Evaluation of SuperLig® 639 Ion Exchange Resin for the Removal of Rhenium from Hanford Envelope A Simulant

by

W. D. King

Westinghouse Savannah River Company

Savannah River Site

Aiken, South Carolina 29808

D. J. McCabe

N. M. Hassan

R. Hayden (Contact)

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DOE Contract No. DE-AC09-96SR18500

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from Hanford Envelope A Simulant**

April 13, 2000

W. D. King, 773-42A ^{WDK}
D. J. McCabe, 773-42A ^{DJM} ^{STW}
N. M. Hassan, 773-A ^{NMH} ^{SS}

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Savannah River Technology Center
Westinghouse Savannah River Company
Aiken, SC 29808

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SUMMARY

Tests have been completed that provide basic information on the removal of pertechnetate ion from Hanford tank waste. This work was conducted in support of the Hanford River Protection Project for BNFL, Inc. SuperLig[®] 639 ion exchange resin has been evaluated for the removal of perrhenate ion from Hanford Envelope A simulant. Perrhenate ion was used as a non-radioactive substitute for pertechnetate ion which is the major form of technetium found in actual Envelope A waste samples. Batch contact and column experiments have been performed using SuperLig[®] 639 resin and 5 M [Na⁺] simulant. The tests were highly successful in demonstrating the effectiveness of this method for removing pertechnetate from Hanford tank waste. These results will provide a critical link between small-scale radioactive tests and pilot-scale simulant tests. Results of testing at each scale will serve as input for the development of an ion exchange model which will be used to predict resin performance in the plant operation. No significant problems were encountered which would prevent use of this resin as planned. Further testing is needed to evaluate methods for reducing the volume and duration of elution.

Although all resin batches satisfy the current BNFL design criteria, significant variability was observed in the capacities of the three batches of SuperLig[®] 639 resin received. Distribution coefficients (K_d) measured in batch contact tests for the "as-received" resins varied by a factor of 2.5 (measured K_d range at 7 mg/L initial [Re]: 200-500 mL/g). Equilibrium adsorption isotherms were determined for two resin batches by varying the initial rhenium concentration. The rhenium breakthrough in column experiments with different resin batches was consistent with the lambda values predicted from the K_d data. (Note: All solutions were passed through the ion exchange columns in the downflow direction.) The 50% Re breakthrough point ranged from 90 to 150 resin bed volumes (BV) in tests performed with a 2.7 cm ID column and a superficial velocity near 0.5 cm/min ([Re] in feed: 13-16 mg/L) depending on the batch of resin tested. For resin batch #980624001DC experiments were performed in a 2.7 cm ID column at three flow rates chosen to span the range of superficial velocities that are planned for facility operation. An additional experiment was conducted using the same sample of resin in a 4.0 cm ID column in order to explore the effects of column geometry on resin performance. At a superficial velocity near 0.5 cm/min, a 39% decrease in the volume of solution processed to reach 50% rhenium breakthrough was observed for the larger diameter column. This result was expected since the residence time and path length (resin bed height) were shorter in the larger diameter column. In the 2.7 cm ID column, floating of the SuperLig[®] 639 resin bed during the feed cycle was not significant ($\leq 5\%$ floating in 5 M [Na⁺] simulant for all resin batches tested). There were no observations of cracking or swelling of the resin bed and no color or fines were observed in the effluent. With the 4.0 cm ID column (batch #980624001DC), as much as 25% of the resin bed floated (probably due to less extensive bridging of the resin beads in the larger diameter column) before the bed was retained with quartz wool and glass beads. Another important objective of this work was to determine the appropriate fluid to use for column elution. Columns were eluted with both 0.5 M nitric acid and deionized water. Deionized water was found to elute the rhenium more quickly than acid, although 16 bed volumes of effluent were required to reach the acceptance criteria (1% of C_0).

1.0 INTRODUCTION

Hanford Radioactive Waste materials have been categorized into four envelopes labeled A through D as specified in the Tank Waste Remediation Contract between BNFL and DOE.¹ Envelopes A, B and C contain only solubilized species and are specified as Low-Activity Waste (LAW). Each envelope is defined based on compositional maximums of chemical and radioactive constituents. Envelopes A and B contain low concentrations of organic species and the primary form of technetium is pertechnetate (TcO_4^-). Envelope C contains higher levels of organic species and technetium which is primarily in the nonpertechnetate form (presumably complexed TcO_2). Envelope D is sludge which has been separated from the supernate and is referred to as High Activity Waste. The current plant design utilizes SuperLig[®] ion exchange resins to remove cesium and technetium (the primary radioactive constituents) from the Hanford LAW. The process is designed to produce a decontaminated waste stream and a concentrated eluate waste stream for vitrification into low and high activity glasses, respectively.

The conceptualized process for pretreatment of Hanford LAW utilizes SuperLig[®] 639 ion exchange resin (a proprietary material) for the removal of Tc-99 (pertechnetate form only). SuperLig[®] 639 is an elutable, organic resin prepared by attaching a highly-selective ion exchange material to preformed polystyrene beads. The resin removes a salt pair (e.g. NaTcO_4 and NaNO_3) from acidic or basic solutions. Nitrate anion concentration in the solid is inversely related to the adsorption of pertechnetate from the contacting solution. Other anions typically present in Hanford waste solutions (e.g. Cl^- , F^- , CO_3^{2-} , NO_2^- , OH^- , SO_4^{2-}) do not significantly affect the adsorption of pertechnetate by SuperLig[®] 639 resin.

