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Modeling Results from Cesium Ion Exchange Processing with Spherical Resins – 11228

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ABSTRACT

Ion exchange modeling was conducted at the Savannah River National Laboratory to compare the performance of two organic resins in support of Small Column Ion Exchange (SCIX). In-tank ion exchange (IX) columns are being considered for cesium removal at Hanford and the Savannah River Site (SRS). The spherical forms of resorcinol formaldehyde ion exchange resin (sRF) as well as a hypothetical spherical SuperLig[®] 644 (SL644) are evaluated for decontamination of dissolved saltcake wastes (supernates). Both SuperLig[®] and resorcinol formaldehyde resin beds can exhibit hydraulic problems in their granular (nonspherical) forms.

SRS waste is generally lower in potassium and organic components than Hanford waste. Using VERSE-LC Version 7.8 along with the cesium Freundlich/Langmuir isotherms to simulate the waste decontamination in ion exchange columns, spherical SL644 was found to reduce column cycling by 50% for high-potassium supernates, but sRF performed equally well for the lowest-potassium feeds. Reduced cycling results in reduction of nitric acid (resin elution) and sodium addition (resin regeneration), therefore, significantly reducing life-cycle operational costs. These findings motivate the development of a spherical form of SL644.

This work demonstrates the versatility of the ion exchange modeling to study the effects of resin characteristics on processing cycles, rates, and cold chemical consumption. The value of a resin with increased selectivity for cesium over potassium can be assessed for further development.

INTRODUCTION

To support tank closures at the SRS and Hanford, a high priority need exists to treat liquid waste contained in the storage tanks. A separation technology called Small Column Ion Exchange (SCIX) is being considered that would allow in-situ treatment of stored wastes. “Small Column” means small relative to industrial sized columns, so that it can completely fit down a waste storage tank riser. Ion Exchange (IX) is a mature technology to remove cesium from liquid wastes.

Resorcinol formaldehyde (RF) resin was patented by the Savannah River National Laboratory [1] to remove cesium from highly alkaline wastes. The resin is a resorcinol ($C_6H_6O_2$) formaldehyde (CH_2O) condensation polymer. This resin has a strong preference for H^+ and can be eluted by using acid to remove Cs^+ and its competitors. Its relative affinities have been estimated to be $H^+ > Cs^+ > Rb^+ > K^+ > Na^+$. The RF resin can withstand multiple cycles of loading and acid elution before its usefulness is exhausted. The original ground-gel form of the RF resin contained shards and irregular shaped particles, causing significant flow resistance and inconsistent results as shards cracked and broke down, creating fines. Because of the poor

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hydraulic performance of the ground-gel form, the RF resin was improved by the Waste Treatment and Immobilization Plant DOE River Protection Project. RF was processed into microspheres by Microbeads AS, in Skedsmokorset, Norway, leading to much better and consistent flow performance [2-4]. In its spherical form the RF resin is referred to as sRF.

IBC Advanced Technologies, Inc. (American Fork, UT) developed a new class of sequestering ligand agents (referred to as SuperLig[®]) that selectively removes various radionuclides (cesium, strontium, etc.) from highly alkaline solutions based on molecular recognition technology and macrocyclic chemistry (covalent bonding). One of these materials (SuperLig[®] 644 or SL644) can remove cesium from simulated and actual Hanford Site alkaline waste even in the presence of excess quantities of sodium and potassium [5]. The chemical and radiolytic stability of SL644 material was investigated [6] and found to be sufficiently stable for cesium ion exchange removal processes in alkaline supernates. The relative affinities of SL644 resin for ion exchange were estimated to be $H^+ \gg Cs^+ \gg K^+ > Na^+$. In general, the SL644 shape is not spherical and can swell upon exposure to liquid solutions of varying alkalinity (i.e., can more than double its size from dry to aqueous conditions). Similar to the RF resin, SL644 resin beds exhibit hydraulic problems in its nonspherical form. Hence, it would be of great interest to explore a potential development of a spherical form of the SL644 resin.

