COMPREHENSIVE SCALE TESTING OF THE ION EXCHANGE REMOVAL OF CESIUM AND TECHNETIUM FROM HANFORD TANK WASTES

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ABSTRACT

Highly selective ion exchange materials will be used to remove radionuclides from tank waste at the Hanford site as part of the River Protection Project. Testing in support of facility design was performed with different sizes of ion exchange columns to provide a basis for comparing results obtained using small-scale with radioactive samples and full design-height (i.e., pilot-scale) with simulant. Results indicate good comparison between small-scale radioactive tests and pilot-scale simulant tests. Because of the cost of performing radioactive tests and the unavailability of large sample volumes, understanding scale-up of performance parameters is critical to ensure that the system will perform as designed. The consistency of scale-up of ion exchange columns using SuperLig® 644 and 639 resins has been demonstrated. Maintaining constant residence time, i.e., Column Volumes per hour, yields similar breakthrough profiles with resin columns ranging from 3.5 cm to 230 cm in height. Experiments performed with flow rates greatly exceeding the design parameters provided valuable information on loading and diffusion parameters. These data will be used, along with a computer model, to permit verification of design and prediction of column performance.

INTRODUCTION

Research at the Savannah River Technology Center (SRTC) aided development of a technical design basis for a Waste Treatment Plant (WTP) to pre-treat and vitrify Hanford tank waste as part of the River Protection Project (RPP). Research performed at SRTC during "Part A" of the Hanford waste treatment effort, provided a basis for preliminary design of a pretreatment and vitrification facility (1). Research completed for "Part B" further supports the design of this facility at Hanford. The research addresses safety concerns, process optimization, and waste form compliance. This program will provide technical data to ensure that the process functions as designed and minimizes costs.

The primary unit processes of the Hanford waste treatment flow sheet are cross-flow filtration, Sr/TRU precipitation, ion exchange, evaporation, and vitrification. Each pretreatment process unit removes the radionuclides from the bulk of the waste and concentrates them into small-volume streams. This High Level Waste (HLW) mixture is vitrified as a high activity glass. The decontaminated aqueous Low Activity Waste (LAW) phase contains the bulk of the waste volume and is vitrified as a low activity glass. The RPP has classified the LAW feed to the WTP into three envelopes; A, B, and C. This paper focuses on the results of testing with Envelope A solutions. The ion exchange design criteria for both cesium and technetium with Envelope A are for the lead column to surpass 100 Column Volumes of treated waste before 50% breakthrough

of the radionuclide is reached. The lag column effluent must also meet the glass form compliance targets for at least 100 Column Volumes.

The research program at SRTC includes both radioactive and simulant tests for all unit processes to ensure consistency and scale-up design factors. Testing has been performed using three sizes of resin columns, small (~5 mL), intermediate (~50 mL), and full-height (~1000 mL) pilot scale. Radioactive testing has been performed with small and intermediate scale columns. Simulant tests have been performed with intermediate and full-length columns. The intermediate scale columns have been used to "bridge the gap" between small-scale radioactive and full-height simulant tests to confirm design assumptions. The results are used for input to computer modeling, which is being utilized to ensure consistent performance, predict the effect of variables, and optimize performance. Scale-up has been shown to be consistent with constant residence time in the column. The optimized process parameters are utilized by the multi-site research team to develop an integrated flow sheet to ensure viability of the process.

Cesium and technetium ion exchange is performed using SuperLig® 644 and 639, respectively. These organic resins have proven effective for removal of the radionuclides. Development of a preconditioning procedure for the cesium resin has been critical to successful operation (4). Water has been shown effective at ambient temperature for elution of the technetium resin, but large volumes are needed. Dilute nitric acid has been shown effective for elution of the cesium resin. Additional testing is ongoing with both resins to examine chemical and radiation stability, optimize performance, reduce eluate volume, and ensure process viability.