As shown in Figure 1, the current phase of the Hanford River Protection Project includes ion exchange testing of actual samples or nonradioactive simulants of each LAW envelope (A, B and C) at the Savannah River Technology Center (SRTC) and the Pacific Northwest National Laboratory. This report describes the intermediate-scale tests performed on simulated Envelope A solutions at SRTC. The principal objective of these intermediate-scale tests (resin bed volume: ~50 mL) was to provide data for computer modeling, allowing comparison of small-scale radioactive tests (resin bed volume: ~6 mL) to pilot-scale simulant tests (resin bed volume: 1 L). The superficial velocities studied span the range of velocities planned for the radioactive and simulant tests. The residence time, i.e., time that the liquid is in contact with the resin, is the same for the small-scale radioactive and large-scale simulant tests, as well as the design basis for the full-scale waste treatment plant. The intermediate-scale tests allow evaluation of the effects of residence time, superficial velocity, and path length by varying the feed flow

Figure 1. SuperLig[®] Ion Exchange Resin Testing Summary - SRTC = Savannah River Technology Center, Aiken, SC; PNNL = Pacific Northwest National Laboratory, Richland, WA; BNFL = British Nuclear Fuels Limited, Inc., Richland, WA; IBC = IBC Advanced Technologies, American Fork, UT; VERSE = ion exchange modeling program available from Prof. Linda Wang, Purdue University.

rate and resin bed geometry, respectively. In addition, the equilibrium capacities of the resin batches were determined to evaluate production batch variability. The rhenium equilibrium batch contact and column data serve as a basis set for the development of an ion exchange model to predict SuperLig[®] 639 column performance in large-scale plant operations designed for the removal of technetium from actual waste solutions. Data from the small-scale radioactive and pilot-scale simulant tests will be used to further refine the model. Results of this modeling will be available in future reports. Another important objective of this work was to determine the appropriate fluid to use for column elution.

Use of perrhenate ion to mimic pertechnetate ion has been previously studied.² While the two ions are not identical, they are sufficiently similar to provide a basis for comparison. The tetrahedral geometries of oxygen atoms in these two species result in nearly identical ion sizes. All isotopes of technetium are radioactive, and use of rhenium as a non-radioactive substitute is significantly less expensive and generates no radioactive waste.

2.0 EXPERIMENTAL

2.1 Materials

Three batches of SuperLig[®] 639 resin were received from IBC Advanced Technologies, American Fork, Utah. All resin samples were composed of tan, spherical beads and were very similar in appearance. Some physical data reported in an earlier document for the 639 resins is provided in Table 1.³ Batch contact tests were conducted for all three resin batches. Most of the column experiments were conducted using the highest density batch of resin (#980624001DC). One additional test was conducted using an equal volume mixture of the two remaining batches.

Table 1. Physical characteristics of "as received" SuperLig[®] 639 resins

Batch #	Resin particle density (g/mL)	Bulk dry density (g/mL)	Water content (%)
980624001DC	1.219	0.489	1.4
981015DHC720011	1.147	0.468	1.3
990420DHC720067	-----	0.479	-----

The Envelope A simulant composition given in Table 2 was based on Hanford Tank 241-AN-105.⁴ The target composition contains approximately 5.0 M [Na⁺], 1.3 M [NO₃⁻], and 1.6 M Free [OH⁻]. The simulant was prepared in 2-L batches and allowed to sit overnight before being filtered through a 0.45 micron Nalgene[®] nylon filter. For the column experiments, numerous 2-L batches of simulant were composited in a large carboy. The simulant was periodically inspected and refiltered when solids were observed. The measured simulant density was 1.23 g/mL. In column experiments, the first 750 mL of simulant exiting the column was discarded and the remainder of the solution was reused for subsequent tests after respiking the solution with sodium perrhenate. The first 750 mL of simulant was discarded because it contained approximately 1 BV of sodium hydroxide solution, which, if mixed with the effluent simulant, would have changed the composition and prevented reuse for subsequent experiments. Freshly-prepared 2 L batches of simulant were added to maintain the volume needed to complete subsequent experiments. The density of the composited simulant was measured periodically to confirm that the bulk composition had not changed significantly. All solutions were prepared using ACS certified, high-purity reagents from Fisher Scientific, Inc. Analysis of the composited simulant prior to use confirmed the basic composition given in Table 2.

Table 2. 241-AN-105 Env. A Simulant Composition

Chemical	Molarity
NaNO ₃	1.16
KNO ₃	0.09
NaNO ₂	1.13
NaOH	1.62
NaAlO ₂	0.69
NaCl	0.12
Na ₂ CO ₃	0.10
Na ₂ SO ₄	3.8 E-03
Na ₃ PO ₄	0.003
Na ₂ SiO ₃	0.004
K ₂ MoO ₄	4.02 E-4
NaReO ₄	7.4 E-05
H ₃ BO ₃	0.002
NaHCOO	0.03
NaCH ₃ COO	0.02
Na ₂ C ₂ O ₄	0.003
HOCH ₂ COOH	0.01

Note: The average Tc-99 concentration reported for the actual AN-105 sample tested at SRTC was 4.4 E-05 M.⁵

2.2 Equipment and Procedures

2.2.1 Batch Contact Experiments

Batch contact tests were performed in duplicate at ambient temperature using a Thermolyne orbital shaker. In a typical experiment ion exchange resin (0.10-0.19 g) and simulant (10-19 mL) were transferred to a 21 mL polyethylene vial and placed on the shaker. The ratio of solution volume to exchanger mass was typically 100 ± 5 . The temperature was measured at the beginning and the end of each experiment (observed range: 19 - 22 °C). After 24 ± 0.5 hours, samples were removed from the shaker and visually inspected. In all cases the resin appeared to be intact indicating that agitation of the vials during the test did not lead to significant material degradation. The amounts of resin floating, suspended and sinking were recorded for each sample. The solution was then filtered through a 0.45 micron Nalgene[®] nylon filter using vacuum. Portions (2-4 mL) of the filtrate and similarly-treated samples of the feed solutions (blanks) were analyzed for rhenium by the Savannah River Technology Center Analytical Development Section using ICP-MS. Data for the batch contact tests are provided in Attachment 1.

