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**DEVELOPMENT OF AN IMPROVED SODIUM TITANATE FOR THE
PRETREATMENT OF HIGH LEVEL NUCLEAR WASTE
AT THE SAVANNAH RIVER SITE**

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ABSTRACT

High-level nuclear waste produced from fuel reprocessing operations at the Savannah River Site (SRS) requires pretreatment to remove ^{137}Cs , ^{90}Sr and alpha-emitting radionuclides (i.e., actinides) prior to disposal onsite as low level waste. Separation processes planned at SRS include caustic side solvent extraction, for ^{137}Cs removal, and sorption of ^{90}Sr and alpha-emitting radionuclides onto monosodium titanate (MST). The predominant alpha-emitting radionuclides in the highly alkaline waste solutions include plutonium isotopes ^{238}Pu , ^{239}Pu and ^{240}Pu . This paper describes recent results to produce an improved sodium titanate material that exhibits increased removal kinetics and capacity for ^{90}Sr and alpha-emitting radionuclides compared to the baseline MST material.

INTRODUCTION

Approximately 140 million liters of high-level nuclear wastes (HLW) are presently stored in 48 underground carbon steel tanks at the Savannah River Site (SRS). Approximately 8 vol % of the waste consists of precipitated metal oxides and hydroxides resulting from caustic additions to acidic waste solutions produced from fuel reprocessing and other operations at the site. The precipitated solids, referred to as sludge, contain about 60% of the radioactivity and settle to the bottom of the HLW storage tanks. The remaining volume of HLW is stored as concentrated liquid and saltcake produced from evaporation of the waste solutions. This fraction of the HLW contains about 40% of the radioactivity and is comprised of principally $^{134,137}\text{Cs}$ with smaller amounts of ^{90}Sr and alpha-emitting isotopes of uranium, plutonium, neptunium and other actinide elements.

Cost effective disposal of the large quantities of high-level radioactive waste solutions requires reducing the radioactive material to the smallest possible volume for incorporation into durable long-term waste forms such as borosilicate glass. Acceleration of waste disposal at SRS requires materials that exhibit increased loading capacities and removal kinetics for ^{90}Sr and alpha-emitting radionuclides compared to the baseline material, MST. Increased loading capacity and removal kinetics would result in

decreased facility footprint and increased throughput for this stage of the pretreatment facility.

Recent laboratory tests identified a promising new sodium titanate material with improved strontium and actinide removal characteristics.[1] This material is chemically similar to monosodium titanate (MST),[2-4] which is the baseline material for the removal of ^{90}Sr and alpha-emitting radionuclides from HLW solutions at the SRS.[5-8] Compared to the baseline MST material, the new sodium titanate materials exhibit higher batch capacities and kinetics. Consequently these materials offer the opportunity to reduce sorbent use and increase throughput in processing facilities. This paper describes results from the continued development of this new material.

EXPERIMENTAL

Evaluation of Sr and Actinide Removal Performance

We evaluated strontium and actinide removal performance by contacting simulated and actual waste solutions with a measured quantity of the new sodium titanate sample. Table 1 provides the composition of the simulated and actual waste solutions used in these tests. We performed batch contact tests with the simulated waste solutions by shaking bottles kept at 25 ± 3 °C in a waterbath. After the addition of the appropriate sample of MST, we sampled each test bottle periodically over a 168-hour test period. All samples were filtered through 0.45- μm nylon-membrane filters to remove MST solids. Measured aliquots of the filtrate were then diluted with an equal volume of 5 M nitric acid. Gamma spectroscopy measured the ^{85}Sr and ^{237}Np content. We measured the plutonium isotopics content by radiochemical separation of the plutonium from neptunium and uranium followed by alpha counting of the extracted plutonium.

Tests with actual waste were carried out in the Shielded Cells Facility of SRNL. The testing protocol followed that described above with simulated waste solutions. Filtration of samples used a 0.1- μm polytetrafluoroethylene syringe filters. We diluted aliquots of the filtrates by approximately a factor of 20 with 2 M nitric acid solution. The higher dilution was required to reduce radiation exposure during subsequent analyses for radiochemical content.