Modeling studies have been conducted for both SL644 and sRF on radioactive alkaline wastes stored at the Hanford Site and the SRS [7-9]. One significant difference between those wastes is the concentration of potassium. In general, SRS wastes have low concentrations of potassium. Potassium is a major competitor of cesium for sites on the sRF resin so its presence will affect loading performance. In this work, a hypothetical (virtual) spherical SL644 was evaluated for removal of cesium from both Hanford and SRS supernates. The spherical SL644 performances were compared to those of sRF to assess the need for further development and testing. Note that at the WM2011 Symposia, Session 33, in which this paper will be presented, also includes other work on spherical resins discussed by Thomas Huff of the Savannah River Remediation, LLC, (paper #2) and Garrett Brown of the Pacific Northwest National Laboratory (paper #3).

MODEL FORMULATIONS

Ion Exchange Column Model

The competition for cation exchange loading at the resin sites is primarily among cesium, rubidium, potassium, and sodium for the cesium-sRF resin system or among cesium, potassium, and sodium for the cesium-SL644 resin system. Based on available batch equilibrium studies, the relative affinities for adsorption have been computed to be $Cs^+ > Rb^+ > K^+ > Na^+$ (RF) [8] and $Cs^+ \gg K^+ > Na^+$ (SL644) [7]. Given this information, early column performance (i.e., the first 5 to 10 bed volumes) generally requires the use of the multi-component formulations. Long-term performance however should be adequately handled using a simpler “effective” single-component formulation as discussed below.

In this model the kinetics associated with local ion exchange at an active resin site are assumed to be very fast (faster than the various liquid mass transfer mechanisms that transport ions to that site). Assuming radial effects to be negligible within the active region of the packed bed (i.e., a

large column-to-particle diameter ratio), a one-dimensional solute (ion) transport equation for the mobile phase (within the bed) becomes

$$\underbrace{\frac{\partial C}{\partial t}}_{\text{storage}} = \underbrace{E_b \frac{\partial^2 C}{\partial z^2}}_{\text{axial dispersion}} - \underbrace{u_o \frac{\partial C}{\partial z}}_{\text{advection}} - \underbrace{\frac{3(1-\varepsilon_b)k_f}{R_p \varepsilon_b} (C - C_{p,r=R_p})}_{\text{liquid film diffusion (mass transfer)}}$$

With boundary and initial conditions

$$\begin{aligned} z = 0: & \quad E_b \frac{\partial C}{\partial z} = u_o (C(t,0) - C_o) \\ z = L: & \quad \frac{\partial C}{\partial z} = 0 \\ t = 0: & \quad C = C(0, z) \end{aligned}$$

Assuming uniformly sized spherical particles with a homogeneous distribution of pores, a one-dimensional species transport equation for the pore phase (within an average sized particle of resin) becomes

$$\underbrace{\varepsilon_p \frac{\partial C_p}{\partial t}}_{\text{storage}} + \underbrace{(1-\varepsilon_p) \left(\frac{\partial Q}{\partial C_p} \right) \frac{\partial C_p}{\partial t}}_{\text{surface adsorption}} = \underbrace{\varepsilon_p \frac{D_p}{r^2} \frac{\partial}{\partial r} \left(r^2 \frac{\partial C_p}{\partial r} \right)}_{\text{Fickian pore diffusion}}$$

$$\begin{aligned} r = 0: & \quad \frac{\partial C_p}{\partial r} = 0 \\ r = R_p: & \quad \varepsilon_p D_p \frac{\partial C_p}{\partial r} = k_f (C - C_{p,r=R_p}) \\ t = 0: & \quad C_p = C_p(0, r) \end{aligned}$$

VERSE-LC Software and Quality Assurance

The VERSE-LC (Versatile Reaction Separation simulator for Liquid Chromatography) Version 7.8 code [10] was chosen based on its availability and accepted use in this field. Prior to applying VERSE-LC to the ion exchange modeling, the code was thoroughly verified by SRNL researchers [11]. The verification process provided quality assurance that the installed PC version of VERSE-LC was capable of adequately solving the above mentioned governing equations and also helped analysts better understand how to accurately use the VERSE-LC code (e.g., mesh refinement requirements and input/output options). For all column results presented in this report, numerical errors associated with the results of VERSE-LC should be very small when compared to the uncertainties associated with various model input parameters (bed density, particle radius, pore diffusion, etc.).