Re distribution coefficients and % removal were calculated for each experiment using the formulas shown in Eqs. 1 and 2, respectively.

$$K_d = [(C_i/C_f) - 1][V/(M \cdot F)] \quad (1)$$

$$\% \text{ removal} = \frac{(100) \cdot (C_i - C_f)}{C_i} \quad (2)$$

C_i = initial [Re] in feed (mg/L)

C_f = final [Re] after contact (mg/L)

V = volume of solution used (mL)

M = mass of "as-received" resin (g)

F = resin dry weight correction factor

F-factors were determined at SRTC to be 98.6% for batch #980624001DC and 98.7% for batch #981015DHC720011 (Table 1). An average F-factor of 98.7% was used to calculate the distribution coefficient for batch #990420DHC720067 since the water content for this batch was not measured.

2.2.2 Column Experiments

Figure 2 shows the ion exchange column design. Columns were constructed from 2.7 and 4.0 cm ID sodium borosilicate glass tubing. Decals were affixed to the outer walls of the columns with 1mm graduations to measure the resin bed height. The outsides of the columns were coated with a layer of polyvinylchloride to reduce hazards associated with potentially pressurizing the apparatus. A 3-way, 6 mm bore Teflon[®] stopcock (#1) was attached to the bottom of the columns. The column head was attached to the column using a Rudivis ground-glass joint. Two 2-way, 6 mm bore stopcocks (#2 and #3) were attached on opposite sides of the column head to serve as feed ports. The column head also contained a pressure gauge, a pressure relief valve, and a fill reservoir that also served as a vent. Stainless steel wire screens (200 mesh) were inserted into the columns to support the ion exchange resin. Colder Products Company polypropylene quick-disconnect couplings were used to connect low-density polyethylene tubing (11/64" ID) to the columns. All solutions were passed through the columns in the downflow direction using Fluid Metering Incorporated QG150 positive displacement pumps with 1/4" and 3/8" piston sizes. Samples were collected either manually or using a Spectrum Chromatography IS-95 Interval Sampler.

Figure 2. Ion Exchange Column Design

The ion exchange column was prepared by soaking the "as-received" resin overnight in deionized water and transferring the resin into the column as a suspension. For most experiments, a 51 mL sample of resin from batch #980624001DC was used. An additional experiment was completed using 50 mL of a 50:50 mixture of batches 981015DHC720011 and 990420DHC720067 in order to model the pilot-scale column tests which were conducted using the same mixture of batches. In each experiment, the resin bed volume was determined by multiplying the measured height of the resin bed by the cross-sectional area. The temperature during the column experiments ranged from 20-23 °C. The bed was pretreated with 3 bed volumes of 1.0 M NaOH over a 1 hour period just prior to beginning each experiment (Pretreatment Cycle). The NaOH liquid level was adjusted so that the volume of liquid above the resin bed was approximately 50 mL (1 bed volume) before simulant solution was pumped into the column. Simulant (13-16 mg/L [Re]) was pumped into the column and approximately 8 mL samples of effluent were collected every 10-20 bed volumes (Feed Cycle). The liquid in the column was then drained to within approximately 1 cm of the top of the resin bed. Sodium hydroxide solution (0.1 M) was pumped into the column until the volume of liquid above the resin bed was approximately 50 mL. The exit line was opened and 2.5-3.5 bed volumes of 0.1

M NaOH was passed through the column in one hour and samples of the effluent were collected every 20 minutes (NaOH Wash Cycle). Subsequently, 2.5-3.5 bed volumes of deionized water were passed through the column in one hour and samples of the effluent were collected every 20 minutes (Post-Feed Water Wash Cycle). (Note: The water wash cycle served to displace residual sodium hydroxide solution from the column prior to elution.) The column was eluted with either deionized water or 0.5 M HNO₃ at a flow rate of 1 or 3 bed volumes per hour and samples were collected every 1-2 hours (Elution Cycle). When eluting with acid, the liquid in the column was drained to within approximately 1 cm of the top of the resin bed following the elution cycle. Deionized water was pumped into the column until the volume of liquid above the resin bed was approximately 50 mL. The column was washed with 3 bed volumes of deionized water during a 1 hour period (Post-Elution Water Wash Cycle). Feed and effluent samples were analyzed for rhenium by the Savannah River Technology Center Analytical Development Section using ICP-MS or ICP-ES. Data from the column experiments are provided in Attachment 2.

The flow rate was monitored during the column experiments by periodically measuring the time required to collect 10 or 25 mL of effluent. The effluent solution during the feed cycle was collected in 3-4 separate reservoirs. The total volume of effluent in each reservoir was measured and corrected for the volume of samples collected and the flow rate was calculated from the time of collection. The overall flow rate was taken to be the weighted average of the flow rates calculated for each collection bottle. The flow rates for the pretreatment, wash, and elution cycles were calculated from the time of collection and the volume of solution collected (after correction for the volume of samples collected). Flow rates measured during each experiment for the feed, wash and elution cycles are provided in Attachment 3. The number of bed volumes of solution processed at each sampling period during column operation was calculated from the frequency of collection and the flow rate. (Note: The column operating procedure was designed to emulate the actual plant operation, where the column is half full of resin and half full of liquid. According to the procedure, the initial simulant that was fed into the ion exchange column during the loading cycle was to be diluted by the 1 BV of 1.0 M NaOH pretreatment solution which remained above the resin bed. Likewise, the post-feed water wash and the eluting solutions were allowed to mix with the liquid head left above the resin from the previous cycle. No attempt was made to correct for mixing of solutions in the column head-space when calculating the number of bed volumes of feed, wash, or eluate processed. Each cycle was considered to start at the moment that the feed line was transferred to the new feed bottle and the pump was activated.) The resin bed volume was monitored throughout the experiments by periodically measuring the height of the bed. The volume varied by less than 5% throughout the experiments.

