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Separation Science and Technology
Publication details, including instructions for authors and subscription information:
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To cite this Article Tranter, T. J. , Tillotson, R. D. and Todd, T. A.(2005) 'Evaluation of Ionsiv™ IE-911 as a Cesium Removal Option for Ineel Acidic Tank Waste', Separation Science and Technology, 40: 1, 157 — 170

To link to this Article: DOI: 10.1081/SS-200041902

URL: http://dx.doi.org/10.1081/SS-200041902

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Separation Science and Technology, 40: 157–170, 2005
ISSN 0149-6395 print/1520-5754 online
DOI: 10.1081/SS-200041902

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Evaluation of Ionsiv^{TM} IE-911 as a Cesium Removal Option for Ineel Acidic Tank Waste

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Abstract: Preliminary tests have been performed with a candidate ion exchange material in order to evaluate the efficacy for removing cesium from simulated Idaho National Engineering and Environmental Laboratory acidic tank waste. The ion exchanger being investigated is the inorganic material crystalline silicotitanate (CST). CST is available commercially in engineered form as IONSIV^{TM} IE-911. Equilibrium and dynamic testing with the CST demonstrated it to have adequate selectivity and capacity for removing cesium from the complex acidic waste matrix. However, to improve the stability of the CST during a continuous column operation, it was necessary to reduce the acidity of the feed to approximately 0.5 M HNO_3. This partial neutralization step was accomplished using 50 wt % NaOH. Formation of a visible precipitate was not observed during this process, and subsequent particle analysis confirmed the absence of any additional particle formation above 0.4 micron. CST was shown to exhibit reasonable, but relatively low, loading capacities for cesium based on a loading per unit mass of exchanger. However, the maximum allowable radiation dose for the spent ion exchanger shipping containers is expected to be the limiting value for cesium loading on the bed material. Therefore, it is anticipated that the candidate ion exchange material would provide acceptable or excess cesium removal capacity.

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Work performed for the U.S. Department of Energy under Contract No. DE-AC07-99ID13727.

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INTRODUCTION/HISTORY

The Idaho National Engineering and Environmental Laboratory (INEEL) currently stores approximately 3.4 million L (900,000 gall) of high-activity liquid waste in underground stainless steel tanks. This waste is predominately acidic aqueous waste resulting from the reprocessing of nuclear fuel from both naval and test reactors. Due to a solvent decontamination wash employed in the extraction process, this waste is also very high in sodium and is often referred to as sodium-bearing waste (SBW). Multiple technologies for treating and removing the liquid waste remaining in several of these tanks are being investigated. As one of these candidate technologies, the cesium ion exchange (CsIX)/Stabilization option would convert liquid SBW into a contact-handled solidified waste that would meet waste acceptance criteria (WAC) for disposal at the Waste Isolation Pilot Plant (WIPP). Liquid SBW would be transferred from Tank Farm Facility (TFF) tanks by existing steam jets to a receiving tank and processed. The liquid SBW would be filtered, passed through ion exchange columns to remove cesium, and then stabilized in a solid form via grouting or absorption on silica gel. Solids would be transferred from the TFF tanks to a solids tank in the treatment facility and processed intermittently over the same time period as the liquid in separate equipment. Solids that are entrained in the liquid waste would be separated, combined with other solids that have settled to the bottom of TFF tanks, and processed separately into a remote-handled waste that meets the WIPP WAC.

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For the solidified SBW to be contact handled, dissolved ^{137}Cs and solid particles, because they contain ^{137}Cs, must be removed from the liquid tank waste. Cesium removal efficiencies of 99.9% have been demonstrated with SBW simulant in small-scale ion exchange tests (1, 2). Assuming an ion exchange removal efficiency of 99.9% for the full-scale treatment facility and the average waste composition, a solids removal efficiency of greater than 92.5% would be required to ensure that the grouted SBW has a surface dose rate less than the contact-handled limit of 200 mREM/hour. Removal of cesium by ion exchange also results in a secondary waste of spent ion exchange material that must be treated and packaged as a remote-handled waste.

