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Experimental Ion Exchange Column with SuperLig®639 and Simulant Formulation

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U.S. DEPARTMENT OF
ENERGY

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ABSTRACT

SuperLig®639 ion exchange resin was tested as a retrieval mechanism for pertechnetate, through decontamination of a perrhenate spiked 5M Simple Average Na⁺ Mass Based Simulant. Testing included batch contacts and a three-column ion exchange campaign. A decontamination of perrhenate exceeding 99% from the liquid feed was demonstrated.

Analysis of the first formulation of a SBS/WESP simulant found unexpectedly low concentrations of soluble aluminum. Follow-on work will complete the formulation.

ABBREVIATIONS

| | |
|--------|--|
| BV | bed volumes |
| DI | Deionized |
| ETF | Effluent Treatment Facility |
| HLW | High Level Waste |
| ICP-ES | Inductively Coupled Plasma Emission Spectrometer |
| ICP-MS | Inductively Coupled Plasma Mass Spectrometer |
| IPS | Interim Pretreatment System |
| LAW | Low Activity Waste |
| SBS | Submerged Bed Scrubber |
| SRNL | Savannah River National Laboratory |
| WESP | Wet Electrostatic Precipitator |
| WTP | Waste Treatment Plant |



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I. INTRODUCTION

Savannah River National Laboratory (SRNL) is engaged in studies to support the construction of a Waste Treatment and Immobilization Plant (WTP) at the Hanford site in Richland, WA . The experiments described in this report are a part of that effort. The baseline plan for this facility called for the High Level Waste (HLW) and Low Activity Waste (LAW) in tanks at Hanford to be vitrified as glass and sealed in canisters. Technical issues continue to surface with the treatment plans for HLW from these tanks. Delays are hampering the speedy retrieval, or processing, of the tanks. Therefore, options are being explored to deal with the LAW portion of the tank waste immediately, circumventing the Pretreatment and HLW portions of the WTP for the time being, and directly feeding the LAW melter from an Interim Pretreatment System (IPS).¹

To accomplish this goal, a treatment process must be chosen for dealing with the condensate waste from the LAW glass plant. The baseline for WTP had called for the condensation in the Submerged Bed Scrubber (SBS) and Wet Electrostatic Precipitator (WESP) to be returned to the Pretreatment Facility.¹ These condensate collectors will generate an estimated 37 million gallons of Recycle waste that contains large concentrations of fluoride, sulfate, and chloride. Halides and sulfates in the stream limit the LAW glass waste loading. If radionuclides can be removed from the Recycle waste stream, the decontaminated liquid can be sent to the Effluent Treatment Facility (ETF) and be purged from the system, decreasing the volume of glass that the LAW facility would create.²

Technetium-99 (Tc-99) is a radionuclide of interest in the tank waste because it is both abundant and long lasting.³ Tc-99 is predominantly found in the tank supernate as pertechnetate⁴ (TcO_4^-) and pertechnetate is the only form of Tc-99 expected in the SBS/WESP condensate.² Pertechnetate is of special concern because of its environmental mobility and water solubility.³ It has been noted to travel at 90% of groundwater velocity in aquifers³ and its availability for uptake into living plants leads to concerns about technetium entering the food chain.⁵ Pertechnetate is also volatile at melter temperatures and so accumulates in the SBS/WESP Recycle waste stream.

This report centers on two separate but related experiments, each conducted to support the design of an interim Waste Treatment Plant for Hanford site. In the first, experimental ion exchange column work continues on using the resin SuperLig®639 to capture and concentrate perhenate, as a non-radioactive substitute for pertechnetate. The selectivity of SuperLig®639 for pertechnetate and perhenate is demonstrated and the three-column series used maintains a high level of effluent decontamination, despite the first column reaching 65% breakthrough at 156 bed volumes (BV). Secondly, the formulation of a simulant for SBS/WESP condensate begins and initial testing reveals an unexpected resistance to soluble aluminum.



II. EXPERIMENTAL

A. Ion Exchange Resin SuperLig®639

1. Equipment

The resin for batch contacts and in the ion exchange columns was SuperLig® 639 resin from lot # 130611552-56, manufactured by IBC Advanced Technologies, Inc., in American Fork, Utah. The resin's properties as received from the manufacturer can be found in the Appendix.

5M Simple Average Na Mass Based Simulant spiked with perrhenate was used for loading. 0.0489g of sodium perrhenate was added to the 4L of premixed simulant (4.74×10^{-5} M $[\text{ReO}_4^-]$). Detailed simulant information can be found in the Appendix.



Figure 1: Three column ion exchange equipment with FMI pump and fractional sampler.

Three ion exchange columns in Figure 1 were connected in series as lead, lag, and polish with sampling points for the effluent of each, as shown in Figure 1. Custom-made, jacketed borosilicate columns, shown in Figure 2, 1.56 cm ID, were used to hold the resin beds. The total height of the resin bed reservoir scale was 10 cm marked in millimeters. Each resin bed rested on top of a screen tack-welded to a stainless steel ring and fitted with an O-ring seal at the 1cm mark in the column. The empty space between the screen and a Teflon® plug in the bottom of each column was filled with 3mm glass beads to remove dead space in the flow path.

