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THE EFFECT OF PRESSURE, HUMIDITY, CAUSTIC PRETREATMENT, AND ORGANIC CONSTITUENTS ON THE CESIUM ION EXCHANGE PERFORMANCE OF IONSIV® IE-911

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THE EFFECT OF PRESSURE, HUMIDITY, CAUSTIC PRETREATMENT, AND ORGANIC CONSTITUENTS ON THE CESIUM ION EXCHANGE PERFORMANCE OF IONSIV® IE-911

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ABSTRACT

We examined ^{137}Cs exchange of crystalline silicotitanate (CST) in simulated waste solution. In particular, we focused on the effect of CST pretreatment on the kinetics and extent of cesium adsorption. We used IONSIV®IE-911 (UOP LLC, Molecular Sieves Division, Des Plaines, Ill), the engineered form of CST. Pretreatment steps examined include soaking IE-911 in 2 mol/L NaOH solution for 3 days, exposing IE-911 to 50% relative humidity for 1 week, and for 3 days, soaking IE-911 in organic-containing simulated salt solution or drying untreated IE-911 in air at 100°C. Some tests were conducted with the sample under 50 and 25 psig of argon. Pretreatment of IE-911 in 2 mol/L caustic solution for 72 hours yielded a slower approach to equilibrium cesium distribution in batch contact tests than the untreated IE-911 samples. Adsorbed

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carboxylates and carbonates likely affected the cesium transport by either increasing the path length or reducing mass transfer rate. However, the effect was completely removed when IE-911 was rinsed with deionized water. Heating IE-911, as received from the vendor, at 100°C for 72 hours significantly degraded its cesium-removal performance by a 40.7% reduction in capacity and a 43% reduction in sorption rate (relative to the untreated IE-911) over 1 week of testing. However, sodium conversion of these samples did not affect cesium sorption. The presence of organic chemicals (e.g., tri-*n*-butyl phosphate, dibutylphosphate, butanol, paraffin, and Dow Corning H-10 defoamer) in simulated salt solution did not affect cesium sorption on pretreated IE-911. Nearly identical (i.e., a difference of only 5.6%) distribution coefficients (K_d) were found between lot no. 9990-9681-0004 and no. 9990-9881-0005. Increasing the atmospheric pressure from 0 to 50 psig had no effect on cesium sorption.

INTRODUCTION

Several million gallons of liquid waste are stored in steel tanks at the Savannah River Site (SRS). The waste contains soluble radionuclides, in particular cesium, along with high concentrations of sodium and potassium. Cesium must be removed before the waste could be removed. Cesium is the primary radionuclide contributing to the activity of the liquid waste.

Current engineering studies examine several processes for removing cesium from (SRS) radioactive waste. One such process uses nonelutable ion exchange with crystalline silicotitanate (CST) particles as the ion exchange media (1). CST is the resulting product of intensive research for a new class of ion exchangers by personnel at Sandia National Labs (SNL) and Texas A&M University. This material shows a large affinity for cesium even in the presence of high sodium concentrations (2–6). CST is a fine powder and cannot be used in column operations. UOP developed a manufacturing process for an engineered form of CST:IONSIV® IE-911. Column design requires knowledge of the equilibrium amount of cesium that adsorbs on IE-911. The typical measure of sorption involves the distribution coefficient (K_d), or the weighted (i.e., volume of salt solution per unit weight of solid) amount of adsorbed cesium relative to the initial cesium concentration in the original salt solution.

$$K_d = \left(\frac{\text{Concentration}_{\text{Initial}}}{\text{Concentration}_{\text{Final}}} - 1\right) \times \frac{\text{Volume of Salt Solution}}{\text{Dry Weight of CST}}$$

The distribution coefficients measured previously varied significantly with the method of IE-911 pretreatment (7). Although the previous work involved CST particles (7), similar effects are expected on the UOP-engineered form because the binder is an inert and minor component of IE-911. The initial water content and the effects of soaking IE-911 in caustic solution significantly affected cesium adsorption. Upon further review of the data, we discovered the need to study other variables. In particular, for use in the proposed application, the effect of pressure and the presence of organic compounds in salt solutions on the cesium distribution coefficient must be understood (8–13). The presence of soluble organic compounds in SRS liquid waste derive from the solvent used to extract uranium and plutonium from the waste by solvent extraction techniques. During service, IE-911 will be placed in a 16-ft column in which the portions of IE-911 at the column bottom may experience pressure in excess of 1.5 atm. This pressure may affect the equilibrium ratio between soluble and absorbed cesium. The IE-911 must also receive caustic solution pretreatment before use. Such pretreatment avoids alumina precipitation inside the pores, helps remove loose materials, and facilitates gas removal from the pores. Questions have been raised about the effect of caustic pretreatment on the kinetics and capacity of cesium sorption on IE-911. Depending on the time of the year, humidity at SRS can vary from 95 to 35 %. This large variation in humidity raised created concerns over the effect, if any, of humidity on cesium sorption. In this work, some of the IE-911 samples were exposed to different humidity extremes (95% and dried at 100°C for 72 hours). The results were compared against the untreated IE-911.