3.0 RESULTS AND DISCUSSION

3.1 Batch Contact Experiments

Batch contact tests were conducted for three batches of SuperLig[®] 639 resin at rhenium feed concentrations ranging from 0.25 to 225 mg/L. All tests for which the initial

rhenum concentration was below 5 mg/L were recontact tests in which the filtrate from earlier contact tests served as the feed solution. The resin particle densities measured for the SuperLig® 639 batches ranged from 1.15 to 1.22 g/mL³ while the density of 5 M [Na⁺] Envelope A simulant was approximately 1.23 g/mL. Close inspection of the samples at the conclusion of the batch contact tests revealed that some resin batches tend to float in the test solution. Varying amounts (0-50%) of the resin were observed to float in the solution, depending on the batch of resin being tested.

Distribution coefficients (K_d) provide a quantitative measure of ion exchange resin equilibrium capacity. As shown in Table 3 and Figure 3, the observed rhenum distribution coefficients varied significantly depending on the resin batch. The K_d values measured for each batch at approximately 7 mg/L initial [Re] indicate that the resin capacity varies by a factor of 2.5 with batch #980624001DC exhibiting the lowest capacity (199 mL/g) and batch #990420DHC720067 exhibiting the highest (512 mL/g). The shapes of the adsorption isotherms in Figure 3 are typical of Langmuir-type adsorption. In general, the distribution coefficients increase as the rhenum equilibrium concentration in solution decreases. At very low concentrations the equilibrium capacity may begin to decrease again as shown in the adsorption curve for batch #981015DHC720011. For each resin which was tested at various rhenum feed concentrations, the K_d values seemed to plateau across a range of rhenum equilibrium concentrations. At final rhenum concentrations ranging from 0.7 to 25 mg/L the distribution coefficients for batch #980624001DC were near 200 mL/g. At rhenum final concentrations ranging from 0.2 to 1.5 mg/L the distribution coefficients for batch #981015DHC720011 were approximately 400 mL/g. Relative to batch #980624001DC the plateau observed in the adsorption isotherm for batch #981015DHC720011 was not observed until the rhenum equilibrium concentration was considerably lower (1.5 versus 25 mg/L final [Re]). (Note: The K_d value measured for batch #980624001DC with a final equilibrium concentration of 3.37 mg/L was anomalous presumably due to experimental or analytical error.) The trends observed in the equilibrium capacities of the resin batches do not correlate with the amount of resin observed to float in the simulant solution. Only batch #981015DHC720011 exhibited a significant tendency to float (30-50% floating) in the simulant at the conclusion of the batch contact tests. As shown in Figure 3, the measured K_d 's for this resin batch were intermediate in magnitude relative to the remaining batches. This observation indicates that sufficient contact was obtained between the solid and the solution in all tests. The amount of resin observed to float in the batch contact tests is also consistent with the resin density measurements shown in Table 3.

Table 3. Rhenium Distribution Coefficients (K_d) Measured for SuperLig® 639 Resin Batches

Batch # 990420DHC720067

Initial [Re] (mg/L)	Final [Re] (mg/L)	Re K_d (mL/g)	% Re Removal	% resin floating
7.18	1.16	512	83.9	0

Batch # 981015DHC720011

Initial [Re] (mg/L)	Final [Re] (mg/L)	Re K_d (mL/g)	% Re Removal	% resin floating
0.26	0.06	351	78.5	30
1.41	0.26	420	81.3	50
6.96	1.41	390	79.8	----
72.78	16.34	338	77.5	----

Batch # 980624001DC

Initial [Re] (mg/L)	Final [Re] (mg/L)	Re K_d (mL/g)	% Re Removal	% resin floating
2.40	0.77	208	67.8	0
7.18	2.40	199	66.6	10
13.99	3.37	315	75.9	10
69.17	24.61	181	64.4	10
152.58	62.75	143	58.9	10
222.70	102.62	117	53.9	0

Figure 3. SuperLig® 639 Batch Comparison - Final [Re] versus K_d

3.2 Column Experiments

Results for the four column experiments performed with SuperLig® 639 batch #980624001DC are shown in Table 4. Three of the four experiments were completed using a 2.7 cm ID column at different volumetric flow rates (Figure 4). The breakthrough profiles follow the expected trends with faster flow rates resulting in more rapid rhenium breakthrough. Decreasing the flow rate from 73.4 to 3.4 BV/hr increases the 50% Re breakthrough point by a factor of 10. In addition, the 50% Re breakthrough point of 90 BV observed at a feed superficial velocity of 0.51 cm/min is very near the value expected based on the batch contact data. The theoretical 50% Re breakthrough point can be predicted from K_d data and the bulk resin density. At a rhenium feed concentration near 15 mg/L the estimated Re K_d for batch #980624001DC is 200 mL/g. The vendor reports the wet bulk resin density for SuperLig® 639 resin to be 0.4 g/mL. The bulk resin density for batch #980624001DC was reported by SRTC to be 0.489 g/mL.³ The number of bed volumes required to reach 50% breakthrough (λ) can be calculated using Eqn. 3.