Equilibrium Cesium Isotherms

To perform column transport simulations, an “effective” single-component isotherm model in an algebraic form must be available to represent the Q-Cp relationship. The VERSE-LC Freundlich/Langmuir Hybrid isotherm model was selected in which the surface loading for cesium on the resin material can be expressed as:

$$Q = \frac{aC_p^{M_a}}{\beta + C_p^{M_b}}$$

The β parameter for cesium depends upon the other ionic competitors for adsorption (i.e., K^+ , Na^+). The β parameter contains the selectivity coefficients making it dependent upon temperature and liquid composition of all of the ionic species in solution. The larger the β parameter the less favorable (lower loadings) an isotherm will be. The a parameter is a product of the degradation factor (η_{df}) and the total resin capacity. The degradation factor is unity when considering a fresh resin and becomes less than unity during chemical exposure (i.e., cycling).

For each particular feed stream at a given temperature, the parameters a , β , M_a and M_b are estimated by fitting the isotherm equation to simulated data generated by the thermodynamic equilibrium CERMOD (for the sRF resin) or TMIXP (for the SL644 resin) codes. Both CERMOD and TMIXP codes were developed at SRNL.

Axial Dispersion

Axial dispersion in packed columns is the result of mechanical dispersion added onto molecular diffusion. For practical flowrates mechanical dispersion dominates. In the Reynolds number range of interest (i.e., 0.001 to 1000), the Chung and Wen correlation is applicable for sufficiently large columns (i.e., large diameter and length) and is expressed as [12]:

$$E_b = \frac{2R_p u \varepsilon_b}{0.2 + 0.011 \text{Re}^{0.48}}$$

where the standard deviation of this correlation based on all available data points was reported to be 46%. The Chung and Wen correlation applies for only sufficiently large columns and correction factors must be considered for columns with small diameters and/or short active bed lengths. The study cases in the SRNL work have the Reynolds numbers ranging from 0.2 to 1.1.

Film Diffusion

VERSE-LC has the Wilson and Geankoplis correlation as an option, and this correlation falls well within the spread of available low Reynolds number data (i.e., 0.0016 to 55). For each ion species considered, the Wilson and Geankoplis correlation is expressed as [13]:

$$J \equiv \left[\frac{k_{fi}}{u \varepsilon_b} \right] Sc_i^{2/3} = \frac{1.09}{\varepsilon_b} \text{Re}^{-2/3}$$

A standard deviation of approximately 25% is reported for this correlation by Wilson and

Geankoplis.

Pore Diffusion

In the VERSE-LC code, the model for intraparticle diffusion assumes that the particles are spheres of uniform radius. To apply the VERSE-LC model it is therefore necessary to determine an average radius from the particle size distribution and an effective pore diffusivity.

Assuming that the pore sizes are large relative to the size of the migrating ions of interest, pore diffusion coefficients should not be significantly lower than their bulk or free stream values. However, some level of reduced diffusion in the pores is expected resulting from bends along the pore paths that are generally accounted for by a particle tortuosity factor τ defined as:

$$D_p = \frac{D_\infty}{\tau}$$

The cesium diffusivity in the bulk feed solution, D_∞ , was calculated using the Nernst-Haskell equation. Tortuosity factors for SL644 and sRF resin have been determined [7, 8] by analyzing experimental data obtained. It was found that using $\tau = 3$ gave reasonable results.

WASTE COMPOSITIONS AND PROPERTIES

In the column simulations performed in this study, feeds at varying compositions from the wastes stored at Hanford and the SRS were considered:

- Hanford: Hot Commissioning (HC), Envelope B (EB), and Subsequent Operations (SO)
- SRS: Tank 3 and Tank 37

SRS waste is generally lower in potassium and organic components than Hanford waste. The feed compositions are listed in Table I. Other feed properties (i.e., diffusivity, density, and viscosity) are shown in Table II.

Table I. Feed Compositions

Species (M)	Hot Commissioning	Envelope B	Subsequent Operations	Tank 3 (SRS)	Tank 37 (SRS)
K ⁺	0.48	0.091	0.15	0.002	0.01
Na ⁺	4.57	4.13	4.60	6.0	6.0
Cs ⁺	2.74x10 ⁻⁵	2.83x10 ⁻⁴	5x10 ⁻⁵	6.35x10 ⁻⁵	1.08x10 ⁻⁴
OH ⁻	1.85	1.28	0.8	0.545	1.97

