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Technical Note

Reduction of Radioactive Waste from Remediation of Uranium-Contaminated Soil

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ABSTRACT

Great amounts of solid radioactive waste (second waste) and waste solution are generated from the remediation of uranium-contaminated soil. To reduce these, we investigated washing with a less acidic solution and recycling the waste solution after removal of the dominant elements and uranium. Increasing the pH of the washing solution from 0.5 to 1.5 would be beneficial in terms of economics. A high content of calcium in the waste solution was precipitated by adding sulfuric acid. The second waste can be significantly reduced by using sorption and desorption techniques on ampholyte resin S-950 prior to the precipitation of uranium at pH 3.0.

1. Introduction

Many nuclear facilities will have to be decommissioned or dismantled in the near future. In Korea, great amounts of radioactive soil and concrete waste had been generated from the decommissioning of two research reactors and a uranium-conversion plant. Volume reduction by an appropriate treatment will decrease the amount of waste to be disposed of, resulting in a reduction in the disposal cost and enhanced efficiency of the disposal site [1].

For the remediation of radionuclide-polluted soil, washing with an appropriate reagent is a simple and effective method. Bicarbonate, strong inorganic acids, and weak organic acids (ascorbic, citric) have been used to remove U(VI) from soil under ambient oxidizing conditions [2,3]. In our laboratory, nitric acid is used for the remediation of uranium-contaminated (U-contaminated) soil. However, it is difficult to decontaminate soil to a clearance radioactivity level by washing with nitric acid alone [4]. For greater than 95% remediation of U-contaminated soil, electrokinetic

technology has been developed to be applied after batch-type washing one or two times. In addition, electrokinetic equipment at a practical scale (512 L of soil/batch) was recently manufactured [5,6]. However, a great amount of acidic waste solution has been generated from the washing processes and the operation of the electrokinetic decontamination equipment [7,8]; it has to be reduced in volume or be recycled. To reduce its volume, the evaporation by heating of water in the waste solution requires too much energy. Thus, a proper method for removing uranium from the waste solution in order to recycle it has been studied. Fig. 1 shows the remediation procedure of U-contaminated soil developed in our laboratory.

To precipitate uranium in the first washing solution shown in Fig. 1, the pH of the waste solution was adjusted to neutral or weak alkaline, and alum and magnetite were also added [9,10]. CaO and NaOH have been considered to increase the pH of the waste solution. While NaOH rapidly dissolves and can easily control the pH of a solution, accumulated sodium ions are hard to remove when the solution is recycled. If CaO is substituted for NaOH, calcium ions can be simply removed from the solution since calcium ions form complexes more easily than sodium ions do, although it takes a longer dissolution time to increase the pH of the solution up to 8.0 because of the lower dissolution rate of CaO.

However, the uranium precipitation method by adding CaO or NaOH generates a great amount of solid radioactive waste (second waste), approximately 10% of the initial soil volume, because the dominant metal ions such as iron and aluminum are also precipitated in the neutral or weak alkaline solution. If uranium is selectively removed from the waste solution, the volume of the second waste will be greatly reduced.

To reduce the volume of the second waste from the remediation of U-contaminated soil, this work examined the use of less acidic washing solution, the removal of dominant ions from the second waste, and the extraction of uranium from the waste solution.

2. Materials and methods

2.1. Elemental analysis of solid and solution

The concentration of uranium in a solid was indirectly analyzed using HPGe γ-spectrometry (Canberra, Genie 2000, Meriden, USA) by measuring the radioactivity of ^{234m}Pa (energy 1001 KeV). Uranium-238 undergoes alpha-particle decay to daughter ^{234}Th (half-life = 24.1 days) to reach secular equilibrium in less than 1 year, and ^{234}Th decays to ^{234m}Pa (half-life = 1.17 minutes) by beta-particle emission [11]. Since the soil was contaminated by natural uranium decades ago, the secular equilibrium among ^{238}U, ^{234}Th, and ^{234m}Pa in this study has already been reached. The radioactivity of ^{238}U corresponds to 48% of the total radioactivity for natural uranium, and 1 Bq/g radioactivity of uranium indicates that 40 mg of natural uranium is contained in 1 kg of sample [12]. The elements dissolved in the solution were analyzed using inductively coupled plasma-atomic emission spectroscopy (ICP-AES, JY Ultima-2C, Jobin Yvon, Palaiseau, France).

