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Filtration of Actual Savannah River Waste Treated with Permanganate or Monosodium Titanate

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Summary

Testing to support the design development of the Hanford Waste Treatment Plant showed the addition of strontium nitrate and sodium permanganate improved filtration rates for their waste. Simulant crossflow-filtration tests performed by the Savannah River Technology Center (SRTC) in 2001 also showed improved filtration rates upon addition of strontium nitrate plus sodium permanganate or sodium permanganate alone to simulated waste when compared to treatment with monosodium titanate (MST). Because of these results, the DOE-SR, through the Tanks Focus Area, requested SRTC to conduct crossflow filter tests with actual SRS waste to compare filter performance between feeds prepared with MST and sodium permanganate.

The authors conducted crossflow filtration tests using 0.1 and 0.5 μ pore size Mott crossflow filters located in the SRTC Shielded Cells. The tests used a feed solution containing 5.6 M sodium and varying concentrations of Tank 40H sludge. Personnel added either MST or hydrogen peroxide and sodium permanganate to the feed. They processed the treated waste through the Cells Unit Filter (CUF) system in tests that varied the axial velocity and transmembrane pressure. Personnel measured filter flux and decontamination factor during the test.

The demonstrations provide the following conclusions.

- The filter flux from feed prepared with permanganate averaged 10% higher (after accounting for transmembrane pressure) than the flux from feed prepared with MST. The improvement proved less than the 100% increase in flux observed during tests with simulated wastes. However, the permanganate feeds contained (~2X) higher concentrations of insoluble solids than the MST containing feeds.
- The filter flux with a 0.1 μ filter proved 30 – 65% higher than the filter flux with a 0.5 μ filter, on average. Plausible causes for this result include the following.
    - The smaller pore-size may prevent small particles from becoming trapped in the filter pores.
    - The sequence of testing may have preferentially removed small particles from the slurry reducing the amount of particles that could plug pores.
    - The testing with the 0.1 μ pore-size filter lacked the history experienced by the 0.5 μ filter. Previous testing shows that process history influences filter flux. In that earlier testing with simulated waste, bypassing the concentration cycle resulted in as much as 80% gain in filter flux.

Keywords: Filtration, Permanganate, Precipitation

Introduction

The Department of Energy selected caustic side solvent extraction (CSSX) as a preferred cesium removal technology for Savannah River Site waste.[1] As a pretreatment step for the CSSX flowsheet, the incoming salt solution that contains entrained sludge is contacted with MST to adsorb strontium and select actinides. The resulting slurry is filtered to remove the sludge and MST. The filtrate receives further treatment in the solvent extraction system.

Testing performed by SRTC and the University of South Carolina with simulated waste and MST showed relatively low filtration rates of 0.03 – 0.08 gpm/ft^2.[2,3,4,5] Additional testing conducted with actual waste showed similar filtration rates.[6] Testing to design the Hanford Waste Treatment Plant showed the addition of strontium nitrate and sodium permanganate improved filtration rates for their waste.[7] Crossflow filtration tests performed by SRTC in 2001, using simulated wastes, also showed improved filtration rates upon addition of strontium nitrate plus sodium permanganate or sodium permanganate alone to the waste rather than MST.[8] Because of these results, the DOE-SR through the Tanks Focus Area requested SRTC to conduct crossflow filter tests with strontium and permanganate addition rather than MST.[9] In addition, they requested SRTC to conduct limited tests with a 0.1 μ filter.

Experiments

The authors conducted crossflow filtration tests using a 0.1 or 0.5 μ pore size, 3/8" ID Mott crossflow filter located in the SRTC Shielded Cells. This filter is referred to as the cells unit filter (CUF). The feed for the tests consisted of slurry containing SRS High Level Waste supernate and sludge treated with either Aqua Air MST or sodium permanganate and hydrogen peroxide to remove soluble strontium and actinides. The tests used a supernate containing 5.6 M sodium.