EXPERIMENTAL

Testing has been performed with three different sized columns: small (~5 mL), intermediate (50-100 mL), and pilot (1000 mL). The pilot-scale columns are identical in height to the full-scale design, but are only 2.57 cm in diameter. Details of each experimental apparatus is provided below:

The small-scale radioactive tests were conducted with Hanford Tank 241- AN-103 salt solution. The solution received from Hanford was diluted with 0.01 M sodium hydroxide solution and filtered with 0.45-µm nylon filters. The solution was 4.99 M [Na^+], as shown in Table I (2). The AN-105 simulant was prepared based on the composition of tank 241-AN-105 at Hanford. The cesium was replenished with cesium nitrate after each simulant experiment. Similarly, the rhenium was replenished with sodium perrhenate after each simulant experiment. The ion exchange materials used for radioactive cesium and technetium removal from 241-AN-103 were SuperLig® 644 (batch # 981020MB48-563) and 639 (batch # 981015DHC720011), respectively. SuperLig® 644 is a polymerized proprietary organic material supplied as 20-70 mesh granules. SuperLig® 639 is composed of polystyrene beads with an attached proprietary organic compound. IBC Advanced Technologies, American Fork, Utah, supplied both resins for testing.

The equipment used for the small-scale ion exchange column tests with radioactive samples was assembled remotely in a shielded-cell in a SRTC laboratory module. Two identical ion exchange columns were used per column test. The columns, which were designated as a lead and lag, were made of a medium wall Pyrex® glass tube with an inside diameter of 11 mm and a total length of

30 cm. The outsides of the column walls were coated with a layer of clear polyvinylchloride to reduce hazards associated with potentially pressurizing the apparatus. The column top assemblies had a fill reservoir, a pressure gauge, a pressure relief valve, and feed inlet port. The fill reservoir on column top assemblies also served as a vent. The top assembly was connected to the lower section by a glass ground joint and was tightly fitted by a screw cap. A ruler affixed to the column wall was used to allow observation of resin bed height changes and liquid level. All tubing connections were made of polypropylene lines that had Teflon® quick-connect fittings attached to each end. After preconditioning, the volumes of the resin in the lead and lag columns were each approximately 6.4 mL and were stored in de-ionized water. The columns were connected in series, and lead column samples were collected between the columns. The decontaminated lag column effluent product was subsequently used in vitrification studies at SRTC.

Intermediate-scale 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. Only a single column was used in the intermediate-scale tests. 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 down flow direction using Fluid Metering Incorporated QG150 positive displacement pumps with ¼" and $^3/_8$" piston sizes. Samples were collected either manually or using a Spectrum Chromatography IS-95 Interval Sampler.

For the intermediate-scale ion exchange column experiments, a 51 mL sample of SuperLig® 639 resin from batch #980624001DC was used. An additional experiment was conducted using 50 mL of a 50:50 mixture of SuperLig® 639 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 column 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 to 23 °C.

The pilot-scale system consisted of two columns hydraulically connected in series. Each column has an inside diameter of 2.57 cm, a total height of 4.61 m, and an approximate swelled resin height of 2.30 m. Therefore, the top half of the column contained only liquid. The total height and the swollen resin height were chosen to duplicate the design of the full-scale facility. The inside diameter of the pilot-scale columns was chosen to satisfy the criterion given by Helfferich (3) that the diameter of an ion exchange column must be at least 30 times as large as the average resin particle diameter (0.75 mm for SuperLig® 639) to eliminate any significant bypass flow at the wall. The transparent columns were double walled for safety. The inner tube was

transparent PVC, and the outer wall was glass, which has excellent transparency. The outer tube was used to contain the resin and simulant in the event of leakage of the PVC tube.

The liquid was pumped using an FMI Lab pump model QV piston pump for which the stroke length was manually adjustable and the frequency was controlled by an external current signal. The pumping rate was controlled by a computer using feedback from a flow meter. The computer compared the flow set point that it had been provided with the measured flow and calculated the required magnitude of the control signal. The computer generated this control signal, which was sent to the pump controller.

The simulant was stored in a polyethylene tank that had a tight, but not sealed, lid. An in-line cartridge filter was used to remove insoluble solids. The simulant was collected in a plastic 55-gallon drum that rested on a 1000-lb. scale. Other liquids were contained in 15 and 50-liter polyethylene carboys. Liquids entered and left carboys (tanks) via polyethylene tubes that passed through slightly oversize holes in the lids that allowed gas venting. All other connections in the facility were made with ¼" o.d. stainless steel tubing. Pressure relief valves set at 50 psig were connected to the tops of both columns.