The high-acidity, radiation, and ionic strength of the liquid tank waste make it very problematic for treatment via conventional ion exchange resins. Consequently, a sorbent or ion exchange material designed for use with this waste stream must be unique in that it must have a very high selectivity for cesium relative to competing metals, as well as good stability in high radiation and acid systems. Inorganic ion exchange materials are usually superior in their ability to satisfy these criteria. However, inorganic ion exchange media typically exist as fine powders, making large-scale use impractical, unless the media can be affixed to an appropriate matrix. The most common matrices utilized for the support structure are organic polymer materials. Supports of this type are very porous and provide good diffusion and hydrodynamic properties. Unfortunately, they are not compatible with high radiation fields or acceptable as final waste forms due to the potential for hydrogen gas generation resulting from radiolytic degradation of the organic matrix.

Crystalline silicotitanate (CST) is a totally inorganic ion exchange material marketed commercially by UOP Inc. This material was originally developed at Sandia National Laboratory, in collaboration with Texas A&M University, for cesium and strontium removal applications in alkaline systems (3). However, previous work has shown that it also has a removal affinity for cesium in acidic systems (2, 4). CST is primarily composed of compounds of Zr, Nb, Ti, Si, and Na and can be purchased in particle sizes engineered for use in a packed column. Scanning electron microscope (SEM) micrographs of the CST material at two different magnifications are shown in Fig. 1.

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EXPERIMENTAL

Waste Tank Simulant

The testing described in this report was performed with a simulant solution in lieu of actual tank waste in order to facilitate testing without the necessity of a hot cell. This simulant was prepared using the major constituents of actual WM-189 tank waste. The WM-189 composition was chosen because it is believed to represent the worst case scenario in terms of acidity and potential interfering metals. The composition of the WM-189 simulant is shown in Table 1.

Neutralization Studies

Prior research has shown that there is some solubility of the CST in INEEL tank waste stimulant, and it was observed that degradation of the ion exchange material increased with an increase in acidity (5). Hence, it will likely be necessary to decrease the acidity of the tank waste in order to maximize stability and performance of the CST. However, there is a limit to the extent of neutralization due to the formation of undesired precipitates as the pH increases.

NaOH solution at a concentration of 50 wt % was selected for neutralization due to ease of process handling and because previous work using calcium resulted in unwanted formation of $CaSO_4$ and $CaHPO_4 \cdot 2H_2O$. A series of partial batch neutralizations was performed using WM-189 simulant

