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Monosodium Titanate in Hydrous Titanium Oxide Spheres for the Removal of Strontium and Key Actinides from Salt Solutions at the Savannah River Site

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Abstract: Fine powders of monosodium titanate effectively remove strontium and plutonium from alkaline salt supernatant. At the Savannah River Site, larger, porous particles with monosodium titanate were desired for continuous column operations. The internal gelation process was used to make hydrous titanium oxide microspheres with 32 and 50 wt% monosodium titanate. With actual supernatant, the microspheres with 50 wt% monosodium titanate produced average batch distribution coefficients of 35,000 mL/g for plutonium and 99,000 mL/g for strontium. These microspheres were tested using a simulant and a flow rate of 5.3 bed volumes per hour. The plutonium removal dropped from 99% to 94% while the strontium removal remained nearly 100%. The microspheres exhibited good flow performance and no particle degradation.

Keywords: Strontium removal, monosodium titanate, hydrous titanium oxide spheres, nuclear waste remediation

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INTRODUCTION

The Savannah River Site (SRS) selected the inorganic adsorbent monosodium titanate (MST) for the removal of strontium, plutonium, and key actinides from its salt waste solutions (1). MST has been used in batch contacts in a 3,700,000 L tank. The micron-sized MST particles, which are created by a sol-gel process, were then removed by filtration of the decontaminated salt solution. Future plans to remediate the nuclear waste at SRS include the use of MST.

The Department of Energy identified a need to incorporate fine MST powder into larger, engineered particles suitable for column use. Use of such material in adsorption columns could substitute for MST batch processing. Engineering MST into nominally 300- to 600-μm particles would allow good flow dynamics and steady column processing of salt solution. Column use of an adsorbent allows maximum loading because the adsorbent is equilibrated with feed levels of adsorbate. Process columns have small footprints relative to those of batch contact tanks and also operate continuously, rather than in batch mode. Process filtration would see lower demand because it would be located upstream of the column and would remove sludge fines without the need for the removal of the MST.

SRS personnel have identified several methods of preparing the engineered MST that are potentially viable options (2). The selection criteria at SRS included the likelihood of commercialization, the potential for good performance, the expected sorbent cost, and the impact on other remediation processes. One of the selected methods was the internal gelation process, which has been used by Oak Ridge National Laboratory (ORNL) researchers to prepare microspheres for the removal of cesium, strontium, and technetium from radioactive salt solutions (3–5). An economic evaluation has shown that microspheres from the internal gelation process are economically viable (6). The internal gelation process provided a unique means for the preparation of composite microspheres in which the MST powder was homogenously dispersed throughout the gel-sphere matrix. Variations in several process parameters can control the type and size of the hydrous metal-oxide gel spheres. The spherules are stable forms and have little or no tendency to degrade under the dynamic conditions of a column operation. A description of the internal gelation process and the optimum process control parameters for the production of hydrous titanium oxide (HTiO) microspheres are provided in the patent *Method for Preparing Hydrous Titanium Oxide Gels and Spherules* (3). The HTiO was selected as the binder for this effort because HTiO microspheres and HTiO powder were able to remove 95.5% of strontium from an alkaline waste simulant at ORNL (5). Therefore, HTiO as well as the MST powder can remove strontium. In addition, HTiO microspheres with a different type of sodium titanate had been made previously using internal gelation (3). A detailed description of the chemistry for the internal gelation process for HTiO microspheres is provided elsewhere (6). It should be noted that the internal gelation process has been used to make microspheres for a wide variety of sorbents and final pH conditions. For example, microspheres with ammonium molybdophosphate powder were made and used to remove cesium from highly acidic waste at the Idaho National Laboratory (7).

Researchers at Savannah River National Laboratory (SRNL) and ORNL prepared and evaluated engineered MST using the internal gelation process. A detailed description of the preparation of the composite microspheres with MST and HTiO at ORNL is provided. After various physical properties of the microspheres were measured at SRNL, batch contact tests were conducted with an actual waste sample as well as with a waste simulant. Since the batch results were promising, two column tests with waste simulant were also performed. The results from the batch and column tests are also provided.