$$\lambda = (K_d) \times (\text{bulk resin density}) \quad (3)$$

The λ value calculated for a feed concentration near 15 mg/L ($K_d = \sim 200$ mL/g) ranges from 80 to 98 BV depending on the value used for the bulk resin density. The observed 50% Re breakthrough point of 90 BV falls within this range, indicating that Re adsorption onto the resin approaches the equilibrium capacity at 3 BV/hr. Operating the plant at this flow rate would fully utilize the resin capacity to process the maximum volume of waste before reaching the 50% Re breakthrough point. The time required for column loading would be maximized and the frequency of elution and regeneration would be minimized relative to operating at faster flow rates. The maximum flow rate

Table 4. SuperLig® 639 Column Re Breakthrough Summary

Batch #980624001DC, 51 mL Resin Bed

Experiment #	Bed Diameter/Height (cm)	Flow Rate (cm/min)	Flow Rate (BV/hr)	50% Breakthrough (BV)
1	2.7/9.0	0.51	3.4	90
2	2.7/9.0	5.2	34.7	30
3	2.7/9.0	11	73.4	9
4	4.0/4.1	0.48	6.9	55

50:50 Mixture of Batches 981015DHC720011 and 990420DHC720067, 50 mL Resin Bed

Experiment #	Bed Diameter/Height (cm)	Flow Rate (cm/min)	Flow Rate (BV/hr)	50% Breakthrough (BV)
5	2.7/8.8	0.43	2.9	150

Note: Experiment numbers indicate the order of completion

Figure 4. SuperLig® 639 Batch #980624001DC Rhenium Breakthrough Profiles (resin bed: 51 mL, 2.7 cm ID)

required to approach equilibrium conditions in the column should be generated by the ion exchange model which will be developed from the data reported herein.

Rhenium elution profiles for the three experiments performed with the 2.7 cm ID column are shown in Figures 4 and 5. In each case the maximum rhenium concentration was observed near the end of the water wash cycle (flow rate: 3 BV/hr) and the tail of the elution profile extended into the elution cycle (flow rate: 1 BV/hr). The profiles shown in Figures 4 and 5 start at the beginning of the water wash cycle. Experiments 1 and 3 utilized 0.5 M HNO₃ as the eluent and deionized water was the eluent for experiment 2. In each experiment the peak rhenium concentration was observed between 2-3 BV after the start of the water wash cycle. Acid significantly increases the volume of solution required to elute the column compared to water. The rhenium concentration in the eluate decreased to <1% of the feed Re concentration within 28 BV when eluting with acid and within 16 BV when eluting with deionized water. (Note: Integration of the area above the loading curves for Experiments 2 and 3 in Fig. 4 indicated that Re loading on the column was nearly identical in these two experiments. Therefore, differences in Re loading on the column in these experiments does not explain the differences in the elution profiles discussed above.)

Figure 5. SuperLig® 639 Batch #980624001DC Elution Profiles (resin bed: 51 mL, 2.7 cm ID; 0-3 BV – deionized water at 3 BV/hr; 4-30 BV – water or 0.5 M HNO₃ at 1 BV/hr)

Figure 6. SuperLig® 639 Batch #980624001DC Elution Profiles – Semi-Log Plot (resin bed: 51 mL, 2.7 cm ID; 0-3 BV – deionized water at 3 BV/hr; 4-30 BV – water or 0.5 M HNO₃ at 1 BV/hr)

After completing three column experiments in the 2.7 cm ID column, the resin was transferred to a 4.0 cm diameter column (Exp. 4). A column experiment was performed at a feed superficial velocity of 0.48 cm/min, which is near the slowest velocity used in the smaller diameter column. During this experiment, it was initially quite difficult to maintain a stable liquid level above the resin during the feed cycle. These problems were not overcome until well into the experiment. On two occasions the liquid level fell below the top of the resin bed briefly. Once the liquid level was stabilized, no obvious air bubbles were observed within the bed. (Note: Floating of the resin bed was probably due to less extensive bridging of the resin beads in the 4.0 cm diameter column, although floating of the resin due to unseen trapped air bubbles cannot be ruled out.) As the simulant solution was passed through the resin bed a significant portion of the resin gradually began to float. After approximately 120 bed volumes of feed solution had passed through the column it was necessary to add quartz wool and glass beads to the top of the bed to keep the resin from floating. Figure 7 compares the rhenium breakthrough profiles observed for the experiments performed in the 2.7 and 4.0 cm ID columns at comparable superficial velocities. Increasing the column ID from 2.7 to 4.0 cm decreases the path length from 9.0 cm to 4.1 cm. The volumetric flow rate was increased from 3.4 (2.7 cm ID column) to 6.9 BV/hr in the 4.0 cm ID column in order to maintain approximately the same superficial velocity (0.5 cm/min). The breakthrough profiles observed follow the expected trend. The Re 50% breakthrough point in the 4.0 cm ID column (55 BV) was 39% lower than the breakthrough observed in the smaller diameter column (90 BV). This result was expected since the residence time and path length were shorter in the larger diameter column.