EXPERIMENTAL

Pretreatment of IONSIV® IE-911

Humidity and Heating Pretreatment

About 0.1 g of an untreated IE-911 (19.6 % water) sample was exposed to 50% relative humidity for 168 hours. At the end of the test, the humidity-treated IE-911 was immediately added to 20 mL of simulated salt solution (described below) for K_d testing. Another 0.1 g of untreated IE-911 sample was heated in an oven at 100°C for 72 hours. When the heat treatment was terminated, the sample was immediately placed in a desiccator and allowed to cool down.

Sodium Pretreatment

Two different IE-911 lots (lot no. 9990-9681-0004 and no. 9990-9881-0005) were examined during testing. Table 1 defines the different IE-911 pretreatment steps examined. First, 0.1 g of untreated IE-911 was placed in a 35-mL polybottle. About 20 mL of 2 mol/L sodium hydroxide was added to the bottle.

Table 1. IE 911 Pretreatment Steps Examined

Treatment	Duration Time
NaOH (2 mol/L)	3 days
50% Relative Humidity	7 days
100°C	3 days
0, 25, and 50 psig during test	72 hours

* Organic components consist of tri-*n*-butyl phosphate, dibutylphosphate, *n*-paraffin, *n*-butanol, and defoamer H-10 (Dow Corning).

(In some tests, simulated salt solution, described below, instead of 2 mol/L sodium hydroxide was added to the IE-911.) The bottle was shaken at 150 rpm on an orbital shaker for approximately 72 hours. At the end of the shaking step, much of the caustic solution was carefully decanted off the bottle without any IE-911 particle loss. At this point the IE-911 sample is considered sodium pretreated. The sample was not allowed to dry. No effort was made to remove fines from IE-911 because previous study in our lab showed that the presence of fines was insignificant to adsorption performance.

Preparation of Simulated Salt Solution

Two liters of simulated salt solution similar to SRS waste were prepared with the components listed in Table 2. The chemicals were added to 1.5 L of water. After the solution was stirred for 48 hours (this time was sufficient for all the chemicals to go into solution), water was added to the solution to increase the total volume to 2 L. The final simulated salt solution contained 19 mg/L cesium (cesium was added as cesium chloride). In addition, the simulated salt solution was traced with ^{137}Cs (about 10^6 decomposition per minute) for counting purposes. In some tests, organic compounds with composition listed in Table 3 were added to the simulated salt solution. This organic compound–containing simulated salt solution was filtered before use.

K_d Test Protocol

For the K_d test, 20 mL of simulated salt solution was added to pretreated sample. In some cases, organic compound–containing simulated salt solution was added to pretreated IE-911. The resulting slurry was shaken with an orbital shaker at a frequency of 150 rpm for 24, 48, 72, and 168 hours. At the end of each test, the liquid was separated from the solids, placed in glass vials, and analyzed for ce-

Table 2. Simulated Salt (Waste) Composition

Component	Concentration (mol/L)
Na^+	5.6
K^+	0.015
Cs^+	1.4×10^{-4}
OH^-	1.91
NO_3^-	2.14
NO_2^-	0.52
AlO_2^-	0.31
CO_3^{2-}	0.16
SO_4^{2-}	0.15
Cl^-	0.025
F^-	0.032
PO_4^{3-}	0.010
$C_2O_4^{2-}$	0.008
SiO_3^{2-}	0.004
MoO_4^-	0.0002

sium by gamma counting spectroscopy. All K_d tests were adjusted for water content in the IE-911 samples (19.68%).

High Pressure K_d Test

For high-pressure K_d testing, we used the device shown in Fig. 1. Approximately 25 mL of simulated salt solution that contained cesium and 0.1 g of untreated IE-911 (19.6% (wt) water) was added to the vessel. The sealed compartment was shaken under pressure (0, 25, and 50 psig) for 72 hours. At the end of the test, the solution was separated from the solids and analyzed for cesium through gamma spectroscopy.

Table 3. Organic Species for Fouling Test

Material	Concentration
Tri-*n*-butyl phosphate	Saturation level (<10 ppm)
DBP and *n*-paraffin	Saturation level for each (2.0 g/L DBP, 10 ppm *n*-paraffin)
Butanol	2 g/L
Evaporator defoamer (Dow Corning Antifoam H-10)	100 ppm

DBP stands for di-butyl phosphate.