Table II. Feed Physical Properties

Feed	T (°C)	Diffusivity (cm ² /min)	Density (g/ml)	Viscosity (cP)
HC	25	4.935x10 ⁻⁴	1.218	2.503
EB	25	6.087x10 ⁻⁴	1.182	1.970
SO	25	5.706x10 ⁻⁴	1.218	1.979

Tank 3	25	6.010×10^{-4}	1.245	1.830
Tank 37	25	4.740×10^{-4}	1.232	2.590

SCIX COLUMN SIMULATIONS

As envisioned, the SCIX process would treat the stored waste using ion exchange columns suspended inside a waste tank. The columns will use a selective elutable resin (e.g., sRF or SL644) to remove cesium from the waste. After sorption of cesium, the loaded cesium is eluted, and the cesium-containing eluate will be further processed in a high-level waste facility (e.g., the DWPF glass melter at the SRS). The eluted resin can be reused in another ion exchange cycle.

SCIX Column Design

The SCIX column design was developed by Oak Ridge National Laboratory and Savannah River National Laboratory. The design overcame earlier issues related to column overheating and long contact time using an annular configuration with an outer diameter of 28" and a 6-inch diameter inner cooling tube. The outer tube has a wall thickness of 5/16" and the inner tube is 6" schedule 40 pipe. The cross-sectional area of the annular ion exchange bed is 0.3575 m^2 . In this study, a column length of 15 ft was chosen.

The proposed design consists of two columns used in series during loading (i.e., a lead column followed by a lag column). When the concentration of cesium in the effluent from the second (lag) column reaches a limit value pre-determined based on regulatory compliance, the ion exchange cycle is stopped. Table III lists the required limit cesium concentrations at the lag column exit for each type of waste feeds. Upon meeting the lag column exit cesium criterion, the first (lead) column is then removed from service, the partially loaded lag column is placed in the lead position, a fresh column is placed in the lag position and the next cycle is started. With sRF or SL644 resin, cesium will be eluted from the loaded column and the eluted column is placed back into service as the lag in the next cycle. The resin can be reused until degradation from the exposure to chemicals and radioactivity becomes too great.

Table III. Cesium Concentration Criterion at the Lag Column Exit

Feed	Lag Column Exit Cs ⁺ Criterion Cs+ (M)	Lag Column Cs ⁺ Concentration Ratio Criterion (C/C _o)
Hot Commissioning	3.7×10^{-8}	1.35×10^{-3}
Envelope B	3.4×10^{-8}	1.20×10^{-4}
Subsequent Operations	3.4×10^{-8}	6.80×10^{-4}
Tank 3	4.7×10^{-9}	7.40×10^{-5}
Tank 37	4.65×10^{-9}	4.30×10^{-5}

Resin Properties

Properties for the sRF resin used in this study are the same as those used previously [8, 9]. For the hypothetical spherical SL644 resin, properties are estimated based on the characteristics and structure of both the granular SL644 resin [7] and the sRF resin. Table IV summarizes the properties for the two resins under consideration. Table V lists the Freundlich/Langmuir isotherm parameters for the sRF resin estimated based on the thermodynamic equilibrium data

generated by the CERMOD code. The isotherms for the spherical SL644 resin are assumed to be the same as those for the granular SL644. The spherical SL644 isotherm parameters obtained from estimation based on the thermodynamic equilibrium data generated by the TMIXP code are shown in Table VI.

Table IV. Na⁺-Form Resin Physical Properties

Property	sRF	Spherical SL644
Bed density ρ_b , g/ml	0.32	0.2792
Solid density ρ_s , g/ml	1.615	1.451
Bed porosity ε_b	0.42	0.42
Particle porosity ε_p	0.6579	0.6682
Average particle diameter, μm	460	460

Table V. Freundlich/Langmuir Isotherm Parameters for the sRF Resin

Feed (@ 25°C)	a	β	M_a	M_b
HC	0.2632	2.076E-3	0.9850	0.8695
EB	0.2401	4.783E-4	0.9727	0.9042
SO	0.2458	8.174E-4	0.9761	0.8941
Tank 3	0.2373	4.805E-4	0.9736	0.9066
Tank 37	0.2345	2.737E-4	0.9706	0.9142