The 4.0 cm diameter column was eluted with water at a superficial velocity of 0.19 cm/min (2.7 BV/hr) which was comparable to the superficial velocity of 0.16 cm/min (1.1 BV/hr) used for the water elution in the 2.7 cm ID column. The elution profiles for the two experiments are shown in Figure 8. In general, the elution profile observed for the 4.0 cm ID column is similar to the profile observed in the 2.7 cm ID column. The maximum rhenium concentration is observed near 4 BV and the width of the elution peak at half-height is comparable to the width observed for the 2.7 cm ID column. However, the elution tail for the experiment in the 4.0 cm ID column is considerably longer than was observed for the 2.7 cm ID column. In the larger diameter column, the Re concentration after 15 BV of eluate had been collected was approximately 8% of the feed concentration. For comparison, in the smaller diameter column the Re concentration was <1% of the feed concentration after 16 BV of eluate had been collected. This observation indicates that residence time is an important processing parameter for rhenium elution. The residence time for the elution of the 4.0 cm ID column decreased relative to the 2.7 cm ID column due to the faster volumetric flow rate. Presumably, the shorter residence time resulted in a longer elution tail, despite the fact that the path length was shorter in the larger diameter column.

Figure 7. SuperLig® 639 Batch #980624001DC Rhenium Breakthrough Profiles – Column L/D Comparison (resin bed: 51 mL, superficial velocity: app. 0.5 cm/min)

Figure 8. SuperLig® 639 Batch #980624001DC Water Elution Profiles – Column L/D Comparison (resin bed: 51 mL; 4.0 cm ID Flow Rate: 0-30 BV at 3 BV/hr; 2.7 cm ID Flow Rate: 0-3 BV at 3 BV/hr, 3-30 BV at 1 BV/hr)

A column experiment was also performed using a 50:50 mixture of SuperLig[®] 639 resin batches 981015DHC720011 and 990420DHC720067 (Exp. 5). This mixture is currently being used in larger, pilot-scale simulant tests at SRTC. The predicted Re 50% breakthrough point based on equation (1) is 213 BV (estimated K_d at initial [Re] 15 mg/L: 450; Note: The K_d value was determined by extrapolating and averaging K_d data for both batches. The average bulk dry density for the mixture of resin batches 981015DHC720011 and 990420DHC720067 was 0.474 g/mL based on the data presented in Table 1). The experiment was conducted in a 2.7 cm ID column at a flow rate of 2.9 BV/hr (0.43 cm/min), which is comparable to the slowest flow rate used for batch #980624001DC. The breakthrough profile for the column containing a mixture of resin batches is compared in Figure 9 to the breakthrough profile for batch #980624001DC at a similar flow rate. The Re 50% breakthrough was observed at 150 BV for the mixture of batches, which is nearly twice the number of bed volumes at which breakthrough was observed for batch #980624001DC (Re 50% breakthrough: 90 BV). This result is qualitatively consistent with the distribution coefficient results.

Figure 10 shows the water elution profile observed for the mixture of resin batches. The column was eluted at 3 BV/hr (0.41 cm/min). The maximum rhenium concentration is observed near 5 BV. The rhenium concentration in the eluate was 22% of the feed concentration after 27 BV of eluate had been collected. Since the adsorption capacities of batches 981015DHC720011 and 990420DHC720067 were significantly different from batch #980624001DC, the elution data cannot be directly compared. However, it is apparent that elution of the mixture of resin batches 981015DHC720011 and 990420DHC720067 at a flow rate of ~3 BV/hr results in a long elution tail. Based on observations regarding the effects of residence time on rhenium elution, elution at slower flow rates would be expected to decrease the volume of eluent required.

Figure 9. SuperLig® 639 Rhenium Breakthrough Profiles – Batch Comparison (resin bed: 51 mL (Exp. 1) and 50 mL (Exp. 5); superficial velocity: 0.51 cm/min(Exp. 1), 0.43 cm/min (Exp. 5))

Figure 10. SuperLig® 639 Water Elution Profile – Mixture of Batches 981015DHC720011 and 990420DHC720067 (resin bed: 50 mL, 2.7 cm ID; Flow Rate: 3 BV/hr)

4.0 CONCLUSIONS

Simulant batch contact and column optimization tests have been completed on three batches of SuperLig® 639 ion exchange resin. Consistent results were obtained for the batch contact and column tests. At feed flow rates near 3 BV/hr the column performance of all resin batches approached or exceeded the current plant design criteria, which requires a minimum of 100 BV of feed to be processed prior to elution and regeneration. However, synthesis of the resin should be modified to increase the density to avoid floating the resin bed while processing these dense feed solutions. Additional research is also needed to identify the manufacturing variables that affect resin capacity and develop methods for its optimization. The columns were most effectively eluted with water at a flow rate of 1 BV/hr. Further work is needed to evaluate methods for reducing column elution duration and volume. These may include heating the water used for elution (as recommended by IBC personnel) or periodically stopping the flow during the elution to allow the resin to continue to desorb technetium without generating more eluate volume.

5.0 DESIGN CHECK

M. C. Thompson

4/18/00
Date

6.0 APPROVALS

Hanford Tank Waste Ion Exchange Column Optimization Task Technical and Quality Assurance Plan (U), BNF-003-98-0032, Rev. 0.

7.0 REFERENCES

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8.0 APPENDIX

8.1 Attachment 1. SuperLig® 639 Batch Contact Data.

Batch #980624001DC

Sample #	BNF-SSIX-17-7	BNF-SSIX-17-7-D	BNF-SSIX-17-5	BNF-SSIX-17-5-D
LIMS #	300127273	300127274	300127269	300127270
Solution Mass (g)	19.249	18.9075	22.022	22.0257
Resin Mass (g)	0.1587	0.1573	0.1803	0.1803
Solution Volume (mL)	15.65	15.37	17.90	17.91
Initial [Re] (mg/L)	2.37	2.42	7.18	7.18
Final [Re] (mg/L)	0.788	0.752	2.37	2.42
solution volume/exchanger mass	98.6	97.7	99.3	99.3
Temperature (C)	22	22	22	22
Shake Time	23.6 hr recontact	23.6 hr recontact	23.8 hrs	23.8 hrs
Kd (mL/g)	198.8	217.6	202.3	196.1
Duplicate Average Kd	208.2		199.2	
Duplicate Average Final [Re] (mg/L)	0.77		2.395	