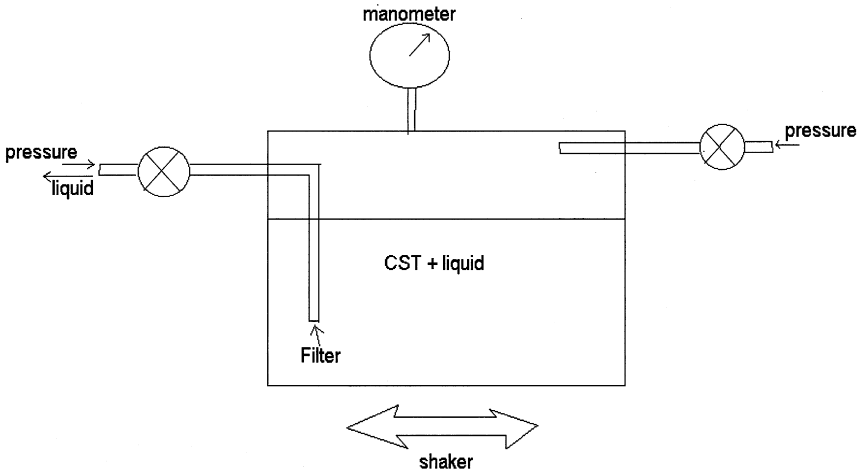


Figure 1. Schematic of the device used for testing the effect of pressure on cesium sorption.

K_d Computation and Error Estimates

The technically correct term for expressing IE-911 cesium capacity under nonequilibrium conditions is D_{Cs}. Because some cesium exchange tests reached steady state, the symbol K_d will be used throughout this paper regardless of whether or not equilibrium conditions had been reached. Computation of the K_d value requires the ratio of the initial to final cesium concentration. Instead, the ratio of the corresponding gamma counts was substituted. This substitution remains valid as long as a linear relationship exists between cesium concentration and gamma counts. Figure 2 provides a calibration curve for the spectrometer and shows the validity of the assumption. Calibration occurred only twice during these experiments: once before measurements and a second time after data collection. In addition to a check for linearity in performance, the performance of the gamma counter was verified through an independent analysis of a set of samples by the Analytical Development Section (ADS) at SRS. Table 4 lists the highest measured concentrations as determined by the ADS equipment and by the equipment used for our tests.

This table lists the highest percentage difference between these 5 samples as 7.3%. Hence the combined error from the variation of the in-house instrument and its inaccuracy approaches 8%. The analysis included measurement of a standard and blank after every third sample tested.

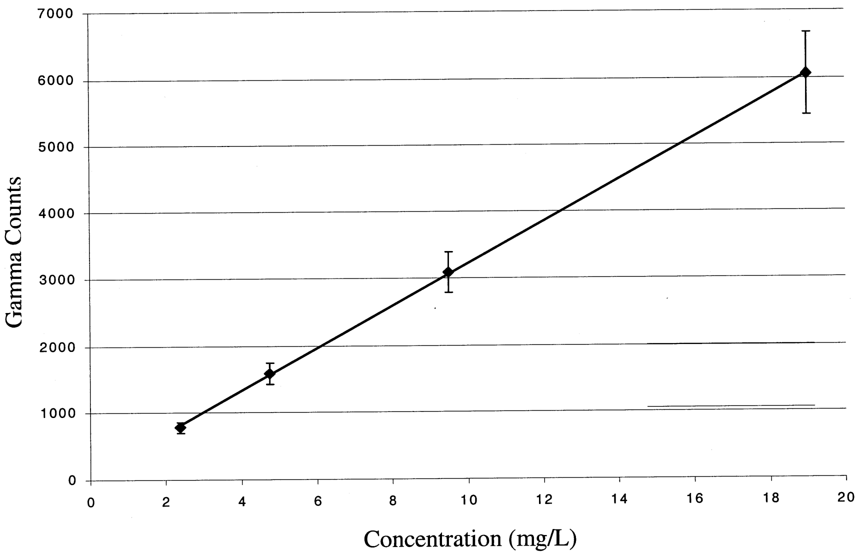


Figure 2. Calibration curve of in-house gamma counter.

Error was determined by the following propagation formula:

$$\left(\frac{\Delta K_\mathrm{d}}{K_\mathrm{d}}\right)^2 = \left(\frac{\Delta C_\mathrm{initial}}{C_\mathrm{initial}}\right)^2 + \left(\frac{\Delta C_\mathrm{final}}{C_\mathrm{final}}\right)^2 + \left(\frac{\Delta V}{V}\right)^2$$

Sample weighing errors (approximately 0.5%) were insignificant compared to errors from gamma counting. Error in gamma counting is determined by the square root of the total gamma count ($\pm$ 1.96 $\times$(counts)$^{0.5}$). The gamma-count percentages of error ranged from 0.2% (for 6000 counts) to 0.8% (for 600 counts). This error is insignificant compared to sampling errors considered through the

Table 4. Disintegration per Minute per mL Comparison Between In-House Counter and the Analytical Division Services (ADS) equipment

ADS Equipment	In-house Counter	% Difference
4643.2	4902	5.6
5045.4	4678	7.3
4174.2	4320	3.3
3617.2	3424.5	5.3
23 127	23 740.5	2.7

Percentage difference relative to ADS.

propagation of error formula. The difference in gamma counting from duplicate samples ranged from 20 counts for consecutive analyses to 450 counts when time between analyses measured 2 hours. These differences represent errors of 1.2 to 9% in the calculated K_d values.