The columns were connected by 1/16" ID fluorinated ethylene propylene (FEP) tubing. Lines entering the columns were secured by Swagelok® Ultra Torr Straight fittings mounted to removable glass heads that allowed the line to be quickly removed and reinserted by loosening the nut. The glass heads were connected to the column by a conical ground glass fitting with a plastic screw tight nut. The tubing was fed out of the columns through Swagelok® fittings mounted in a Teflon® plug.

Fluid Metering (FMI) pumps provided controlled flow rates. Two different pumps were used for fast and slow stages.

Feed temperatures were monitored with three type-T thermocouples, inserted through a port in the heads, read by a 12 channel, DigiSense® scanning thermometer.

A Neslab® RTE-111 water bath kept the column temperature constant during elution. Teflon® quick-connect fittings and flexible PVC tubing were used to connect the water bath to the column jackets.

Analysis of samples was done with an inductively coupled plasma emission spectrometer (ICP-ES).

2. Procedure

a. SuperLig®639 Testing

The resin was received as dry spherical beads, as shown in Figure 3. Bulk density measurements of the resin were taken by weighing 20 mL of loosely packed resin into a graduated cylinder then tapping the side until the volume of the resin no longer changed.

Batch contacts were conducted in target phase ratios, simulant volume (mL) to resin mass(g), of 100:1 and 20:1. Resin portions were held in 60 mL polypropylene bottles as noted in Table 1. Sample 1 was a control

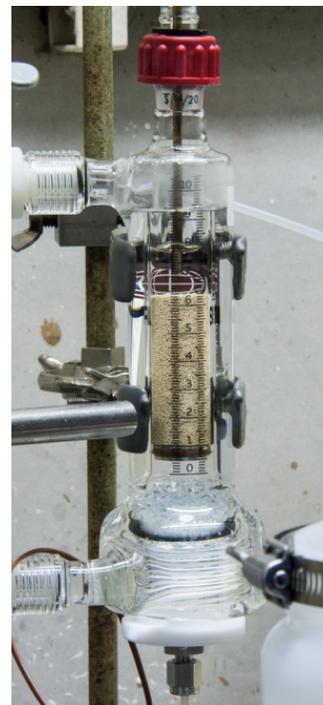


Figure 2: The column bed levels and liquid levels are easily noted by the use of the scale on the inner chamber.

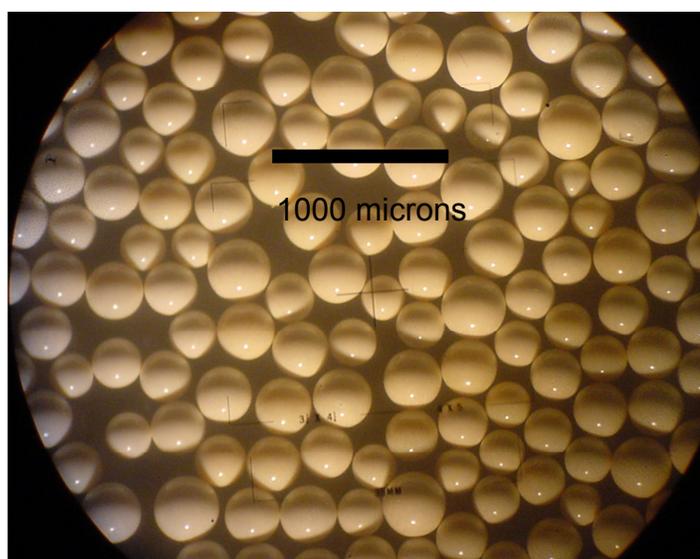


Figure 3: Photo of SuperLig®639 beads under a microscope.

bottle with no resin.

The bottles were agitated in a $26.6 \pm 1^\circ\text{C}$. shaker oven at 175 rpm for the first 13 hours and 140 rpm for the following 38.3 hours.

Resin batches 6 and 7 were subjected to a $65 \pm 1^\circ\text{C}$ deionized (DI) water pretreatment before the batch contact. DI water was added to both bottles, in a resin to water ratio of 1:10, and the bottles were submerged in a hot water bath for 1.67 hours the first day and 6 hours the next. The pretreated resins were air filtered before the simulant was applied.

