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An Assessment of Inorganic Ion-Exchange Materials for the Removal of Strontium from Simulated Hanford Tank Wastes

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ABSTRACT

Several inorganic ion-exchange materials were evaluated for the removal of strontium from two simulated Hanford tank wastes (NCAW and 101SY-Cs5) using static batch experiments. Sodium titanium silicate, $Na_2Ti_2O_3SiO_4 \cdot 2H_2O$ (NaTS), was the best material in NCAW with a K_d of 2.7×10^5 mL/g at a volume-to-mass ratio of 200:1. In the 101SY-Cs5 simulant, strontium extraction was more difficult due to the presence of complexants and consequently K_ds were greatly reduced. Sodium nonatitanate, NaTi, performed best in the presence of these complexants and gave a K_d of 295 mL/g, though none of the materials performed particularly well. Pellets suitable for column studies were synthesized and the ion exchangers evaluated in column studies. Breakthrough curves correlated well with the K_ds obtained from batch experiments with the sodium titanium silicate performing best in NCAW and a pelletized form of sodium nonatitanate performing best in 101SY-Cs5. Both the sodium titanate and the sodium titanosilicate performed better than IONSIV IE-911, a commercially available ion exchanger, in the NCAW simulant, and consequently could be used for the removal of ^{90}Sr from highly alkaline tank wastes.

INTRODUCTION

A series of titanosilicate and titanate inorganic ion-exchange materials have been developed at the Department of Chemistry, Texas A&M University, in

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conjunction with AlliedSignal for the selective removal of strontium from aqueous tank waste simulants.

Over the last 50 years, large amounts of aqueous high level radioactive wastes (HLW) have been produced at the Hanford nuclear site in Washington State as a result of chemical extraction processes designed to extract ^{239}Pu from irradiated uranium fuel. At present the HLW is stored in 177 steel-lined tanks and has an estimated volume of approximately 65 million gallons. The composition of the waste varies considerably from tank to tank; however, previous studies have shown that all of the tanks are believed to contain three distinct phases (1). These phases consist of salt cake at the top of the tank, an alkaline supernate, and a metal hydroxide sludge at the bottom. ^{90}Sr predominantly occurs in the hydroxide sludges but is also found entrained within the salt cake and in the alkaline supernate.

In order to dispose of this tank waste, it has been proposed by the Department of Energy (DOE) to separate the majority of the radioactivity from the supernate and salt cake, thus enabling the residual salts to be classed as low level waste (LLW) and minimizing the amount of HLW for vitrification. One method of attaining this objective is to use ion exchange to selectively remove radioisotopes such as ^{137}Cs and ^{90}Sr from the high concentrations of inert salts. We have evaluated inorganic materials (2–5) since they have higher chemical, radiation, and thermal stabilities than conventional organic resins. In addition, they typically exhibit greater capacities and selectivities for a wide range of monovalent and divalent ions (6–13), and desired properties can be "fine-tuned" into the materials by varying such synthetic parameters as reaction times and temperatures.

In previous papers (14, 15) we presented some preliminary batch experiments using a number of tank waste simulants in order to identify suitable materials for column experiments. In this paper we report the performance of selected ion-exchange materials in column tests and compare the results with IONSIV IE-911, a commercially available ion-exchange material.

EXPERIMENTAL

Materials

All reagents were of analytical grade and were used without further purification. ^{89}Sr (269 mCi/g total Sr) was used for NCAW experiments and ^{89}Sr (69.3 mCi/g total Sr) was used for 101SY-Cs5. Both batches of isotope were purchased from Isotope Products Laboratory. ^{89}Sr activities were measured using a Wallac 1410 liquid scintillation counter and a Fisher Scientific Scintisafe Plus 50% scintillation cocktail.

Samples of the titanosilicate pharmacosiderites, sodium titanosilicate, and sodium titanate were prepared according to established methods which were described in previous papers (4, 14–16). The materials chosen for evaluation were as follows:

Sodium nonatitanate, $Na_4Ti_9O_{20}\cdot xH_2O$, “NaTi” (batch GMG-I-1)
Potassium titanosilicate pharmacosiderite, $HK_3(TiO)_4(SiO_4)_3\cdot 4H_2O$, “KTS-Ph” [batch EAB-III-71, synthesized according to the solution method (15)]
Hydrogen titanosilicate pharmacosiderite, $H_4(TiO)_4(SiO_4)_3\cdot 8H_2O$, “HTS-Ph” (batch EAB-II-25H)
Sodium titanosilicate, $Na_2Ti_2O_3SiO_4\cdot 2H_2O$, “NaTS” (batch RC-5-23A). This compound is similar to the pure unpelletized version of IONSV IE-911