The piping for the pilot-scale facility included thirty-five diaphragm type solenoid valves that were controlled by the computer and fourteen manual valves. By opening and closing the appropriate valves, either the left hand column (#1) or the right hand column (#2) could be the lead column and flow could be either upward or downward in the columns. Liquid flowing out of the lead column can be directed through the lag column or directly to the collection tanks. For most of the steps in the process, the flow was downward in both columns. However, for the resin post-elution rinsing and regeneration steps, the direction of flow was reversed in the upward direction. Liquid samples having a volume of 5 mL were collected using an automatic sampler system. A Dell Optiplex GXIP computer using LabView Version 5.1 software was used to control the tests and to display and record experimental data. SuperLig® 639 resin came from two batches, 640.57 g (1.3 L dry) from batch 981015DHC720011 and 624.0 g (1.3 L dry) from batch 990420DHC720067.

For all experiments, fresh resin batches of SuperLig® 644 were pretreated according to protocol developed by Savannah River Technology Center (4). This protocol (see below) included an acid-caustic cycle that resulted in a fully swollen resin bed in the sodium hydroxide form. The resin generally swells by approximately 100% volume versus the dry received form.

New SuperLig® 644 Resin Pretreatment Procedure:

- Soak in 1.0 M NaOH (~10:1 vol/:vol) for 2 hrs (gentle shaking)
- Decant NaOH and add de-ionized water
- Slurry resin into column while tapping side of column
- Pump 3 column volumes (CV) of 0.5 M HNO_3 in 1 hour
- Pump 3 CV of de-ionized water in 1 hour
- Pump 6 CV of 0.25 M NaOH in 2 hours
- Pump 3 CV of de-ionized water in 1 hour (only if stored >48 hours)

All experiments utilizing new SuperLig® 639 resin included soaking the resin in 1.0 M NaOH, and slurrying the resin into the columns. The resin beds were then pretreated with 3 Column Volumes (CV = resin bed volume) of 1.0 M NaOH over one hour period just prior to beginning each experiment (Pretreatment Cycle).

For both cesium and technetium columns, the NaOH liquid level was adjusted so that the volume of liquid above the resin bed was approximately 1 Column Volume before salt solution was pumped into the column. (**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. Accordingly, the initial salt solution that was fed into the column at the beginning of the loading cycle was diluted by the 1 CV of NaOH pretreatment solution, which remained above the resin bed and that in the polymer tubing. 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 column 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. Salt solution was pumped into the columns and analytical samples of the effluent were collected every 10-20 column volumes (Feed Cycle). At the conclusion of the feed cycle, the resin beds were washed with 0.1 M sodium hydroxide solution. The dilute sodium hydroxide solution was pumped into the columns to displace the salt solution and is made alkaline to prevent aluminum hydroxide precipitation (for small and intermediate-scale tests, most of the salt solution was drained from the column headspace prior to addition of dilute sodium hydroxide). The resin beds were flushed with 2.5 to 3.5 column volumes of 0.1 M NaOH in one hour and analytical samples of the effluent were collected. Subsequently, 2.5 to 3.5 column volumes of deionized water were passed through the columns in one hour and analytical 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 columns prior to elution.) The columns were eluted with either deionized water (technetium columns) or 0.5 M HNO_3 (SuperLig® 644 columns) at a flow rate of 1 column volume per hour and analytical samples were collected every 1 to 2 column volumes (Elution Cycle). Feed and effluent samples were analyzed by the SRTC Analytical Development Section.

The flow rate was monitored during the column loading and elution experiments by periodically measuring the time required to collect a known volume of the effluent. The pilot-scale tests utilized an on-line flow rate meter, which was confirmed by weighing the mass of the solution collected. The solution density was measured at the start of the test.