Waste Sample Preparation

Table 1 shows the targeted insoluble solids concentration for each test. The initial insoluble solids loading target equaled ~ 1.15 g/L (approximately 0.10 wt %) for the baseline sludge/MST feed. For the permanganate tests, the experimental design maintained the sludge loading the same as the baseline flowsheet (~ 0.6 g/L).[10] The demonstration with more concentrated slurries targeted 0.67 wt % sludge and either 0.62 wt % MST or 0.22 M sodium permanganate (2.1 wt %). These concentrations match the concentrations in the 2001 test.[6] The actual concentrations varied because the supernate density equaled 1.18 g/ml rather than 1.25 g/mL and the current tests added dilution water with the hydrogen peroxide, sodium permanganate, and sludge.

Table 1. Insoluble Solids for Actual Waste Filter Tests

Sludge/MST	$NaMnO_4$
0.051 wt %sludge 0.047 wt % MST	0.051 wt %sludge 0.24 wt % $NaMnO_4$ (0.148 wt % as MnO_2)
0.71 wt % sludge 0.66 wt % MST	0.58 wt % sludge 2.1 wt % $NaMnO_4$ (1.3 wt % as $NaMnO_2$)

The testing used samples of SRS High Level Waste supernate from Tanks 37H, 44F, 26F, and 46F. Personnel mixed the salt solutions to prepare four identical supernate samples. Personnel combined 180 mL of Tank 37H/44F supernate from one source, 85 mL of Tank 37H/44F supernate from a second source, 30 mL of Tank 26F supernate, and 65 mL of Tank 46F supernate. Personnel then added 435 mL of 1.6 M NaOH to adjust the sodium concentration to 5.6 M. To each feed solution, they also added 15 mL of 2.4 x 10^6 dpm/mL ^{238}Pu in nitric acid. Technicians mixed the solutions and measured the final density. They then collected samples from each of four equal volume aliquots, filtered them, and analyzed the filtrate for sodium (by ICPES), anions (by IC and Free OH), plutonium (by PUTTA), uranium (by ICPMS), neptunium (by ICPMS), and strontium-90 (by Eichrom Sr-Spec based extraction and liquid scintillation counting).

Personnel then added 2.6 grams of 18.4 wt % Tank 40H sludge to each solution and mixed the resulting slurries. Following the mixing, technicians collected a sample, filtered it, and analyzed for plutonium, uranium, neptunium, and strontium-90. Each sample mixed for another two days, at which time personnel collected another sample, filtered it, and analyzed the filtrate for plutonium, uranium, neptunium, and strontium-90.

Treatment with MST or Sodium Permanganate

Personnel added 0.55 g MST/liter of solution to the first slurry sample. Following the addition of MST, the feed solution mixed for 24 hours and technicians collected a sample, filtered it, and analyzed for ^{90}Sr and actinides. Personnel transferred the slurry to the filter feed tank and processed it through the crossflow filter. Personnel varied the axial velocity and transmembrane pressure so that this data provides direct comparison with previous filter test data.[2,3,5,6,9] Table 2 shows the operating parameters – axial velocity and transmembrane pressure (TMP) – for each test.

Table 2. Planned Real Waste Test Conditions

Axial Velocity (ft/s)	TMP (psi)
14	41
6	15
14	15
6	41
10	28
10	28

Following completion of the first test, personnel observed a very high axial pressure drop. Personnel stopped the test, and investigated the cause of this high pressure drop. They identified the cause as a plug in the filter tube. They drained the system and removed the filter tube. They found a black object of unknown origin in the tube, which they removed by inserting a rod into the tube and dislodging the object. They then inserted the filter back into the apparatus, and checked the system by circulating dilute (0.01 M) sodium hydroxide. The axial pressure drop equaled 1 – 2 psi, as expected. The technician drained the sodium hydroxide, added the waste slurry to the feed tank, and resumed the test.

Following this filtration test, personnel increased the nominal sludge and MST concentrations to 0.71 wt % and 0.66 wt %, respectively.