Table 1. Composition of Simulated and Actual Waste Solutions

<u>Analyte</u>	<u>Unit</u>	<u>Simulant</u>	<u>Actual Waste</u>
NaOH	M	1.36 (0.14)	2.10 (0.0070)
NaNO ₃	M	2.44 (0.24)	2.47 (0.015)
NaNO ₂	M	0.116 (0.012)	0.648 (0.0063)
NaAl(OH) ₄	M	0.503 (0.050)	0.423 (0.0068)
Na ₂ CO ₃	M	0.016 (0.010)	0.566 (0.0758)
Na ₂ SO ₄	M	0.551 (0.055)	0.0518 (0.0010)
Total Na	M	5.2 (0.52)	5.48 (0.367)
⁸⁵ Sr	dpm mL ⁻¹	1.65E+05 (3.22E+03)	-
Total Sr	μg L ⁻¹	484 (32)	1,310 (242)
Total Pu	μg L ⁻¹	218 (13)	275 (56)
²³⁷ Np	μg L ⁻¹	461 (90)	131 (19)
Total U	μg L ⁻¹	9,550 (330)	11,100 (1,740)

* Numbers in parenthesis are single standard deviation of replicate measurements.

Evaluation of Filtration Characteristics

We performed filtration tests in a stirred cell filtration apparatus with a simulated waste solution having the chemical composition as reported in Table 1 without the radioactive components. We added the appropriate MST sample to the solution to provide a solids concentration of 0.55 g L⁻¹. We poured approximately 60 mL of the feed suspension into the stirred cell, agitated the cell contents, pressurized the cell to 30 psi, and measured the filtrate volume as function of time. Tests evaluated the filtration characteristics of the MST samples with the following filter media: 0.1 μ TruMem[®] ceramic (old rotary filter baseline), 0.1 μ Mott sintered SS (ARP baseline), 0.1 μ Pall sintered SS (SWPF baseline), 0.5 μ Pall sintered SS (rotary microfilter). The Mott and Pall pore sizes are nominal. We performed two sets of tests with the 0.1 μ Mott and 0.1 μ Pall media using a fresh feed suspension for the 2nd set of tests.

RESULTS AND DISCUSSION

Simulated Waste Tests

Tests evaluated a wide variety of preparation conditions to prepare the sodium titanate material with the best combination of strontium and actinide removal characteristics. From these studies we selected conditions for larger laboratory scale syntheses and prepared three separate batches of the new sodium titanate material. The larger laboratory preparations represented a 25 – 100 fold increase in batch size from earlier laboratory syntheses.

We tested the strontium and actinide removal performance of the three batches of sodium titanate using the simulated waste solution (see Table 1) at sorbent concentrations of 0.1 and 0.2 g L⁻¹. These tests also included a sample of the baseline MST. Duplicate tests for each of the batches revealed very similar performance among all of the samples.

Table 2 provides a summary of the average normalized decontamination factors (DF) for strontium, plutonium and neptunium over a range of batch contact times. The decontamination factor is determined by dividing the initial sorbate concentration by the concentration at the indicated sampling time. A normalized DF value the ratio of the measured DF value of the new sodium titanate material to that of the baseline MST sample. The results indicate that the new sodium titanate samples exhibit much improved strontium and plutonium removal performance compared to the baseline MST sample. For example, the normalized strontium DF values for the new sodium titanate samples were consistently about a factor of four greater than that of the baseline MST. Plutonium removal performance proved even higher as the normalized DF values ranged from 4 to 70.

Table 2. Average Normalized DF Values for the New Sodium Titanate Samples

Time (h)	Normalized DF - Strontium		[MST] = 0.2 g/L	
	[MST] = 0.1 g/L		Average	Std Dev
2	3.80	0.22	4.75	0.14
4	4.10	0.21	4.79	0.24
6	3.23	0.18	5.31	0.66
12	4.45	0.20	5.16	0.23
24	4.43	0.22	4.93	0.40
168	4.30	0.19	5.30	0.39

Time (h)	Normalized DF - Plutonium		[MST] = 0.2 g/L	
	[MST] = 0.1 g/L		Average	Std Dev
2	3.59	0.25	11.8	1.25
4	8.69	0.50	25.6	2.90
6	13.0	1.41	33.6	6.18
12	23.3	1.28	53.5	1.92
24	35.3	2.88	65.3	4.92
168	16.7	3.16	70.5	5.20

Time (h)	Normalized DF - Neptunium		[MST] = 0.2 g/L	
	[MST] = 0.1 g/L		Average	Std Dev
2	1.26	0.049	1.31	0.094
4	1.30	0.123	1.28	0.211
6	1.26	0.131	1.70	0.241
12	1.81	0.262	1.46	0.124
24	1.58	0.079	1.67	0.230
168	1.75	0.186	2.58	0.611

Normalized DF values calculated by dividing the measured DF value for the new sodium titanate to that measured for the baseline MST sample (Optima 00-QAB-417) at the same test condition.