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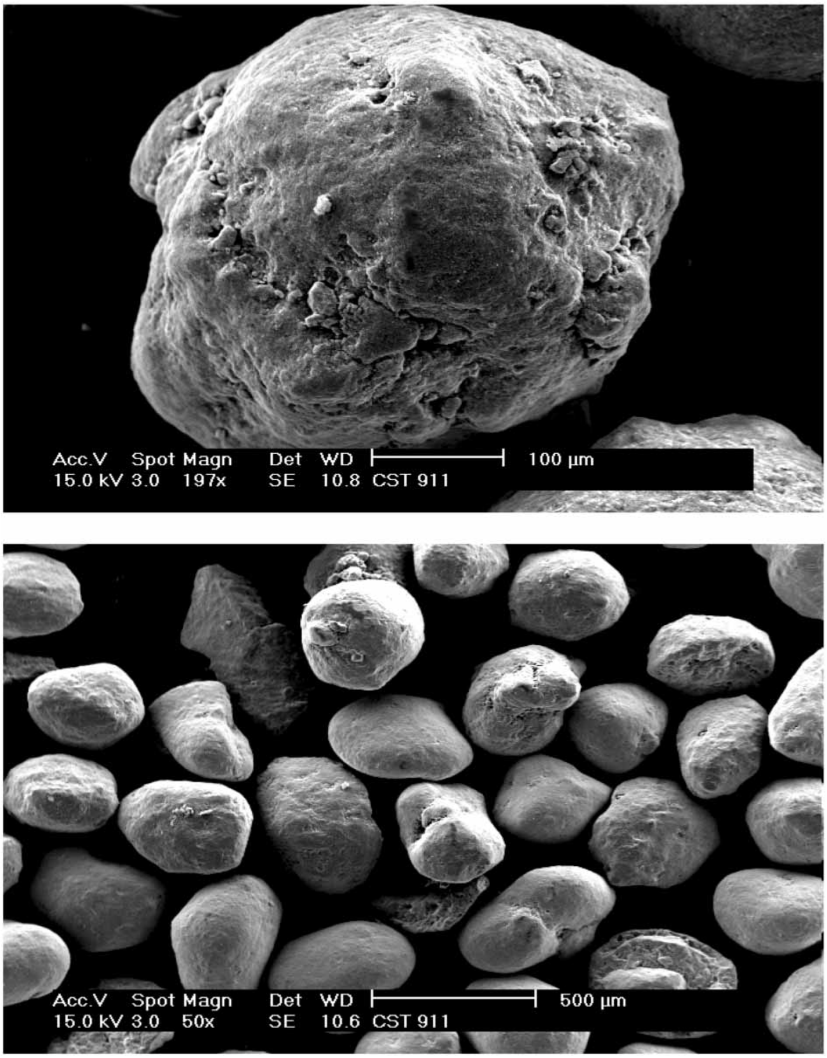

Figure 1. SEM micrographs of CST sorbent particles.

subsequent to filtering the simulant through a 0.45 micron filter. This filtering step is typically performed 24 h after simulant make-up to remove solids, which are an artifact of the simulant preparation procedure. Three aliquots of 100 mL each were removed from the filtered WM-189 and placed in glass beakers. The aliquots were then adjusted to 1.0, 0.5, and 0.2 M HNO_3 by the addition of 50 wt % NaOH. Subsequent to NaOH addition and mixing, a small sample (~0.1 mL) was removed and titrated to ensure the

Table 1. WM-189 sodium-bearing waste simulant

Species	Desired molarity	Species	Desired molarity
Al	7.11E−01	K	2.25E−01
B	2.12E−02	Mg	2.21E−02
Ca	7.30E−02	Mn	1.95E−02
Cd	3.91E−03	Na	2.04E + 00
Cl	2.06E−02	Ni	2.32E−03
Cr	5.64E−03	NO_3	6.52E + 00
Cs	2.68E−05	Pb	1.16E−03
Cu	9.54E−04	PO_4	2.07E−03
F	1.38E−02	Si	3.08E−04
Fe	2.68E−02	Sr	1.41E−04
H	2.86E + 00	SO_4	1.07E−01
Hg	6.50E−03	Zn	1.07E−03
		Zr	3.57E−04

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target acid molarity was reached. The samples then stood overnight at ambient temperature to allow sufficient time for precipitate formation. After this time period (~18 h), each partially neutralized fraction, as well as an unadjusted WM-189 feed sample, was visually inspected, photographed, and analyzed for particle size distribution via particle analyzer (Coulter Model LS230).