As personnel started the next filter test, they observed a leak in the system and some of the solution drained into the catch pan. Personnel repaired the leak. Since some of the feed solution leaked from the equipment, technicians added 300 mL of supernate, which contained approximately 0.6 g/L Tank 40 sludge, from a second slurry to the filter feed tank. Personnel then performed the next filter test.

Personnel prepared the slurry for the second test by taking a feed sample and adding hydrogen peroxide (0.045 moles peroxide/liter feed solution), and sodium permanganate (0.01 moles permanganate/liter feed solution). Following the addition of sludge, permanganate and peroxide, the feed solution mixed for 4 hours. Other researchers – doing work related permanganate treatments concurrently – discovered that the hydrogen peroxide used may have partially decomposed. Because of this uncertainty, the technicians performed a second peroxide addition (0.045 moles peroxide/liter feed solution). They mixed the feed solution for an additional four hours, collected a sample, filtered it, and submitted the filtrate for strontium and actinide analyses.

Results from the plutonium analyses showed no plutonium removal. Personnel added additional hydrogen peroxide (0.045 moles peroxide/liter feed solution) and sodium permanganate (0.01 moles permanganate/liter feed solution). They mixed the solution for four hours, collected a sample, filtered, and submitted for analysis. The technician added the treated waste slurry to the filter feed tank and started filtration. The filtrate had a dark, purple color. Personnel added more hydrogen peroxide (0.045 moles peroxide/liter feed solution) and the filtrate turned clear within a few minutes.

The technician processed the waste through the crossflow filter measuring flux as a function of axial velocity and transmembrane pressure using the test conditions from Table 2.

Following this filtration test, personnel added additional sludge, permanganate, and peroxide to the feed solution to reach cumulative concentrations of 0.58 wt % and 0.22 M, and 0.99 M, respectively. They mixed the waste and processed it through the filter. The filtrate appeared purple. The technician added more peroxide (0.99 moles peroxide/liter feed solution), but the filtrate remained purple. Personnel allowed the slurry to sit over the weekend. Upon starting the filter again approximately 62 hours later, the filtrate had a greenish color, indicating some reduction of permanganate had occurred. Personnel added sodium formate (0.99 moles formate/liter feed solution) to the feed solution. After one hour, the filtrate appeared clear and the filter testing continued.

Upon starting the pump, the flow tube plugged and leaked filtrate from the overflow tube. (See Figure 1 for details.) Apparently the trace residues that dried in the horizontal section over the weekend provided sufficient pressure drop to prevent flow. The trace residue and the slurry that filled the plugged line had a deep purple color, indicating presence of unreduced permanganate. When the technician loosened the fittings and rotated the device slightly counterclockwise, the flow passage cleared.

Following these tests, personnel cleaned the equipment and replaced the 0.5 μ filter with a 0.1 μ filter. The technician processed the slurries containing the elevated MST and the elevated permanganate through the 0.1 μ filter. Personnel did not clean the filter between these tests. They measured filter flux as a function of operating parameters without collecting filtrate samples for analysis.

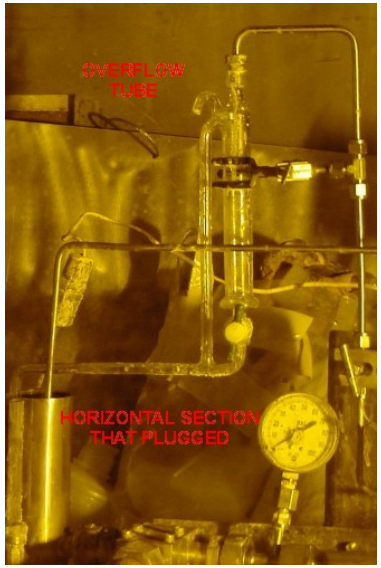

Figure 1. Details of flow tube that plugged during final demonstration with concentrated permanganate slurry.