2.2. Using less acidic washing solution

If a less acidic solution is used in the first and second washing processes shown in Fig. 1, the second waste will be reduced. To identify the difference in decontamination efficiency at a pH range of 0.5–1.5, U-contaminated soil was washed with various pH solutions in the following sequence.

(1) Four solutions were prepared by adding 150 g of U-contaminated soil with 29 Bq/g and 300 g of the washing solution into a 1.0 L Erlenmeyer flask.
(2) The pH of the solutions was adjusted to 0.47, 0.86, 1.16, and 1.49 by adding less than 1 mL of concentrated nitric acid or sodium hydroxide solution.
(3) The solutions were shaken for 10 minutes at 100 rpm.
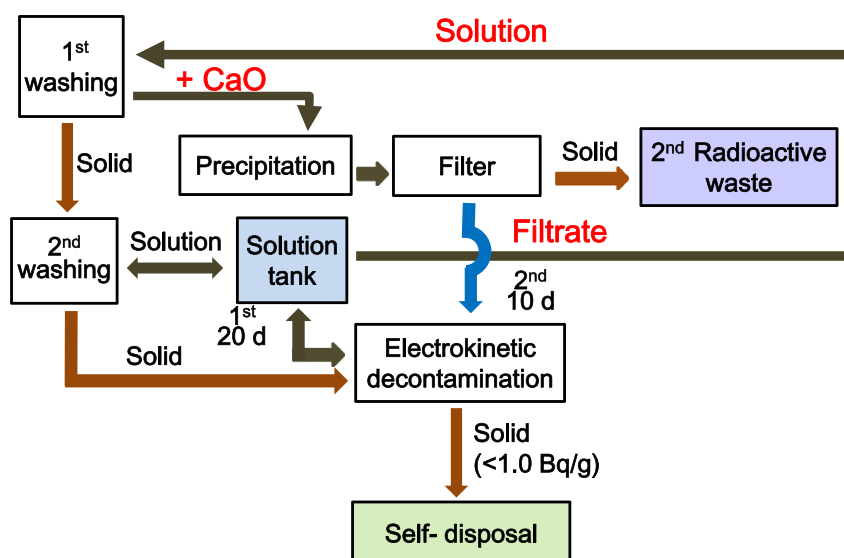(4) The pH of the solutions was readjusted to 0.54, 0.85, 1.06, and 1.58 in order, respectively.

Fig. 1 – A remediation procedure for U-contaminated soil.

(5) After shaking for 20 hours at 100 rpm, the pH of the solutions was measured.
(6) Process (5) was performed again.
(7) The solutions were passed through a Whatman 4 filter paper with a pore size of approximately 20 μm.
(8) The filtered soil was dried for 2 hours at 110 °C, and its radioactivity was measured.

2.3. Removal of dominant elements

2.3.1. Identification of dominant elements in the second waste

When the composition of the second radioactive waste was analyzed, calcium was found to be a major element (Table 1). To discover whether calcium is precipitated with an increasing pH, the concentration of calcium in the various pH waste solutions was measured by the following method.

(1) Waste solution (pH 0.49) was added to four 200-mL plastic bottles.
(2) Small amounts of NaOH were added to three of plastic bottles to prepare various pH solutions.
(3) After shaking for 18 hours at 200 rpm, the pH of the solutions was measured.
(4) The pH of the solutions was measured again after additional shaking for 2 hours at 200 rpm to check the pH variation.
(5) The solutions were filtered with a 0.2-μm-pore filter.
(6) The concentrations of calcium and uranium in the filtrate were analyzed using ICP-AES.

As another experiment, CaO was added until the pH of the washing solution reached 8.1. Then, a portion of the solution was filtered with a 0.2-μm-pore filter and another portion was centrifuged for 10 minutes at 2,000 rpm. The concentrations of calcium and uranium in the filtrates were analyzed.