The above materials were compared with IONSIV IE-911, a pelletized form of the sodium titanosilicate manufactured and marketed by UOP, Des Plaines, IL. This material contains about 20% binder and is marketed as a cesium and strontium selective ion exchanger (17, 18) for use in the treatment of alkaline tank wastes. All of the selected materials and the IE-911 are synthesized under highly basic conditions ($pH > 14$) and consequently will be stable in the highly alkaline wastes encountered within the Hanford tanks. Hence, exchanger breakdown will not be a factor when evaluating these materials.

Batch Experiments

Two waste simulants, NCAW and 101SY-Cs5, were prepared according to information supplied by Pacific Northwest National Laboratory (PNNL). The compositions of these two wastes are given in Table 1. NCAW is a relatively simple waste simulant and contains no complexants and is representative of waste in tank 102-AZ diluted to 5 M Na^+ and is 1.68 M in NaOH. Hence, strontium present in the aqueous phase is likely to occur as $Sr(OH)^+$ which should be readily extracted by ion exchange. 101SY-Cs5 is representative of tank waste 101-SY, also diluted to 5 M Na^+, and contains significant amounts of chelating agents such as EDTA and nitrilotriacetate which will complex any strontium present, making removal by ion exchange far more difficult.

Between 0.05 and 0.01 g of ion exchanger was accurately weighed into a scintillation vial to give V:m ratios of 200 and 1000, respectively, and contacted with 10 mL of the waste simulant which had been spiked with ^{89}Sr. Sufficient ^{89}Sr tracer was added to ensure that there was enough activity present in solution to allow it to be measured with statistical accuracy on a liquid scintillation counter. The total strontium concentration for these batch tests was <1 ppm, thus ensuring that no precipitation occurred. The vials were capped and placed on a rotary shaker for approximately 24 hours to ensure that equilibrium was reached. The mixtures were then filtered using Whatman No. 42 filter paper (which had previously been shown to be sufficient to retain any

TABLE 1
The Composition of the Waste Simulants NCAW and 101SY-Cs5

Species	Concentration (M) NCAW	Concentration (M) 101SY-Cs5
Al	0.43	0.42
Ca	0	4.20E-03
Cs	5.00E-04	4.19E-05
Fe	0	1.96E-04
K	0.12	0.034
Mo	0	4.20E-04
Ni	0	2.50E-04
Na	4.99	5.1
Rb	5.00E-05	4.20E-06
Sr (total)	2.70E-07	2.90E-07
Zn	0	5.00E-04
Carbonate	0.23	0.038
Fluoride	0.09	0.092
Hydroxide	3.4	3.78
Hydroxide (free)	1.68	2.11
Nitrate	1.67	1.29
Nitrite	0.43	1.09
Sulfate	0.15	4.75E-03
Phosphate	0.025	0.02
Citric acid	0	5.00E-03
Na_4 EDTA	0	5.00E-03
Iminodiacetic acid	0	0.031
Na_3 nitrilotriacetate	0	2.50E-04
Sodium gluconate	0	0.013
Theoretical pH	14.5	14.4

fines generated) and the residual activity in the solutions was determined using liquid scintillation counting. Distribution coefficients (K_ds) were then calculated using

$$K_d = [(C_0 - C_f)/C_f]V/m \tag{1}$$

where C_0 = initial activity of solution (cpm/mL)
C_f = final activity of solution (cpm/mL)
V = volume of solution (mL)
m = mass of exchanger (g)

All K_ds quoted are a mean of two or more separate determinations. Duplication of results was excellent, with repeat K_d determinations generally being within 5% of each other.

Column Experiments

Materials which showed good K_ds were further evaluated using column techniques. The exchangers typically consisted of fine powders (<2 μm) which needed to be combined with an alkali-resistant inorganic binder to produce pellets suitable for use in column experiments. This pelletizing was achieved using titania as a binder according to a proprietary method developed by AlliedSignal Corp., Des Plaines, IL, and will not be discussed further. The granular material produced was sieved to obtain 40–60 mesh particles (0.25–0.42 mm) and then slurried into a Bio-Rad column (0.66 cm i.d., 5 cm length) with deionized water to obtain a bed volume of between 1 and 1.5 mL. To avoid any unwanted hydrolysis of the exchangers resulting from contact with the deionized water, the column was connected to Tygon tubing and 1 M sodium hydroxide passed through to condition the column and ensure proper bed settling for columns containing NaTS, NaTi, and IE-911. KTS-Ph was conditioned using 1 M potassium hydroxide to ensure that it was in the K^+ form.

