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Loading during Low Curie Salt Use**

W. R. Wilmarth
V. H. Dukes
J. T. Mills

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Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

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Reactivity of Crystalline Silicotitanate (CST) and Hazardous Metal/Actinide Loading during Low Curie Salt Use

W. R. Wilmarth, V. H. Dukes, and J. T. Mills
Waste Processing Technology
Savannah River National Laboratory
Westinghouse Savannah River Company

SUMMARY

Crystalline Silicotitanate (CST) in its engineered form (IONSIV[®] IE-911) continues to be studied for possible use for removing radioactive cesium from several types of waste solutions at the Savannah River Site. This study involved deriving information about spent CST that assists in determining possible disposition alternatives. Results for this work include:

- After passing 3000 column volumes of a dissolved saltcake simulant containing RCRA hazardous metals, the spent CST passed a TCLP test and is RCRA non-hazardous.
- The spent CST was found to have transuranic concentrations greater than the TRU limit of 100 nCi/g. The triplicate measurement showed TRU levels > 4000 nCi/g.
- Studies involving simulating storage of ground CST in sludge slurries indicated no detrimental effects on the measured yield stress or viscosity of the slurries when stored for up to 4 months at 50 °C.
- During the storage testing, there was no indication of significant degradation of the CST as measured by in growth of CST-specific elements in the liquid phase of the slurry.
- Also, during storage tests minor desorption of cesium from the ground CST material was observed.

INTRODUCTION

An option for processing some of the saltcake stored at the Savannah River Site following dissolution is the Low Curie Salt (LCS) process. In this process, an identified saltcake tank is drained of its interstitial supernate to remove a significant (~40%) portion of the radioactive cesium inventory. A second step may involve washing of this drained saltcake by a suitable interstitial fluid to further remove cesium. Finally, water is added to dissolve the saltcake for final disposal in the grout produced by the Saltstone Facility.¹ Modification to the Saltstone requirements have been proposed to increase specific radionuclide limits including cesium.² However, depending on the degree of drainage and washing, the dissolved saltcake may possess levels of radioactive cesium above the modified limits. Therefore, a final polishing step, such as ion exchange, would be advantageous to aid in meeting facility requirements.

Prior studies examined crystalline silicotitanate (CST, IONSIV IE-911, UOP, Des Plaines, IL) for removing cesium from Savannah River Site waste.^{3,4,5,6,7} CST proved very effective at removing radioactive cesium from SRS wastes, but researchers identified leaching and chemical stability issues. Subsequent studies made progress toward alleviating this instability. However, the applicability to the LCS CST use is not known.

Additionally, multiple options exist for disposal of the spent CST resin from a polishing operation. Prior testing did not extensively examine areas such as the environmental hazards of either the spent resin or its affinity for transuranic species. Previous work demonstrated affinity for actinides.^{8,9,10} Therefore, this study examined three primary objectives as listed below.^{11,12,13}

- Determine the reactivity and physical changes that CST (granular and ground) undergoes during storage in SRS high level waste. Determine if the changes impact the slurry rheology or ability to pump CST or sludge solids.
- Determine if CST is hazardous versus the Resource Conservation and Recovery Act (RCRA) requirements, after being loaded with prototypical levels of Ag, As, Ba, Cd, Cr, Hg, Pb and Se and to determine the level of sorption of these metals.
- Determine the loading of U and Pu on CST after passing prototypical volumes of dissolved saltcake simulant saturated with the actinides.

During the course of this work, the development of CST as a cesium polishing process has been supported by the EM-21 office. The concept^{14,15} involves the use of an in-riser column followed by a second processing step to grind the engineered form of CST to an appropriate size for feeding the Defense Waste Processing Facility. The results from this work are applicable for determining properties of the disposed CST from the small column deployment.

EXPERIMENTAL DETAILS

Task 1. CST Reactivity¹⁶

Previous use of inorganic ion exchange materials in the tank farms led to mound formation due to material binding and clumping.¹⁷ Therefore, a risk for agglomeration of spent CST material during storage in a sludge tank exists based on waste chemistry and CST chemistry. The following experiments investigated this risk. Parameters to be examined include temperature (35 and 50 °C, ground or as-received CST, supernate composition, and whether the CST and sludge are well mixed or the CST is added in layers.