Table VI. Freundlich/Langmuir Isotherm Parameters for the Spherical SL644 Resin

Feed (@ 25°C)	a	β	M_a	M_b
HC	0.1441	5.1474E-4	1	1
EB	0.1418	1.4192E-4	1	1
SO	0.1376	2.4279E-4	1	1
Tank 3	0.1326	2.552E-4	1	1
Tank 37	0.1444	1.3707E-4	1	1

Column Performance Study Cases

The objectives of this work were, through modeling, to predict the performance (i.e., number of processing cycles) of both sRF and spherical SL644 resins for the removal of cesium from Hanford and SRS wastes. The study investigated the column performance in the following cases:

1. Nominal flow rate (10 gpm)
2. High flow rate (20 gpm)
3. Low flow rate (5 gpm)

For the SRS wastes stored in tanks 3 and 37, simulations were carried out until the tanks were empty. For Hanford wastes, since specific tanks are not known, the cases were simulated sufficiently long (e.g., 1800 bed volumes [BV]) to obtain meaningful number of cycles.

COLUMN MODELING RESULTS

All column study cases were carried out for the Hanford and SRS waste feeds to predict the performance of both sRF and spherical SL644 resins for cesium removal. Note that during the first cycle, both columns contain fresh resin. After the first cycle in all subsequent cycles, the partially loaded lag column is placed into the lead position and a clean (eluted) column is placed into the lag position at the start of the cycle. Since, after the first cycle, a partially loaded column is in the lead position, less waste volume is processed per cycle before the cesium concentration operating limit is reached.

For illustration, the results for the Hot Commissioning and Tank 37 feeds in the nominal flow rate case are plotted in Figures 1 through 4 for both sRF and spherical SL644 resins. In these figures, the cesium concentrations relative to the feed concentrations (C/C_0) are displayed at the exit of the lead column (red solid line) and the lag column (blue dashed line), respectively. As described under the "SCIX Column Design" section, the ion exchange cycle is stopped, when the effluent concentration of cesium in the lag column reaches the limit value. The lead column is then removed from service; the lag column becomes the lead column. A fresh column is placed in the lag position and a new cycle begins. Therefore, in Figures 1 through 4, a complete "spike" represents a full cycle.

Table VII provides a summary of the number of processing cycles predicted for both resins at the nominal, high and low flow rate conditions. For clarity, the results for nominal, high and low flow rates are highlighted with yellow, green and brown backgrounds, respectively. In Table VII, the resin performance is predicted based on 1800 BVs for the Hanford wastes and on the tank capacity for the SRS wastes. The ratios of number of sRF cycles and spherical SL644 cycles are computed for comparison of the resin performances. Spherical SL644 performs better when the ratio is larger than 1, while the less-than-1 ratio indicates a better performance for sRF. As discussed above, the relative affinities for adsorption were computed to be $Cs^+ > Rb^+ > K^+ > Na^+$ for the sRF resin and $Cs^+ \gg K^+ > Na^+$ for the SL644 resin. That means K^+ would compete more against Cs^+ for adsorption onto sRF than onto SL644. Consequently, the presence of high K^+ concentration in a waste would eventually decrease cesium sorption on the sRF resin, resulting in more processing cycles. Table VII confirms this phenomenon at all flow rates.