RESULTS AND DISCUSSION

The compositions of the radioactive composite filtrate (Tank 241-AN-103) and Envelope A simulant (Tank 241-AN-105) are shown in Table I (5). Although the solutions are not identical, they are sufficiently similar to allow a comparison of the ion exchange data. Tanks 241-AN-103 and 241-AN-105 are within the "Envelope A" category of tank supernates. The AN-103 sample was a composite sample of the entire contents of tank 241-AN-103, and is representative of the average tank contents. Simulation of the AN-105 tank was chosen because it is typical of the Envelope A tank wastes that will be processed in the RPP-WTP. Perrhenate was used as a non-

radioactive surrogate for pertechnetate ion in the simulant testing, as it was not feasible to use radioactive materials in the pilot-scale system and there are no non-radioactive isotopes of technetium.

Table I. Radioactive composite filtrate (241-AN-103) analysis and Envelope A (241-AN-105) simulant compositions.

Constituent ions [M]	Tank-241-AN-103 filtrate [M]	AN-105 Simulant [M] primary constituents
[Na^+]	4.99	5.0
[Al]	0.84	0.69
[Ca]	0.0002	0
[Cr]	0.0014	0
[P]	0.0099	0.003
[Si]	0.0034	0.004
[K^+]	0.117	0.09
[Mo]	3.56E-4	4.02E-4
[B]	0.00012	0.002
Anions, M		
[NO_2^-]	0.866	1.13
[NO_3^-]	0.998	1.25
[Cl^-]	0.085	0.12
[SO_4^{-2}]	0.009	0.0038
[PO_4^{-2}]	0.0059	0.003
[C_2O_4=]	0.0069	0.003
Free OH-	1.869	1.62
Radionuclides		
[Cs-137] Cs total	200 uCi/ml 6.7E-5 M	 6.2E-5 M
[Tc-99] Re	3.1E-5 M	 6.87E-5
TIC	0.31	0.13
TOC	0.047	0.066

The simulant formulation shown in Table I includes 0.066 M Total Organic Carbon (TOC), which is a combination of sodium formate (0.02 M), sodium oxalate (0.003 M), and glycolic acid (0.01 M).

Results of the cesium ion exchange tests at three different scales are shown in Figure 1. All experiments significantly out performed the minimum design criteria of 100 CV to 50% breakthrough at 3.0 CV/hr (CV: Column Volume is defined as the resin volume swollen in salt solution). The lead column breakthrough profiles are shown for the small-scale radioactive sample of AN-103, and the intermediate and pilot-scale tests with AN-105 simulant. The intermediate-scale testing was performed at 6.4 CV/hr, and all other tests were conducted at 3.0-3.5 CV/hr. The breakthrough of the intermediate-scale test is slightly faster (as shown in Figure 1) between 100 and 175 column volumes, but the results are otherwise comparable. The superficial velocities range by 40X, but the results are similar. There was insufficient volume of salt solution to continue the small-scale radioactive test beyond 160 CV, and it was impractical to reduce the size of the column further.

The intermediate-scale column tests were designed to cover the range of superficial velocities (volumetric flow rate/resin bed cross-sectional area) and contact times (Column Volumes per

hour), and are not intended to be used as a recommended mode of operation. While operating at the design superficial velocity of about 13 cm/min, the data generated using the intermediate-scale column are effectively equivalent to sampling the full-height column at a depth of 8.8 cm. This allows observation of the development of the mass transfer zone. These results were utilized to determine diffusion and absorption kinetic parameters that are input for the ion exchange computer model, VERSE-LC (6). The column dimensions and experimental parameters are summarized in Table II for all three scale experiments.

Table II. SuperLig® 644 and 639 Column Breakthrough Summary.