1. Control test 1, sludge alone at 50 °C
2. Control test 2, CST in 1 M NaOH, 50 °C

3. Sludge, ground CST, washwater, slurry mixed, 50 °C
4. Sludge, ground CST, washwater, slurry mixed, 50 °C, shaken periodically
5. Sludge, ground CST, washwater, layered, 50 °C
6. Sludge, ground CST, supernate, slurry well mixed, 50 °C, shaken periodically
7. Sludge, ground CST, supernate, slurry well mixed, 35 °C, shaken periodically
8. Sludge, ground CST, supernate, slurry layered, 50 °C
9. Sludge, CST, washwater, slurry mixed, 35 °C

Each test with CST utilized cesium-loaded resin. Two control tests were performed with the first containing only sludge and the second containing only CST in sodium hydroxide solution. For the tests where the vessel will be periodically mixed, the vessel was placed into a pre-heated orbital shaker at a setting of 300 rpm for 10 minutes and returned to the thermo-sated oven. Initial plans called for rheology measurements to be taken on the following frequency: 1 week, 1 month, 2 months, 3 months, 6 months, 9 months and 1 year. Testing was terminated following the 3rd month sample plus one sample taken after the 4th month due to funding concerns. Additionally, aqueous samples were analyzed by atomic absorption for cesium desorption, and inductively coupled plasma – emission spectroscopy for elements of the CST matrix in the final two sets.

Task 2. Hazardous Metal and Actinide Loading¹⁸

Spent CST resin will require characterization prior to disposal as a waste. The final disposal path remains undefined. Therefore, this task is designed to determine two specific aspects of the spent resin characteristics. The actinide and hazardous metal content was determined after a prototypical number of column volumes of a dissolved saltcake simulant passes through a bed of CST.

- In the first sub-task, researchers prepared a non-radioactive dissolved saltcake simulant based on the SRS Tank 41H composition¹⁹ as outlined in Table 1. Ag, As, Ba, Cd, Cr, Hg, Pb and Se were added to levels measured in actual dissolved saltcake. Personnel prepared an approximately 2 mL column of CST and 6 liters of the waste was passed through the 2 mL column, thus representing 3000 column volumes. The superficial velocity was maintained at 7 cm/min. At the completion of the test, the solid CST was washed with 10 column volumes of inhibited water and submitted for the Toxic Chemical Leach Procedure in the Analytical Development Section (ADS). It should be noted that ADS is not certified by the Environmental Protection Agency for regulatory analysis.
 - In the second sub-task, researchers added actinides (e.g., uranium and plutonium) in lieu of the hazardous metals. The actinides concentrations were verified to be stable at the start of the test. In other words, samples were taken from the stock solutions and analyzed until actinide activities became constant. The test used similar volumes of waste and CST. At the conclusion of the loading test, samples of the washed CST were digested in nitric acid and submitted for mass spectrometry and alpha counting.
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Table 1. Composition (M) of SRS Waste Simulants

Component	Supernate	Washwater ²⁰	Dissolved Saltcake ²¹
Na ⁺	7.5	1.6	6.65
OH ⁻	3.5	0.6	1.9
NO ₃ ⁻	2.0	0.30	2.6
NO ₂ ⁻	1.5	0.46	0.9
AlO ₂ ⁻	0.31	0.1	1.2
CO ₃ ²⁻	0.1	0.05	0.05
SiO ₃ ²⁻	0.004	0.004	0.004

RESULTS AND DISCUSSION

CST Reactivity

Shown in Figure 1 and Figure 2 are the measured yield stresses as a function of time for the CST/sludge slurries for the washwater and supernate samples respectively. Each data point represents an average of two extrapolated y-intercepts of the shear stress – shear rate plots generated from replicate rheology measurements. Additionally, yield stress data was obtained from the down flow curve, i.e. data from the rheogram when the rotator cup is being slowed. This is believed to provide the best indication of the ability to be able to pump the mixed slurry. This study did not examine the yield strength of the stagnant, settled slurry as would have been measured using a so called vane measurement. In other words, these tests did not examine the force required to re-suspend a settled slurry.

