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Selective Removal of Strontium and Cesium from Simulated Waste Solution with Titanate Ion-exchangers in a Filter Cartridge Configurations-12092

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ABSTRACT

Experimental results for the selective removal of strontium and cesium from simulated waste solutions with monosodium titanate (MST) and crystalline silicotitanate (CST) laden filter cartridges are presented. In these proof-of-principle tests, effective uptake of both Sr-85 and Cs-137 were observed using ion-exchangers in this filter cartridge configuration.

At low salt simulant conditions, the instantaneous decontamination factor (D_F) for Sr-85 with MST impregnated filter membrane cartridges measured 26, representing 96% Sr-85 removal efficiency. On the other hand, the Sr-85 instantaneous D_F with co-sintered active MST cartridges measured 40 or 98% Sr-85 removal efficiency.

Strontium-85 removal with the MST impregnated membrane cartridges and CST impregnated membrane cartridges, placed in series arrangement, produced an instantaneous decontamination factor of 41 compared to an instantaneous decontamination factor of 368 for strontium-85 with co-sintered active MST cartridges and co-sintered active CST cartridges placed in series. Overall, polyethylene co-sintered active titanates cartridges performed as well as titanate impregnated filter membrane cartridges in the uptake of strontium.

At low ionic strength conditions, there was a significant uptake of Cs-137 with co-sintered CST cartridges. Tests results with CST impregnated membrane cartridges for Cs-137 decontamination are currently being re-evaluated.

Based on these preliminary findings we conclude that incorporating MST and CST sorbents into membranes represent a promising method for the semi-continuous removal of radioisotopes of strontium and cesium from nuclear waste solutions.

INTRODUCTION

Commercial processes frequently use ion exchange materials in a continuous process featuring either a packed column or a cartridge filter configuration. To prevent hydraulic problems such as high pressure drops across the column, the particle size of the ion-exchanger must be relatively large, which reduces particle packing and, therefore, overall efficiency. In a cartridge filter design, the ion-exchanger is incorporated into an inert, porous material that allows faster solution flow. Relative to the packed column, the cartridge design can employ an ion-exchanger with much smaller particle size, which enhances denser particle packing. Given the smaller particle size and denser packing, the diffusion distances between particles is expected to be minimized leading to more efficient adsorption. The net effect is greater utility of the ion exchanger and higher throughput.

In an earlier study, membranes incorporating both MST and CST particles had been successfully produced and found to be capable of selectively decontaminating strontium and cesium from simulated radioactive waste stream.^{i,iii} The task involved incorporating MST and CST particles with acceptable particle size distribution into a porous membrane, passing a salt solution containing cesium and strontium through membrane discs having a diameter of 47 mm and thickness of 1.5-2.0 mm and analyzing the effluent. Because of the success attained in the initial phase with the MST and CST-impregnated membranes, it was decided to fabricate and test the performance of titanate filtration cartridges incorporating membranes produced by both 3M and POREX[®] Filtration Division and housed in Pentair water filter units.

EXPERIMENTAL

The polytetrafluoroethylene (PTFE)-based and polyethylene (PE)-based membranes containing MST and CST particles were prepared by commercial partners using their proprietary fabrication technologies. Industrial partners included 3M for the PTFE/titanate loaded membrane designs, POREX[®] Corporation, Filtration Division for the PE/titanate sintered cartridge designs and Pentair Water for titanate cartridge wrapping to form cartridges with 3M titanate-loaded membranes.

CST pellets having a particle size distribution of 300 to 500 microns were obtained from UOP (Des Plaines, Illinois). Since the working particle size range for the CST had to be less than 80 microns (3M manufacturing requirement), the CST material was ground and wet vacuum sieved to obtain the desired particle size distribution between 45 and 75 microns. MST powder, having a particle size distribution of about 1 – 35 microns, was obtained from Optima Chemical Group, LLC (Douglas, GA) as a 15 wt% aqueous suspension. Additional details for preparing of the MST and CST materials for incorporating into porous membranes are presented in references ⁱ

After preparing the MST and CST particles to meet the required particles size distribution, the processed ion-exchange materials were shipped to the commercial partners for incorporation into the PTFE and PE membranes. The POREX[®] finished product included both MST and CST membranes and MST and CST cartridge filters based on their patented “sintered” technology. The technology involves co-sintering polyethylene particles with the MST or CST ion exchange particles and forming a molded and structured porous matrix, which constitutes the cartridge filter.. The ion exchange materials tend to be fixed into place with minimal leaching/extraction.