Equilibrium Isotherms

Batch experiments were performed with CST to determine the equilibrium cesium concentration in the solid phase as a function of equilibrium cesium concentration in the feed. A National Institute of Standards (NIST) traceable ^{137}Cs tracer was added to the WM-189 simulant to yield a feed solution of approximately 600 Bq/mL. WM-189 simulant used for the CST batch contacts was also neutralized to 0.5 M HNO_3 prior to the tests to lower the acidity due to sorbent stability concerns as described previously. The tracer was allowed to equilibrate with the stable cesium in the simulant solution for 24 h prior to beginning each experiment. The CST was washed with deionized water to remove fines and then dried to constant weight. Batch experiments were initiated by quantitatively weighing and transferring amounts of dry CST into 20 mL borosilicate glass test tubes. The particle sizes of the CST were in the range of 210–350 micron. Fifteen milliliter aliquots of feed solution were then quantitatively dispensed into each test tube containing known weights of CST. The tubes were attached to a mixing wheel (ROTO-TORQUE Model 7637-01), and the solid and liquid fractions were mixed for contact times of 48 ± 4 h at ~5 rotations per minute. The results of previous rate experiments reported for CST indicate that the 48 h contact is sufficient time for cesium equilibrium to be established between the aqueous and solid phases (6). Tests were performed at a temperature of 32 ± 3°C. After the 48 h contact period, the tubes were removed from the mixer and centrifuged for 5 min. Aliquots (5 mL) were removed from the aqueous fraction and filtered through a Gelman® Acro disk (0.45 μm) syringe filter to remove any solids suspended in the liquid. No visible suspended solids were present before or after filtering. Analyses of the aqueous feeds for total cesium and ^{137}Cs concentrations, respectively, were done via inductively coupled plasma spectroscopy (ICPS) and gamma spectrometry (Canberra® HPGE) coupled to a (SUN® Microsystems Sparc 10) computer workstation. Analysis of the equilibrated fractions for ^{137}Cs was done by gamma spectrometry. Total measurement uncertainties varied as a function of analyte concentration but were typically within ± 5%. The total cesium concentration in the equilibrated aqueous fractions was then determined by multiplying the ratio of ^{137}Cs in the equilibrated fractions and the feed by the total cesium concentration in the feed. The analyte concentration in the solid phase was calculated by material balance.

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Bench-Scale Dynamic Column Tests

In order to assess the efficacy of CST for cesium removal in a fixed-bed system, small-scale column tests were performed to obtain kinetic and loading capacity data. These data were obtained by generating breakthrough curves at various combinations of flow rate and bed volume. These column data can then be used to elucidate column capacity and kinetic parameters, which under appropriate conditions, can be used for pilot scale column design.

Breakthrough tests with simulated WM-189 tank waste feed were performed at a temperature of 32 ± 4°C. These breakthrough tests were accomplished by running the feed through the column at a constant rate and monitoring the effluent until the Cs concentration out of the column (C) was approximately equal to the Cs concentration into the column (C_0). Similar to the batch tests, radioactive ^{137}Cs (Isotope Products Inc.) was added to each of the feed solutions to bring the ^{137}Cs activity to between 600 and 1600 Bq/mL, and the spiked feed solutions were allowed to equilibrate for at least 24 h prior to testing. The WM-189 simulant used for the CST column feed was also neutralized to 0.5 M HNO_3 with 50 wt % NaOH to mitigate CST stability concerns as previously discussed. After the equilibration time, aliquots of the adjusted feeds were removed, and ^{137}Cs and total cesium were quantified by gamma spectrometry and ICPS, respectively.

The columns used for each of the tests were 9.5-mm inside diameter with a maximum possible bed height of 48 mm. The inlet, outlet, and base of the column were stainless steel with the bed portion made of transparent

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Plexiglass, which allowed visual inspection of the bed throughout the tests. The tests were performed with bed volumes ranging from 2–4 cm^3. Particle sizes of the CST were 210–350 micron. A Teflon® frit was used below and above the ion exchange bed to fix it in place. Air-free beds were obtained in each test by utilizing a careful and consistent technique for loading the ion exchange material into the column. A schematic representation of the experimental column setup is shown in Fig. 2.

The CST was washed with deionized water to remove fines and then dried to constant weight. The desired volume of dry CST was quantitatively weighed and then transferred to a column charged with dilute HNO_3. The pump and associated tubing had also been previously charged with dilute HNO_3 to remove air. The CST was allowed to gravity settle, and then the column was tapped lightly to facilitate leveling of the bed. A Teflon® frit was inserted until it came in contact with the top of the ion exchange bed, but the bed was not compressed. The columns were visually monitored during each test, and air bubbles or bed disruptions were not observed.