Results

Feed Characterization

Table 3 shows the characterization of the filtrate from four identical feed slurries for these tests. The plutonium analyses prior to treatment with MST or permanganate show good agreement, indicating that the prepared slurry reached an equilibrated state prior to start of experiments. The strontium analyses do not agree as well as the plutonium analyses. One plausible explanation is the strontium in the Tank 40H sludge. The measured strontium concentration in the sludge was 9.9 x 10^9 dpm/g dried sludge.[11] Since the feed solutions in Table 1 contained 0.051 wt % sludge, the sludge added approximately 6 x 10^6 dpm/mL feed slurry. The strontium may not have equilibrated as quickly as the plutonium.

Table 3. Feed Solution Characterization

	Feed 1	Feed 2	Feed 3	Feed 4	Average
Density (g/mL)	1.185	1.187	1.180	1.180	1.183 ± 0.004
Sodium (M)	4.6	4.6	4.6	4.6	4.6
Hydroxide (M)	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08
Nitrate (M)	7.8	5.6	6.6	7.6	6.9 ± 1.0
^{238}Pu initial (d/m/mL)	16,000	19,000	13,000	14,000	15,000 ± 3000
^{238}Pu after sludge addition (d/m/mL)	24,000	22,000	18,000	26,000	23,000 ± 3000
^{238}Pu two days after sludge addition and prior to start (d/m/mL)	21,000	18,000	20,000	20,000	20,000 ± 1300
^{90}Sr initial (d/m/mL)	30,000	29,000	40,000	25,000	31,000 ± 6400
^{90}Sr after sludge addition (d/m/mL)	265,000	80,000	75,000	122,000	136,000 ± 89,000
^{90}Sr two days after sludge addition and prior to start (d/m/mL)	112,000	69,000	90,000	60,000	85,000 ± 28,000

The density proved slightly lower than the density during the 2001 tests (1.20 – 1.22 g/mL).[5] As the density of solutions containing salts such as sodium hydroxide, sodium nitrate, sodium nitrite decreases, the solution viscosity also decreases.[12] Classical filtration theories predict filter flux to increase with decreasing fluid viscosity.[13]

Filter Flux

Figure 2 shows the filter flux as a function of transmembrane pressure (TMP) at low solids concentration during these tests. The figure also shows filter flux data from the tests conducted in 2001 for comparison.[6] The average filter flux during the MST tests measured 0.15 gpm/ft^2. The average flux during the permanganate tests also equaled 0.15 gpm/ft^2. The average TMP during the permanganate tests proved 10% lower than in the MST tests. The filter permeance (filter flux/TMP) proved higher in the permanganate test. In the permanganate test, two permanganate additions occurred which increased the insoluble solids concentration, and thereby decreased the filter flux.

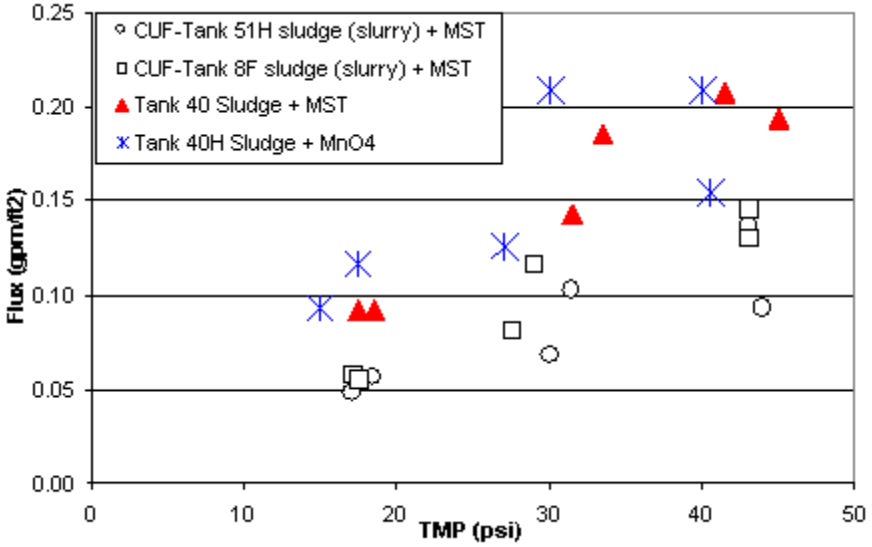

Figure 2. Filter Flux as a Function of Transmembrane Pressure (TMP) at Low Insoluble Solids Concentration