The new sodium titanate samples exhibited much faster removal kinetics compared to the baseline MST particularly for strontium and plutonium. Figure 1 provides a plot of the average plutonium concentration versus time for the tests with the simulated waste solution. The control test contained no added sorbent and served as a measure of sorbate removal by a mechanism other than sorption onto the sodium titanate such as precipitation or sorption onto bottle walls. Clearly plutonium removal proceeded much faster with the new sodium titanate compared to the baseline MST. For example, after 2 hours, the new sodium titanate at a concentration of 0.2 g L^{-1} reduced the plutonium concentration from about $200 \text{ } \mu\text{g L}^{-1}$ to less than $10 \text{ } \mu\text{g L}^{-1}$. At the same sorbent concentration the baseline MST reduced the plutonium concentration from about $200 \text{ } \mu\text{g L}^{-1}$ to $100 \text{ } \mu\text{g L}^{-1}$. Thus, the rate of plutonium removal with the new sodium titanate measured about 10 times that of the baseline MST.

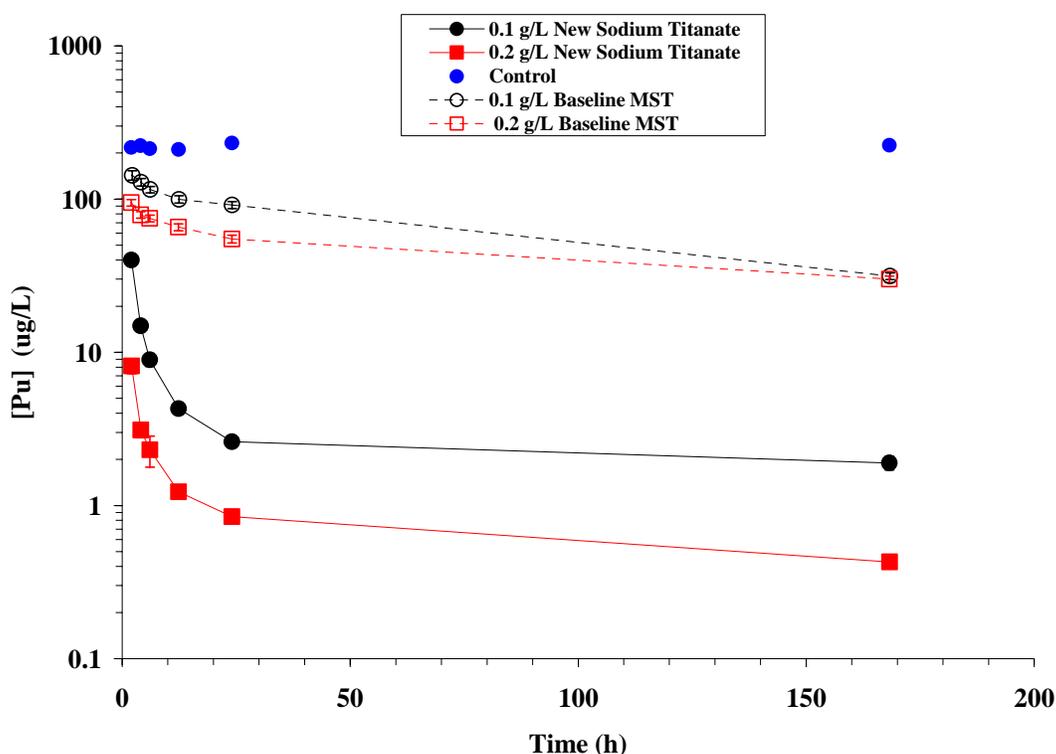


Figure 1. Average Plutonium Concentration versus Time upon Contact of Simulated Waste Solution with New Sodium Titanate and Baseline MST Samples.