SuperLig® 644 cesium column; Batch #981020mb48-563

Experiment #	Bed Height/Diam. (cm)	Column Volume (mL)	Flow Rate (cm/min)	Flow Rate (CV/hr)	50% Breakthrough (CV)
Small Rad AN-103	6.4/1.1	6.1	0.32	3.0	~300
Intermed Sim #3	8.2/2.7	46.3	0.87	6.4	240
Intermed Sim #1	7.5/2.7	42.3	6.0	48.4	135
Intermed Sim #2	7.5/2.7	42.3	10.6	85.5	100
Intermed Sim #4	3.5/4.0	44.0	0.83	14.0	240
Pilot Sim #1	185/2.6	960	12.9	3.5	270

SuperLig® 639 perrhenate column; Batch #980624001DC

Experiment #	Bed Height/Diam. (cm)	Column Volume (mL)	Flow Rate (cm/min)	Flow Rate (CV/hr)	50% Breakthrough (CV)
Intermed Sim #1	9.0/2.7	51	0.51	3.4	90
Intermed Sim #2	9.0/2.7	51	5.2	34.7	30
Intermed Sim #3	9.0/2.7	51	11.0	73.4	9
Intermed Sim #4	4.1/4.0	51	0.48	7.1	55

SuperLig® 639 perrhenate column; 50:50 Mixture of Batches 981015DHC720011 and 990420DHC720067

Experiment #	Bed Height/ Diam (cm)	Column Volume (mL)	Flow Rate (cm/min)	Flow Rate (CV/hr)	50% Breakthrough (CV)
Small Rad AN-103*	5.9/1.1	5.6	0.27	3.0	270
Intermed Sim #5	8.8/2.7	50	0.43	2.9	150
Pilot Sim #9	230/2.6	1193	12.5	3.4	160

*The small radioactive test with AN-103 used only batch #981015DHC720011

The effects of flow rate on the cesium loading profile for the intermediate-scale tests are shown in Figure 2. The first three tests utilized 2.7 cm inside diameter columns, and the fourth test utilized the same resin sample slurried into another column with an inside diameter of 4.0 cm. The results indicate that the resin has relatively fast kinetics, meeting the design criteria even at 85.5 CV/hr. The effect of the flow rate is relatively small, with the 6.4 and 14.0 CV/hr flow rates

yielding nearly identical results. This suggests that the RPP-WTP facility could operate the columns at a higher flow rate and still meet the design parameters of 100 CV to 50% breakthrough. However, at these high velocities the overall decontamination factor would be lower, and the computer model can be used to optimize the process.