2.3.2. Removal of calcium from filtrate

When a filtrate was recycled in the electrokinetic equipment in Fig. 1, the high concentration of calcium in the filtrate caused some problems. Thus, the removal of calcium from the filtrate by adding sulfuric acid to form a $CaSO_4$ precipitate was studied as follows:

(1) 5, 10, 20, 40, and 60 mL of 98% sulfuric acid were slowly added to 500 mL of the filtrate;
(2) the solution was left for 6 hours;
(3) after removal of the supernatant, the solution was centrifuged for 10 minutes at 2,500 rpm;

(4) the solid was gathered and dried for 2 hours at 110 °C;
(5) the precipitate was weighed; and
(6) the calcium concentration in the filtrate before and after adding sulfuric acid was measured.

2.4. Removal of uranium from the washing solution

2.4.1. Sorption of uranium by an ampholyte resin

The sorption of uranium from the electrokinetic waste solution (pH = 2.5 at 20 °C) by an ampholyte resin (S-950, Purolite Co., 150 Monument Rd #202, Bala Cynwyd, PA 19004 U.S.A.) with aminodiphosphonic groups was tested [13,14]. Fe, Al, Ca, and Mg were the dominant metals dissolved in the waste solution. Uranium sorbed on the S-950 resin was desorbed by a batch-type method. The detailed procedure for the sorption and desorption of uranium by S-950 is as follows:

(1) The waste solution was filtered with a Whatman 4 filter paper.
(2) Ten grams of S-950 was added to 100 mL of filtrate.
(3) The solution was stirred for 2 hours at 100 rpm, and then left for longer than 12 hours.
(4) The solution was filtered with a Whatman 4 filter paper.
(5) The resin was dried for 24 hours at room temperature.
(6) Among dried resin, each 4 g of the resin was added into two 500-mL Teflon bottles.
(7) A 50-mL volume of 0.5 M and 1.0 M Na_2CO_3 solutions were added to the bottles, respectively.
(8) The bottles were shaken for 5 hours at 60 rpm in a shaker adjusted to 60 °C.
(9) The solution was filtered with a Whatman 4 filter paper.
(10) Thirty-five percent H_2O_2 was added to the filtered 0.5 M Na_2CO_3 solution to adjust to approximately 1.0 M H_2O_2.
(11) To precipitate uranium, the pH of the solution was adjusted to pH 3.0 ± 0.1 by adding nitric acid or sulfuric acid.
(12) After 16 hours, the solutions were filtered with a Whatman 4 filter paper and a 0.20-μm-pore syringe filter.
(13) The uranium concentration in the filtrates sampled from (9) and (12) was analyzed using ICP-AES.

For the regeneration test of the S-950 resin, 2 g of the resin was added into 1 L of the waste solution to saturate its reactive sites, and the resin was then washed with 50 mL of 0.5 M Na_2CO_3 solution using the procedure of (1) to (8) in the experiment above. After filtering, the resin was stirred in 20 mL of 0.1 M NaOH for 10 minutes and washed with a small amount of demineralized water. Finally, the washed resin was shaken in 100 mL of the waste solution for 2 hours. The uranium concentration in the solution was analyzed before and after sorption on the resin.

2.4.2. Sorption of uranium by an anion exchanger resin

Since the adsorption of uranyl sulfate anions by a strong anion exchanger was reported [15], the soil was washed with a 1.0 M sulfuric acid solution instead of nitric acid, and the solution was filtered. Then, a sorption test by IRA 910, a strong anion exchanger, was performed with the filtered solution as follows: (1) IRA 910 resin was activated in the dilute NaOH

Table 1 – The dominant metals in second waste.	
Element	Composition (wt%)
Ca	10.0 ± 0.5
Al	2.5 ± 0.2
Fe	1.8 ± 0.2
Si	2.4 ± 0.2
U	0.20 ± 0.02

Solution	pH					Radioactivity of washed soil (Bq/g)
	Initial adjustment	After shaking for 10 min	Readjustment	After shaking for 20 h	After again shaking for 20 h	
1	0.47	0.95	0.54	0.71	0.82	4.67 ± 0.45
2	0.86	1.17	0.84	0.91	0.92	5.05 ± 0.50
3	1.16	1.40	1.06	1.27	1.24	5.13 ± 0.50
4	1.49	1.51	1.58	1.58	1.43	5.12 ± 0.50

Table 2 − The pH variation in washing solutions and the radioactivity of soil after washing.

solution and washed with demineralized water; (2) 1.0 g of activated IRA 910 resin was put into 50 mL of soil washing sulfuric acid solution; (3) after 2 hours of shaking, the supernatant was collected; and (4) the activity of the solution was measured.