Actual Waste Tests

Given the excellent performance of the new sodium titanate samples with simulated waste solutions we tested performance with actual waste supernate obtained from the SRS. In this set of tests we contacted the actual waste supernate (see Table 1) with the samples of the new sodium titanate at 0.1 and 0.2 g L^{-1} . The baseline MST test featured a sorbent concentration of 0.2 g L^{-1} . We also included a test in which a small amount of the solvent planned for use in Caustic Side Solvent Extraction process was added in

addition to the new sodium titanate. Note that the strontium, plutonium and uranium concentrations in this waste are close to the respective solubility limits. Thus, this waste supernate represented a significant challenge to demonstrate good performance.

Test results confirmed that the new sodium titanate exhibited improved performance for the removal of strontium and actinides compared to the baseline MST. Figure 2 provides a plot of the total plutonium activity versus time upon contact of the actual waste solution with the new sodium titanate and baseline MST. As we observed with simulated waste, the plutonium activity decreased rapidly upon addition of the new sodium titanate.

The current waste acceptance criteria (WAC) for the disposal of supernate waste solution to the Saltstone facility at SRS limits the total alpha activity to no more than 22,500 pCi mL⁻¹. The baseline MST sample reduced the plutonium activity to about 200,000 pCi mL⁻¹ after 24-hours of contact, which remains well above the WAC limit. Thus, an increased quantity of the baseline MST material would be required to successfully treat waste at this alpha activity. At the same sorbent concentration, the new sodium titanate reduced the plutonium activity below the WAC limit after 12 hours of contact.

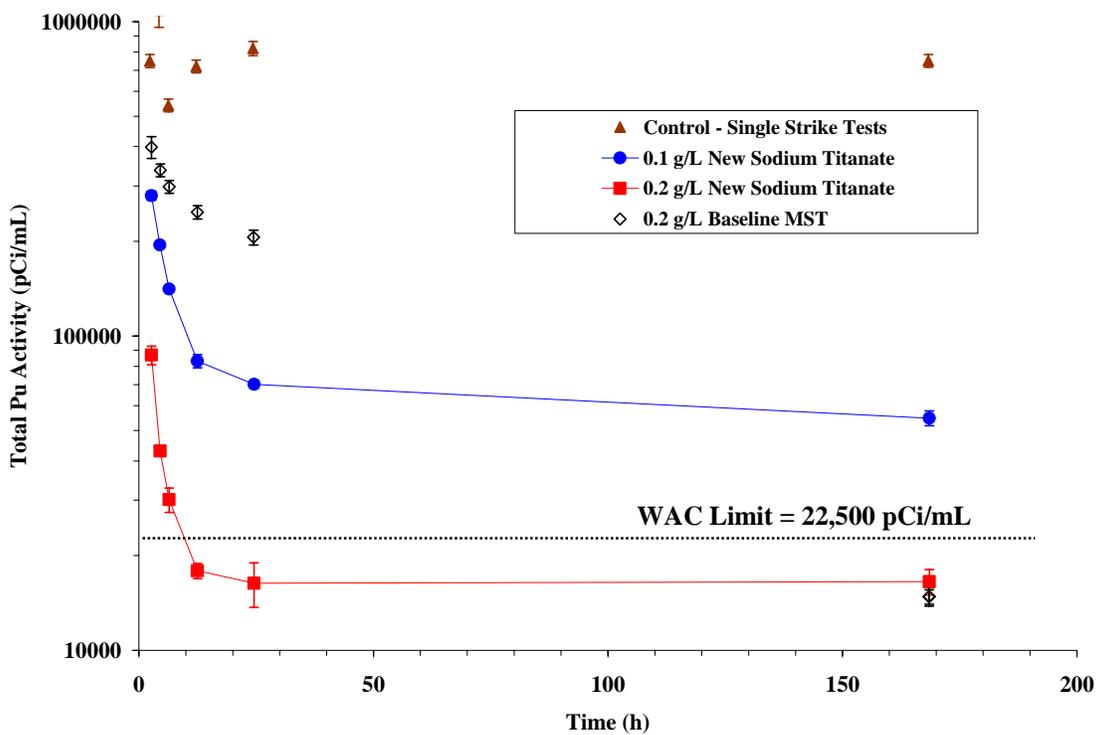


Figure 2. Total Plutonium Activity versus Time upon Contact of Actual Waste with New Sodium Titanate and Baseline MST