EXPERIMENTAL

The preparation of the HTiO microspheres with MST involved several steps. First, the slurry of the MST was passed through a sieve to remove large particles, and the MST content in the slurry was then determined. Next, a stock solution of titanium oxychloride ($TiOCl_2$) in hydrochloric acid (HCl) and a stock solution of hexamethylenetetramine (HMTA) and urea were prepared. The stock solutions were then chilled in an ice water box and then combined. The MST slurry was mixed and transferred to the combined stock solutions. The solution with MST, $TiOCl_2$, HMTA, and urea was then placed into internal gelation equipment (most of which has been described elsewhere) (8, 9). The chilled feed solution was dispersed as droplets into silicone oil at 90°C to cause gelation to occur. After the microspheres were collected, they were aged in the hot silicone oil, washed with trichloroethylene (TCE), washed with 0.5 M ammonium hydroxide (NH_4OH), and air-dried at ambient temperature. A description of the preparation of the HTiO microspheres with MST is provided next.

Apparatus to Form Gel Microspheres

As shown in Fig. 1, the laboratory-scale apparatus that was used to make the HTiO/MST gel spheres was a modification of an apparatus used to produce uranium and plutonium microspheres in a glove box. The use of MST required a few minor modifications, because of the presence of an insoluble solid. First, a glass (rather than a stainless steel) broth pot was used to ensure that the broth solution was adequately mixed and that the suction line was at the proper depth. Second, the glass broth pot was placed on a magnetic stir plate so a magnetic stir bar could be used to keep the MST suspended. Third, the position of the micropump gear pump was changed so

Figure 1. Apparatus for the preparation of the HTiO/MST microspheres.

that the broth solution was pumped from the broth pot through the gear pump to the vibrator. Due to concerns with precipitation, the pump head was also cooled to 5°C.

The apparatus consisted of a reservoir for the hot silicone oil, a pump to circulate the silicone oil through the gel-forming apparatus, a chilled broth pot, a vibrating nozzle system to control the size of the broth droplets, a glass gel-forming column, a downstream polyvinylchloride (PVC) transport line to provide a residence time for the gel spherules to hydrolyze and solidify, and a stainless steel mesh product collector to catch and age the gel spheres and to separate the oil from the gel spheres.

The silicone oil reservoir was a stainless steel open-top rectangular container. Two 250-W stainless steel heating blades were positioned at the rear of the reservoir to heat the oil at 90°C. A thermocouple that was positioned in the basket at the bottom and near the front of the reservoir was connected to a temperature controller. A Lightnin® mixer with its stainless steel shaft positioned between the two heating blades and its stainless steel impeller located near the bottom of the reservoir was used to mix and maintain the oil at the desired temperature. A stainless steel wire-mesh (100 mesh) basket was placed in the front space in the reservoir to prevent any spilled gelled spheres from being pumped out of the reservoir into the circulating pump. An Eastern Centrichem centrifugal pump was used to move the hot oil from the reservoir through a stainless steel line to the vertically positioned glass gelation column. The flow from the pump was divided into two streams. One stream was routed through a vertically positioned PVC tube; the open end was positioned about 7.5 cm down into the gelation column. Oil from this tube provided most of the flow through the gel-forming tube and downstream serpentine PVC tube to the collection basket. The typical flow rate was between 250 and 300 mL/min. A small hole was punctured through the PVC tube about 5 cm above the top of the gelation column to accommodate a blunt-end droplet forming needle. During a production run, the tip of the needle was positioned in the center of the PVC tube. The flow of the hot silicone oil across the tip of the needle could be regulated to help set the size of the droplets.

The components of the broth-droplet-forming system consisted of a chilled Pyrex broth vessel, a magnetic stirrer positioned under the broth vessel, a peristaltic pump, a vibrator and controller from Alpha-M Corp, and a blunt-end electropolished stainless steel 20-gage needle from Popper & Sons. The peristaltic pump was used to transfer the chilled broth-MST mixture from the broth vessel through a 3/32-in. Tygon tube to the droplet-forming needle at a rate of 15 mL/min. The tube passes through the vibrator at approximately 30 cm from the needle. The frequency of the vibrator was set at 180 to 200 Hz to help produce droplets with the desired diameter. The goal was to obtain air-dried microspheres with diameters in the range of 300 to 600 μm.