The 3M finished product included only the MST and CST membrane sheets. Pentair Water (Brookfield, WI) was provided sheets several meters in length of the impregnated MST and CST membranes from 3M, which they wound into filter cartridges (MST or CST cartridges). These filter cartridges were produced by wrapping the membrane sheets around a central core three times with specific lengths of each MST and CST membrane sheet and sealing the ends of the cartridge. These 3M and POREX[®] cartridges were put into commercially available cartridge housings which met the cartridge configurations shown in Table 1.

The dimensions of these cartridges (length, inner diameter and outer diameter) were standard values which fit into any Pentair Water's commercial filter housing with end-caps. The nominal dimensions of the cartridges along with weight percent and actual weights of the active ion exchange materials per cartridge are shown in Table 1. Representative MST and CST cartridges from both 3M and POREX[®] were dismantled and the weight of the active titanates determined based on the weight percent information of the MST and CST active sorbent materials as provided by 3M and POREX[®].

Filter cartridge unit configuration and testing methodology

The experimental configuration unit consists of a transparent box (45.72 cm x 30.48 cm x 20.32 cm) made of Lexan[®] polycarbonate sheets containing two transparent filter housing units connected in series. Each filter housing unit contains one filter cartridge. Thus, two filter housing units contains two filter cartridges in series with the MST cartridge the lead cartridge followed by the CST cartridge. The lead MST cartridge sees the incoming feed solution delivered by the peristaltic pump before the CST cartridge. The system contains three pressure gauges; one at the influent, the other between the two cartridge units and the last one placed after the CST cartridge, just before the effluent line. The unit was equipped with two valve sampling ports. The first sampling port valve was located between the two cartridges and the last one after the CST cartridge (effluent end).

The simulated waste solutions used for these tests featured salt solutions having two different ionic strengths and spiked with known levels of Sr-85, Cs-137 and Np-237. The simulated waste did contain uranium and plutonium, but the concentrations of these components were not measured in these tests. The lower ionic strength simulant featured a total sodium concentration of 0.29 M (derived from a simple dilution of the 5.6 M Na⁺ simulant), while the second simulant, known as the high-salt simulant, featured a total sodium concentration of 5.6 M. and 1.33 M free-OH. This solution was chiefly comprised of sodium salts of nitrate, hydroxide, nitrite, aluminate, sulfate and carbonate.

The simulant and radionuclide compositions are shown in Tables 2 Reagent grade chemicals (Fisher Scientific) and in-house ultra-pure water (MilliQ Element) were used in the preparation of the simulants. After the simulant preparations, the resulting solutions were stirred at ambient laboratory temperature for about three weeks, filtered through 0.45-micron disposable Nalgene[®] filters, and filtered solutions stored in tightly-capped polyethylene bottles until use.

The salt solution was pumped through the filter cartridges using a peristaltic pump at a flow rate of 80 ± 1 mL/min for the 3M cartridges and a flow rate of 67.0 ± 1 mL/min for the POREX[®] cartridges. Simulant flow through POREX[®] cartridges were maintained at lower flow rates to reduce pressure differences across the filter cartridges in series. For the 3M cartridges and the 0.29 M Na⁺ simulant, it took 26 ± 1 minute for the simulant to completely fill the lead cartridge housing. After this time the effluent from the

lead MST cartridge started flowing into the second cartridge housing containing the CST cartridge. At this time, sampling at the sampling port located between the two cartridge housings was initiated to determine the performance of the lead cartridge. It took another 19 ± 1 minutes or a total of 45 ± 1 minutes from when the pump was first turned on to have the second cartridge housing (CST cartridge) completely filled. Sampling of the effluent after the CST cartridge occurred using the second port. The pressure difference between the cartridges, at this flow rate, was less than 2.5 psi. The tests were repeated with the 3M cartridges using the 5.6 M sodium salt solution.

Tests with the POREX[®] cartridges were performed with both types of simulants at the same flow rates. The POREX[®] cartridges were more densely packed and, therefore, required a higher operating pressure of approximately 7 psi to obtain reasonable flow of 67 ± 1 mL/min. Thus, a time of 45 minutes was required to fill the lead MST cartridge housing and a total of 85 minutes to fill the second CST cartridge before sampling from the second or effluent sampling port could be started.

The sampling sequence included collecting two aliquots immediately after the solution came out of the lead MST cartridge and then two additional aliquots after the solution exited the CST cartridge. Thereafter, additional samples were collected after every five minutes of simulant flow through the cartridges at each of the two sampling ports. Only one of the duplicate samples was analyzed while the second was held in reserve in case of discrepancies.