3. Results and discussion

3.1. Washing with less acidic solution

The uranium concentration (0.1−0.5 g/L) in the solution with pH of approximately 0.5 after the first washing shown in Fig. 1 was much less than the solubility (approx. 2×10^{-2} mole/L) of UO_2 at pH 1.5 [16]. Thus, although the soil is washed with a less acidic solution, the washing efficiency will not be significantly decreased. The increase of the pH in the washing solution decreases the dissolution of metals from the soil, and less acid is needed. Moreover, a small amount of CaO or NaOH is required to neutralize the acidic washing solution for the precipitation of uranium, which would finally reduce the second waste.

Table 2 shows a comparison of the decontamination efficiency of the soil at a pH range of 0.5−1.5. The radioactivity of the soil decreased to 4.67, 5.05, 5.13, and 5.12 Bq/g after two 20-hour washings in the solutions with a final pH of 0.82, 0.92, 1.24, and 1.43, respectively. This result shows the trend of higher decontamination efficiency in a more acidic solution. However, the difference in radioactivity in the four washed soils was very

slight compared to the 29 Bq/g of the soil before washing. Therefore, the washing at pH 1.5 will be more advantageous than that at pH 0.5 for reduction of the second waste.

3.2. Removal of a dominant element

One method for the reduction of the second waste is to remove the dominant elements in it. Calcium was found to be a dominant element from the analysis of the second waste (Table 1). The high content of calcium may be due to either the precipitation of calcium with an increasing pH of the solution or the excess addition of CaO for the neutralization of the acidic waste solution. Fig. 2 was obtained from a precipitation test in various pH solutions from 0.49 to 8.0 to find whether the high content of calcium is due to the precipitation of calcium with increasing pH. When the pH of the solutions was measured after additional shaking for 2 hours at (4) in section 2.3, it was maintained without any remarkable variation. The red color of the solutions with pH 1.71 and 3.11 may be caused by iron compounds such as iron hydroxide. When the pH 8 solution was filtered, its color was lost. Although the concentration of calcium was a little decreased at pH 8.0, Fig. 2 indicates that approximately 7 wt% of calcium in the waste solution is not precipitated in the pH range of 0.5−8.0.

To change the pH of the washing solution to 8.1, 3.75 g of CaO was added to 50 mL of the waste solution. If all CaO is dissolved in the solution, the concentration of calcium will increase by approximately 5.4%, and a value of 12.4% was obtained in the centrifuged solution. However, only 2% in the

Filtration
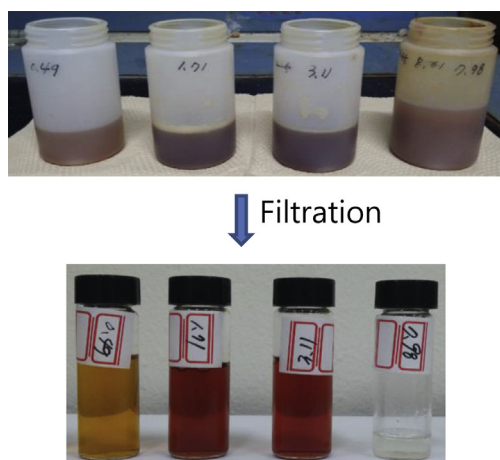
pH	Ca (wt%)	U (mg/L)
0.49	6.9	476
1.7	7.3	485
3.1	7.4	473
8.0	6.0	0