Microscopy and Spectroscopy Equipment

The IE-911 samples were analyzed by scanning electron microscopy (SEM) and energy dispersed X-ray fluorescence spectroscopy through the use of an ISI DS130 dual stage microscope. All samples were rinsed with water to remove salt residues at the surface of the particles. Selected samples were analyzed by molecular infrared spectroscopy (IR) with a Nicolet 220 spectrometer. For IR analysis, IE-911 samples were rinsed with distilled water to remove residual salts at the particle surface. Then the sample was placed in a nitrogen purge bag and ground with a mortar and pestle. The grounded sample was mixed with potassium bromide and placed inside a Diffuse Reflectance Infrared Fourier Transform accessory for analysis.

RESULTS AND DISCUSSION

The Effect of Heating and Relative Humidity

Figure 3 shows the effect of heating the untreated IE-911 for 72 hours at 100°C, and other pretreatment steps on the cesium removal rate. Inspection of the figure suggests most experiments showed a region where the rate of cesium exchange appeared relatively constant (i.e., zero-order kinetics). One can approximate the rates from the slopes of the lines in the figure. Table 5 shows the cesium ion exchange rate as determined from the 24- and 48-hour data points of the untreated, heated, and pretreated IE-911.

Heating the IE-911 at 100°C for 72 hours greatly reduced cesium ion exchange rate (12.7 mL/(g·h)) and extent (1241 mL/g) relative to that for the nontreated material. The reduced capacity may reflect irreversible pore size reduction during dehydration (6) that significantly reduces the number of available absorption sites. The same dehydration effect is expected to occur in other pretreatments of IE-911.

As shown in Fig. 3, humidity had no effect on cesium sorption.

The Effect of Caustic Treatment

Soaking IE-911 in 2 mol/L NaOH for 24 hours converts the material to the sodium form. According to literature, the cesium capacity should change slightly

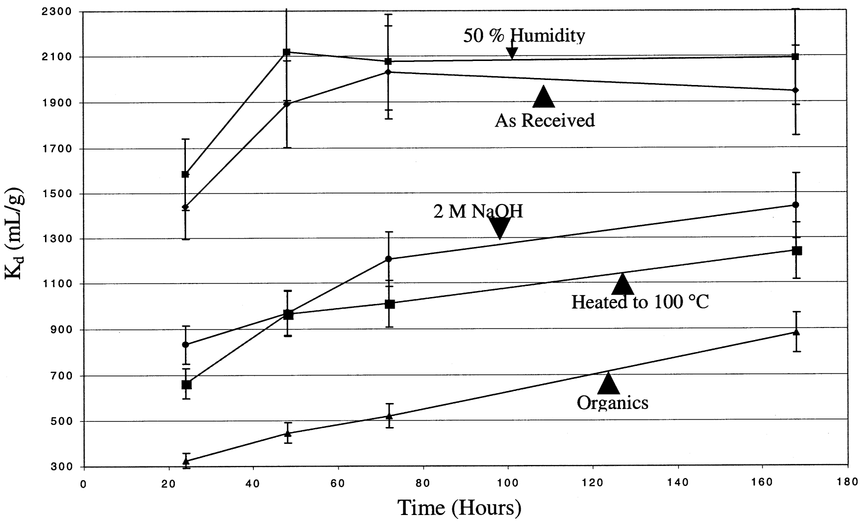


Figure 3. The effect of different IE-911 pretreatments on the cesium K_d values.

after it is soaked in 2 mol/L caustic solution (1). However, the observed cesium ion exchange rate and capacity in these tests after caustic pretreatment proved slower and lower than the untreated IE-911 (see Fig. 3 and Table 5). This indicates that the cesium-sodium exchange has a different capacity than the cesium-hydrogen exchange. This difference in the cesium K_d values between the sodium and the untreated IE-911 may be due to an inherently low thermodynamic equilibrium between sodium and cesium. Another possible explanation is fouling of the pretreated IE-911. To check the validity of this hypothesis, surface analysis of the inside of the pretreated IE-911 (ground particles) may reveal absorbed species. Molecular spectroscopy (IR difference spectrum) identified carboxylate and carbonates on pretreated IE-911 (Fig. 4).