The filter flux for the MST containing feed (average of 0.15 gpm/ft^2) slightly exceeds the flux measured in the 2001 test (0.091 gpm/ft^2) at a similar solids loading. The standard deviation of measured filter flux from different feed solutions was 27% during the 2001 tests. The difference between the flux in the current test and that from 2001 is more than two standard deviations. Several factors contribute to this difference. The feed in this test contained supernate from Tanks 37H, 44F, 26F, and 46F. The supernate in the 2001 tests came from Tanks 37H and 44F. The sludge in the current test came from Tank 40H. The sludge in the previous test came from Tanks 8F and 51H. The supernate density in the current test ranged between 1.18 and 1.19 g/mL. The supernate density in the previous test ranged between 1.20 and 1.22 g/mL. A lower supernate density is indicative of a lower supernate viscosity.[12] Since classical filtration theories predict filter flux to vary inversely with viscosity [13], a lower viscosity solution will correlate with faster filtration. The MST in both tests came from the same source, but prior to the 2001 test, personnel ground the MST to reduce the particle size. These tests did not use ground MST. Classical filtration theories predict filter flux to increase with increasing particle size.[13]

Figure 3 shows the filter flux as a function of transmembrane pressure during the tests with high solids concentration. The figure also shows filter flux data from the tests conducted in 2001 for comparison.[6] The average filter flux during the MST tests equaled 0.082 gpm/ft^2. The average filter flux in

the permanganate tests measured 0.087 gpm/ft^2, or slightly higher than in the MST tests. The filter flux for the MST containing feed exceeded the flux measured in the 2001 test (0.047 gpm/ft^2 average).

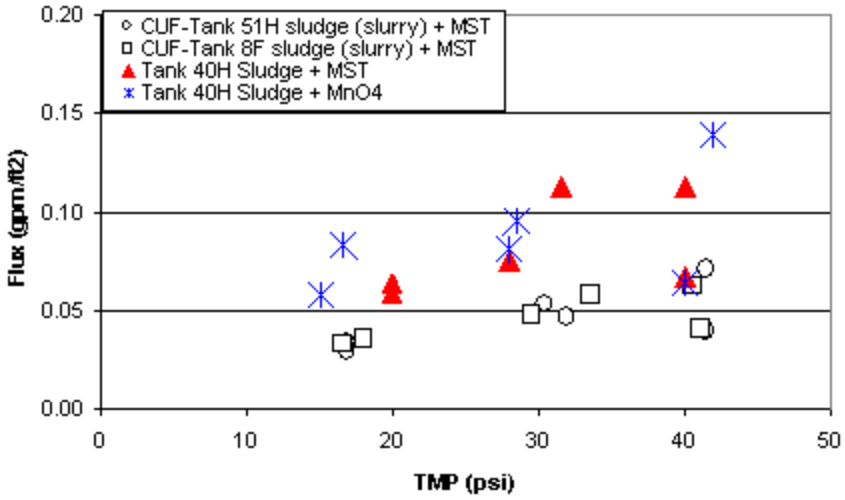

Figure 3. Filter Flux as a Function of Transmembrane Pressure (TMP) at High Insoluble Solids Concentration

The permanganate addition did not produce the level of improvement seen in the simulant filtration test.[8] The multiple additions of reductant may have affected the solid particle formation and produced particles more difficult to filter than the ones produced in the simulant test. In addition, the permanganate feed contained a higher insoluble solids loading than the MST containing feed.