Preparation of the MST/HTiO Spheres

After an MST sample arrived from SRNL, it was passed through a stainless steel 150 mesh sieve. Deionized water was used to remove large particles, which could potentially plug the 20-gage needle. Only a very small amount of solids failed to pass through the sieve. The slurry filtrate was collected in a plastic beaker and allowed to settle for 7 days. Next, the water above the settled powder was removed by decantation. The quantity of MST in the slurry was determined to be 26.42 wt%.

A solution of 1.71 M $TiOCl_2$ in 1.4 M HCl and a solution of 3.2 M urea in 3.2 M HMTA were made for use in the preparations. The densities of the $TiOCl_2$/HCl and HMTA/urea solutions were 1.23 and 1.14 g/mL, respectively. In the preparation with 50 wt% MST, 520.2 g of broth was prepared at 5°C with the following composition: 0.78 M Ti^{4+}, 0.64 M H^+, 1.75 M HMTA, and 1.75 M urea. Then, 246 g of MST slurry, which contained 65 g of air-dried MST, was added to the chilled broth while it was mixed to keep the MST powder in suspension. This chilled mixture was pumped through the vibrator and the needle into a flowing stream of hot silicone oil. The short transport time of the broth to the heated oil made it possible for the MST to remain in suspension. The composite gel spheres were collected in two 80 mesh stainless steel baskets and aged for 20 min. After the oil had been drained, the gel spheres were batch washed four times with TCE to remove silicone oil from the surface of the spheres. The spheres were then batch washed with 0.5 M NH_4OH to remove the internal HMTA, urea, and ammonia chloride. The batch washing sequence was continued until the measured conductivity of the spent wash solution was constant. The microspheres were allowed to air-dry in stainless steel pans.

One problem during the preparation of the 50 wt% MST was the tendency of the HTiO/MST microspheres to bond tightly together upon drying. A large fraction of the microspheres could not be separated easily when they were sieved. The clumps had to be physically broken apart, which led to many cracked and irregularly shaped spheres, as shown in Fig. 2. Figure 3 shows the image of a HTiO microsphere with 50 wt% MST from a scanning electron microscope (SEM). The SEM image shows that the microsphere has a significant amount of surface area. This preparation yielded approximately 78 g of microspheres in the 300- to 1000-μm-diameter range. This problem did not occur during the preparation of the 32 wt% MST spheres, as shown in Fig. 4. The only difference in the preparations of the 32 and 50 wt% samples was the amount of MST slurry added to the broth.

Physical Characterization

The particle size distributions of the microspheres were measured in water using a Microtrac® S3000 laser scattering analyzer. The mean diameter for the 50 wt% MST microspheres was 573 μm with a standard deviation of 105 μm, while the mean diameter for the 32 wt% MST spheres was 520 μm with a standard deviation of 81 μm. The standard deviation in microns describes the width of the measured particle size distribution.

Bed density and weight percent of MST are also important properties for column calculations since the amount of active adsorbent per bed volume is needed. For the bed density measurement, the microspheres were poured into a 10-mL graduated cylinder and gently tapped until no further change in settling was observed. The average dry bed densities of the 50 and