The data from these plots shows little or no increase in yield stress for the duration tested (up to 4 months). The yield stress of the washwater/CST/sludge slurries were very low ranging between 0.5 and 2 Pa. The experimental protocol “washed” the sludge with the washwater simulant thrice prior to initiating the testing. This data does indicate that operation was successful. There does appear to be an experimental artifact associated with the data obtained from the 3-month samples. The measured yield stresses nearly doubles either the measured values at either the 2 or 4 month sampling. The rheology data and the experimental compositions were checked and found to be accurate. However, a physio-chemical explanation for this phenomenon is not understood. In reality, the magnitude of the yield stresses is very low and poses no engineering issue associated with the ability to be able to move the resultant slurry. It should be noted that the abnormality at the 3-month sampling, also, occurs in the supernate samples as well. There also does not appear to be a difference between the rheological behavior of the samples stored at 35 °C or 50 °C.

With respect to the yield stress data from the supernate-based slurries, the data are very similar. Previous work at higher temperatures by Poirier²² and Taylor²³ had shown

increased yield stress or yield strength. The results reported herein do not show a large temperature effect. However, there does appear to be a slight increase in yield stress with storage time observed in Figure 2.

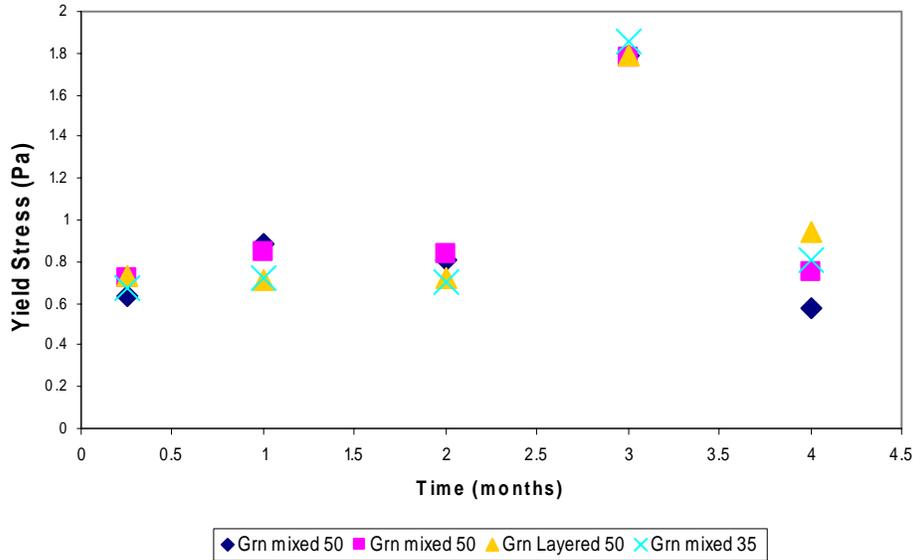


Figure 1. Yield Stress Data (Pa) for the Washwater/Sludge/CST Slurries

Figure 3 and

Figure 4 show the measured plastic viscosities (mPa-s or cP) for the CST/Sludge slurries that had been stored at elevated temperatures. In both simulants there is a large degree of scatter in the data taken at the different time intervals. Conversely, there is very good agreement for the measured plastic viscosities for each of the CST/sludge simulant samples within a given sampling interval. This indicates that a large fraction of the variability in the data is due to the experimental variability in the sample preparation rather than the response to the storage time at elevated temperature.

In the washwater simulants the measured viscosities ranged from 3 to 6 cP and 5 to 10 cP in the supernate-based CST/sludge slurries. These values are expected for the different chemistries of the supernate and washwater simulants. It is also indicative of the viscosity being dominated by the fluid viscosity rather than the sludge/CST weight percent insoluble solids.