Figure 4 shows the filter flux as a function of filter pore size during these tests. The average filter flux with the 0.5 µ filter equaled 0.082 gpm/ft^2 for MST containing feed and 0.087 gpm/ft^2 for manganese containing feed. The filter flux with the 0.1 µ filter averaged 0.135 gpm/ft^2 for MST containing feed and 0.116 gpm/ft^2 for manganese containing feed. The measured filter flux proved higher (30 ◆ 65%) with the 0.1 µ filter than with the 0.5 µ filter.

Several possible reasons may account for this result. The smaller pore size filter allows fewer fine particles to become trapped in the pores, minimizing any loss of flux with the smaller pore-size filter. McCabe et al. found similar results in previous testing with a 0.1 µ Graver filter.[14,15] That study tested a 0.1 µ Graver filter and a 0.5 µ Mott filter with Hanford and Oak Ridge simulated sludge. The Graver filter produced higher flux (~ 20%) with 0.1 wt % sludge, while the 0.5 µ Mott filter produced higher flux (~100%) with 5 wt % sludge.

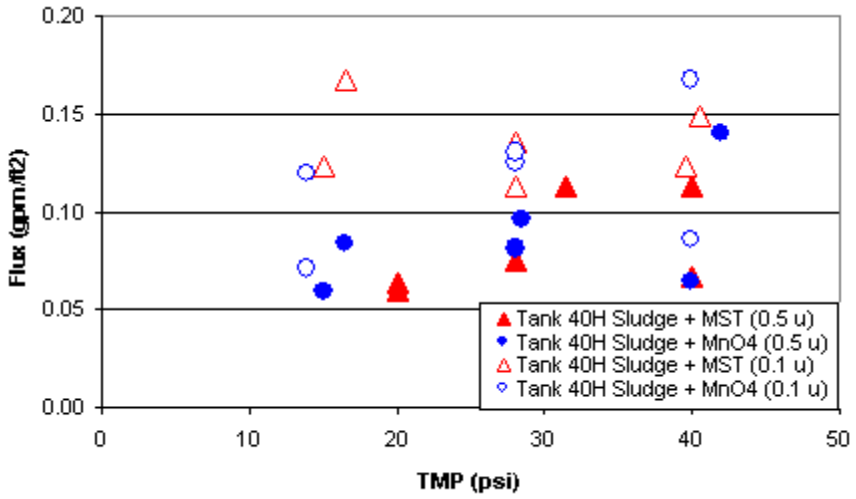

Figure 4. Filter Flux as a Function of Filter Pore Size

A second contributing factor for the observation in the current study involves the process history experienced by the filter. Since this demonstration used the slurry from tests with the 0.5 μ pore-size filter prior to tests with the 0.1 μ pore-size filter, the fine particles may have become trapped in the 0.5 μ pore-size filter and removed from the feed solution. Such removal of fine particles from the slurry would reduce any filter fouling in the subsequent test.

A third reason involves another attribute of the process history. The testing exposed the 0.5 μ pore-size filter to waste with both low and high solids content. The 0.1 μ filter only processed the waste with high solids content. The low solids feed would cause some fouling of the filter in the tests with the 0.5 μ filter. The high solids loading increases the slurry resistance for fines to be transported to the filter surface (similar to the hindered settling observed with concentrated slurries). Since the fine solids had not been transported to the filter pores, they would not contribute to filter fouling in this test. Additionally, the higher solids concentration may increase the number of collisions between particles. Since the sludge particles show a tendency to coagulate, the increased number of collisions could lead to more agglomeration and a larger mean particle size, which can increase filter flux.