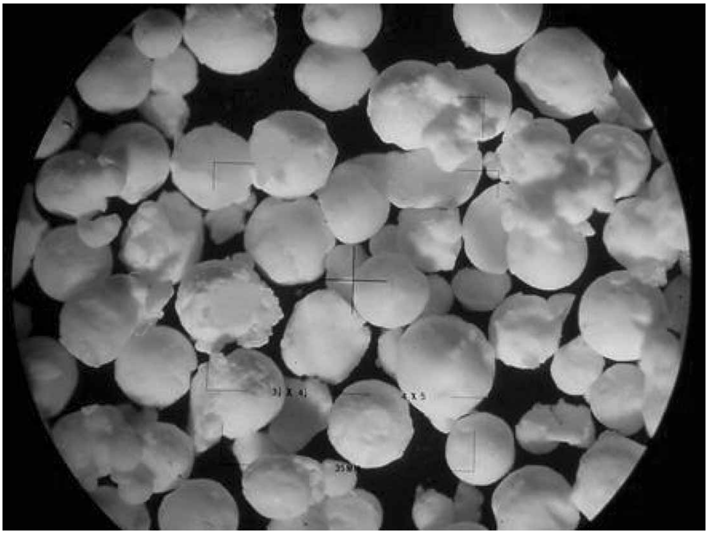

Figure 2. Photograph of HTiO microspheres with 50 wt% MST.

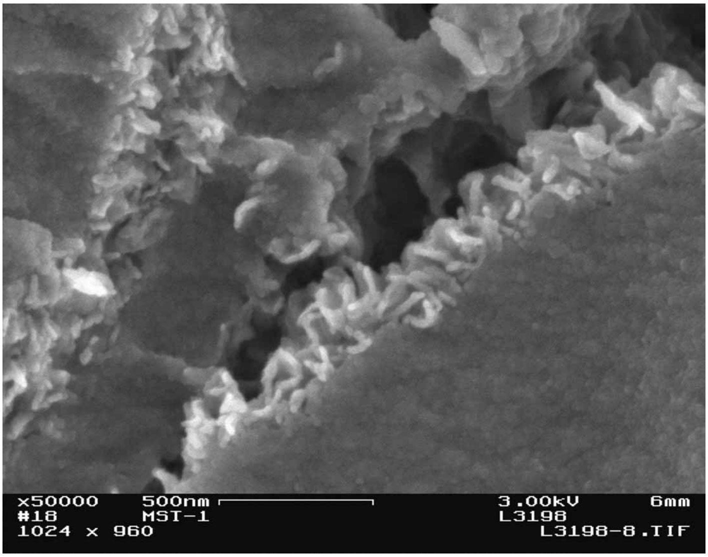

Figure 3. Image of a 50 wt% MST microsphere from a scanning electron microscope.

32 wt% MST spheres were 0.991 and 1.053 g/mL, respectively. The weight percent of MST was confirmed by dissolution of a known quantity of each sample, and the titanium content was then measured by inductively coupled plasma emission spectroscopy (ICP-ES). The sodium peroxide fusion method using nickel crucibles was successfully used to dissolve the microspheres. The ICP-ES results confirmed that very little MST was lost during the preparation of the microspheres.

The average crush strength for HTiO microspheres with and without MST was determined dynamometrically. The crush strength is the weight in grams needed to break a single microsphere. For these measurements, 20 microspheres of each composition were randomly selected. Each microsphere was placed on a Mettler PM1200 analytical balance and then crushed using the flat smooth surface of the stainless steel rod from a modified micrometer. The crush strengths of the HTiO microspheres with and without MST were in excess of 2000 g. Therefore, the strength of these microspheres is more than sufficient for the proposed column operations at SRS.

Salt Solutions for Batch Contact and Column Tests

Table 1 lists the measured concentrations of the salt solution used for all spiked simulant tests. This simulant recipe has been used uniformly for most of the MST work at SRNL (10). The simulant was spiked with ^{85}Sr, ^{239}Pu/^{240}Pu, ^{237}Np, and U. Table 2 presents the measured concentrations of