The IR difference spectrum results from the subtraction of the IR spectrum data of the untreated IE-911 from the IR spectrum data of the pretreated IE-911.

Table 5. The Effect of Pretreatment on the Initial Cesium Ion Exchange Rate

Pretreatment	Initial Rate (mL/(g·h))	K_d (mL/g) at 168 hours
None	18.8	1949
50% Relative humidity	22.3	2094
100°C heated	12.7	1241
2 mol/L NaOH	7.7	1440
Organic compounds in salt solution	3.8	883

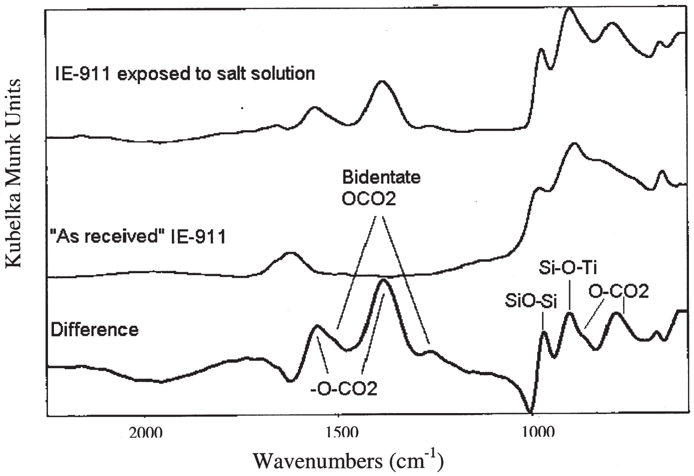
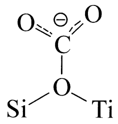
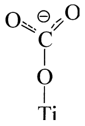
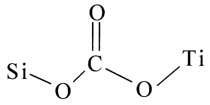


Figure 4. The IR difference spectrum of IE-911 after 4000 bed volumes of salt solution was applied to the system.

The IR difference spectrum (Fig. 4) shows absorption near 1550 cm^{-1}, 1365 cm^{-1}, 877 cm^{-1}, 768 cm^{-1}, and 679 cm^{-1}. The first 2 bands at 1550 cm^{-1} and 1365 cm^{-1} represent the asymmetric and symmetric stretch of the —CO_3^{2-} molecule, and the other bands are associated with bending motions. The behavior at 905 cm^{-1} represents the surface Si—O—Ti bands perturbed by the sorption of sodium ions. The loss of absorption near 3600 cm^{-1} arises from the loss of hydroxyl surface hydrogen molecules due to carbonate absorption, which was described in the caustic pretreatment section.







A carbonate molecule bonding via either a titanium or silicon atom best explains the absorption bands at 1532 and 1362 cm^{-1} (Fig. 4). The data in Fig. 4 imply that the absorbed molecule has a relative high stability. No nitrogen was detected by energy dispersed X-ray fluorescence on the ground IE-911 samples; these results mean that neither nitrate nor nitrite salts caused the reduced sorption performance.

After prolonged exposure (i.e., 168 hours) of IE-911 to simulated waste, alkaline earth carbonates and bidentate carbonates form. These compounds can form by exposure to carbon dioxide as follows:

$$\mathrm{Ti} - \mathrm{OH} + \mathrm{NaOH} + \mathrm{CO_2} = \mathrm{Ti{-}O} \rightarrow \mathrm{CO_2^-}\ \mathrm{Na^+} + \mathrm{H_2O}$$

The absorbed carbonates may cause steric hindrance of cesium transport. Pretreating the particles may cause carbonates and carboxylates to accumulate at a level that influences kinetics as well as capacity. IR analysis of the “as received” particles showed that the untreated samples had no foreign substance on its surface or interior implying that no carbonate was present before the pretreatments (sample preparation was done under nitrogen).

Another potential explanation involves an increase in film diffusion resistance (which cannot be accounted for by particle fouling). According to the literature, the effect of increased film diffusion resistance manifests itself as a linear relationship between K_d and time (6). The pseudolinear relationship seen in Fig. 3 could indicate an increase in film diffusion resistance as a consequence of caustic pretreatment. The limited data available do not allow us to verify the hypothesis of film diffusion resistance or to quantify the resistance of it. However, when pretreated IE-911 was rinsed with deionized water, the cesium sorption rate and capacity was similar to that of the untreated IE-911 indicating that the effects are removable.