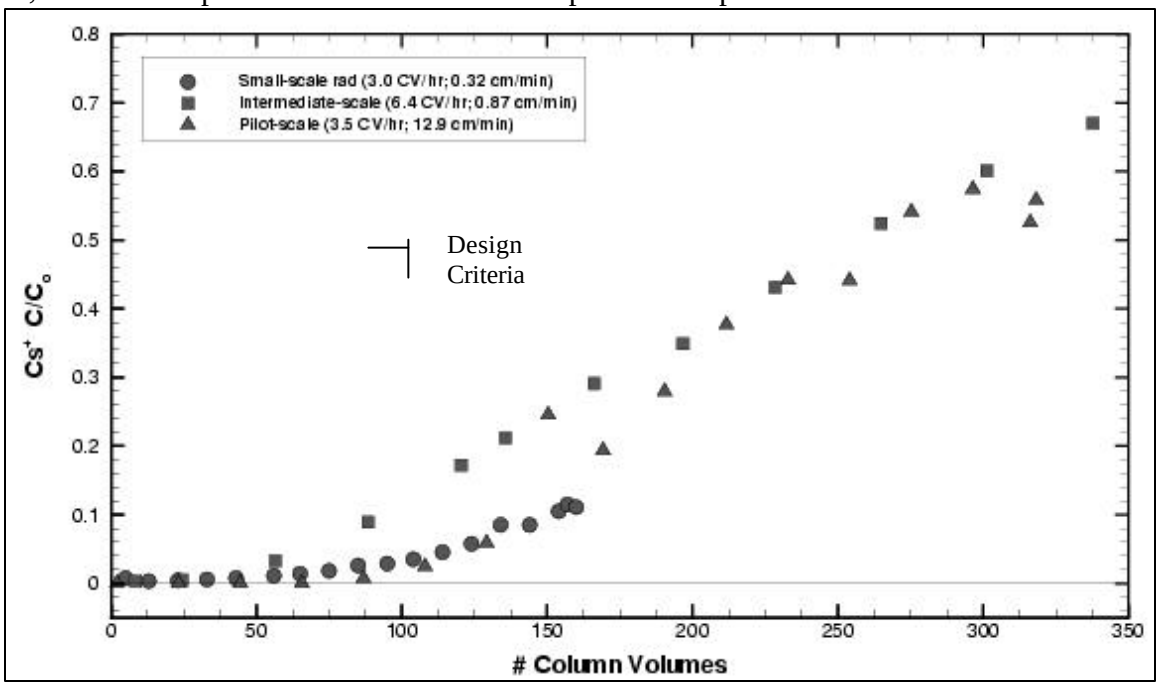

Fig 1. Cesium Breakthrough Profiles for Small-Scale Radioactive and Intermediate and Pilot-Scale Simulant Tests

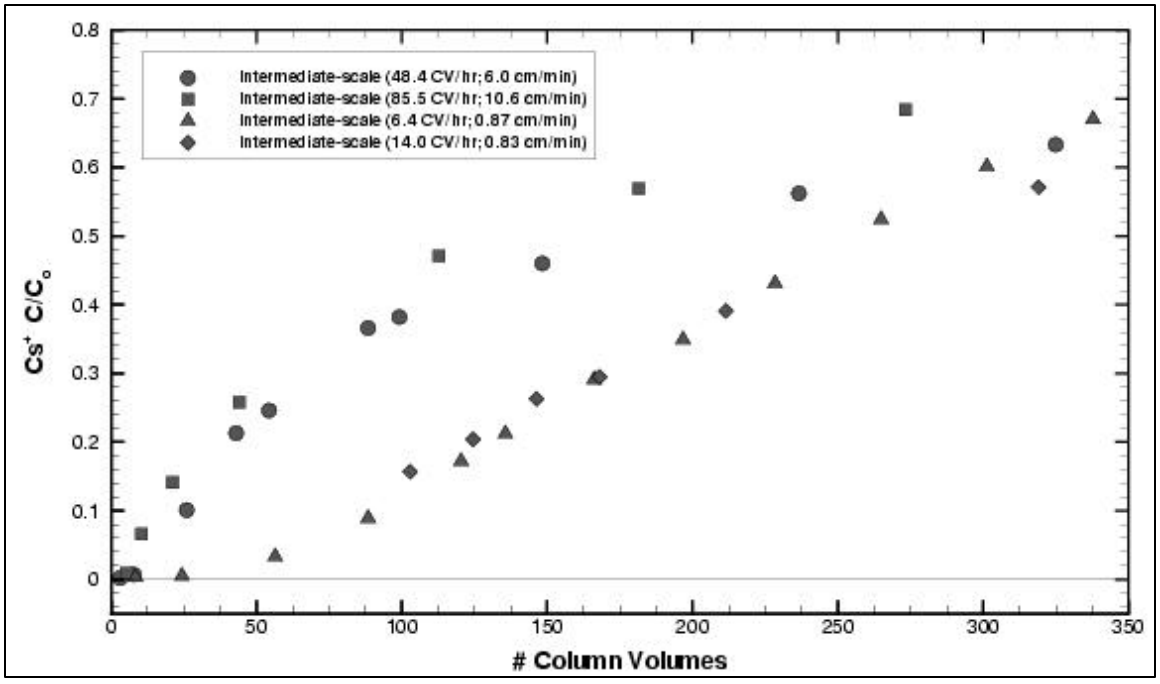

Fig 2. Cesium Breakthrough Profiles for Intermediate-Scale Simulant Tests at Various Flow Rates

The technetium breakthrough profile for SuperLig® 639 using Hanford Tank 241-AN-103 sample is shown in Figure 3, along with rhenium breakthrough profile from intermediate and pilot-scale tests with 241-AN-105 simulant. The data show that the system outperforms the facility design criteria for each condition. The column performances for the intermediate and pilot-scale tests with simulant are similar to each other, but the radioactive sample performance in the small-scale column is significantly better. This suggests that the resin is more effective for pertechnetate than for perrhenate, despite their chemical similarities. The nitrate to pertechnetate molar ratio in the radioactive AN-103 sample (3.22E4) was higher than the nitrate to perrhenate ratio in simulant (1.82E4). Nitrate is the principal competitor with pertechnetate for the active sites on the resin, so a higher ratio would be expected to yield a lower removal efficiency, if the ions were identical. Both of these effects then yield a conservative projection for the resins' performance with this waste type in the RPP-WTP. (Note that some other Hanford waste tanks have a higher nitrate to pertechnetate ratio, and would have poorer performance)