Table 3 provides a summary of the average normalized DF values for strontium and plutonium in the actual waste tests. Strontium removal proved higher with the new sodium titanate sample than that with the baseline MST sample. At the sorbent

concentration of 0.2 g L^{-1} , the DF values for the new sodium titanate sample measured about 2.7 times higher than those of the baseline MST sample. Tests with the new sodium titanate at a sorbent concentration of 0.1 g L^{-1} resulted in strontium DF values comparable to those of the baseline MST sample. We also observed that the strontium DF values in the test with the new sodium titanate and CSSX solvent proved very similar to those without the CSSX solvent. Thus, we conclude that the presence of the CSSX solvent did not adversely influence strontium removal by the new sodium titanate sample.

In earlier tests with simulated waste solution, the new sodium titanate exhibited a factor of about 5 higher strontium removal than the baseline MST sample (see Table 2). We attribute the lower increase in strontium removal performance in the actual waste tests to the higher initial total sorbate concentration, which results in greater overall loading of the sorbent. The actual waste solution contained a total cation equivalent concentration for the four sorbates of $128 \pm 21 \text{ }\mu\text{M}$, which is 32% higher than that of $97 \pm 15 \text{ }\mu\text{M}$ for the simulated waste solution.

As with strontium, the new sodium titanate sample exhibited increased plutonium removal compared to the baseline MST. At a sorbent concentration of 0.2 g L^{-1} the DF values of the new sodium titanate sample measured between 5 and 11 times higher than those of the baseline MST sample (see Table 3). The 168-hour result for the baseline MST tests showed a large increase in plutonium removal compared to the earlier sampling times (see Table 7). Note that we did not observe a similar increase in strontium removal. Additional analyses confirmed the low plutonium result. We have not observed this type of behavior with the baseline MST in other tests with simulants or tank wastes. Thus we conclude this result is in error. Tests with the new sodium titanate at a sorbent concentration of 0.1 g L^{-1} resulted in plutonium DF values between 1.5 and 3.5 times higher than those of the baseline MST sample at 0.2 g L^{-1} . Thus, we conclude that the new sodium titanate sample clearly demonstrated improved plutonium removal performance compared to the baseline MST sample.

We also observed that the plutonium DF values in the test with the new sodium titanate and CSSX solvent proved very similar to those without the CSSX solvent. Thus, we conclude that the presence of the CSSX solvent did not adversely influence plutonium removal by the new sodium titanate sample.

In earlier tests with simulated waste solution, the new sodium titanate exhibited a factor of 11 to 70 times higher in the plutonium DF value than the baseline MST sample (see Table 2). For the actual waste tests the new sodium titanate sample exhibited increases in the DF values of between 5 and 11 times that of the baseline MST sample. As with strontium, we attribute the lower increase in plutonium removal performance in the actual waste tests to the higher initial total sorbate concentration in the actual waste. The actual waste solution measured about 25% higher in plutonium concentration than that in the simulant (275 ± 56 versus $218 \pm 13 \text{ }\mu\text{g L}^{-1}$). These findings confirm that the new sodium titanate sample clearly exhibits increased capacity for plutonium compared to the baseline MST sample.

Table 3. Average Normalized Decontamination Factors for New Sodium Titanate

Time (h)	Normalized DF			
	Strontium		Plutonium	
	Average	Std Dev	Average	Std Dev
2.6	2.61E+00	1.45E-01	5.21E+00	3.14E-01
4.5	2.70E+00	7.29E-01	5.54E+00	3.99E-01
6.4	>2.65E+00		6.53E+00	1.12E+00
12.4	>1.66E+00		1.13E+01	4.06E-01
24.4	>2.35E+00		8.88E+00	2.87E+00
168.4	>1.44E+00		8.02E-01	2.67E-01

Normalized DF values calculated by dividing the measured DF value for the new sodium titanate to that measured for the baseline MST sample (Optima 00-QAB-417) at the same test condition.