The feed was pumped through the columns by a walveless metering pump (FMI Lab Pump Model QVG-50). Consistent flow rates were maintained by controlling the pump with a 10-turn potentiometer (FMI Stroke Rate Controller Model V100). Specified feed rates ranged from 5 to 10 bed volumes (BV) per hour but were consistent for each test. Column effluent samples were collected at constant intervals throughout each run by using a fraction collector (Spectra-Chrom Model CF-1), which enabled discrete fraction collection at preset time intervals. The amount of ^{137}Cs in each effluent sample was measured via gamma spectrometry. Total measurement uncertainty varied as a function of analyte concentration but was typically within ±5%. The total cesium concentration in the effluent fractions was determined by

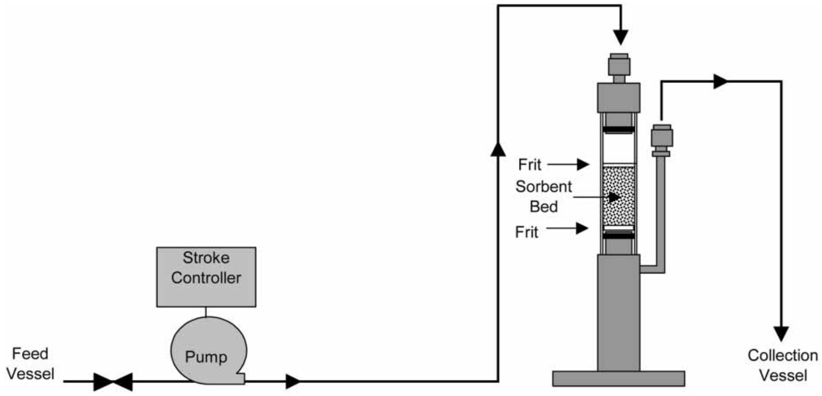

Figure 2. Diagram of experimental column and associated equipment.

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multiplying the ratio of ^{137}Cs in the effluent samples and the feed by the total cesium concentration in the feed.

Dynamic Stability Tests

These tests were performed to assess the extent of CST dissolution under flow conditions during the bench-scale column tests. These tests were designed to look for evidence of ion exchange or support material breakdown by detecting an increase of decomposition products in the column effluent.

The CST from UOP is normally in the acid form [i.e., $H_2NaSi_2(Nb_{0.3}Ti_{0.7})_4O_{13}(OH)4H_2O + 0.93Zr(OH)_4$]. The composition of this form of CST is approximately 3% Na, 17% Ti, 7% Si, 11% Zr, and 14% Nb (7, 8). In previous static testing, Nb, Ti, and Zr concentrations were measured in the aqueous phase as indicators of CST degradation. This same suite of metals was used to quantify CST breakdown in the dynamic experiments.

The extent of CST dissolution was determined using the effluent stream from the same column experiments described previously. Thus, equipment and test parameters are as defined in the Bench-Scale Dynamic Column Tests section. At the completion of each column test, an aliquot was removed from the total effluent volume and analyzed for the selected metals discussed previously. The concentration of these metals relative to any amount initially present in the feed was then used to calculate the fraction of the ion exchange material that dissolved during the test period.

RESULTS AND DISCUSSION

Neutralization Studies

The formation of a visible precipitate was not observed in any of the neutralized fractions of WM-189 simulant. The results of the particle size distribution analysis did not indicate the presence of any solids above the size detection limit of the instrument, which is approximately 0.4 micron. These data suggest that partial neutralization of the WM-189 simulant from 2.88 to approximately 0.5 molar acid does not result in any solids formation above 0.4 micron. However, the effect of partial neutralization on any solids below this size range cannot be determined from these experiments.