The tank waste simulant was spiked with ^{89}Sr to give total strontium concentrations of 2.7×10^{-7} M (0.024 ppm) and 4.1×10^{-6} M (0.36 ppm) for the NCAW and 101SY-Cs5 simulants, respectively. (The strontium concentration in 101SY-Cs5 was considerably greater than the 2.90×10^{-7} M suggested in Table 1. This was necessary due to the lower specific activity of the source of ^{89}Sr used for the 101SY-Cs5 work.) The waste simulants were then passed through the column at a rate of approximately 20 bed volumes (BV) per hour for NCAW and 5 BV/h for the 101SY-Cs5 simulant. Twenty BV/h was chosen for NCAW due to the high K_ds and perceived long duration of the experiment. A more realistic 5 BV/h (19) was selected for the 101SY-Cs5 work. Eluant fractions were collected twice a day for the NCAW and approximately once per hour for the 101SY simulant and analyzed for ^{89}Sr using liquid scintillation counting.

Percentage breakthrough (BT) was then calculated using

$$\%BT = (A_f/A_0) \times 100 \tag{2}$$

where A_f = activity of solution exiting column (cpm/mL)
A_0 = activity of solution entering column (cpm/mL)

RESULTS AND DISCUSSION

The K_ds and percentage ^{89}Sr removal for the NCAW and the 101SY simulants are given in Tables 2 and 3, respectively.

Sodium titanosilicate, NaTS, was the best material for the removal of Sr from NCAW simulant and showed only a slight decrease in the measured K_d as the $V{:}m$ ratio was increased from 200 to 1000. Ion-exchange theory predicts

TABLE 2
The K_ds and % Sr Removal from NCAW Simulant

	$V{:}m = 200$		$V{:}m = 1000$	
Sample	K_d (mL/g)	% Removal	K_d (mL/g)	% Removal
HTS-Ph	4.48×10^4	99.55	3.70×10^3	78.09
KTS-Ph	2.02×10^4	99.80	5.00×10^3	83.85
NaTi	2.35×10^5	99.92	3.96×10^4	97.52
NaTS	2.70×10^5	99.93	2.26×10^5	98.09
IE-911	2.93×10^4	99.30	2.06×10^4	95.38

that the K_d will be independent of the $V{:}m$ ratio (for trace exchange), so this result suggests that the capacity for ^{89}Sr is not greatly affected by the volume increase. Sodium titanate also exhibited a high K_d of 2.35×10^5 mL/g at a $V{:}m$ ratio of 200 but this decreased dramatically to only 3.96×10^4 mL/g at a $V{:}m$ ratio of 1000 (Table 2), suggesting a slightly lower capacity for strontium in comparison to the sodium titanosilicate. The pharmacosiderites, HTS-Ph and KTS-Ph, had K_ds of 4.48×10^4 and 2.02×10^4 mL/g, respectively, at a $V{:}m$ of 200, but at the higher $V{:}m$ ratio the K_ds dropped to 5000 mL/g or less. These results indicate that the pharmacosiderites and the sodium titanate are less selective toward ^{89}Sr and are absorbing a greater proportion of other ions from solution in addition to the desired $Sr(OH)^+$. In comparison, the commercially produced material, IE-911, maintained a K_d in excess of 20,000 mL/g as the $V{:}m$ ratio was increased, suggesting that there is little interference with ^{89}Sr uptake from the other ions present in solution. This is surprising since this material is known to be selective for Cs^+ from tank wastes (17) which would be expected to compete for the available ion-exchange sites and thus reduce the number of sites available for exchange with ^{89}Sr. From these

TABLE 3
K_ds and % Sr Removal from 101SY-Cs5 Simulant at a
$V{:}m$ Ratio of 200

Sample	K_d (mL/g)	% Removal
HTS-Ph	164	45.6
KTS-Ph	31	13.2
NaTi	295	61.1
NaTS	231	54.7
IE-911	210	53.6

data it would be expected that the NaTS would perform best in column experiments. However, since the waste solutions are highly alkaline, the strontium is present as $Sr(OH)^+$. These ions may be able to fit into the framework sites as well as the tunnels, and therefore Cs^+ and K^+ uptake, which fills only tunnel sites (4, 5), would not interfere with uptake of $Sr(OH)^+$ in the framework sites. X-ray diffraction studies are underway to establish the strontium-exchange sites.