To determine the average sorbate (Sr-85 or Cs-137) picked up by the cartridge during each sampling time, the corresponding measured activity for that sorbate was subtracted from the initial sorbate concentration in the simulant solution. This difference was normalized to give the average activity per gram sorbate [(dpm/mL)/g] using the weight of the sorbate per cartridge. Similarly, the normalized processed simulant volume was determined by dividing the volume of simulant in mL which had gone through each cartridge at each sampling event (using the liquid flow rate through the cartridge) by the total volume of simulant in the cartridge and cartridge housing (1, 500 mL). Thus, the adsorption performance graph presentations are based on a plot of the product of the average activity per gram sorbate and simulant flow rate in mL per minute (adsorption rate [dpm/min].g) versus the normalized processed volume. The sorbate activity changes with processed simulant volume are shown as plots of sorbate permeate activity (concentration at any time divided by initial sorbate concentration) versus normalized processed volume.

Batch Contact Test

A batch contact test was also performed with the “as-received” and ground CST materials used in the fabrication of the CST cartridges. In this batch experiment, about 0.1 grams of the respective sorbent samples were contacted with 10 mL of a 5.6 M Na⁺ salt solution containing Sr-85, Cs-137, Np-237, U-235/238 and Pu-239/240 for 24-hours at ambient laboratory temperature. The CST solids were removed by filtration and the filtrate analyzed by gamma spectroscopy to determine Sr-85, Cs-137 and Np-237 activities. The concentrations of U and Pu were not measured in these batch contact tests.

Table 1. A summary of 3M and POREX[®] filter cartridge configuration information.

	3M		POREX [®]	
	MST	CST	MST	CST
Absorbent weight per cartridge (Average), g*	7.99 (32 wt %)	28.76 (85 wt %)	60.4(32 wt %)	74.17(40 wt %)
Cartridge height (cm)	25.4	25.4	25.4	25.4
Cartridge diameter (cm)	5.715	5.715	5.715	5.715
Membrane length (cm)	10.16	10.16	NA	NA
Membrane thickness (cm)	4.57E-02	5.08E-02	NA	NA
Membrane wraps per cartridge	3	3	NA	NA
Average total weight of filter and active IX materials, g	24.96	33.84	188.74	185.41

NA: Not applicable. POREX[®] cartridges were based on sintered design. * PTFE and PE used as filler. Cartridges did not contain the same total mass of absorbent.

Table 2. Analytical Result for Simulant Composition

Analytes	0.29 M Na ⁺ solution	5.6 M Na ⁺ solution	Units
Al	1.26E+02± 8.49E-01	1.16E+04± 4.24E+01	mg/L
B	<9.2E+00	3.07E+01± 7.50E+00	mg/L
Ca	3.07E+00± 1.94E+00	9.23E-01± 8.34E-02	mg/L
K	<1.20E+00	8.85E+01± 2.69E+00	mg/L
Mg	5.06E-01± 0.00E+00	1.78E-01± 6.22E-03	mg/L
Mo	<5.34E+00	<5.34E-01	mg/L
Na	6.64E+03± 2.83E+01	1.28E+05± 5.66E+02	mg/L
S	<2.70E+01	1.93E+04± 3.96E+02	mg/L
Si	<1.19E+01	2.32E+01± 8.49E-01	mg/L
Zn	<1.06E+00	1.66E-01± 2.31E-02	mg/L
Cs-137	1.30E+05± 1.13E+03	3.59E+05 ± 2.97E+04	dpm/mL
Sr-85	1.00E+05± 7.07E+02	9.65E+04± 2.69E+03	dpm/mL
Pa-233	3.31E+02± 7.07E+00	4.95E+02± 9.19E+01	dpm/mL
Np-237	8.21E+02 ± 4.38E+01	1.05E+03± 1.13E+02	dpm/mL

RESULTS AND DISCUSSION

Filtration cartridges incorporating titanate ion-exchange sorbents, MST and CST, respectively, were fabricated using two different porous matrices and their performance for the removal of strontium and cesium were evaluated in low (0.29M Na⁺) and high (5.6M Na⁺) ionic strength salt solutions. The PTFE-based cartridges were assembled by Pentair Waster using titanate-loaded membrane sheets comprised of PTFE impregnated with MST or CST produced by the 3M Corporation. The PE-based cartridges were produced by the POREX[®] Corporation. Characteristics of the filter cartridges are summarized in Table 1.

The strontium-85 permeate activity performance overlay graphs (effluent concentration profile) are shown in **Figure 1 insert A** for both POREX[®]-produced MST and 3M produced MST in a low ionic strength salt solution simulant. Both graphs indicate a steady decrease in the Sr-85 activity with increase in process volume for MST-CST in a series configuration. Figure 1 insert B shows the plot of Sr-85 uptake rate with both MST-loaded filter cartridges from the 0.29 M Na⁺simulant. Both the 3M and POREX[®]-produced MST cartridges show a gradual and steady approach to steady-state adsorption of Sr-85 with increasing volume of simulant processed. The 3 M MST-cartridges exhibited 96% removal

efficiency for Sr-85 with an instantaneous decontamination factor of 26. The Sr-85 removal with POREX[®] cartridge exhibited 97% removal efficiency with an instantaneous decontamination factor of 40.