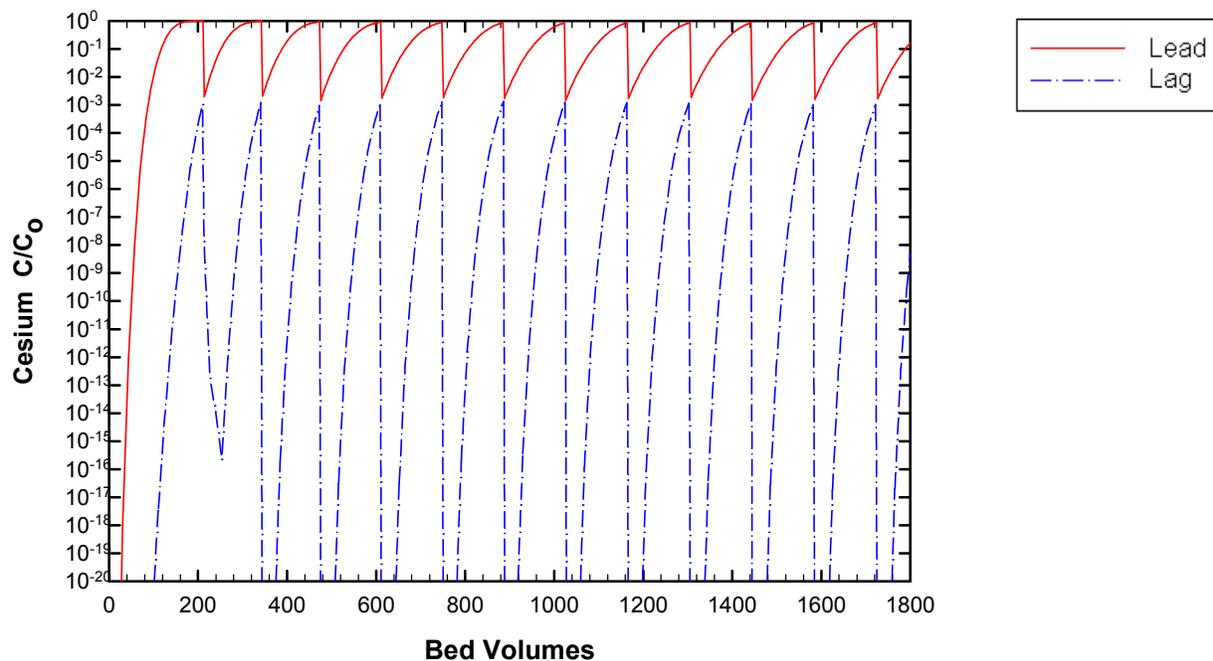


Fig. 1. Cesium breakthrough prediction for Hot Commissioning Operations feed at nominal condition (flow rate: 10 gpm, temperature: 25°C) with 460 μm diameter RF spheres.

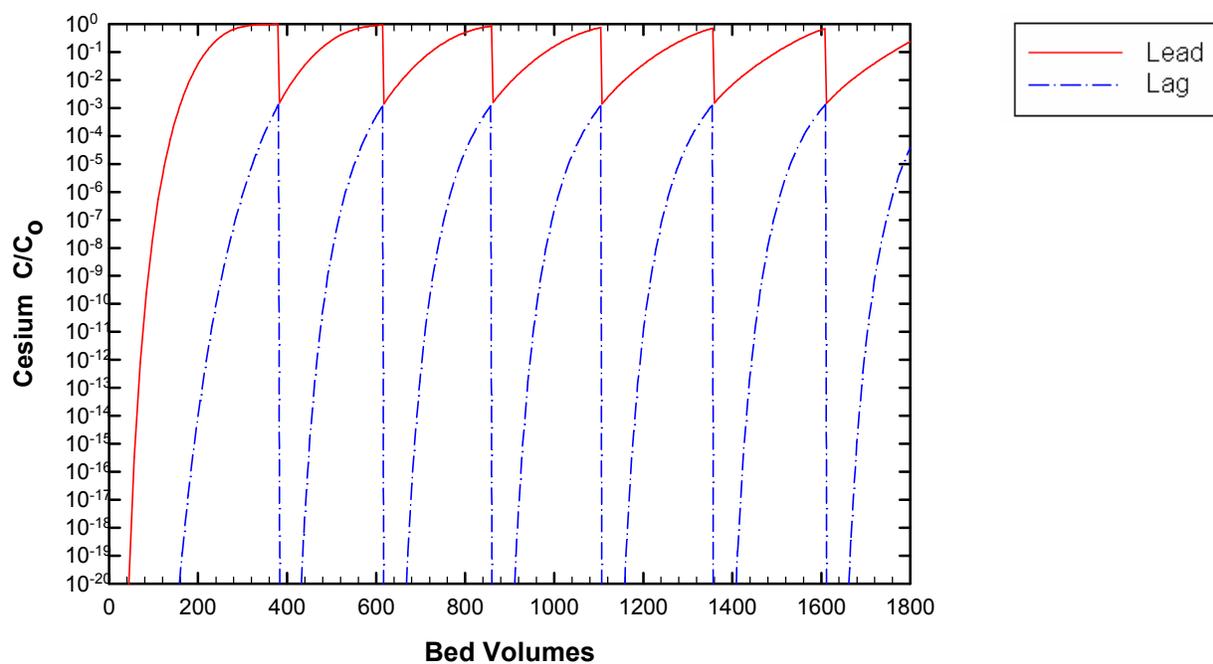


Fig. 2. Cesium breakthrough prediction for Hot Commissioning Operations feed at nominal condition (flow rate: 10 gpm, temperature: 25°C) with 460 μm diameter SL644 spheres.