Table 1: Batch contact target and actual values⁶.

| Sample number | Actual Resin Mass (g) | Actual Volume of Simulant (mL) | pretreatment | Target Phase Ratio (mL/g) | Actual Phase Ratio (mL/g) |
|---------------|-----------------------|--------------------------------|-----------------|---------------------------|---------------------------|
| 1 | 0.0000 | 19.9318 | none | NA | NA |
| 2 | 0.2001 | 19.9191 | none | 100 | 99.546 |
| 3 | 0.2001 | 19.8986 | none | 100 | 99.443 |
| 4 | 1.0013 | 19.9156 | none | 20 | 19.890 |
| 5 | 1.0007 | 19.9085 | none | 20 | 19.895 |
| 6 | 0.2002 | 19.9150 | 65.0°C DI water | 100 | 99.476 |
| 7 | 1.0008 | 19.9274 | 65.0°C DI water | 20 | 19.911 |

b. Preparing the Ion Exchange Equipment

When the columns were first assembled and filled with fluid, an air pocket was trapped under the stainless steel screen. This bubble was removed by back-flushing the system. The end of the line exiting each column was lowered until it was filled with water then raised to force the water back into the column, pushing the air out.

Flow rate calibration measurements for the pumps were taken with DI water and 0.1M NaOH before the run. A target flow rate of 1.0 BV/hr was used for pump 5 and 3.2 BV/hr for pump 4. For each, a mass flow rate was determined and converted to a volume flow rate using the standard density for the feed at 19°C . Flow rates were found to be linearly connected to dial settings as shown in Figures 4 and 5. These measurements were used to determine the scale of pump adjustments as needed during column runs.

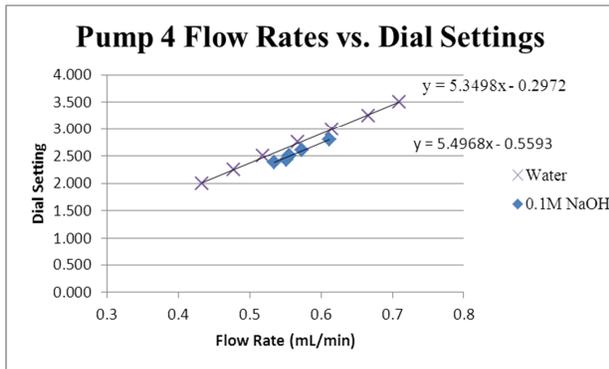


Figure 4: Pump 4 calibration measurements with water and 0.1M NaOH at 19°C.⁶

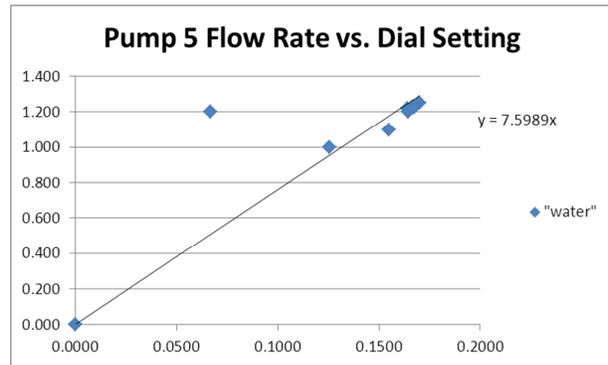


Figure 5: Pump 5 calibration measurements with water at 19°C.⁶

c. Ion Exchange Treatment

The ion exchange column campaign consisted of pretreatment, caustic flow, loading, displacement, and elution. During caustic flow and loading, feed was pumped to the lead column only and gravity fed to the other columns. The columns were then disconnected from each other, displaced and eluted separately. The test matrix in Table 2 was developed for 65% breakthrough on the lead column and an assumed bed volume of 10 mL.

Table 2: Target Ion Exchange Matrix.⁶

| Treatment Stage | Rate (BV/hr) | Rate (mL/min) | Duration (hours) | Volume (mL) | Temperature (°C) |
|-----------------|--------------|---------------|------------------|-------------|------------------|
| Pretreatment | 1.0 | 0.1667 | 6 | 60 | 25 |
| Loading | 3.2-3.4 | 0.5667 | 48 | 1632 | 25 |
| Displacement | 3.0 | 0.5000 | 1 | 30 (each) | 25 |
| Elution | 1.0 | 0.1667 | 20 | 200 (each) | 65 |

Flow rates were found as mass flow rates and converted to volume flow rates using feed densities. Flow rates were verified during the course of a run by timed sampling of the exiting effluent. An accumulated average mass flow rate was found for each stage using total change in effluent or feed bottle used during that stage.

Liquid levels for the columns were continuously monitored to ensure the bed was covered by at least 1 cm of fluid.

Pretreatment

The pretreatment procedure for the fresh resin involved soaking the resin in hot DI water. Resin was measured into individual vials for each column in the proportions shown in Table 3. filled with ASTM water, shaken vigorously to discourage floating, and placed in a hot water bath. The bath was heated to $65\pm 7^{\circ}\text{C}$ for 3 hours. The resins were left in the bath for another 21 hours. The resins were then agitated by stirring for an hour before being transferred to their respective columns.