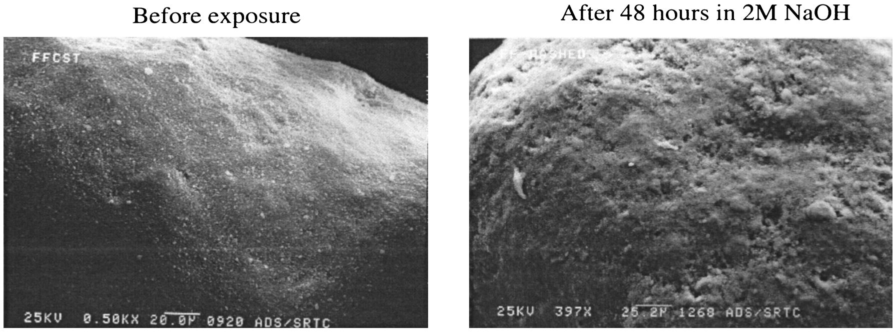
The IE-911 particles were also investigated for physical and chemical changes. Observations of the IE-911 particle surface via SEM showed pits and craters after the exposure to caustic solution (Fig. 5). The pretreatment results in larger surface area and loss of mass per particle. This finding indicates some degree of chemical instability of the IE-911 in SRS waste solution. These effects could translate into weaker particles and loss of capacity; therefore, the effects merit more extensive study.

Experiments were also conducted to investigate the effect of drying pretreated IE-911 on the resulting cesium ion exchange ability. IE-911 was placed in caustic solution for 2 days. At the end of 2 days, the solution was decanted and the solids maintained at room temperature for 24 hours. Table 6 and Fig. 3 show the result from a subsequent K_d test conducted on the dried material.

Although the percent difference decreased with contact time, the data indicate that drying IE-911 after treatment reduces the ion exchange ability. Drying of pretreated material adversely impacts performance. Hence, circumstances that could lead to drying during interim storage of IE-911 samples should be avoided.

The Effect of Simulated Salt Solution Exposure on IE-911 Performance

Samples of sodium treated IE-911 (0.1 g) were placed in simulated salt solution (with no cesium) for 2 days. The simulated salt solution was removed from the



*White spots are binder-enriched regions.

*This picture shows the formation of pits and craters after caustic solution treatment.

Figure 5. SEM picture of the surface of IE-911 (a) Before treatment. White spots are binder-enriched regions. (b) After 48 hours in 2 mol/L caustic solution. The pits and craters formed after caustic solution treatment can be seen.

sorbent and then the sorbent was subsequently placed in a simulated salt solution containing cesium. The test yielded a K_d of 1340 mg/L (not shown in Figure 3) after 1 week. Although we did not measure the K_d throughout the week, the low value indicates slow kinetics relative to the untreated material. The sorbent was then dried and investigated for physical and chemical changes. Microscopic observation again showed pits and craters as the result of exposure to the salt solution. Examination of the molecular insides of the particle showed the presence of absorbed carbonates and carboxylates. No nitrogen was detected by energy dispersed X-ray fluorescence, ruling out nitrate or nitrite salts causing the reduced sorption performance.

Table 6. K_d Values of IE-911 Dried and Wet After Caustic Treatment

Time (hours)	Dried After 2 mol/L NaOH Treatment (mL/g)	Wet After 2 mol/L NaOH Treatment (mL/g)	% Difference
24	610	834	26.8
48	774.5	973	20.4
72	995.5	1206	17.4

Percentage difference relative to wet sample.

The Effect of Organic Compounds on K_d Values

Some samples of pretreated IE-911 were placed in simulated salt solution that was saturated with selected organic compounds (Table 3) for 24 hours. Figure 3 shows the cesium K_d values of samples subjected to organic-compound solutions as a function of time. The K_d values increased linearly within a 168-hour period. At the end of this period, the K_d value of 883 mL/g (Fig. 3) fell well below the value obtained from the untreated material during the same time period. However, the K_d value of 883 mL/g is not statistically different from the sodium pretreated IE-911 K_d value. We made efforts to identify physical and chemical changes in the material that correlated with material performance. According to SEM, the washed particle showed pits and craters just as in the case of the IE-911 in salt solution. IR analysis of the inter-structure of the IE-911 indicated the presence of carboxylates and carbonates. The analyses showed no hydrocarbons or phosphate on or in the particles.

An effort was made to isolate the effect of the internal carbonate by washing the material several times with distilled, deionized water. The cesium K_d value of the washed IE-911 reached a value of 1357 mL/g after 1 week. Because this value falls below those of the untreated material, the inorganic material situated on the particles may affect kinetics and capacity. The low cesium ion-exchange capacity of the unwashed material (883 mL/g) results from the deposition of material on the IE-911 samples. The linearity between K_d and time could serve as evidence for deteriorating mass transfer rate as a result of the pretreatment.

The Effect of Pressure on K_d Values

Samples of untreated IE-911 particles were contacted with cesium-containing salt solution under argon at various pressures (0, 25, and 50 psig). Figure 6 shows that pressure has negligible effect on cesium K_d values. The figure suggests no correlation of distribution coefficient with applied pressure, which is in agreement with published literature (4).