We observed similar neptunium DF values for the new sodium titanate sample in each of the tests. The baseline MST sample exhibited higher DF values for neptunium than the new sodium titanate at each of the sampling times. In previous simulant tests we observed that the new sodium titanate sample exhibited slightly higher DF values than the baseline MST sample. The initial neptunium concentration in the actual waste solution measured more than one-third lower than that in the simulant tests (131 ± 19 versus $461 \pm 90 \mu\text{g L}^{-1}$). Given the lower neptunium concentration and the previous findings with the simulated waste solution, the results with the new sodium titanate sample are surprising. Perhaps the higher loading of strontium and plutonium decreased sorption of neptunium onto the new sodium titanate.

We observed no measurable removal of uranium in any of the tests. Previous testing with simulated waste solutions at low sorbent concentrations also showed no measurable uranium removal. Given the similar initial uranium concentrations in both the actual waste ($11,100 \pm 1,730 \mu\text{g L}^{-1}$) and simulated waste ($10,200 \pm 2,040 \mu\text{g L}^{-1}$) solutions, the lack of measurable uranium removal is not unexpected. Higher sorbent concentrations are needed to determine uranium removal performance of the new sodium titanate sample with actual waste solutions.

Filtration Characteristics

We evaluated the filtration characteristics of new sodium titanate samples using a stirred cell filtration apparatus that we had previously shown could be used to provide a good qualitative comparison of the filterability of different feed slurries.^{1,2} We evaluated the filtration characteristics of the MST samples with the following filter media: 0.1 μ TruMem[®] ceramic, 0.1 μ Mott sintered stainless steel, 0.1 μ Pall sintered stainless steel, and the 0.5 μ Pall sintered stainless steel. Samples tested included the baseline MST (Optima 00-QAB-471) and the three new sodium titanate samples prepared at the larger laboratory scale (LS1, LS2 and LS3).

We observed no difference in filtrate rate between the baseline MST and the new sodium titanate samples with the TruMem[®] media. Figure 4 shows the results from the tests conducted with the 0.1 μ Mott media, which is the filter media planned for processing

facilities at the SRS. We performed two sets of tests with this filter media. In general we observed a decrease in filtration rate in the second test set compared to the first set. However, within each test set, we observed no difference in filtrate rate between the baseline MST and the new sodium titanate.

We observed no difference in filtration rates with the 0.1 μ Pall filter media, but did find that the new sodium titanate filtered slower with the 0.5 μ Pall filter. Measurement of the particle size distribution of the baseline MST and the new sodium titanate samples revealed no significant differences among the sample. The new sodium titanate samples appeared to have a higher fraction of fines around 0.5 μ in size than the baseline MST. Perhaps this higher fraction of fines leads to reduced filtration with the larger pore filter media. However, since the processing facilities plan on using the smaller 0.1 μ sized filter, we conclude that filtration characteristics of the new sodium titanates should be similar to those of the baseline MST.