In the 101SY-Cs5 simulant, the K_ds for ^{89}Sr were several orders of magnitude lower than the K_ds obtained in NCAW. The best material was sodium nonatitanate, NaTi, with a K_d of only 295 mL/g, which corresponds to the removal of 61.1% of the strontium present. NaTS and IE-911 also had K_ds $>$ 200 mL/g, but both pharmacosiderites performed poorly. This lower performance is due to the presence of large amounts of complexants in this particular simulant which effectively chelate the strontium, making it unavailable for ion exchange.

From the batch data we decided to evaluate the sodium titanosilicate, the sodium nonatitanate, the pharmacosiderite, KTS-Ph, and UOP's IE-911 using column techniques.

Column Experiments

The breakthrough curves obtained for the materials using the NCAW simulant are given in Fig. 1. It was attempted to run all of the columns to 50% breakthrough, but in the majority of cases this proved impossible due to the gradual precipitation of solids in both the column and the tubing. This precipitation also caused a gradual reduction in the flow rate from an initial 20 BV/h to approximately 13 BV/h over the duration of the experiment. The problem stemmed from the fact that both simulants were nearly saturated with dissolved solids at room temperature. Even allowing bottles of the simulant solutions to stand in the laboratory for several weeks resulted in minor amounts of crystals forming at the bottom of the containers. Consequently, it is not surprising that some nucleation around the ion-exchange particles occurred over the course of the NCAW column experiments. This precipitation problem needs to be taken into account when designing a process for the large-scale treatment of the Hanford tank wastes. The reduction in flow rate increased the bed residence time and consequently may have led to a better performance than if the flow rate had been maintained at 20 BV/h. Ideally, it would have been preferable to perform the experiment at 60°C to minimize precipitation, but the safety hazards associated with the use of radioisotopes prevented our doing this.

The titania binder used to produce pellets suitable for column studies was also found to remove some strontium from the NCAW solutions. However, the K_ds were at least an order of magnitude lower than the ion-exchange ma-

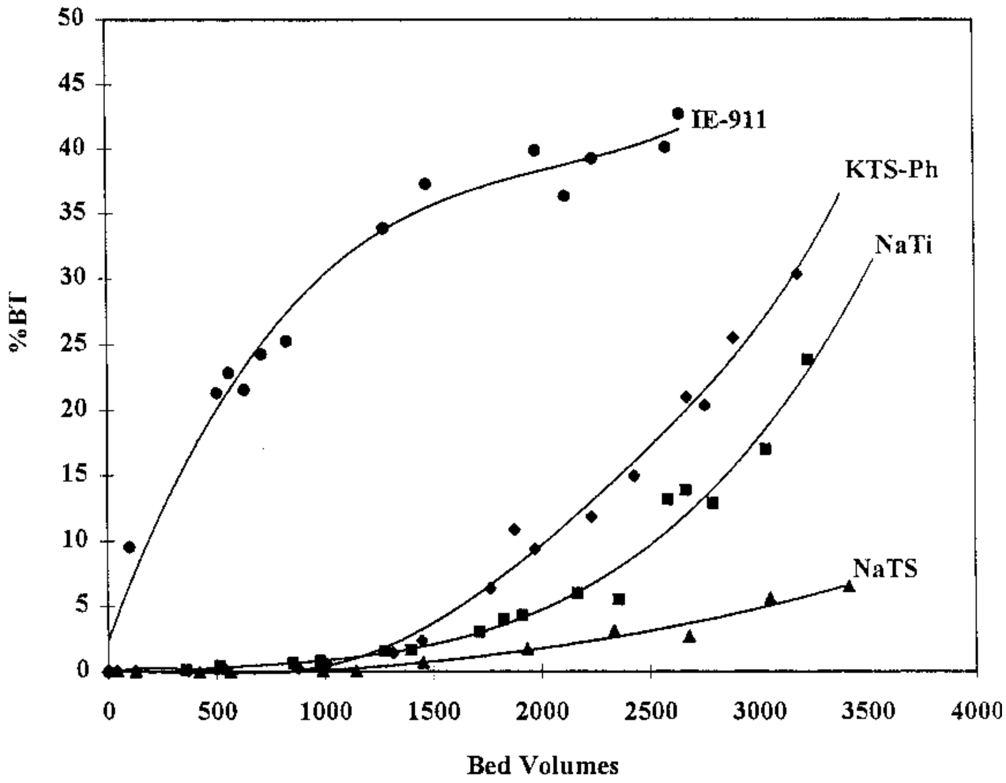

FIG. 1 Breakthrough curves for the NCAW simulant.

terials, so the performance of the beads will be related more to the ion exchanger than to the binder. Consequently, as the percentage binder increases, the performance of the beads will decrease. All three of the samples prepared "in-house" at Texas A&M contained approximately 85 wt% active material and 15 wt% binder, allowing direct comparison of the pelletized exchangers.