Variability of Cesium Sorption Between Manufacturer Lots

Figure 7 shows cesium sorption data from experiments on 2 different lots of material: lot 9990-9681-0004 and lot 9990-9881-0005. The tests showed good agreement (5.6% difference) between the 2 lots in the untreated state and in flowing simulated-salt solution that contained organic compounds. However, lot 9990-9681-0004 exhibited reduced cesium ion exchange capacity (28% differ-

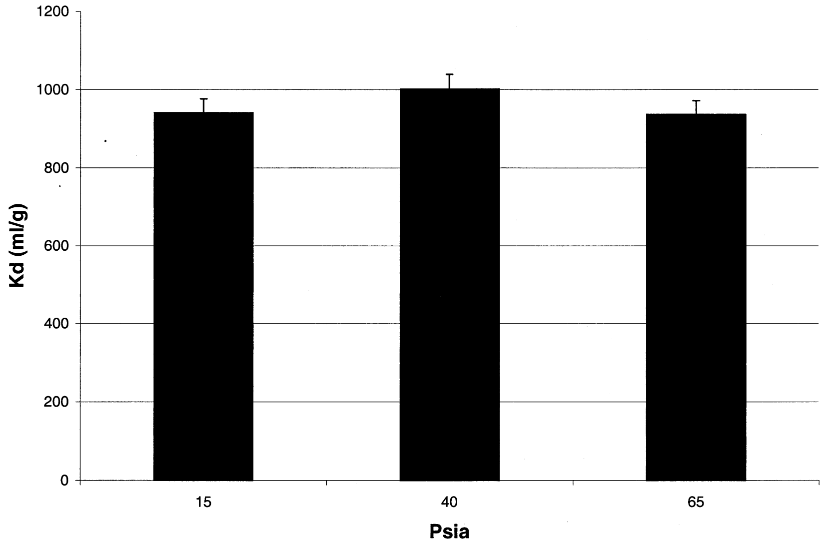


Figure 6. The effect of pressure on cesium K_d values.

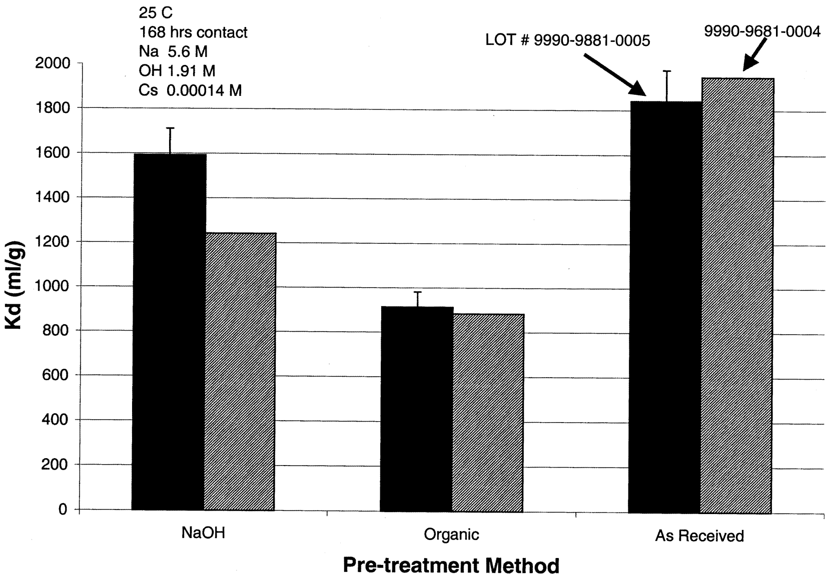


Figure 7. The combined effect of lot variability and pretreatment on cesium K_d values.

ence) when treated in 2 mol/L NaOH compared to the samples in the untreated state.

IE-911 Stability in Simulated Salt and Caustic Solution

Samples of IE-911 were placed in simulated salt and caustic solutions for 2 weeks. In one case, the test continuously flowed 4000 bed volumes of salt solution (over 2 weeks) through a column packed with the IE-911 (at a superficial velocity of 5 cm/min). After contacting the IE-911 with the solution, the particles were washed and analyzed for topography and chemical composition. Surface topography images revealed the pits and craters; similar surface changes had occurred with materials exposed to caustic solution (2 mol/L). The eroded fines could cause column plugging. Microscopy analysis shows that the chemical attack and pitting appears limited to the surface, which could imply no significant reduction in column performance. However, formation of small fines may result in passage of radio-cesium from the column, thereby detracting from performance. Additional longer-term exposure with subsequent column testing will provide insight into process limitations.