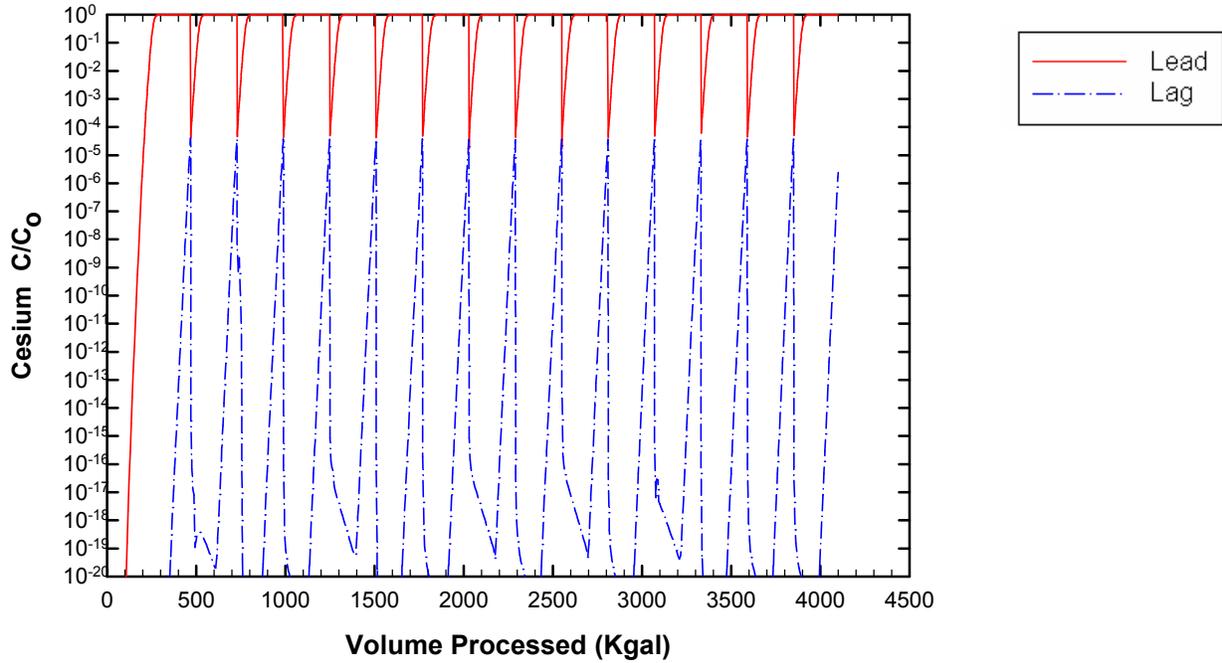


Fig. 3. Cesium breakthrough prediction for Tank 37 feed at nominal condition (flow rate: 10 gpm, temperature: 25°C) with 460 μ m diameter RF spheres.