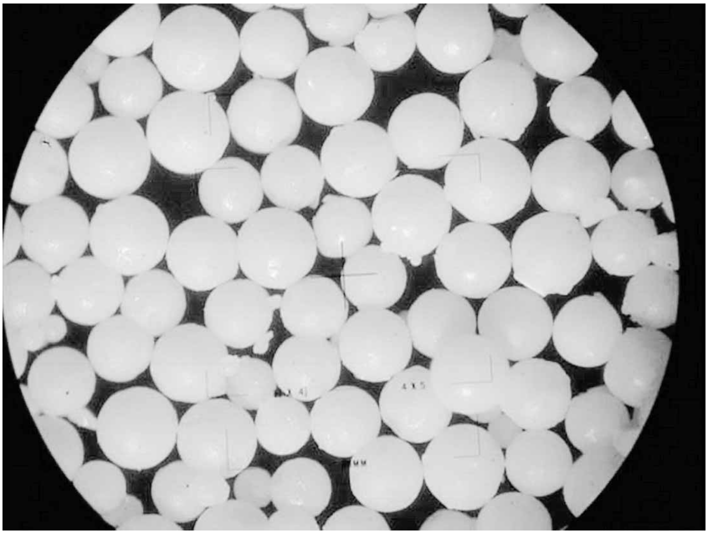

Figure 4. Photograph of HTiO microspheres with 32 wt% MST.

^{239}Pu/^{240}Pu and ^{237}Np for the simulant for the indicated number of days after makeup (11).

The performance of the MST/HTiO microspheres was also tested on a composite of actual tank supernatants from Tanks 11H, 30H, 32H, and 39H. The composite solution was diluted to 5.8 M in sodium using 1.66 M sodium hydroxide. The composite also contained 9350 mg/L of aluminum. The diluted composite was then allowed to stand. Table 3 shows the changes in radiochemical analyses (11).

Batch Contact Procedures

The batch contact procedure for this work was closely aligned with past procedures for MST, except that batches of engineered material required a

Table 1. Composition of the salt solution simulant

Component	Molarity
Free hydroxide	1.33
Aluminum	0.429
Sulfate	0.521
Total sodium	5.6
Nitrate	2.60
Nitrite	0.134
Carbonate	0.026

Table 2. Measured actinide concentrations in the salt solution simulant

Day	[^{239}Pu/^{240}Pu] (μg/L)	[Np] (μg/L)	[U] (μg/L)
0	179	530	[a]
11	197	549	[a]
27	194	568	[a]
48	211	508	12,500

[a] Not measured.

change in the sampling procedure (10). To provide the typical phase ratio of 2500 mL/g (or 0.4 g of MST per liter), 250 mL of the salt solution was contacted with microspheres that contained 0.1 g of MST. For the simulated salt solutions, 500-mL bottles were used to provide sufficient headspace for good agitation of each batch at the shaker cycle rate of 175 rpm. Oven shaker temperature was controlled to 27 ± 2°C and was monitored by the oven thermocouple and a separate calibrated thermocouple.

Prior to any sampling of the simulant, the agitation was stopped, and the solids were permitted to settle for a minimum of 0.5 h. Approximately 5 mL of salt solution was then withdrawn and filtered through 0.45-μm nylon syringe tip filters. Next, 4 mL of the filtrate was pipetted into 4 mL of 5 M nitric acid. The combined solutions were agitated for a minimum of 2 h to allow complete acid dissolution. Three milliliters of acidified filtrate was measured into gamma vials for counting, and 5-mL samples were prepared for plutonium analysis using thenoyltrifluoroacetone extraction as well as for ^{85}Sr gamma counting.

For tests with actual tank wastes, the contents of the bottle were agitated using a five-station magnetic stirrer and small Teflon® magnet bars. Batch bottles with actual salt solutions were 250 mL because headspace was not needed for good agitation. A water bath provided temperature stability at

Table 3. Radionuclide concentrations in the composite of actual tank supernatants

Time after compositing (days)	[Total Pu] (μg/L)	[^{239}Pu/^{240}Pu] (μg/L)	[^{90}Sr] (Bq/L)	[U] mg/L
2	105	94	6.38×10^{4}	3.0
9	102	91	6.60×10^{4}	2.6
21	89	78	6.62×10^{4}	2.6
21 (filtered)	52	47	1.68×10^{4P}	2.1
48	98	88	6.47×10^{4}	[a]
48 (filtered)	64	58	2.05×10^{4P}	[a]

[a] Not measured.