Equilibrium Isotherms

Values of solid phase equilibrium cesium concentration obtained from the batch contacts with CST were plotted against the corresponding equilibrium aqueous phase concentration values to generate equilibrium isotherms. Since each of the isotherm plots was of a Type I shape, a fit of the Langmuir model was performed for each of these data sets. The Langmuir equation is of the form

$$q_e = \frac{KQ_0C_e}{1 + KC_e} \tag{1}$$

where

q_e = equilibrium solid phase Cs concentration, mg/g,

C_e = equilibrium aqueous phase Cs concentration, mg/L,

Q_0 = asymptotic maximum solid phase Cs concentration, mg/L,

K = equilibrium constant, L/mg

A user-defined algorithm was used in conjunction with commercial statistical software (Table Curve®, Jandel Scientific) to fit the Langmuir model to each data set. Equilibrium data and the corresponding Langmuir fits are presented for the CST/WM-189 simulant system in Figs. 3 and 4. Figure 3 includes data at higher equilibrium values than Fig. 4, which was intended as more of a region of interest (ROI) plot. Thus, Fig. 3 gives a higher value for Q_0 due to additional experimental data points at higher feed concentrations. However, in the Cs concentration region of interest for INEEL tank waste (1–10 ppm), both curve fits predict solid phase concentrations that are in reasonable agreement.

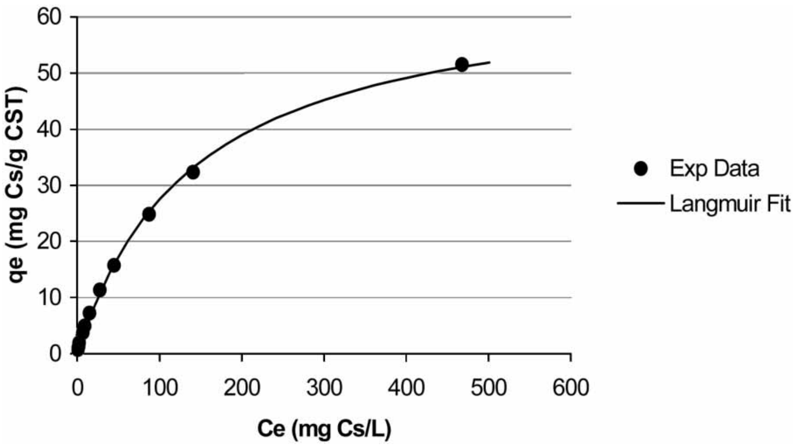

Figure 3. Isotherm for CST/WM-189 simulant system, $K = 0.007$, $Q_0 = 66.57$.

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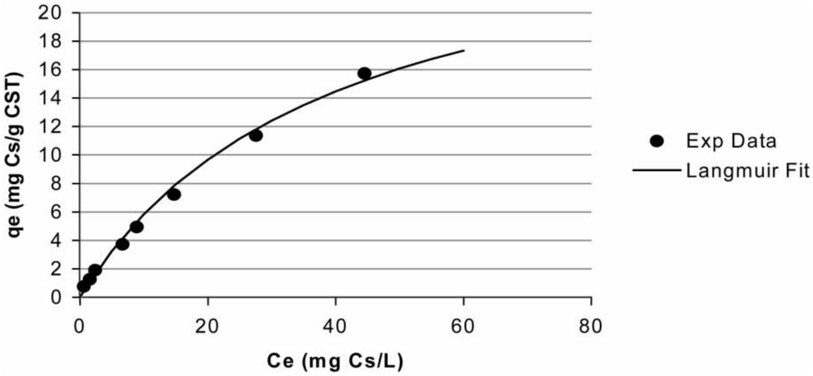

Figure 4. Isotherm for CST/WM-189 simulant system, $K = 0.025$, $Q_0 = 28.7$.

Finally, it should be noted that care was taken in each of the isotherm experiments to obtain experimental equilibrium data points that will bracket the expected Cs concentration in the actual INEEL waste tanks.

Bench-Scale Dynamic Column Tests

Breakthrough curves for each of the dynamic column tests were generated by plotting the fractional column cesium breakthrough (C/C_0) against the number of bed volumes of feed processed. Cesium breakthrough curves for the CST column tests are given in Figs. 5 and 6 for column volumes of 2 cm^3, and feed rates of 10 BV/hour and 5 BV/hour, respectively.