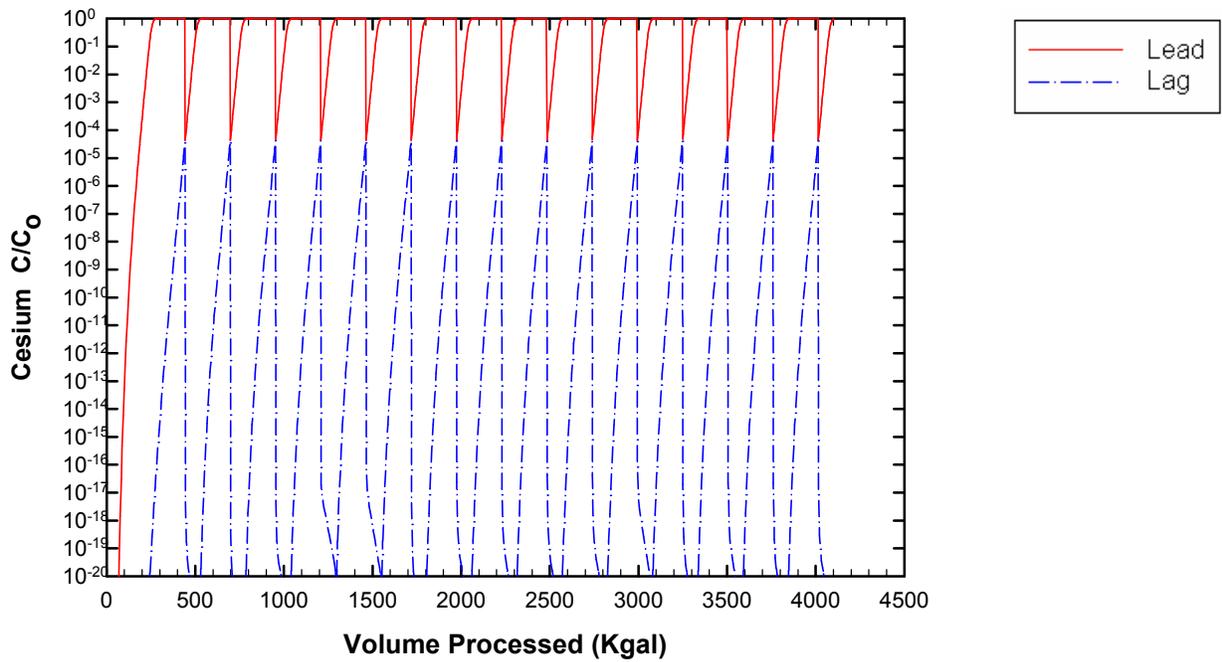


Fig. 4. Cesium breakthrough prediction for Tank 37 feed at nominal condition (flow rate: 10 gpm, temperature: 25°C) with 460 μ m diameter SL644 spheres.

Table VII. Performances for the sRF and Spherical SL644 Resins.

	Number of Cycles		Ratio of Number of Cycles sRF/Spherical SL644
	sRF	Spherical SL644	
Nominal HC	12	6	2.00
Nominal EB	5	4	1.25
Nominal SO	4	3	1.33
Nominal Tank 3	9	11	0.82
Nominal Tank 37	14	15	0.93
High-flow HC	13	7	1.86
High-flow EB	5	4	1.25
High-flow SO	5	3	1.67
High-flow Tank 3	9	11	0.82
High-flow Tank 37	15	15	1.00
Low-flow HC	12	6	2.00
Low-flow EB	5	4	1.25
Low-flow SO	4	3	1.33
Low-flow Tank 3	9	10	0.90
Low-flow Tank 37	14	15	0.93

CONCLUSIONS

Both sRF and spherical SL644 performance was simulated with Hanford and SRS wastes at various flow rate conditions (i.e., 5 gpm, 10 gpm, and 20 gpm). The following conclusions were drawn:

- Spherical SL644 is superior to sRF for high-potassium feeds (Hanford wastes).
- Spherical SL644 and sRF perform equally well for low-potassium feeds (SRS wastes).
- Reduced cycling results in reduction of nitric acid consumption (resin elution) and sodium addition (resin generation), therefore, significantly reducing life-cycle operational costs.
- Further development and testing of a spherical form of SL644 are recommended.

NOMENCLATURE

a	Freundlich/Langmuir Hybrid isotherm model coefficient, mol/L
C	Bulk phase solute concentration, mol/L
C _p	Pore phase solute concentration, mol/L
C _o	Column inlet solute concentration, mol/L
D _p	Pore diffusivity, cm ² /min
D _∞	Bulk diffusivity, cm ² /min
E _b	Axial dispersion coefficient, cm ² /min
J	Mass transfer (Wilson and Geankoplis correlation)
k _f	Film mass transfer coefficient, cm/min
L	Column length, cm
M _a	Freundlich/Langmuir Hybrid isotherm model exponent

M_b	Freundlich/Langmuir Hybrid isotherm model exponent
Q	Solid-phase solute concentration, mol/L _{BV}
r	Radial position from center of adsorbent particle, cm
Re	Reynolds number
R_p	Resin particle radius, μm
Sc	Schmidt number
t	Time, min
u_o	Linear interstitial velocity, cm/min
z	Position in axial direction, cm
β	Freundlich/Langmuir Hybrid isotherm model coefficient
ϵ_b	Bed porosity
ϵ_p	Particle porosity
ρ_b	Bed density, g/cm ³
ρ_s	Solid density, g/cm ³
τ	Particle tortuosity factor

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