27 ± 2°C. Then, 1.5 g of filtrate was measured into 60-mL plastic sample bottles that contained premeasured 25-mL portions of 3 M nitric acid. Acidified filtrate samples sat for more than 2 h before further sample pouring and cell exit for chemical and radiochemical analyses.

Adsorption Column Procedures

Two adsorption column tests on the HTiO microspheres with 50 wt% MST were conducted to remove plutonium and strontium from simulated waste solutions containing 5.6 M sodium. The simulant recipe was the same as for batch testing, and ^{85}Sr was used as a radiotracer. Multiple feed analyses were taken to determine the plutonium content. The MST microspheres were soaked in nonradioactive strontium-free simulant for at least 5 days before being loaded into the column. The MST/HTiO microspheres swelled to twice their original size in the first 2 days (or less) of soaking in caustic simulant solution. The material did not exhibit a change in size after the initial soaking period or during testing.

The column tests treated the simulated waste in a once-through fashion and operated continuously using the same equipment. The column and tubing were rinsed with water and with nonradioactive simulant solution between tests. The first column test lasted 23 days and treated 10 L of simulated waste solution. The second test continued for 5 days at a higher flow rate, which is reported in bed volumes (BV)/h. Most of the solution (8 L) from the first test was recovered and spiked again with plutonium and strontium for the second test. Details for the different column tests, such as the ratio of column length (CL) to diameter (d) ratio, are provided in Table 4. Though the second test did not contain significant total strontium, all tests contained sufficient ^{85}Sr radiotracer for measurement purposes. The jacketed column had a diameter of 0.833 cm. The circulating water temperature through the jacket was maintained between 23.0 and 26.0°C for the two tests.

For the two column tests, ^{85}Sr was measured by a low-temperature germanium detector. However, the tests did not use the same detector. Both detectors yielded values with no more than 10% uncertainty. Due to the

Table 4. Characteristics of column tests using microspheres with 50 wt% MST

Test number	Sorbent mass (g)	Sorbent volume (mL)	CL/d ratio	Feed rate (BV/h)	Pu (μg/L)	Total Sr (μg/L)
1	1.86	3.38	7.2	5.3	108	0.72
2	0.92	2.0	4.3	15	120	[a]

[a] Not measured.

relatively short half-life of ^{85}Sr, measured values were time adjusted based on the natural decay of ^{85}Sr and the time between sampling and analysis. All of the ^{85}Sr values represent activity at time of sampling. The ^{85}Sr data were normalized to a constant feed level of 100 to facilitate comparison.

RESULTS AND DISCUSSION

Batch Contacts

Batch contact tests provide information on relative kinetics; an equilibrium point on the isotherm; and, depending on phase ratio, an estimate of sorbent capacity. The analyses for these tests covered sorption of strontium, plutonium, and neptunium. The basic parameters for the batch contact tests are the initial and final concentrations of adsorbates, the sorbent mass, and the solution volume. Decontamination factor (DF) is calculated with the following equation:

$$\mathrm{DF} = \frac{(\text{initial concentration})}{(\text{final concentration})}$$

Distribution coefficient (K_d) is determined via the following equation.

$$K_d = (\mathrm{DF} - 1) \times \frac{(\text{solution volume in mL})}{(\text{total mass of sorbent in g})}$$

Sorbent performances in the caustic salt simulant and the actual tank solution for strontium and plutonium are compared with MST powder (11) in Tables 5 and 6, respectively. Batch distribution coefficients (K_d) are calculated using total sample mass. Therefore, the performance of the microspheres with 32 wt% MST was lower than the microspheres with 50 wt% MST. Duplicated experiments with 50 wt% MST are indicated by parenthetical numbering (i.e., #1 and #2).

Table 5. Batch contact performance for strontium removal using MST/HTiO spheres

		Simulant solution		Actual tank solution	
Sample	Adsorbent (g)[a]	Sr DF	Sr K_d (mL/g)	Sr DF	Sr K_d (mL/g)
MST powder	0.10	291	7.24×10^5		
50 wt% MST (#1)	0.20	79	9.73×10^4	74.5	9.21×10^4
50 wt% MST (#2)	0.20	83	1.02×10^5	86.5	1.07×10^5
32 wt% MST	0.31	56	4.41×10^4		

[a] Equivalent MST = 0.1 g.

