.3 by bicarbonate at elevated temperatures, 80-90°C) produces a strong reducing environment and has been widely used in soil science application for the removal of amorphous coatings on crystals of free oxides from soils and sediments.[12] Its principal mechanism is reductive dissolution of ferric oxide minerals to ferrous forms following

complexation of the ferrous ions by citrate into a water soluble phase. Quite likely a similar mechanism is responsible for uranium removal, namely, reductive dissolution of uranium(VI) phase to uranium(IV) and subsequent complexation of uranium(IV) by citrate and Tiron into water soluble forms. In both cases, dithionite with Tiron and the CBD, considerable iron is removed along with uranium, generating considerable quantities of iron as secondary waste.

Identification of Mineralogical Uranium Forms in Soils

Uranium contamination of soils at Fernald resulted as a consequence of liquid and particulate discharges from uranium processing facilities. Particulate forms of uranium varied widely (e.g., metallic, oxides, and fluorides). Liquid forms were generally those present in nitric acid raffinate discharges (principally uranyl nitrate and possibly complexed forms of uranium tributyl phosphate). These uranium compounds accumulate in soil as weathered products of the original particulate forms or as insoluble secondary reaction products as a result of soluble uranium compounds being precipitated as discrete uranium minerals or occluded onto soil minerals as they interact with soluble constituents of soil. Buck et al.[13] have identified a number of uranium-bearing phases in the Fernald soils prior to and after extraction by carbonate-citrate-, and Tiron-based extraction. Uranium-bearing phases identified prior to treatment included uranium absorbed onto iron oxides, uranium silicates, uranium phosphates (autunites), uranium oxides (UO_{2+x}), calcium uranium oxide, and uranium contained within a calcium fluorite phase. Analysis of the Fernald incinerator soil following carbonate extraction revealed that >65% of the residual uranium was in highly insoluble uranium phosphate and calcium uranium phosphate form and ~20% as a uranium oxide form. These uranium phases appear to be present in the treated soils as discrete particles. These data strongly suggest that the best decontamination strategy is not to remove such uranium forms by chemical extraction processes (i.e., using sulfuric acid or reductive dissolution processes that generate large levels of soluble iron in the water-soluble effluent phase), but rather, follow carbonate extraction with a physical separation step such as aqueous biphasic extraction. In this manner, the uranium(VI) forms which appear to be largely associated with or adsorbed onto iron oxides can be removed by carbonate extraction and the uranium(IV) forms which tend to be present in discrete mineral forms that are more resistant to chemical extraction can be removed by the aqueous biphasic extraction. Efforts along these lines are being conducted. For example, samples of the Fernald incinerator soil previously extracted by carbonate/$KMnO_4$ are being sent to Agronne National Laboratory where they can be treated in a columnar counter-current aqueous biphasic extractions.[14]

RISK ASSESSMENT

One important consideration in selecting and designing soil treatment technologies is the cleanup limit which must be attained. To establish a uranium cleanup limit that is protective of human health and the environment, several site-specific parameters must be characterized. Not only are the concentrations and locations of uranium important, but data to support predictions of environmental transport and risk assessments are also required.

Several site-specific parameters that must be quantified to assist in predicting the environmental transport of uranium as well as to support the development of uranium cleanup limits are listed in Table 1. Traditionally, several of these parameters are determined during site characterization approaches; however, slight modifications to the data collection process can produce a more robust data set, which provides a greater degree of technical evidence upon which to base informed decisions. For example, when uranium concentration data are collected, the selection of proper analytical techniques to determine the isotopic composition of uranium in a soil sample provides additional information. These isotopic data are essential to estimate the potential radiological effects encountered when exposed to contaminated soils because some isotopes are more potent than others. In addition, the isotopic data can be used to differentiate between site-related contamination and regional background uranium concentrations.

Table 1. Uranium Characterization Data to Support Site-Specific Risk Assessments

Characterization Data	Data Use	Justification
Concentration of Uranium Isotopes	Dose/Risk Calculations	Accurate estimates of potential adverse health effects
Soil Particle Size Distribution and Associated Uranium Concentration	Exposure Assessment Calculations	Provides uranium concentrations of respirable soil particles for inhalation pathway evaluation
Form of Uranium (Chemical Speciation)	Environmental Transport Modeling	Determines potential for migration in surface/ground water and subsequent ingestion calculations
Fraction of Soluble/Insoluble Uranium in Soil	Environmental Transport Modeling; Exposure Assessment Calculations; Biokinetic Modeling/Toxicity Assessment	Determines potential for migration in surface/groundwater; provides estimates of uranium absorbed by organs and tissues; estimates of adverse effects on organs and tissues

Other parameters that assist in quantitative risk assessment include the soil particle-size distribution and the insoluble/soluble fractions of uranium associated with contaminated soil. There is potential for radiological damage to lung tissue when uranium is inhaled either as a particulate or adsorbed to a soil particle. Determination of the uranium concentration associated with various soil particle sizes is important because particle sizes less than 20 microns carry the greatest inhalation risk.

Likewise, knowledge about the chemical form of uranium and the soluble fraction of uranium in a given soil are key inputs for risk assessment calculations. The solubility of uranium species indicates the potential for migration in surface or ground water. Based on the hydrologic and geologic configuration of the site, contaminated water may subsequently be ingested and cause adverse health effects. The solubility of uranium is also important for human exposures via inhalation and dermal contact. Based on the solubility and specific exposure factors, uranium doses to particular organs can be estimated. These estimates are used as model inputs to predict health risks (i.e., cancer risks and nephrotoxicity).

The final consideration for proper site characterization is establishing of site-specific or regional background concentrations for uranium. Without a knowledge of the background level of uranium in soil, it is not possible to evaluate whether or not a cleanup limit has been achieved.

CONCLUSIONS

The development of efficient and economic soil treatment technologies is important for future remediation of all DOE sites. DOE sites having the potential need for decontamination of uranium from soils include Fernald, Oak Ridge Reservation, Paducah, Pantex, Los Alamos, Richland, Rocky Flats, Portsmouth, and Idaho Falls. These radiologically contaminated sites pose a formidable problem given the heterogeneity of the soils and the complex interactions that can take place between the soils and the contaminant. As such, no single treatment technology will be effective for all soils nor can such technologies be applied without site specific information on the chemical and physical

characteristics of the soil and waste materials. In support of this need the USID has established an approach that integrates the efforts of site characterization, soil decontamination, and risk assessment. Although costs associated with many of the characterization and decontamination strategies described are very high, such costs are still very small in comparison to employing remediation strategies that are ineffective or marginally effective.

In this paper, we have outlined an approach for the characterization and remediation uranium contaminated soils. A number of examples are given in this paper that are based on USID efforts conducted at Fernald. The basic approach calls for site characterization to be conducted early in the remediation process. These data are used in the screening of alternative soil treatment strategies and for performing base-line design and risk assessment calculations. Bench-scale treatability studies are then conducted to evaluate and refine the extraction capabilities of the selected treatment technologies. Treatability studies are supported with both pre- and post-treatment characterization of samples. Comparison of the results helps identify those uranium phases for which the treatment is effective as well as those phases unaffected by the treatment. Utilizing site-specific data, risk assessment calculations are performed to assess the toxicity and migration potential of uranium in both the treated and untreated soils. Such an approach is critical in establishing cleanup criteria which are both realistic and protective of human health and the environment.

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