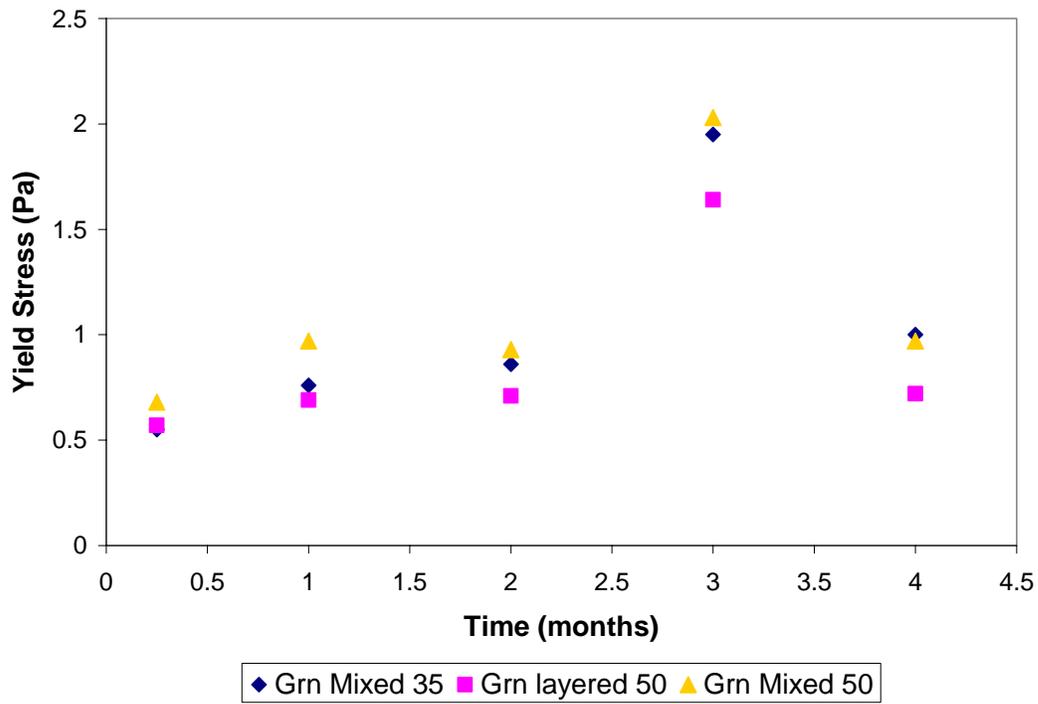


Figure 2. Yield Stress Data (Pa) for the Supernate/Sludge/CST Slurries

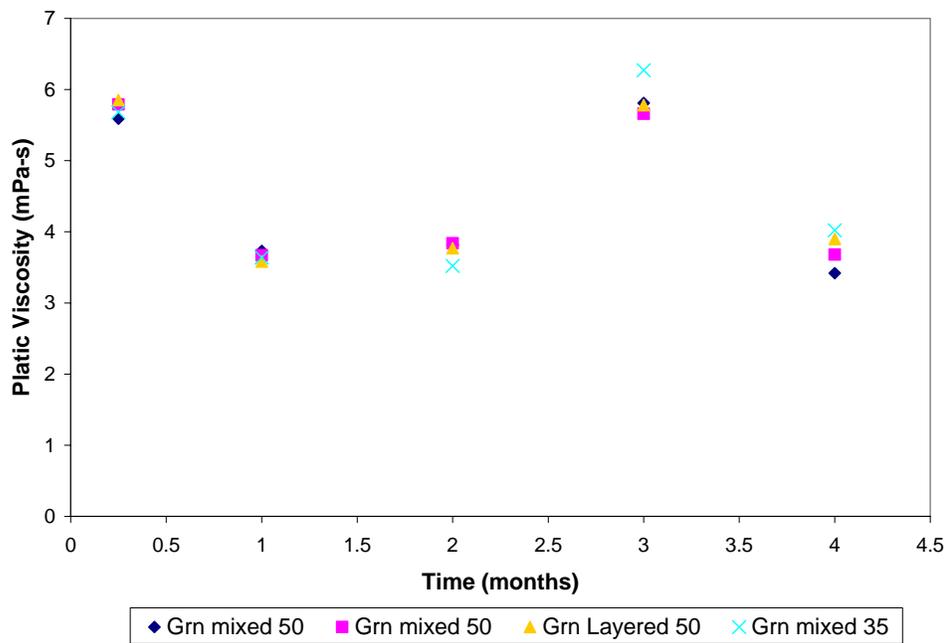


Figure 3. Plastic Viscosities of CST/Sludge Slurries in Washwater Simulants

For the samples at the three and four month duration, liquid samples were submitted for elemental analysis for CST components (i.e., Si, Ti, Zr, and Nb) and cesium. The purpose of these samples was to determine if the CST was degrading (or dissolving) and whether the absorbed cesium was desorbing from the resin. Table 2 provides the measured values of the selected elements for the two control samples (i.e., sludge alone and ground CST in 1 M sodium hydroxide solution) and the seven CST/Sludge/Simulant slurries.