Table 3: Resin added to each column.⁶

| Column | Mass of as received resin (g) |
|--------|-------------------------------|
| Lead | 4.9001 |
| Lag | 4.899 |
| Polish | 4.9024 |

Initial Caustic Flow to Columns

0.25M NaOH was conducted through the series of columns at an accumulated average rate of 0.89 BV/hr for 6 hours. The flow rate was increased after the first flow rate verification, three hours into this stage, from 0.152 mL/min (0.87BV/hr) to 0.162 mL/min (0.923BV/hr). The effluent from the 0.25M NaOH flush was collected en mass and retained.

Loading

The loading phase flow rates were increased to fit the swelled bed volume of 10.89 mL. Loading took place at an average flow rate of 3.311 ± 0.108 BV/hr^A. During the first 13.67 hours pump ran at a slower of 2.83 BV/hr. The pump speed was increased after the first sampling. The effluent from the first 17 hours of the loading stage was discarded. Effluent from the remainder of the run was retained for future use.

Sampling of the loading stage was conducted approximately every 10 BV. Samples were collected for 10 minutes per column. Collection began with a sample from column 3 and continued upstream so that sampling did not affect the function of columns not yet sampled. Beginning with the second sampling set, the sampling valves for columns 1 and 2 were allowed to drain into a beaker before samples were collected to displace any residual fluid from the last sample. During each sampling set, column measurements were collected: temperature, bed heights, and liquid levels.

^A Determined from column 3 samples. The volume of effluent collected in the 10 minute sampling interval was determined using the density of the simulant reported by the lab, 1.2354 g/mL.

Column 1 developed a slow leak during the loading stage as evidenced by a salt deposit that formed on its exiting line and the plastic sheath for the supply line beneath it. No attempt was made to correct this during loading.

The column was drained and dismantled to apply Teflon® tape to the union. After the tape was applied and the fitting replaced, the fluid retained from column 1 was replaced and fresh feed simulant was added to bring the liquid level past the top of the bed. Altogether, 5.3g of fresh feed fluid was added during the mending process. The air under the screen was displaced by back-flushing fluid through the column. Finally, the 0.1M displacement solution was applied to the top of the column and pressing any floating beads back into contact with the rest of the bed.

The loading feed bottle and the effluent bottle mass changes were compared to determine that the amount of fluid lost due to the leak was negligible.

Displacement

The columns were displaced separately using 0.1M NaOH solution. The change in feed fluid densities, from 1.2354 g/mL to 1.00 g/mL, required the exit lines to be adjusted lower than their loading positions. The pump was set to a slower flow rate for this stage than for loading and connected to each column for one hour before they were eluted. Accumulated average flow rates for each column were 3.042 BV/hr, 2.985 BV/hr, and 2.971BV/hr, for columns 1, 2, and 3 respectively.^B Displacement effluent from each column was collected en mass.

Elution

The columns were eluted immediately following their displacements, beginning with column 3. Columns were eluted separately with 65±3°C ASTM water for 20 hours at 0.9515 BV/hr, 0.9988 BV/hr, and 1.0238 BV/hr, for columns 1,2, and 3 respectively. Elution effluents for columns 2 and 3 were collected in a single bottle. However, column 1 effluent was routed to a fractional sampler, set to collect samples in ½ hour increments. All elutions were started in the afternoon and set to run overnight. This presented no problems for columns 2 and 3. However, the sampling mechanism for column 1 malfunctioned during the unsupervised night shift, presumably due to an air bubble caught in the line, and most of the elution samples were lost as spillage.

^B As found from the change in feed bottle mass.

B. SBS/WESP Simulant Formulation

1. Equipment

The simulant was formulated in a Pyrex borosilicate volumetric flask. DI water was used to rinse simulant ingredients into the flask. The constituents were weighed with a four-place balance with the exception of sodium chromate, sodium fluoride, boric acid, lithium carbonate, and silica which were weighed with a three-place balance located in the hood. Teflon stir bars were used with a magnetic hot plate for the stirring and heating. An electronic pH meter was used to measure pH except where noted.

Subsequent heating of the simulant was accomplished with a beaker full of tap water on the same hot plate as a water bath. Water temperature readings were taken with a thermometer.

2. Procedure

Simulant was mixed in two stages with care given to the order that chemicals were combined. First, ionic compounds meant to simulate the SBS/WESP condensate from tank waste contributors were combined, in the order given below:

1. Sodium fluoride (weighed in hood) and aluminum nitrate nonahydrate added, pH reading taken.
2. Sodium chloride, potassium chloride, and sodium chromate (weighed in hood) added, pH reading taken.
3. Sodium nitrate and sodium nitrite added, pH reading taken.
4. Sodium phosphate dibasic hydrate, ammonium nitrate, and ammonium sulfate added, pH reading taken.

Then, the minerals from glass formers expected in the condensate were added. The target amounts and actual measured amounts of each chemical addition are given in Table 4.