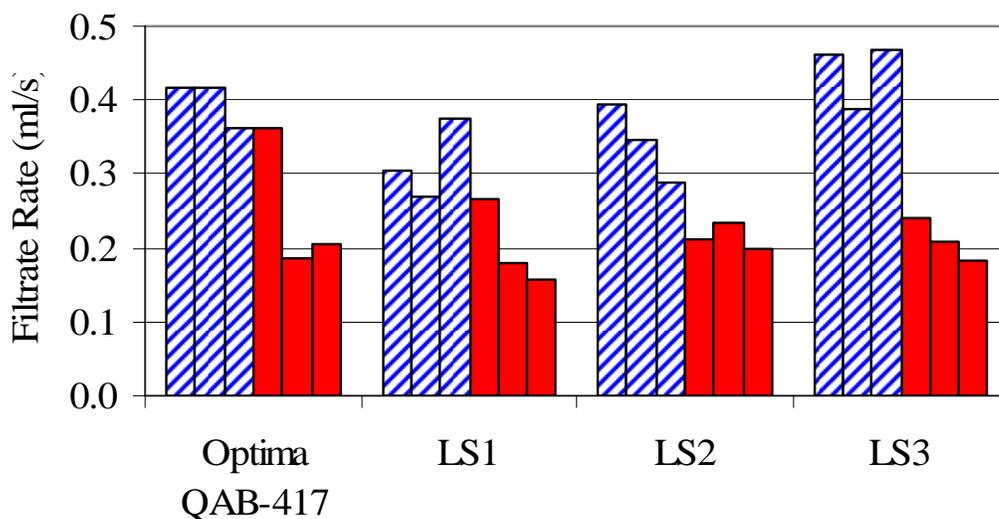


Figure 4. Filtration Rate with 0.1- μ m Mott Media

Shelf-Life

The baseline MST appears to have a very long shelf-life when stored as an aqueous suspension. For example, the sample of MST used in this study for comparison to the new sodium titanate samples was produced about 10 years ago and has shown no loss in strontium and actinide separations performance during this time period. Given the good shelf-life of MST, we evaluated the shelf-life of the new sodium titanate samples by measuring strontium and actinide removal performance after storing at ambient laboratory conditions as an aqueous slurry for six months. We used the same simulant (see Table 1) that we used when we first tested the performance of the new sodium titanate samples. For these tests we limited the new sodium titanate testing to a single MST concentration (0.2 g L^{-1}) in duplicate for each sample with sampling events at 6 and 12-hours.

Table 4 provides the average and standard deviation of the strontium, plutonium and neptunium DF values for the new sodium titanate and baseline MST samples at both testing dates. Note, we did not test the performance of the baseline MST sample at 0.4 g L^{-1} at the initial time date. Figure 5 provides a plot of the plutonium concentration for the new sodium titanate and baseline MST samples at both the initial test set and the set after 6-months of storage.

Inspection of Table 4 indicates that the removal of strontium and neptunium was not affected by storage of the new sodium titanate for 6 months at ambient laboratory temperature. For strontium, we observed that the new sodium titanate exhibited an average DF value 5 times greater than that of the baseline MST sample after 6 and 12-hours of contact at a 0.2 g L^{-1} sorbent concentration for both the initial and 6-month testing dates. Comparison of the new sodium titanate results at 0.2 g L^{-1} with that of the baseline MST at the higher concentration of 0.4 g L^{-1} revealed that the new sodium titanate exhibited a strontium DF value of 1.5 times that of the baseline MST.

Testing results indicated lower average plutonium DF values at the 6-hour and 12-hour sampling times after storing the modified MST samples for six months (see Table 4). At the 95% confidence level the range of plutonium DF values at the initial and 6-months dates overlap indicating the DF values are not statistically different. Thus we cannot absolutely conclude that the plutonium removal performance has changed over the 6-month storage time.

Plotting the individual data points for each trial reveals that the 6-month plutonium concentrations are consistently higher than those initially with the exception of a single trial result in each data set. This trend suggests that the material has lost a small fraction of capacity. However, the plutonium removal remains quite high. For example, the plutonium DF values after 6-months measured between 23 and 43 times higher than the baseline MST added at 0.2 g L^{-1} and 13 to 23 times higher than the baseline MST added at 0.4 g L^{-1} . Thus, after 6-months of storage, the modified MST provides excellent removal characteristics for strontium and actinides.

Table 9. Strontium, Plutonium and Neptunium DF Values for the Modified and Baseline MST Samples at the Initial and 6-month Storage Times

	Strontium DF											
	New Sodium Titanate @ 0.2 g/L				Baseline MST @ 0.2 g/L				Baseline MST @ 0.4 g/L			
	6-hours		12-hours		6-hours		12-hours		6-hours		12-hours	
	Average	Std Dev	Average	Std Dev	Value	Uncertainty	Value	Uncertainty	Value	Uncertainty	Value	Uncertainty
Initial	1.13E+02	1.40E+01	1.28E+02	5.80E+00	8.50E+00	2.09E-01	6.88E+00	1.79E-01	nd	-	nd	-
6-months	1.10E+02	3.50E+00	1.37E+02	5.74E+00	2.36E+01	5.97E-01	2.78E+01	7.47E-01	7.35E+01	2.02E+00	9.00E+01	3.44E+00
	Plutonium DF											
	New Sodium Titanate @ 0.2 g/L				Baseline MST @ 0.2 g/L				Baseline MST @ 0.4 g/L			
	6-hours		12-hours		6-hours		12-hours		6-hours		12-hours	
	Average	Std Dev	Average	Std Dev	Value	Uncertainty	Value	Uncertainty	Value	Uncertainty	Value	Uncertainty
Initial	9.57E+01	1.76E+01	1.72E+02	6.16E+00	1.85E+00	1.27E-01	2.11E+00	1.63E-01	nd	-	nd	-
6-months	6.38E+01	6.14E+00	1.43E+02	2.66E+01	2.82E+00	1.82E-01	3.31E+00	2.43E-01	5.08E+00	3.23E-01	6.22E+00	4.66E-01
	Neptunium DF											
	New Sodium Titanate @ 0.2 g/L				Baseline MST @ 0.2 g/L				Baseline MST @ 0.4 g/L			
	6-hours		12-hours		6-hours		12-hours		6-hours		12-hours	
	Average	Std Dev	Average	Std Dev	Value	Uncertainty	Value	Uncertainty	Value	Uncertainty	Value	Uncertainty
Initial	1.77E+00	2.52E-01	1.83E+00	1.55E-01	1.30E+00	8.21E-02	8.94E-01	7.11E-02	nd	-	nd	-
6-months	1.83E+00	3.93E-01	1.24E+00	2.07E-01	1.10E+00	2.52E-01	7.44E-01	2.19E-01	1.24E+00	1.90E-01	2.37E+00	1.80E-01