There appears to be little degradation of the CST material in that little of the IONSIV IE-910 elements are detected (Si, Ti, Nb). Silicon is detected at concentrations as high as 35 mg/L in the filtered supernate from Test No. 7. However, it should be noted that the washwater and supernate simulants as prepared should have 0.004 M Si or 112 mg/L. Silicon loss may have been from formation of aluminosilicates. The binder material (Zr) in the CST (IONSIV IE-911) was measured in the supernate-containing samples but not in the washwater-containing samples.

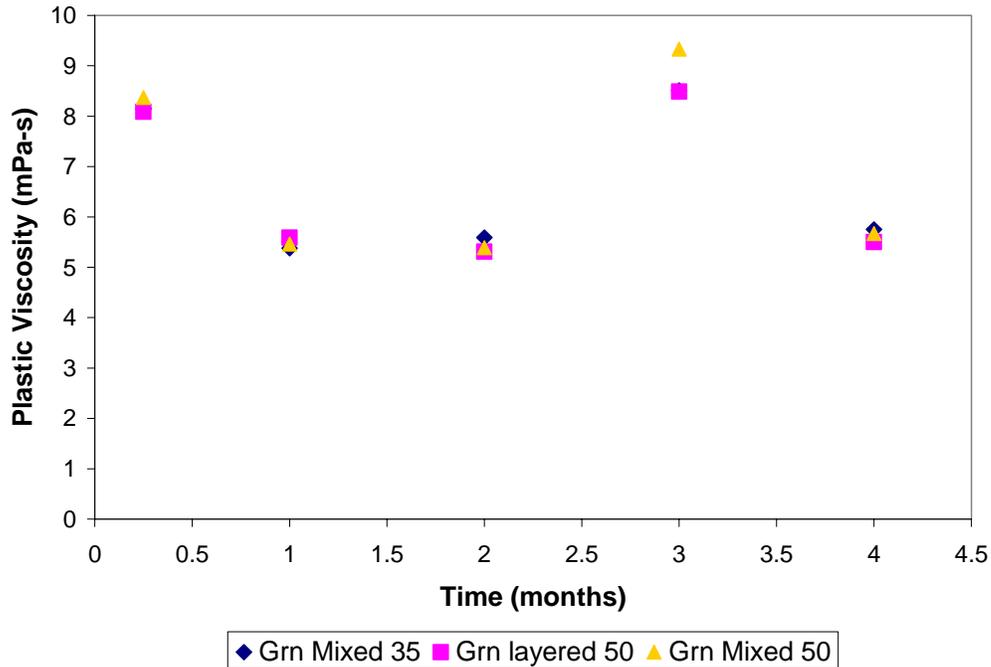


Figure 4. Plastic Viscosities of CST Sludge Slurries in Supernate Simulants

Table 2. Elemental Analysis of CST/Sludge Testing

Test No.	1	2	3	4	5	6	7	8	9
Fluid	Sludge	CST	Washwater	Washwater	Washwater	Supernate	Supernate	Supernate	Washwater
Temp (°C)	50	50	50	50	50	50	35	50	35
Mixing			Mixed	Mixed	Layered	Mixed	Mixed	Layered	Mixed
3 month									
Si (mg/L)	< 4	33	7.5	7.8	8.5	17.5	35	21	5
Ti (mg/L)	< 0.3	0.14	< 0.1	< 0.1	< 0.1	0.1	< 0.1	< 0.1	< 0.1
Zr (mg/L)	< 0.3	0.52	< 0.1	< 0.1	< 0.1	1.3	1.1	0.6	0.1
Nb (mg/L)	< 0.7	2.2	< 0.14	< 0.14	< 0.14	< 0.14	< 0.14	< 0.14	< 0.14
Cs (mg/L)	0.1	0.64	0.7	0.6	0.74	2.5	1.7	4.1	0.5
Cs-137 (Ci/gal)	0.011	0.071	0.077	0.066	0.082	0.276	0.188	0.453	0.055
4 month									
Si (mg/L)	0.65	29	6.5	6.7	5.6	15	34	4.4	4.6
Ti (mg/L)	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06
Zr (mg/L)	< 0.1	0.4	< 0.1	< 0.1	< 0.1	1.1	1.03	< 0.1	< 0.1
Nb (mg/L)	< 0.1	1.5	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5	< 0.1	< 0.1
Cs (mg/L)	< 0.1	0.24	0.255	0.27	0.24	1.32	1.21	1.3	0.235
Cs-137 (Ci/gal)		0.027	0.028	0.030	0.027	0.146	0.134	0.144	0.026

Cs-137 values are calculated.