The flask was filled to the target volume after the addition of all glass formers. The pH of the solution was measured with indicator paper and adjusted with 0.5486g of 0.1N nitric acid from a pH of 7.6 to a pH of 7.0-7.2 and sampled. The simulant was then placed in a $55 \pm 5^\circ\text{C}$ water bath for seven hours, left to stir in the same bath overnight, and sampled. Finally, the simulant that remained after sampling had soluble aluminum nitrate added to it. 33.2 mg of aluminum nitrate nonahydrate was dissolved in water and added to the remaining 40mL of simulant.

Formulation of this simulant is ongoing.

Table 4: Actual and target concentrations of simulant additives.⁷

| Chemical Name | Target Concentration (g/L) | 100mL Measured Value Solution 1 (g) | Solution 1 Concentration (g/L) |
|----------------------------------|----------------------------|-------------------------------------|--------------------------------|
| sodium fluoride | 3.2090 | 0.321 | 3.21 |
| aluminum nitrate-nonahydrate | 0.3990 | 0.0396 | 0.396 |
| sodium chloride | 1.395 | 0.1400 | 1.400 |
| potassium chloride | 0.2189 | 0.0221 | 0.221 |
| sodium chromate | 0.2834 | 0.028 | 0.28 |
| sodium nitrate | 1.221 | 0.1222 | 1.222 |
| sodium nitrite | 0.0160 | 0.0017 | 0.017 |
| sodium phosphate-dibasic hydrate | 0.0403 | 0.0040 | 0.040 |
| ammonium nitrate | 4.759 | 0.4759 | 4.759 |
| ammonium sulfate | 3.220 | 0.3220 | 3.220 |
| kyanite | 0.7450 | 0.0745 | 0.745 |
| borax | 0.0123 | 0.0011 | 0.011 |
| boric acid | 1.430 | 0.143 | 1.43 |
| wollastonite | 0.7721 | 0.0776 | 0.776 |
| iron oxide | 0.4296 | 0.0428 | 0.428 |
| lithium carbonate | 0.3918 | 0.037 | 0.37 |
| forsterite olivine | 0.2571 | 0.026 | 0.26 |
| sodium carbonate | 0.0033 | 0.003 | 0.03 |
| silica | 2.857 | 0.286 | 2.86 |
| rutile | 0.1142 | 0.0109 | 0.109 |
| zinc oxide | 0.2858 | 0.0284 | 0.284 |
| zircon | 0.3721 | 0.0369 | 0.369 |

III. RESULTS

A. Ion Exchange Resin SuperLig®639

1. Batch Contact

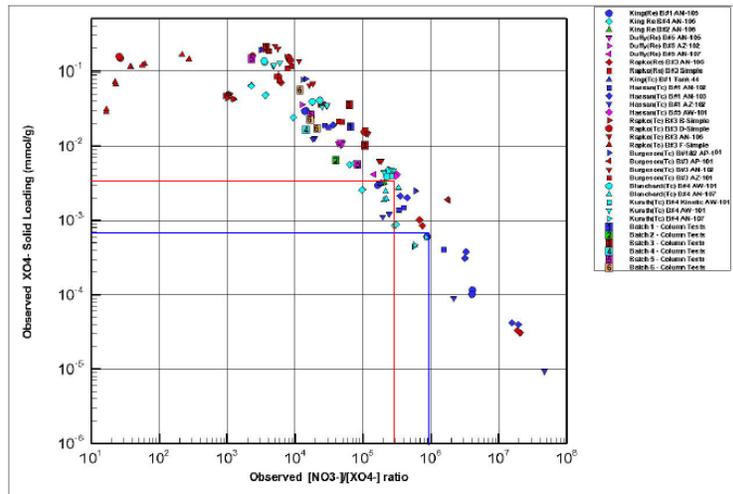


Figure 6: The resin loading values are overlaid on this compilation of 150 SuperLig®639 data sets; averaged values from batches with a phase ratio of 20 are shown in blue and phase ratio 100 batches are shown in red.⁸

As shown in Figure 6 above, taken from a recent modeling report that combines experimental data from a total of 150 data sets, “Upgrade to Ion Exchange Modeling for Removal of Technetium from Hanford Waste Using SuperLig®639 Resin,” the values found in these batch tests are well within the range of values determined in other tests,⁸

Table 5: Batch contact for SuperLig® 639, 26.6°C, 51.3 hour exposure.⁶

| Sample number | Actual Phase Ratio (mL/g) | Rhenium in solution (mg/L) | Rhenium on solid (mmol/g) | Batch Kd (mL/g) | $\frac{[NO_3^-]}{[ReO_4^-]}$ |
|---------------|---------------------------|----------------------------|---------------------------|-----------------|------------------------------|
| 1 | NA | 5.93 | NA | NA | |
| 2 | 99.55 | 1.49 | 2.37E-03 | 296.299 | 5.79E+04 |
| 3 | 99.44 | 1.54 | 2.34E-03 | 284.401 | 5.62E+04 |
| 4 | 19.89 | 0.410 | 5.89E-04 | 267.892 | 2.10E+05 |
| 5 | 19.89 | 0.324 | 5.98E-04 | 344.481 | 2.66E+05 |
| 6 | 99.48 | 1.51 | 2.36E-03 | 290.851 | 5.71E+04 |
| 7 | 19.91 | 0.553 | 5.74E-04 | 193.619 | 1.56E+05 |