A portion of the technetium in the AN-103 sample may be in a non-pertechnetate form, although batch contact tests indicated that more than 97% of the technetium was removable by this resin, which is selective for pertechnetate only. The higher than expected 8% breakthrough during the first 60 CV (Figure 3) (and lag column data not shown here) may be due to a number of factors, including that the resin floated during the early portion of the experiment. The floating was caused by nearly identical resin and salt solution densities. The floating was remedied by inserting quartz wool and glass beads to restrain the bed.

The effects of the flow rate on the loading profile for intermediate-scale columns with SuperLig® 639 resin are shown in Figure 4. The first three tests utilized 2.7 cm diameter columns, and the fourth test utilized the same resin sample sluiced into another column with a 4.0 cm diameter. The fifth test utilized the 2.7 cm diameter columns and a combination of two different batches of ion exchange resin and was identical to that used in the pilot-scale tests. These two batches had better performance than that used in tests 1-4. The resin batch used in the radioactive test was one of the two used in the fifth and pilot-scale test. The results indicate that the resin has relatively slow kinetics, and one batch marginally reaches the design criteria at 3.4 CV/hr. The fifth test, using the mixture of resin batches exceeded the minimum design criteria. The effect of residence time is relatively large, with faster flow rates yielding significantly faster breakthroughs. However, the results are conservative with respect to the facility design, for the reasons cited above.

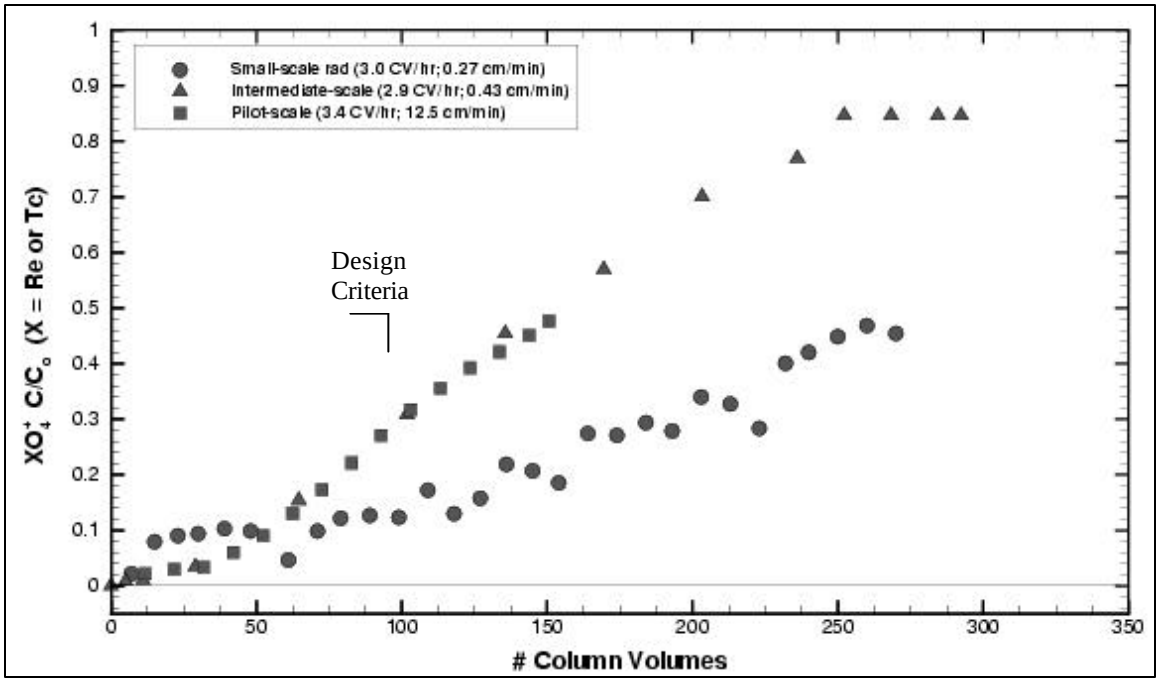

Fig 3. Pertechnetate Breakthrough Profile for Small-Scale Radioactive and Perrhenate Breakthrough Profiles for Intermediate and Pilot-Scale Simulant Tests