CONCLUSIONS

Pretreatment of IE-911 affects the rate at which equilibrium is obtained. Depending on the treatment, the rate of cesium ion exchange may decline sharply. IR spectroscopy of the pretreated IE-911 samples identified carbonate species that could cause internal fouling. These species developed as a result of the caustic pretreatment. In particular, IE-911 in salt solution with organic addition (e.g., tri-*n*-butyl phosphate, dibutylphosphate, butanol, paraffin, and Dow Corning H-10 defoamer) showed a linear temporal behavior and the most detrimental impact on performance. However, the presence of organic compounds did not statistically affect the cesium sorption on sodium-treated IE-911 samples. Different batches of IONSIV® IE-911 exhibited nearly identical cesium distribution coefficient (K_d). However in the case of IE-911 treated with caustic solution, rinsing with distilled and deionized water improved cesium sorption.

During 2 weeks of contact between IE-911 and a simulated salt solution, the particles developed pits and craters. These observations raise questions regarding the long-term stability of the IE-911 in contact with waste solutions. Future tests should examine ion-exchange column performance after prolonged contact with waste solution.

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REFERENCES

1. Anthony, R.G.; Dosch, R.G.; Gu, D.; Phillip, C.V. Use of Silicotitanates for Removing Cesium and Strontium from Defense Waste. Ind. Eng. Chem. Res. **1994,** *33* (11), 2702–2709.
2. Anthony, R.G.; Philip, C.V.; Dosch, R.G. Selective Adsorption and Ion Exchange of Metal Cations and Anions with Silico-Titanates and Layered Titanates. Waste Man.. **1994,** *13,* 503–512.
3. Zheng, Z.; Gu, Z.D.; Anthony, R.G. Estimation of Cesium Ion Exchange Coefficients for Concentrated Electrolytic Solutions When Using Silicotitanates. Ind. Eng. Chem. Res. **1995,** *34* (6), 2142–2147.
4. Zheng, Z., Zheng, R.A.; Miller, J.E. Modeling Multicomponent Ion Exchange Equilibrium Utilizing Hydrous Crystalline Silicotitanates by a Multiple Interactive Ion Exchange Site Model. Ind. Eng. Chem. Res. **1997,** *36* (6), 2427–2434.
5. Gu, D.; Nguyen, L.; Philip, C.V.; Huckman, M.E.; Anthony, R.G.; Miller, J.E.; Trudell, D.E. Cs^+ Ion Exchange Kinetics in Complex Electrolyte Solutions Using Hydrous Crystalline Silico-Titanates. Ind. Eng. Chem. Res. **1997,** *36* (12), 5377–5383.
6. Huckman, M.E.; Latheef, I.M.; Anthony, R.G. Ion Exchange of Several Radionuclides on the Hydrous Crystalline Silicotitanates, UOP IONSIV IE- 91 1. Sep. Sci. Technol. **1999,** *34* (6 & 7), 1145–1166.
7. Zheng, Z.; Philip, C.V.; Anthony, R.G. Ion Exchange of Group I Metals by Hydrous Crystalline Silicotitanates. Ind. Eng. Chem. Res. **1996,** *35* (11), 4246–4256.
8. Klavetter, E.A.; Brown, N.E.; Trudell, D.E.; Anthony, R.G.; Gu, D.; Thibaud-Ekey, C. Performance of Crystalline Silicotitanates for Cesium Removal from Hanfor Tank Waste Simulants, presented at the Waste Management Conference, Phoenix, Ariz, March 11–15, 1994.
9. Anthony, R.G.; Philip, C.V.; Dosch, R.G. Selective Adsorption and Ion Exchange of Metal Cations and Anions with Silicotitanates and Layered Titanates, Proceedings of the Gulf Coast Hazardous Research Center's Fifth Annual Symposium on Emerging Technologies: Metals, Oxidation and Separation, Lamar University, Beaumont, Texas, February 25–26, 1993.
10. Braun, R.; Dangiery, T.J.; Fennelly, D.J.; Sherman, J.D.; Schwerin, W.C.; Willis, R.R.; Brown, N.E.; Miller, J.E.; Anthony, R.G.; Phillip, C.V.; Bray, L.A.; Brown, G.N.; Lee, D.D.; Borek, T.T.; Connors, W. J. Crystalline Sil-

icotitanates—Novel Commercial Cesium Ion Exchangers. Spectrum 96, August 21, 1996, Seattle, Washington.

11. Harland, C.E. *Ion Exchange, Theory and Practice,* Second Ed.; Royal Society of Chemistry Paperbacks, 1993.
12. Phillip, C.V.; Anthony, R.G. *Competitive Ion Exchange Between the Proton Form of Second Generation TAM-5 and Cesium Cation*. Special Report #2, Sandia National Laboratories, July 26, 1994.
13. Marsh, F.S.; Svitra, Z.V.; Bowen, S.M. *Effects of Soluble Organic Complexants and Their Degradation Products on the Removal of Selected Radionuclides from High-Level Waste, Part III, Part IV,* Los Alamos National Laboratories, Report # LA-13000 UC-940, 1995.

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