2. Ion Exchange Treatment

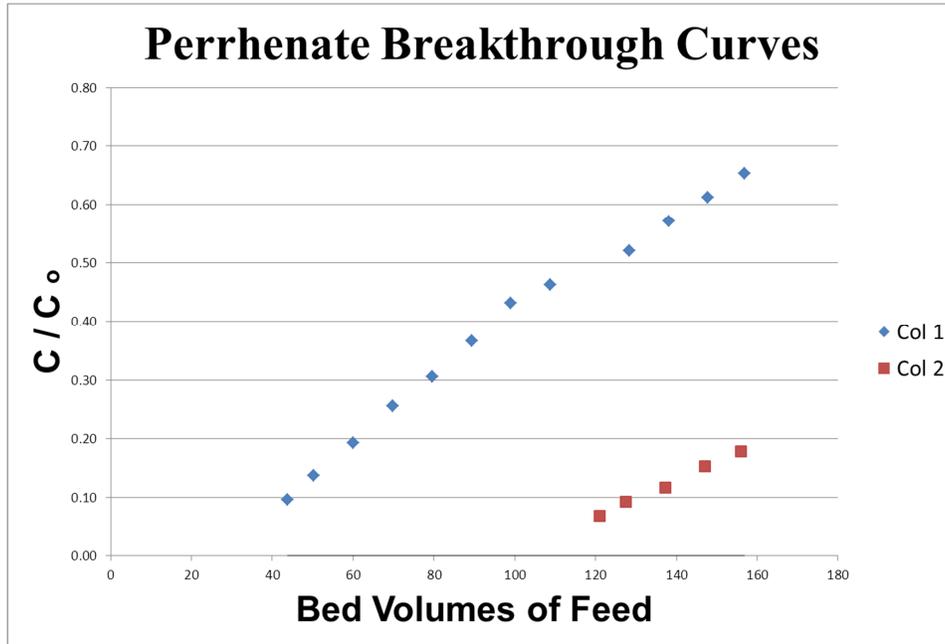


Figure 7: Perrhenate breakthrough from 25°C loading. Results from ICP-ES.⁶

During loading of the columns, 156 BV were processed. Breakthrough was expected to be significant and column 1 reached 65% breakthrough by the end of loading. ICP-ES analysis of column 3 data did not detect any measurable rhenium in any of the samples, as shown in Figure 7.

The breakthrough curves have a gradual slope, indicating the mass transfer rate of the resin was quite slow. Decontaminated fluid was able to move past the resin beads although they had not reached capacity.

A decontamination factor of 117 was shown by Inductively Coupled Plasma Mass Spectrometer (ICP-MS); results from columns 2 and 3 are shown in Figure 8, on the next page.

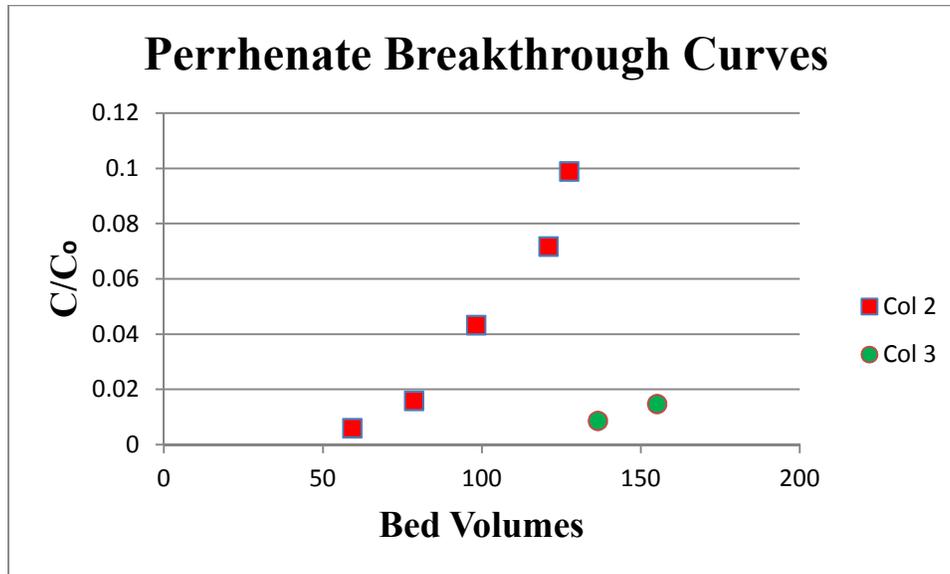


Figure 8: Perrhenate breakthrough from 25°C loading. Results from ICP-MS.⁶

Analysis of the samples also indicates that the resin is very selective for perrhenate. As shown in Table 6 below, there was very little change in other feed ion levels.