Table 6. Batch contact performance for plutonium removal using MST/HTiO spheres

		Simulant solution		Actual tank solution	
Sample	Adsorbent (g)[a]	Pu DF	Pu K_d (mL/g)	Pu DF	Pu K_d (mL/g)
MST powder	0.10	58.6	1.44×10^5		
50 wt% MST (#1)	0.20	20.5	2.44×10^4	31.8	3.85×10^4
50 wt% MST (#2)	0.20	25.1	3.01×10^4	25.6	3.08×10^4
32 wt% MST	0.31	19.6	1.49×10^4		

[a] Equivalent MST = 0.1 g.

Both strontium and plutonium were relatively easy to remove, and the K_d values easily exceeded 1.E+04. Strontium removal performance for the 50 wt% MST microspheres was consistent between simulant and the actual tank solution, while the plutonium removal performance was slightly better with the actual tank solution. The 50 wt% MST microspheres did not show solution matrix effects for plutonium.

The MST powder removed 99.7% of the strontium, while the HTiO microspheres with 50 and 32 wt% MST removed 98.8 and 98.2% of the strontium, respectively. In comparison, the HTiO microspheres without MST removed 95.5% of the strontium from a comparable alkaline waste at ORNL (3). These results confirmed a key assumption that was made prior to this effort. The performance of the HTiO microspheres would improve as the weight percent of MST in the microspheres increased. Because the performance of the HTiO microspheres with MST exceeded the SRS requirements for strontium and plutonium removal, the evaluation of the microspheres proceeded to column tests.

Column Tests

The HTiO microspheres with MST successfully removed plutonium and strontium. The plutonium content in the feed was similar for each test. However, since the strontium activity in the feed solutions for the two tests was sufficiently different, the strontium data were normalized to reflect a strontium feed content of 100. For the 50 wt % MST microspheres at a flow rate of 5.3 BV/h, plutonium removal was 99% initially and 94% after 2900 BV. Therefore, the microspheres had not yet reached their capacity. When the flow rate was increased from 5.3 to 15 BV/h, the plutonium adsorption performance was not significantly reduced. At a flow rate of 5.3 BV/h, the 50 wt% MST microspheres removed essentially all of the strontium throughout 2900 BV. However, the increase in the flow rate to 15 BV/h did lower the strontium removal to approximately 95% at 1600 BV.

During the column tests, no noticeable fines were observed, and no operational problems such as a pressure buildup were reported. Even though the column tests used microspheres with irregular shapes, the flow performance was still good. Because the crush strengths of the HTiO microspheres were very high, no problems are anticipated when the SRS remediation effort begins to use larger columns of the engineered MST.

CONCLUSIONS

MST is an inorganic adsorbent that effectively removes strontium, plutonium, uranium, and other trace elements from alkaline salt supernatant. The internal gelation process was used to produce an engineered form of the MST, which had a nominal particle diameter of 300 to 600 μm. This internal gelation process provided a unique means for the preparation of composite microspheres in which the MST powder can be homogenously dispersed throughout the gel-sphere matrix. The amount of MST in the HTiO microspheres can be varied. Microspheres with 32 and 50 wt% were made and tested in this study. Batch tests of these microspheres used actual SRS tank supernatant as well as a salt solution spiked with stronium and plutonium. In tank waste tests, the HTiO microspheres with 50 wt% MST produced batch distribution coefficients of 35,000 mL/g for plutonium and 99,000 mL/g for stronium. These results were confirmed by the results from the simulant batch tests. Finally, HTiO microspheres with 50 wt% MST successfully removed Pu and Sr in column tests. At a flow rate of 5.3 BV/h, plutonium removal was 99% initially and 94% after 2900 BV while essentially all of the strontium was removed throughout 2900 BV. Finally, these results clearly demonstrate that engineered MST with reasonable performance and physical properties can be made.

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