Measured [Re] in Feed (mg/L)	2.37	2.42	7.18
Sample #	BNF-SSIX-17-5	BNF-SSIX-17-5-D	BNF-SSIX-7-D-1
LIMS #	300123956	300123957	300127275

Sample #	BNF-SSIX-17-1	BNF-SSIX-17-1-D	BNF-SSIX-17-2	BNF-SSIX-17-2-D
LIMS #	300126842	300126843	300126845	300126846
Solution Mass (g)	14.7093	14.7396	14.7016	14.7136
Resin Mass (g)	0.1203	0.1213	0.1198	0.1202
Solution Volume (mL)	11.96	11.98	11.95	11.96
Initial Conc. (mg/L)	13.992	13.992	69.173	69.173
Final Conc. (mg/L)	3.6199	3.1291	24.555	24.672
solution volume/exchanger mass	99.4	98.8	99.8	99.5
Temperature (C)	22	22	22	22
Shake Time	24.0 hrs	24.0 hrs	24.0 hrs	24.0 hrs
Kd (mL/g)	286.0	344.3	182.0	180.2
Duplicate Average Kd	315.2		181.1	
Duplicate Average Final [Re] (mg/L)	3.3745		24.6135	

Measured Re in Feed (mg/L)	13.992
Sample #	BNF-SSIX-P-1
LIMS #	300126844

69.173
BNF-SSIX-D-2
300126847

Attachment 1. SuperLig® 639 Batch Contact Data (cont.).

Batch # 980624001DC

Sample #	BNF-SSIX-17-3	BNF-SSIX-17-3-D	BNF-SSIX-17-4	BNF-SSIX-17-4-D
LIMS #	300126848	300126849	300126851	300126852
Solution Mass (g)	14.7095	14.7074	14.7158	14.721
Resin Mass (g)	0.1205	0.1206	0.1199	0.1205
Solution Volume (mL)	11.96	11.96	11.96	11.97
Initial [Re] (mg/L)	152.58	152.58	222.7	222.7
Final [Re] (mg/L)	61.996	63.513	102.03	103.21
solution volume/exchanger mass	99.2	99.1	99.8	99.3
Temperature (C)	22	22	22	22
Shake Time	24.0 hr recontact	24.0 hr recontact	24.0 hrs	24.0 hrs
Kd (mL/g)	145.6	139.6	118.5	115.5
Duplicate Average Kd	142.6		117.0	
Duplicate Average Final [Re] (mg/L)	62.7545		102.62	

Measured [Re] in Feed (mg/L)	152.58
Sample #	BNF-SSIX-P-2
LIMS #	300126850

222.7
BNF-SSIX-P-3
300126853

Batch #990420DHC720067

Sample #	BNF-SSIX-17-6	BNF-SSIX-17-6-D
LIMS #	300127271	300127272
Solution Mass (g)	22.0561	22.0191
Resin Mass (g)	0.1833	0.1832
Solution Volume (mL)	17.93	17.90
Initial [Re] (mg/L)	7.18	7.18
Final [Re] (mg/L)	1.12	1.19
solution volume/exchanger mass	97.8	97.7
Temperature (C)	22	22
Shake Time	23.8 hrs	23.8 hrs
Kd (mL/g)	530.9	493.3
Duplicate Average Kd	512.1	
Duplicate Average Final [Re] (mg/L)	1.155	

Measured [Re] in Feed (mg/L)	7.18
Sample #	BNF-SSIX-D-1
LIMS #	300127275

Attachment 1. SuperLig® 639 Batch Contact Data (continued).

Batch #981015DHC720011

Sample #	BNF-SSIX-10-1	BNF-SSIX-10-1-D	BNF-SSIX-10-3	BNF-SSIX-10-3-D
LIMS #	300123956	300123957	300124008	300124012
Solution Mass (g)	21.9902	21.8143	17.9917	17.5916
Resin Mass (g)	0.1809	0.1801	0.1499	0.1503
Solution Volume (mL)	17.88	17.74	14.63	14.30
Initial [Re] (mg/L)	6.9605	6.9605	1.3958	1.4198
Final [Re] (mg/L)	1.3958	1.4198	0.2632	0.26313
solution volume/exchanger mass	98.8	98.5	97.6	95.2
Temperature (C)	20	20	19	19
Shake Time	24.1 hr	24.1 hr	24.0 hr recontact	24.0 hr recontact
Kd (mL/g)	395.2	385.4	421.2	419.6
Duplicate Average Kd	390.3		420.4	
Duplicate Average Final [Re] (mg/L)	1.41		0.263	

Measured [Re] in Feed (mg/L)	6.96		
Sample #	BNF-SSIX-10-1-FD		
LIMS #	300123958		

Sample #	BNF-SSIX-10-5	BNF-SSIX-10-5-D	BNF-SSIX-10-2	BNF-SSIX-10-2-D
LIMS #	300124167	300124168	300123959	300123960
Solution Mass (g)	13.9225	13.0358	21.8462	21.8045
Resin Mass (g)	0.1113	0.1184	0.1827	0.1816
Solution Volume (mL)	11.32	10.60	17.76	17.73
Initial Conc. (mg/L)	0.2632	0.26313	72.781	72.781
Final Conc. (mg/L)	0.056	0.057	16.119	16.561
solution volume/exchanger mass	101.7	89.5	97.2	97.6
Temperature (C)	20	20	20	20
Shake Time	24.0 hr recontact	24.0 hr recontact	24.1 hrs	24.1 hrs
Kd (mL/g)	377.4	324.7	342.8	332.4
Duplicate Average Kd	351.0		337.6	
Duplicate Average Final [Re] (mg/L)	0.0565		16.34	