Table 6 :Samples taken at the end of the run, from each column, show very little change in the levels of other ions in the simulant.⁶

| Sample ID | Al | K | Na | P | S | Re |
|-----------------------|------|------|--------|------|------|-------|
| 5MH2-Col1-Feed 13 (A) | 8760 | 1330 | 117000 | 1800 | 3320 | 4.91 |
| 5MH2-Col1-Feed 13 (B) | 8720 | 1320 | 118000 | 1800 | 3320 | 4.90 |
| 5MH2-Col2-Feed 13 (A) | 8860 | 1350 | 118000 | 1800 | 3320 | 1.36 |
| 5MH2-Col2-Feed 13 (B) | 8740 | 1330 | 118000 | 1800 | 3330 | 1.32 |
| 5MH2-Col3-Feed 13 (A) | 8790 | 1310 | 118000 | 1820 | 3360 | <1.00 |
| 5MH2-Col3-Feed 13 (B) | 8800 | 1320 | 119000 | 1820 | 3360 | <1.00 |
| Simulant Feed (A) | 8810 | 1340 | 120000 | 1790 | 3330 | 7.61 |
| Simulant Feed (B) | 9020 | 1310 | 119000 | 1790 | 3330 | 7.53 |

B. SBS/WESP Simulant Formulation

Soluble aluminum levels were lower than expected in the 100mL batch of SBS/WESP simulant. Aluminum was present in the solid phase, but as shown in Table 7 below, no aluminum was detected in the supernate after the initial combination of materials or heating of the simulant. Even after dissolved aluminum nitrate nonahydrate was added, the soluble aluminum levels were undetectable. Formation of aluminum complexes with boron, lithium, or silica is suspected.

Table 7: ICP-ES results from first batch of simulant, no soluble aluminum is detected.⁷

| Element | predicted concentration (mg/L) | 100mL initial makeup | | 100 mL | 100 mL after |
|---------|--------------------------------|----------------------|-----------------------|-------------------------------------|--|
| | | liquids (mg/L) | supernate only (mg/L) | after heating supernate only (mg/L) | addition of soluble Al supernate only (mg/L) |
| Al | 28.7 | 70.7 | <1.00 | <1.00 | <1.00 |
| Cr | 91 | 78.85 | 80.35 | 78.95 | 101 |
| K | 114.77 | 152.5 | 129 | 109 | 161 |
| Na | 2691 | 2440 | 2460 | 2225 | 2565 |

IV. FUTURE WORK

A. Ion Exchange Resin SuperLig®639

Radioactive batch contact testing and ion exchange work will be performed.

Further testing with this combination of simulant and batch of resin in the three column arrangement is planned, beginning with a run where loading is performed at 45°C.

B. SBS/WESP Simulant Formulation

Analysis of samples by ICP-ES from a second batch of this simulant is underway. The results will be used to determine when the aluminum is no longer soluble and which glass formers affect the change.

V. APPENDIX

Pump Flow Calibrations⁶:

Table 8: Pump 4 measured flow rates with ASTM water.

| Dial Setting | time (min) | mass of water(g) | volume (mL) | Flow rate (mL/min) |
|--------------|------------|------------------|-------------|--------------------|
| 2.000 | 10.00 | 4.3181 | 4.32502 | 0.432502 |
| 2.250 | 10.00 | 4.7665 | 4.774139 | 0.477414 |
| 2.500 | 10.00 | 5.1787 | 5.186999 | 0.5187 |
| 2.750 | 10.00 | 5.6595 | 5.66857 | 0.566857 |
| 3.000 | 10.00 | 6.1382 | 6.148037 | 0.614804 |
| 3.250 | 10.00 | 6.6572 | 6.667869 | 0.666787 |
| 3.500 | 10.00 | 7.0886 | 7.09996 | 0.709996 |

temp=19C
density=0.9984 (g/mL) at 19°C

Table 9: Pump 4 measured flow rates with 0.1M NaOH.

| Dial Setting | time (min) | mass of NaOH(g) | volume (mL) | Flow rate (mL/min) |
|--------------|------------|-----------------|-------------|--------------------|
| 2.800 | 10.00 | 6.1171 | 6.1171 | 0.61171 |
| 2.610 | 11.00 | 6.3089 | 6.3089 | 0.573536 |
| 2.500 | 10.00 | 5.5439 | 5.5439 | 0.55439 |
| 2.500 | 10.00 | 5.5567 | 5.5567 | 0.55567 |
| 2.390 | 10.00 | 5.342 | 5.342 | 0.5342 |
| 2.430 | 10.00 | 5.5165 | 5.5165 | 0.55165 |