As for the desorption of cesium from the CST resin, Table 2 shows the measured cesium concentration in the liquid phase from each sample. The CST used in these tests had been preloaded with cesium and equilibrated the CST with a solution of 2 g/L cesium nitrate in 1 M sodium hydroxide for 1 week at a liquid to volume ratio of 10. Also included in Table 2 are calculated Cs-137 activities. These activities are calculated in units of Ci/gallon as specified by assuming that one third of the soluble cesium is Cs-137 and a specific activity of 87 Ci/g.²⁴

The calculated Cs-137 activities resulting from desorbed cesium from the CST range from very low levels (~ 0.03 Ci/gal) as in Test No. 9 with washwater and 35 °C to high levels (~ 0.5 Ci/gal) as in the supernate Test No. 8 at 50 °C. At first glance, this phenomenon shows significant need for downstream processing of several of these matrices to remove that desorbed cesium prior to disposal in SRS Saltstone. An artifact of this testing is that the simulants did not contain cesium in their compositions. In actual operation, the supernate or washwater would contain soluble cesium and an equilibrium would be established between the cesium bound in the CST and the soluble cesium concentration in the aqueous phase. In the actual waste case, the presence of cesium, the equilibrium shift will limit the amount of cesium that desorbs. If disposal of the CST is to a sludge tank, the desorbed cesium contained in the washed sludge tank will be transferred to DWPF.

CST Affinity for Hazardous Metals and Actinides

The first subtask of this portion of the study was to examine the fate of RCRA metals upon contact with the crystalline silicotitanate resin. Work by Marsh²⁵ with some early batches of Sandia National Laboratory-produced crystalline silicotitanate showed some affinity for metals other than cesium in batch equilibrium contacts. Divalent metals such as strontium, cobalt, zirconium and manganese were sorbed slightly. Trivalent metals such as americium, cerium and iron also showed affinity. Other than chromium, the RCRA metals were not examined. In order to dispose of the spent CST material following treatment of SRS liquid waste, it must be known whether the material is hazardous from the RCRA perspective.

A column test was performed which passed a very large quantity (~ 2000 column volumes) of simulated dissolved saltcake loaded with the RCRA hazardous metals at levels previously observed in actual dissolve SRS saltcake. Table 3 shows the results of a modified Toxic Chemical Leaching Procedure test on the spent, washed CST. The modification was simply a reduction in the amount of solids in the TCLP test while keeping the solid to liquid ratio constant. Three values are shown in the table for each RCRA metal (Ag, As, Ba, Cd, Cr, Hg, and Pb; Se was also requested). These values are the RCRA Limit concentration, measured TCLP results as an average of three loaded CST analyses, and the result from a single TCLP test of as-received CST.

Only barium and chromium were above the detection limit in the liquid phase of the TCLP leachate and had values of 0.67 ± 0.15 and 0.43 ± 0.17 mg/L, respectively. Although measurable, these metals (Ba and Cr) were at least a factor of 10 below the RCRA limit concentration. The remaining metals (Ag, As, Cd, Hg, Pb, and Se) were below their detection limit. These results indicate strongly that spent CST resin following cesium removal operations with SRS dissolved saltcake will be non-hazardous with respect to RCRA. This allows more flexibility in determining the ultimate disposal location.

Table 3. TCLP Results of Spent CST Resin

Analyte	RCRA Limit (mg/L)	TCLP Result (mg/L)	CST Blank (mg/L)
Ag	5	< 0.3	< 0.3
As	5	< 0.045	< 0.045
Ba	100	0.67 ± 0.15	0.95
Cd	1	< 0.02	< 0.02
Cr	5	0.43 ± 0.17	< 0.05
Hg	0.2	< 0.11	< 0.11
Pb	5	< 0.8	< 0.8
Se	1	< 0.045	< 0.045

The TCLP test only examines the environmental hazard of the spent material for the leaching behavior of each of the hazardous metals. It is not intended to determine the loading of each metal onto the CST resin. Following the column test using a large volume of simulated waste, the CST resin was digested and analyzed for the amount of RCRA metal. If one assumes that the large volume of waste was sufficient to fully load the resin, then the measured values indicate the CST capacity in that given chemical matrix.