Measured [Re] in Feed (mg/L)	6.96
Sample #	BNF-SSIX-10-1-FD
LIMS #	300123958

72.78
BNF-SSIX-10-2-FD
300123961

8.2 Attachment 2. SuperLig® 639 Column Data.**SuperLig 639 Batch #980624001DC****Exp. 1****0.51 cm/min, 2.69 cm ID Column**

ICP-MS Analysis

Feed Cycle

Water/Acid Elution

Feed 16 mg/L Re

# BV	C/Co	# BV	[Re] (mg/L)
4.2	0.001	1.7	27.7
8.5	0.002	2.9	309.1
29.6	0.050	5.1	27.7
48.8	0.160	7.4	22.2
68.0	0.331	9.7	21.0
87.2	0.489	27.7	0.1
107.9	0.675	29.7	0.1
128.6	0.806		
149.3	0.838		
170.0	0.894		
190.7	0.906		
211.4	0.913		
226.4	0.913		
236.3	0.944		
246.1	0.956		
256.0	0.950		
265.8	0.925		
275.6	0.931		
285.5	0.925		
295.3	0.913		
305.2	0.919		

Exp. 2**5.2 cm/min, 2.69 cm ID Column**

ICP-ES Analysis

Feed Cycle

Water Elution

Feed 15.9 mg/L Re

# BV	C/Co	# BV	[Re] (mg/L)
2.3	0.103	1.2	21.800
12.2	0.328	2.3	195.000
31.5	0.540	3.6	230.000
41.7	0.697	4.8	114.000
62.2	0.774	5.9	40.400
82.7	0.801	7.1	12.400
103.2	0.847	8.2	3.500
123.7	0.850	9.3	1.600
144.2	0.869	9.4	0.630
164.7	0.924	10.5	0.463
		11.6	0.323
		12.8	0.255
		13.9	0.208
		16.1	0.145
		18.4	0.110
		20.6	0.093
		22.8	0.075
		25.1	0.059

Attachment 2. SuperLig® 639 Column Data (cont.)

Exp. 3

11.0 cm/min, 2.69 cm ID Column

ICP-ES Analysis

Load Cycle

Water/Acid Elution

Feed

16 mg/L Re

# BV	C/Co	# BV	[Re] (mg/L)
2.6	0.126	1.3	18.533
5.1	0.348	2.0	20.700
9.1	0.506	3.0	86.733
10.1	0.539	5.0	32.000
12.7	0.618	7.0	30.000
19.0	0.628	9.0	26.067
21.7	0.589	11.0	20.700
24.3	0.650	13.0	14.567
27.4	0.598	15.0	9.303
35.5	0.650	17.0	5.297
45.5	0.725	19.0	3.100
55.6	0.800	21.0	1.510
65.6	0.781		
75.7	0.756		
85.7	0.775		
95.8	0.800		
105.8	0.825		
125.9	0.794		
146.3	0.813		
166.8	0.850		
187.2	0.844		
207.6	0.875		
289.3	0.925		
330.1	0.881		

Exp. 4

0.48 cm/min, 4 cm ID Column

ICP-ES Analysis

Load Cycle

Water Elution

Feed

14 mg/L Re

# BV	C/Co	# BV	[Re] (mg/L)
0.3	0.048	1.0	34.0
0.4	0.004	2.0	39.0
11.3	0.065	3.0	116.0
22.0	0.273	4.0	327.0
32.5	0.312	5.0	242.0
43.0	0.403	6.0	54.0
53.6	0.487	8.0	9.7
85.2	0.649	10.0	2.9
106.3	0.706	12.0	2.8
124.4	0.723	15.0	1.2
179.1	0.824	18.0	0.5
		27.0	0.3
		29.0	0.1

Attachment 2. SuperLig® 639 Column Data (cont.)

SuperLig® 639 Mixture of Batches 981015DHC720011 and 990420DHC720067

Exp. 5

0.43 cm/min, 2.69 cm ID Column

ICP-ES Analysis

Load Cycle

Water Elution

Feed 14 mg/L Re

# BV	C/Co	# BV	[Re] (mg/L)
5.0	0.008	1	13
11.0	0.010	2	18
29.0	0.034	3	89
64.6	0.154	4	391
101.8	0.308	5	403
135.6	0.454	6	303
169.5	0.569	7	199
203.3	0.700	8	134
236.1	0.769	10	55
252.2	0.846	11	41
268.3	0.846	12	28
284.4	0.846	15	11
292.4	0.846	25	4
		27	2.8

8.3 Attachment 3. Flow Rate Analysis for SuperLig® 639 Column Feed, Wash and Elution Cycles.

Bed Diameter cm	Feed cm/min (BV/hr)	Feed Range cm/min	Post-feed water wash cm/min (BV/hr)	Elution* cm/min (BV/hr)
2.7	0.51 (3.4)	0.42 - 0.53	0.54 (3.6)	0.16 (1.1) A
2.7	5.2 (34.7)	5.2 - 5.7	0.54 (3.6)	0.16 (1.1) W
2.7	11.0 (73.4)	10.9 - 11.2	0.48 (3.2)	0.15 (1.0) A
4.0	0.48 (6.9)	0.42 - 0.56	0.19 (2.7)	0.19 (2.7) W
2.7	0.43 (2.9)	0.41 - 0.48	0.41 (2.8)	0.41 (2.8) W

* The letters A and W indicate whether acid or water were used for elution, respectively.