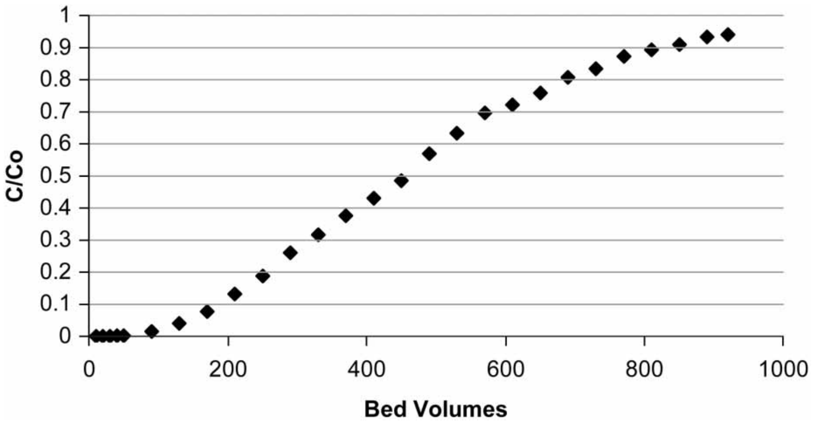

Figure 5. CST column test with WM-189 simulant. BV = 2 cm^3, flow rate = 10 BV/hour.

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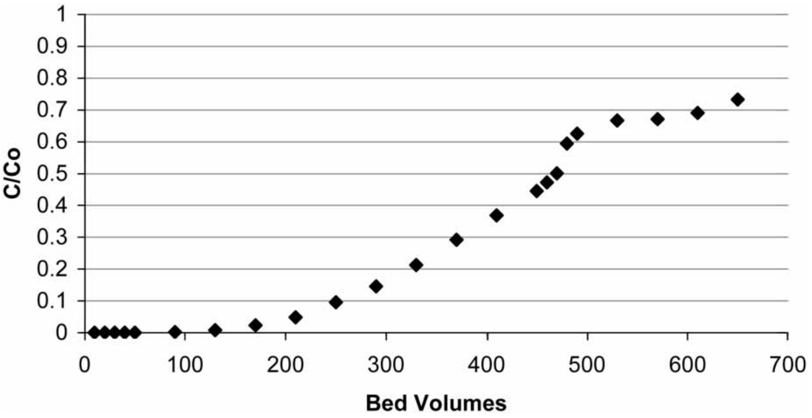

Figure 6. CST column test with WM-189 simulant. BV = 2 cm^3, flow rate = 5 BV/hour.

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The term dynamic capacity, expressed as mass of Cs exchanged per mass of ion exchanger, is used to describe the capacity of the entire column for a specified design breakthrough. For example, Cs capacity for a column considered "exhausted" at 0.1% breakthrough would be substantially different than the capacity if the column remained in service to 100% breakthrough. Dynamic capacity is determined by the following relationship:

$$DC = \int_0^v \frac{(C_0 - C)dv}{M} \tag{2}$$

where

v = volume at specified breakthrough, L,

C_0 = Cs concentration in feed, mg/L,

C = Cs concentration in column effluent at v, mg/L,

M = mass of ion exchange material, g.

A commercial statistical software package (Table Curve®, Jandel Scientific) was used to perform a "best fit" equation to each of the breakthrough curves for CST. The integral of these curves was evaluated numerically with upper limit values of v corresponding to 100% column exhaustion. This value was then subtracted from the total area to give the mass of cesium exchanged. The total cesium removed was divided by the weight of the CST to give a capacity in terms of mg Cs/g CST.