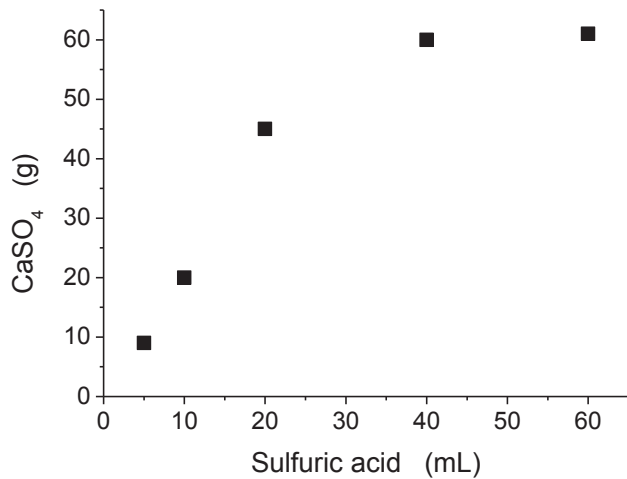Fig. 2 − The color of the solution, and the concentration of calcium and uranium at various pHs waste solution.

Fig. 3 – The amount of CaSO₄ precipitate as a function of the added volume of sulfuric acid: 98% sulfuric acid was added into 500 mL of the filtrate in Fig. 1.

of metallic hydroxides to the surface of the cathode electrode. In addition, the thick layer of metallic hydroxides on the surface of the electrode increases the electrical resistance, and results in an enhancement of the temperature of the electrolyte solution.

For the removal of calcium from the filtrate, sulfuric acid was added to the filtrate since the solubility of $CaSO_4$ in water is low (approx. 0.2 g in 100 mL water at 20 °C [17]). When greater than 40 mL of 98% sulfuric acid was added to 500 mL of filtrate, approximately 60 g of $CaSO_4$ precipitate was generated, as shown in Fig. 3. This means that 3.5% of calcium was removed from the filtrate. The measurement of calcium concentration in the filtrate before and after adding sulfuric acid using ICP-AES also showed a similar reduction of the concentration of calcium from 3.8% to 0.08% after adding 60 mL of sulfuric acid. Therefore, the calcium from the filtrate can be removed by the addition of sulfuric acid. The recycling of a calcium-removed waste solution finally reduces the amount of second waste.

calcium concentration was increased in the solution filtered with a 0.2-μm-pore filter. This result means that a fine Ca compound is precipitated at pH 8.1 when greater than 9 wt% of calcium exists in the solution. From these results, the high content of calcium in the second waste might be due to the excess addition of CaO because of the very slow dissolution of CaO in the waste solution.

After the first washing of soil shown in Fig. 1, the solution was adjusted to near pH 8 by adding CaO in order to precipitate the uranium. The precipitate was filtered using a filter press with a 25 μm pore size, and the filtrate could be recycled. However, when a filtrate with a high concentration of calcium was recycled in the electrokinetic equipment, the low permeability of the filtrate from the anode cell to the cathode cell created several problems, such as a weakening of the fabric tamis, the corrosion of electric wires, and the adhesion

3.3. Sorption of uranium by exchanger resins

If uranium is selectively removed from an acidic waste solution by a resin and desorbed from the resin by a proper reagent, the solution can be reused without an increase of pH to precipitate the uranium, and a very small amount of second waste will be generated. From the sorption experiment of uranium by S-950 in the washing solution, Table 3 was obtained. This table shows that the relative sorption efficiency for uranium is greater than those of other dominant ions in the waste solution. Some 97% of uranium was sorbed on S-950, and 90% of uranium was desorbed from the resin by a batch-type washing with a 0.5 M Na_2CO_3 solution at 60 °C, although the desorption rate by washing with a 1.0 M Na_2CO_3 solution was unexpectedly slightly decreased. When the 0.5 M Na_2CO_3 solution with 1.0 M H_2O_2 was adjusted to pH 3.0 ± 0.1, most of the uranium in the solution was removed, as shown in Table 3. Nitric acid was more effective than sulfuric acid for

Table 3 – The concentration of ions in solution before and after sorption, after desorption by S-950 resin, and after precipitation.