The best material was NaTS which exhibited less than a 10% ^{89}Sr breakthrough after 3500 BV. Both NaTi and KTS-Ph also showed insignificant breakthrough up to approximately 1000 BV. However, after this volume, both materials showed a relatively rapid breakthrough as the available ion-exchange sites began to saturate with sodium and potassium ions. All three columns eventually became blocked due to precipitation, but it can be clearly seen that the performance of the materials closely followed the K_ds in batch experiments with NaTS showing the least breakthrough and KTS-Ph the greatest.

Extrapolation of the curve for KTS-Ph to 50% breakthrough indicated an approximate total strontium capacity in NCAW of 3.0×10^{-3} meq/g while the capacity for NaTi was probably around 10% greater than this. Insufficient breakthrough for NaTS had occurred to allow an approximation of the capacity to be calculated, but it was clearly significantly greater than the capacities for KTS-Ph and NaTi.

The IE-911 performed very poorly in the column experiments and exhibited a ^{89}Sr breakthrough of almost 10% immediately. Breakthrough continued to increase rapidly and reached almost 40% after 2000 BV. Repeated column experiments using IE-911 confirmed this observation, thus eliminating poor bed packing as an explanation for the rapid breakthrough. From the batch K_ds it was expected that the IE-911 would breakthrough after KTS-Ph but before the NaTi. This unexpected early breakthrough may well be due to poor exchange kinetics in comparison with the other exchangers.

The breakthrough data for the 101SY-Cs5 simulant are given in Fig. 2. As was expected from the far lower K_ds, 50% breakthrough was reached in just a few days as opposed to the NCAW experiments which took several weeks to perform. In 101SY-Cs5, strontium absorption by the binder was negligible, allowing direct comparison of the materials. In these samples there was some variation in the binder content which is outlined in Table 4. An industrially produced sample of pelletized sodium nonatitanate (Batch 8212-149) donated by AlliedSignal was also evaluated. This material was essentially the same as

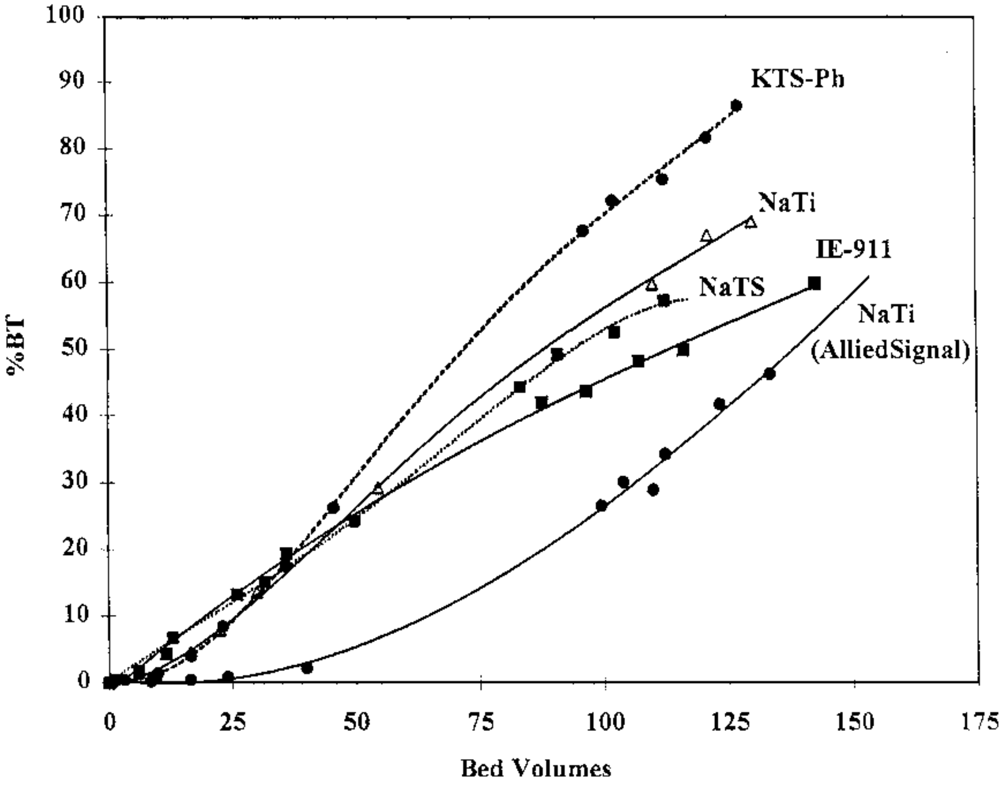

FIG. 2 Breakthrough curves for the 101SY-Cs5 simulant.