Values for C_0 and CST capacities corresponding to 100% bed exhaustion are given in Table 2. Predicted values of CST capacity calculated from the

Table 2. CST cesium capacity data

Cs Feed concentration C_0	Column capacity	Capacity predicted from langmuir isotherm (high range)	Capacity predicted from langmuir isotherm (ROI)
3.7 mg/L	1.9 mg Cs/g CST	1.7 mg Cs/g CST	2.5 mg Cs/g CST
3.7 mg/L	1.8 mg Cs/g CST	1.7 mg Cs/g CST	2.5 mg Cs/g CST

Langmuir equation coefficients presented in the previous section are also listed in the table for comparison.

The CST capacities calculated from the column tests are also in good agreement with those calculated from the isotherm data. It is recognized that in some instances the calculated equilibrium capacity of the column is slightly higher than that predicted from the isotherms. Obviously, it is not possible for column performance to exceed static equilibrium limits. This discrepancy may be an artifact of the analytical measurement uncertainty and curve fitting bias associated with each of these values. However, it may also be attributable to the fact that solid phase equilibrium changes very little with time as the value approaches 100%. Since the batch tests were approximately 48 h and the column tests were much longer ($>$100 h), it may be that the isotherm tests did not achieve the last few percent of true equilibrium. This would explain the slightly higher cesium concentrations achieved in the columns, which were operated to exhaustion and in constant contact with fresh feed for a much longer time period. In either case, the Langmuir equation coefficients derived from the fits to the isotherm data predicted the column performance very well and should be acceptable for estimating column capacity for a specified Cs concentration in the feed.

Dynamic Stability Tests

As explained previously, an estimate of bed dissolution during the dynamic column tests was derived from the increase in the effluent concentration of specific metals, which are primary constituents of the CST material. These results for each of the column tests described previously are given in Table 3.

The CST material has been shown in previous investigations to be unstable in high acid concentrations, with nearly 100% of the Zr fraction dissolving during long contact times at acid concentrations of $\geq$2 M HNO_3 (5). Thus, the values of 15% and 18.5% dissolution based on Zr are not a surprise, even though the feed was neutralized to 0.5 M H^+. Zirconium is

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Table 3. CST dynamic stability test Results

Feed rate BV/hour	Bed vol Cm^3	[H$^+$]	Dissolution based on Ti	Dissolution based on Zr
10	2	0.5	2.5%	15.2%
5	2	0.5	2.8%	18.5%

a primary constituent that binds the CST particle. Therefore, dissolution of the Zr compound would be expected to produce a proportional amount of fines in the bed.

CONCLUSIONS/RECOMMENDATIONS

Based on tests with simulated INEEL tank waste solution, this paper describes results of preliminary investigations of the CST ion exchanger material as a candidate for removing cesium from INEEL tank waste. CST has been shown to have reasonable Cs removal properties in the WM-189 simulant solution tested. It suffers from the characteristic of being unstable in acid systems. However, the bench-scale tests with CST produced acceptable results with ~17% dissolution of the $Zr(OH)_4$ binder during the column runs. Since dissolution of the Zr compound would be expected to produce a proportional amount of fines in the bed, further studies are needed to optimize the relationship between feed acid adjustment and in-service time, so that CST particle degradation is minimized.

Neutralization tests performed with the WM-189 simulant using 50 wt % NaOH did not result in the formation of a visible precipitate at acid concentrations down to 0.2 molar. Additionally, a particle analysis subsequent to partial neutralization confirmed the absence of additional particle formation above 0.4 micron. Therefore, a precolumn feed adjustment with NaOH should be a viable option for increasing the chemical stability of the CST. However, neutralization tests with actual INEEL waste tank solution should be performed to confirm the feasibility of this approach.

The maximum allowable radiation dose for the spent ion exchange shipping containers is expected to be the limiting value for cesium loading on the bed material. Thus, the results obtained in this preliminary study suggest that the candidate ion exchanger exhibits acceptable or excess cesium exchange capacity. However, a prudent risk mitigation strategy would encourage additional testing with the CST at semi- and pilot scale. It is also recommended that future investigations include small-scale testing with the CST material using actual tank waste. Although it is unlikely that any significant differences in performance will be encountered with actual waste, it is prudent to test this assumption as soon as practical.

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