	Concentration (g/L)				
	Fe	Al	Ca	Mg	U
Sorption solution					
Before sorption	0.31 ± 0.03	9.8 ± 1.0	11.7 ± 1.0	2.0 ± 0.2	0.32 ± 0.03
After sorption	0.29 ± 0.03	7.5 ± 0.8	11.5 ± 1.0	2.0 ± 0.2	$9.4 \times 10^{-3} \pm 1 \times 10^{-3}$
Desorption solution					
0.5 M Na₂CO₃ solution					0.22 ± 0.02
1.0 M Na₂CO₃ solution					0.18 ± 0.02
Filtrate[a]					
Nitric acid					
Whatman 4					$5.0 \times 10^{-3} \pm 5 \times 10^{-4}$
0.2 μm					$1.7 \times 10^{-3} \pm 2 \times 10^{-4}$
Sulfuric acid					
Whatman 4					$9.8 \times 10^{-3} \pm 1 \times 10^{-3}$
0.2 μm					$4.3 \times 10^{-3} \pm 5 \times 10^{-4}$

[a] After adding H_2O_2, the 0.5 M Na_2CO_3 solution was adjusted to pH 3.0 ± 0.1 to precipitate uranium by adding nitric acid or sulfuric acid. The solution was filtered with a Whatman 4 filter paper or a 0.20-μm-pore syringe filter.

the precipitation of uranium. Kim et al [18] reported uranium in the 0.5 M Na_2CO_3 solution with H_2O_2 precipitates as $UO_2(O_2)\cdot 4H_2O$ at pH 3–4.

The decrease of only 10% of uranium concentration in 1 L of waste solution in the regeneration test of the S-950 resin indicates that all reactive sites of the resin are saturated. After the desorption of uranium by 0.5 M Na_2CO_3 solution, the resin was washed with 0.1 M NaOH and demineralized water. When the washed resin was added into the waste solution, the concentration of uranium decreased from 320 mg/L to 140 mg/L. This result means that the resin was well regenerated.

Compared with the uranium precipitation method by adding CaO or NaOH, the selective removal of uranium from the waste solution and brief regeneration of the used resin can remarkably reduce the second waste volume.

Rahmati et al [15] reported that uranyl sulfate anion complexes such as $UO_2(SO_4)_2^{2-}$ and $UO_2(SO_4)_3^{4-}$ are formed in 0.02–9.0 M of a sulfuric acid solution, and 70–80% of these complexes were adsorbed by IRA 910, a strong anion exchanger. However, the concentration of uranium in a soil washing solution with 1.0 M sulfuric acid was not significantly changed after the sorption by IRA 910. The little sorption of uranium onto the strong anion exchange resins in 0.1–2.0 M sulfuric acid may be due to the low formation of $UO_2(SO_4)_2^{2-}$ (aq) and $UO_2(SO_4)_3^{4-}$ (aq) [19] and/or the hindrance of sulfate ions [20].

4.   Conclusion

To reduce the volume of the second waste from the remediation of U-contaminated soil, washing with less acidic solution and the removal of dominant ions and uranium from the waste solution have been studied. Although the decontamination efficiency slightly increased with a more acidic solution in a pH range of 0.5–1.5, the increase of pH in the washing solution from 0.5 to 1.5 will be more advantageous owing to the need for less acid and alkaline reagents and the lesser dissolution of metals from the soil. The high content of calcium in the second waste solid might be due to the excess addition of CaO because of its very slow dissolution in the waste solution. Calcium can be removed as $CaSO_4$ from the waste solution by adding sulfuric acid. Uranyl ions in the acidic waste solution were sorbed on S-950, which is an ampholyte resin with a high sorption efficiency for uranium, and desorbed from S-950 by a batch-type washing with a 60 °C heated 0.5 M Na_2CO_3 solution. The uranium ions in a 0.5 M Na_2CO_3 solution were precipitated at pH 3, and the S-950 resin used was regenerated by 0.1 M NaOH. Consequently, the second radioactive waste volume will be significantly reduced by the removal of uranium as a very small amount of precipitate from the acidic waste solution and a brief regeneration of the resin used.

Conflicts of interest

All authors have no conflicts of interest to declare.

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