TABLE 4
Inorganic Binder Content of Pelletized Samples Used in Column Studies with 101SY-Cs5 Simulant

Sample	% Binder
KTS-Ph	23
NaTi	41
NaTS	19
IE-911	Not known
AlliedSignal titanate	Not known

the NaTi but had been produced and pelletized on an industrial scale by AlliedSignal.

The breakthrough characteristics for the ion-exchange columns followed the trend observed with the batch K_ds. All of the materials, except the AlliedSignal pelletized titanate, showed immediate strontium breakthrough which was expected due to the very low K_ds in the batch experiments. The AlliedSignal material showed a smoother breakthrough curve and only reached 50% breakthrough after approximately 125 BV, which was at least 50 BV more than any of the other exchangers, including IE-911. The improved performance relative to the NaTi synthesized at Texas A&M University may be due to either a reduced level of binder, better diffusion kinetics, or a combination of both.

Sodium titanosilicate has a framework structure that creates tunnels parallel to the *c*-axis (4). There are two types of ion-exchange sites in this compound with half of the sodium ions located in framework sites and half within the tunnels. Ions significantly larger than Na^+, such as K^+, Rb^+, and Cs^+, cannot fit in the framework sites and are thus confined to the tunnel sites (5). The very high selectivity exhibited for Cs^+ by the titanosilicate stems from the fact that Cs^+ in the tunnel sites forms Cs—O bonds that are close in bond distance to the sum of their ionic radii. The reason for the high selectivity toward strontium in alkaline solution is not yet known as the structure of the strontium phase is as yet undetermined. Our preliminary data indicate that strontium is present as $Sr(OH)^+$ and that it may be present in both the tunnel and the framework sites.

Competition for the two types of sites would come mainly from Na^+, K^+, and Rb^+ in the NCAW waste. The latter two ions would occupy the tunnel sites only. Sodium ions can also fill the tunnels, but selectivity for these sites is low. The preferred sodium ion site is in the framework where the sum of the ionic radii are very close to the Na—O bond distance (4). Thus, given the high level of competing ions in the NCAW simulant, the level of removal of Sr^{2+} is quite good.

The pharmacosiderites utilized in this study are cubic and also have framework structures (2). Tunnels of equal size run parallel to the three unit cell directions and the univalent ions are located near the cube face centers. Sr^{2+} is slightly preferred to Cs^+ but, as the K_d values show, not as strongly as these ions are preferred by sodium titanium silicate.

Sodium nonatitanate has a layered structure and is poorly crystalline (20). Although its affinity for Sr^{2+} or $Sr(OH)^+$ in strongly alkaline solution is high, little is known about its preference for the other ions in the NCAW solution.

CONCLUSIONS

These experiments have shown that strontium present in NCAW simulant can readily be removed by sodium nonatitanate (NaTi), pharmacosiderite (KTS-Ph), and sodium titanosilicate (NaTS). The best material was clearly NaTS with less than 10% ^{89}Sr breakthrough, even after passing 3500 BV through the pelletized material at a rapid flow rate of up to 20 BV/h. KTS-Ph and NaTi were less effective, but all three materials performed better than IE-911. Column blocking proved to be a problem due to the high content of dissolved solids. This needs to be addressed when designing a process for the economic treatment of the Hanford tank wastes. A possible solution would be to further dilute the tank supernates or to operate the ion-exchange process at an elevated temperature to minimize any precipitation.

In 101SY-Cs5, all of the exchangers performed poorly though a sample of sodium titanate produced industrially by AlliedSignal did not reach 50% ^{89}Sr breakthrough until after approximately 125 BV. Consequently, remediation of tank wastes containing high concentrations of complexants by ion exchange is likely to prove difficult. Precipitation in the columns did not occur in the 101SY-Cs5 experiments due to the very rapid ^{89}Sr breakthrough.

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