Tests conducted at the University of South Carolina in 2001 fed a 4 wt % simulated sludge and MST slurry to a clean filter. The average filter flux during that test proved approximately 80% higher than the average filter flux during tests with a 4.2 wt % simulated sludge and MST slurry and a filter that had processed lower concentrations of insoluble solids. During the 2001 filter tests with actual waste, personnel processed a 4.7 wt % simulated sludge solution – previously used at the University of South Carolina – in the CUF. The measured flux during those tests proved approximately 80% higher than predicted from simulant test results.[6]

Alpha and Strontium Removal

Table 4 shows the radionuclide concentrations in feed and filtrate samples during this test. Filtrate samples were collected from the filtrate sample valve of the CUF and by filtering samples with a 0.45 μ syringe filter. The neptunium concentration in all samples was less than the detection limit.

Table 5 shows the calculated decontamination factors. The MST addition produced a DF of 19 for plutonium and 3 for strontium. The permanganate addition produced a DF of 5 for plutonium, with no significant difference between the samples collected from the CUF and samples that were prepared with a syringe filter. The strontium DF was 1.3 for the CUF samples and 3.9 for the sample prepared with a syringe filter. Additional analyses are needed to quantify the uranium removal and to better quantify the plutonium and strontium removal.

The analyses for the filtered material show a large amount of variability suggesting possible cross contamination of samples. Known contamination events happened during the demonstration with MST treated waste (at low and high solids concentration.) Furthermore, the ICP-MS equipment remained broken for an extended period of time leaving a large number of samples unanalyzed. As a result of these factors, the data set for evaluating actinide and strontium removal efficiency in this test remains rather sparse with a high degree of uncertainty.

Table 4. Radionuclide Concentrations in Feed and Filtrate

Solution	Pu (nCi/g)	Pu (µg/mL)	Sr (nCi/g)	U (µg/L)	Np (nCi/g)
Supernate	6.4	0.288	11.8	1660	< 2.15
Supernate + Sludge	8.8	0.088	51.6	NA	NA
Supernate + Sludge (aged 2 days)	7.6	0.064	31.5	NA	NA
MST Treated					
Treated Material (CUF)	0.4	0.010	11.1	1700	
Treated Material (Syringe Filter)	NA	NA	NA	3700	< 0.70
Permanganate Treated					
Treated Material (CUF)	1.6	0.032	23.8	2800	< 0.14
Treated Material (Syringe Filter)	1.5	0.031	8.1	2123	< 0.70

NA = Not available.

Table 5. Radionuclide DF (Based on Activity)

	Pu	Sr

MST Treated		
Treated Material (CUF)	18.8	2.9
Treated Material (Syringe Filter)	NA	NA
Permanganate Treated		
Treated Material (CUF)	4.8	1.3
Treated Material (Syringe Filter)	4.9	3.9

NA = Not Available.

Conclusions

The demonstrations provide the following conclusions.

- The filter flux from feed prepared with permanganate averaged 10% higher than the flux from feed prepared with MST. The improvement proved less than the 100% increase in flux observed during tests with simulated wastes. However, the permanganate feeds contained (~2X) higher concentrations of insoluble solids than the MST containing feeds.
- The filter flux with a 0.1 μ filter proved 30 – 65% higher than the filter flux with a 0.5 μ filter, on average. Plausible causes for this result include the following.
    - The smaller pore-size may prevent small particles from becoming trapped in the filter pores.
    - The sequence of testing may have preferentially removed small particles from the slurry reducing the amount of particles that could plug pores.
    - The testing with the 0.1 μ pore-size filter lacked the history experienced by the 0.5 μ filter. Previous testing shows that process history influences filter flux. In that earlier testing with simulated waste, bypassing the concentration cycle resulted in as much as 80% gain in filter flux.

Recommendations

The authors recommend the following future work.

- Continue development of optimal permanganate treatment recipe with particular emphasis on optimizing the ratio or reducing agent (e.g., hydrogen peroxide) to permanganate.
- Perform additional tests with 0.1 and 0.2 μ filters using simulated waste.
- After development of an optimal permanganate recipe, conduct an additional filter test with actual waste.
- Perform rheological measurements of solutions containing incomplete reaction of permanganate.
- Perform additional analytical work to quantify the uranium removal and to better quantify the plutonium and strontium removal.

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