Table 4 shows the results of triplicate analyses of the spent CST resin along with a single measurement of as-received CST resin. All of the RCRA metals were absorbed onto the CST resin to some degree. Selenium analysis was complicated by a spectral interference that was not resolved in this testing and the selenium capacity is not reported. As expected, divalent metals are absorbed well by the CST resin. Lead was the metal with the highest capacity approaching 6 mg Pb/g of CST with barium and mercury below a concentration of 1 mg/g of resin. Chromium is also absorbed by the CST with a capacity approaching 1 mg/g. However, in general, these loadings are low indicating these species would stay in the liquid phase and be disposed in Saltstone.

Another aspect of the use of CST in the processing of cesium-containing solutions is the fate of the actinides in those solutions. As previously mentioned, several authors have studied the actinide affinity for the CST resin. In general, the affinity is low with typical batch distribution coefficients, K_d 's, on the order of 20 to 200 mL/g. The test was designed to determine the plutonium loading on spent CST in order to ascertain whether the CST is transuranic, i.e., Pu activity > 100 nCi/g of CST. Additionally, the uranium loading is important to understand with regards to criticality safety when processing enriched uranium-containing solutions. Therefore, testing was conducted

Table 4. Hazardous Metals Capacity for CST

Analyte	Effective Capacity (µg/g)	CST Blank (µg/g)
Ag	< 60	< 60
As	10.8 ± 0.1	10.7
Ba	607 ± 96.5	27
Cd	23.2 ± 9.9	11.7
Cr	775 ± 122	44
Hg	209 ± 33.3	21.3
Pb	5940 ± 304	427
Se	NM	NM

NM: Selenium was not measured due to interference.

whereby ~ 3000 column volumes of a dissolved saltcake simulant was passed through a small CST column and the spent CST resin was digested and analyzed for its actinide content. In this testing, the actinide-containing simulant was prepared and allowed to age for one month to ensure that the actinides were not super-saturated prior to passing through the column.

At the conclusion of the actinide run, the column was washed with 200 column volumes of inhibited water to remove all traces of the actinide-containing dissolve saltcake simulant. Triplicate samples were submitted following a nitric acid microwave digestion for plutonium alpha measurement and uranium concentrations by inductively coupled plasma – mass spectrometry. The plutonium results showed an average plutonium-239 activity of 4062 ± 131 nCi/g of CST resin. This shows the CST to be transuranic under these conditions. Therefore, any disposal option for the spent CST resin outside of the DWPF sludge option must take this categorization into consideration, such as disposal in the Waste Isolation Pilot Plant (WIPP). Contrary to the plutonium loading, the uranium loading was low and averaged $360 \mu\text{g/g}$ of CST. At this level, it is doubtful that the uranium would pose a criticality hazard.

CONCLUSIONS

The use of crystalline silicotitanate to remove radioactive cesium in a polishing step prior to final disposal of waste solutions stored at the Savannah River Site is technologically feasible. This work supports that effort by examining the chemical and radionuclide content of the spent material to assist in identifying potential disposal pathways. Additionally, the study show no measurable effects of storing CST mixed with sludge and washwater or supernate for periods up to 4 months and temperatures up to 50°C . A small desorption of cesium from the CST resin was observed in these tests.

Column ion exchange testing to determine whether the spent CST was either hazardous according to the RCRA requirements or transuranic was conducted. The results showed that the spent resin was not RCRA hazardous but exhibited plutonium concentrations well exceeding the TRU limit for disposal as low level waste. In addition, the spent resin was found to contain very minor amounts of uranium and several RCRA metals showed affinity for the CST resin.

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²⁵ S. F. Marsh, Z. V. Svitra, and S. M. Bowen, "Distribution of 14 Elements on 63 Absorbers from Three Simulant Solutions (Acid-Dissolved Sludge, Acidified Supernate and Alkaline Supernate) for Hanford HLW Tank 102-SY," LA-12654, Rev. 1, August 1994.