temp=19C
density=1.00 (g/mL) at 19°C

Table 10: Pump 5 Measured Flow Rates

| Dial Setting | time (min) | mass of water(g) | volume (mL) | Flow rate (mL/min) |
|--------------|------------|------------------|-------------|--------------------|
| 0.000 | 10.00 | 0.0000 | 0.0000 | 0.0000 |
| 1.000 | 5.00 | 0.6268 | 0.6278 | 0.1256 |
| 1.100 | 5.00 | 0.7728 | 0.7740 | 0.1548 |
| 1.200 | 5.00 | 0.3342 | 0.3347 | 0.0669 |
| 1.200 | 5.00 | 0.8206 | 0.8219 | 0.1644 |
| 1.250 | 8.00 | 1.3585 | 1.3607 | 0.1701 |
| 1.220 | 5.00 | 0.8183 | 0.8196 | 0.1639 |
| 1.226 | 5.00 | 0.8306 | 0.8319 | 0.1664 |
| 1.228 | 5.00 | 0.8331 | 0.8344 | 0.1669 |
| 1.227 | 5.00 | 0.8360 | 0.8373 | 0.1675 |

temp=19C
density=0.9984 (g/mL) at 19°C

SuperLig® 639 May 2013 4+ Liter Batch (M)

Resin Properties Requirements and Results Table

| <u>Property</u> | <u>Unit</u> | <u>Specification</u> | <u>Result</u> |
|---|-------------------|----------------------------------|---------------------|
| <u>Obtained</u> | | | |
| SuperLig® 639 resin total purity/capacity | mole/kg | ≥0.8 | 1.0 |
| F-factor (moisture content) | wt% | <5% | <0.2% |
| Resin Density (Avoidance of Flootation) | g/cm ³ | 1.25 | 1.258 ^a |
| Re Distribution Coefficient (Re Kd) | mL/g | >200 | 316 ^b |
| Leachable Total Organic Carbon | mg/L | 100 maximum | 72.7 |
| Leachable Halides (F/Cl/Br) | mg/L | 100 maximum | 33 |
| | | Note: only chloride was detected | |
| Resin fines content | wt% | <0.1% | <0.01% ^c |
| Particle Size: Less than 212 microns | wt% | <1% | <0.01% ^c |
| Particle Size: Greater than 1000 microns | wt% | <1% | 0.2% ^d |

^aLess than 10% of resin by volume floats in 4.9 M NaNO₃ or mixture of 5 and 4.9 M NaNO₃ or 4.8 M NaNO₃ or in simulated waste solution, but slightly more than 10% of resin by volume floats in 5 M NaNO₃ after wetting and coming to equilibrium over several hours.

^bKd value in simplified 3 M NaOH/2 M NaNO₃/0.1 M KNO₃ was 287 mL/g.

^cData reported are for dry sieving and no particles could be collected or weighed under 212 microns. Wet Coulter Laser Counter analysis also confirmed no particles below 100 microns and very low percentage under 212 microns and over 1000 microns. Metal spatula had to be used to sift resin through 1000 micron screen due to static and clumping of resin in very dry form.

^d0.103 g out of 50.1 grams total could be weighed that were on top of the 1 mm screen. An additional 0.482 grams out of 50.1 grams total were collected in the pores of the 1 mm screen itself.

Simulant reported values from before the perrhenate was added ⁶:

Table 11: Reported 5M Simple Average Na Mass Based Simulant constituents.

| <u>Compounds</u> | <u>Actual Weight (g)</u> |
|-------------------|--------------------------|
| Water | 2804.2 |
| Potassium Nitrate | 13.2601 |
| Sodium Chloride | 9.8603 |
| Sodium Sulfate | 48.8603 |
| Sodium Hydroxide | 892.2 |
| Aluminum Nitrate | 460.7 |
| Sodium Phosphate | 74.5002 |
| Sodium Acetate | 20.9604 |
| Sodium Carbonate | 116.2 |
| Sodium Nitrate | 227.2 |
| Sodium Nitrite | 156.0 |

Table 12: Reported 5M Simple Average Na Mass Based Simulant measured properties

| <u>Sample</u> | <u>Density (g/mL)</u> | <u>% wt solids</u> |
|---------------|-----------------------|--------------------|
| 1 | 1.23544 | 27.10 |
| 2 | 1.23542 | 27.21 |

Total volume=4L

Table 12: Lab analysis of perrhenate spiked simulant in ppm.

| <u>Sample ID</u> | <u>Lab ID</u> | <u>Al</u> | <u>K</u> | <u>Na</u> | <u>Re</u> | <u>F</u> | <u>Cl</u> | <u>NO2</u> | <u>NO3</u> | <u>SO4</u> | <u>PO4</u> |
|----------------------|---------------|-----------|----------|-----------|-----------|----------|-----------|------------|------------|------------|------------|
| HTWOS 5M Initial (A) | 13-0978 | 8587 | 1359 | 110549 | 7.47 | <100 | 1520 | 28800 | 99400 | 8130 | <100 |
| HTWOS 5M Initial (B) | 13-0978 | 8427 | 1318 | 111818 | 7.44 | <100 | 1510 | 28600 | 99300 | 8060 | <100 |



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VII. REFERENCES

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