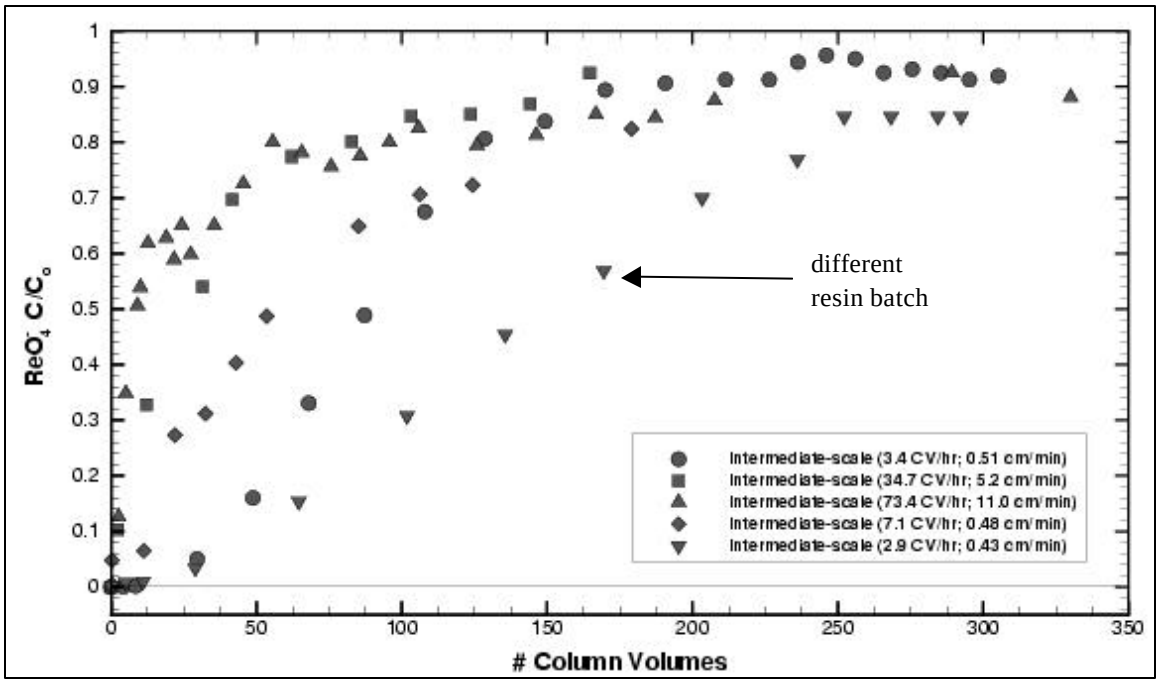

Fig 4. Perrhenate Breakthrough Profiles for Intermediate-Scale Simulant Tests at Various Flow Rates and with Different Resin Batches

Other aspects of the feasibility of using these resins in the RPP-WTP are being investigated, including the radiation stability, chemical stability, elution behavior, and performance with other waste envelopes. These results will be reported in separate documents.

CONCLUSIONS

The consistency of scale-up of ion exchange columns using SuperLig® 644 and 639 resins has been demonstrated. Maintaining constant residence time, i.e., Column Volumes per hour, yields similar breakthrough profiles with column sizes ranging from 3.5 to 230 cm in length. Experiments performed with flow rates greatly exceeding the design parameters provided valuable information on loading and diffusion parameters. These data will be input to a computer model to permit optimization of design and prediction of performance. Further details of the experimental procedures and results can be found in references 7-10.

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