nd = not determined

New sodium titanate results are average and standard deviation of six trials

Baseline MST results are single determinations with reported analytical uncertainty

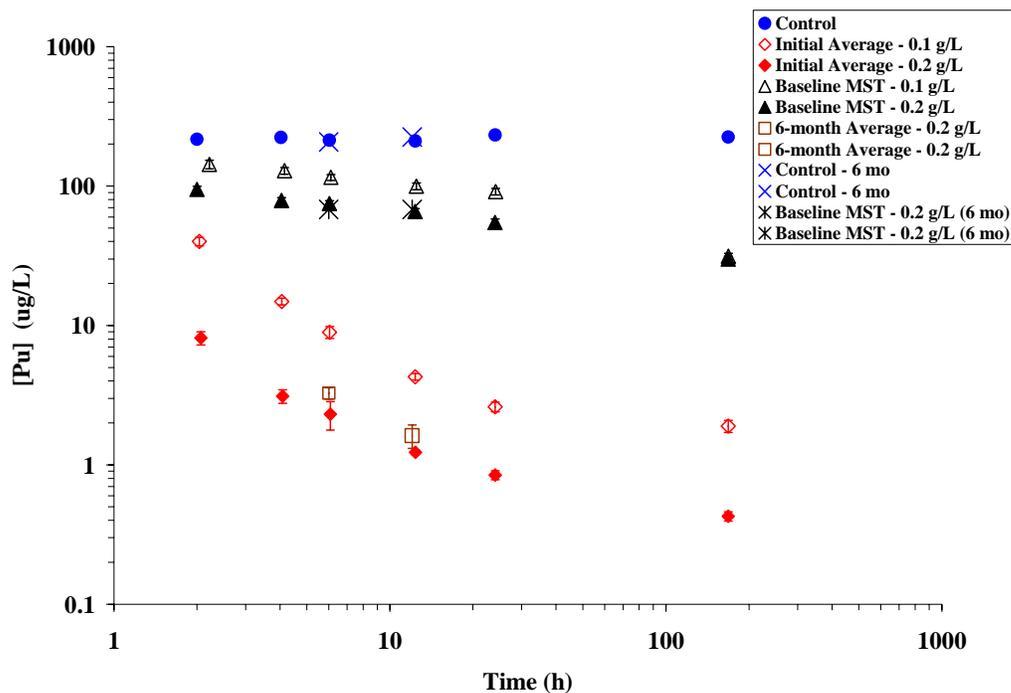


Figure 5. Plot of Average Plutonium Concentration versus Time for Tests with Modified and Baseline MST Samples at the Initial and 6-month Storage Times

CONCLUSIONS

Testing demonstrated that the preparation of an improved inorganic sorbent can be reproduced at a larger laboratory scale. In addition to excellent removal characteristics with simulated waste solutions, the new sodium titanate material demonstrated improved performance with actual tank waste. Filtration characteristics of the new sodium titanate proved similar to that of the baseline MST. Also, after 6 months of storage, the new sodium titanate continues to show excellent strontium and actinide removal performance. Based on these results we conclude that the new sodium titanate material appears an excellent candidate for replacing the baseline MST for waste processing at the SRS.

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