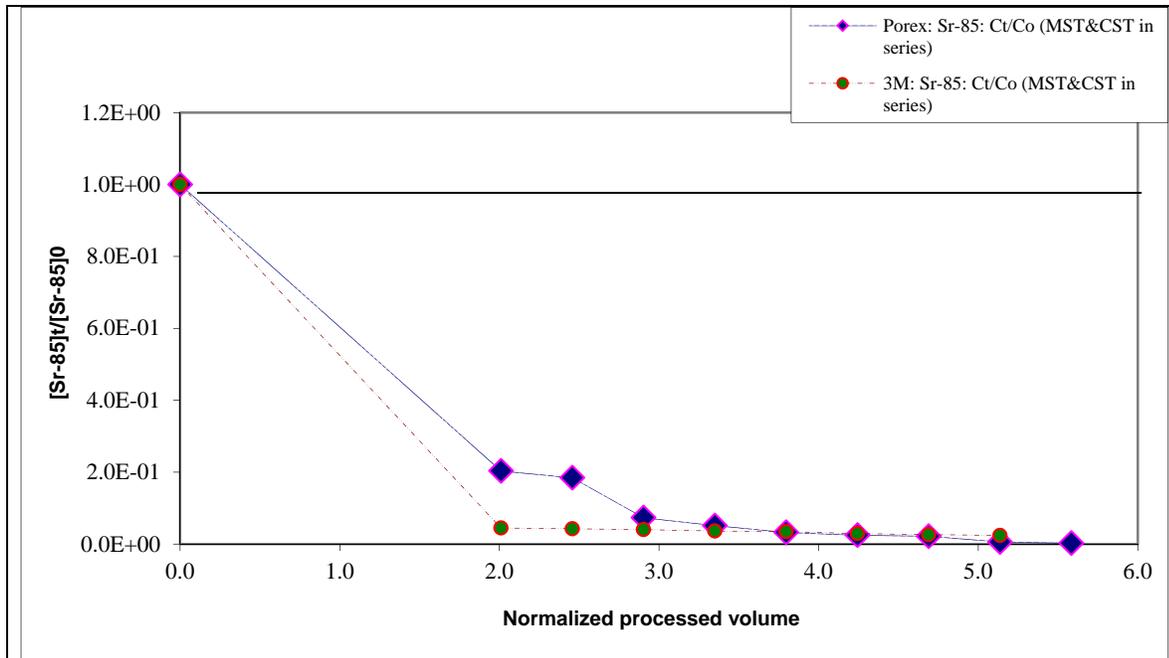
Interestingly, the 3M-cartridge showed a remarkable large uptake rate of Sr-85 compared with the POREX[®]-based cartridge even though the 3M-cartridge contained only about 13% of the MST quantity found in the POREX[®]-cartridges. Since these tests were not carried out to full saturation, it is difficult to ascertain whether the POREX[®]-based MST cartridges, with significantly more MST sorbate than the 3M cartridges, would have processed more simulant without overloading of the active titanate in comparison with the 3M-based MST cartridges.

The strontium-85 permeate activity performance overlay graph is shown in Figure 2 insert C for both 3M produced MST and CST in a low ionic strength salt solution. This effluent concentration profile overlay graphs indicate a steady decrease in the Sr-85 activity with increase in simulant volume processed for MST only and CST as well. Thus, although CST is designed mainly for the uptake of Cs-137, the graph also shows that a measurable amount of Sr-85 is sorbed by CST. Figure 2 insert D shows the uptake rate of Sr-85 on the lead MST cartridge and the lagging CST cartridge (3M designed cartridges). As expected, the CST-cartridge removed additional Sr-85 that was not removed by the preceding MST cartridge. The uptake rate of Sr-85 by the CST-cartridge appeared to be constant with increase simulant volume treated. After passing through both cartridges, the overall removal efficiency measured 97.6% with an instantaneous decontamination factor of 41, which is significantly higher than sampling immediately after the lead MST-cartridge (96% removal efficiency with instantaneous DF of 26). The increase in Sr-85 performance data is even much more pronounced with the POREX[®] MST and CST cartridges in series. After the lead MST-cartridge, the Sr-85 removal efficiency measured 97% (DF is 40). After passing through both cartridges, the Sr-85 removal efficiency measured 99.7% (DF of 368).

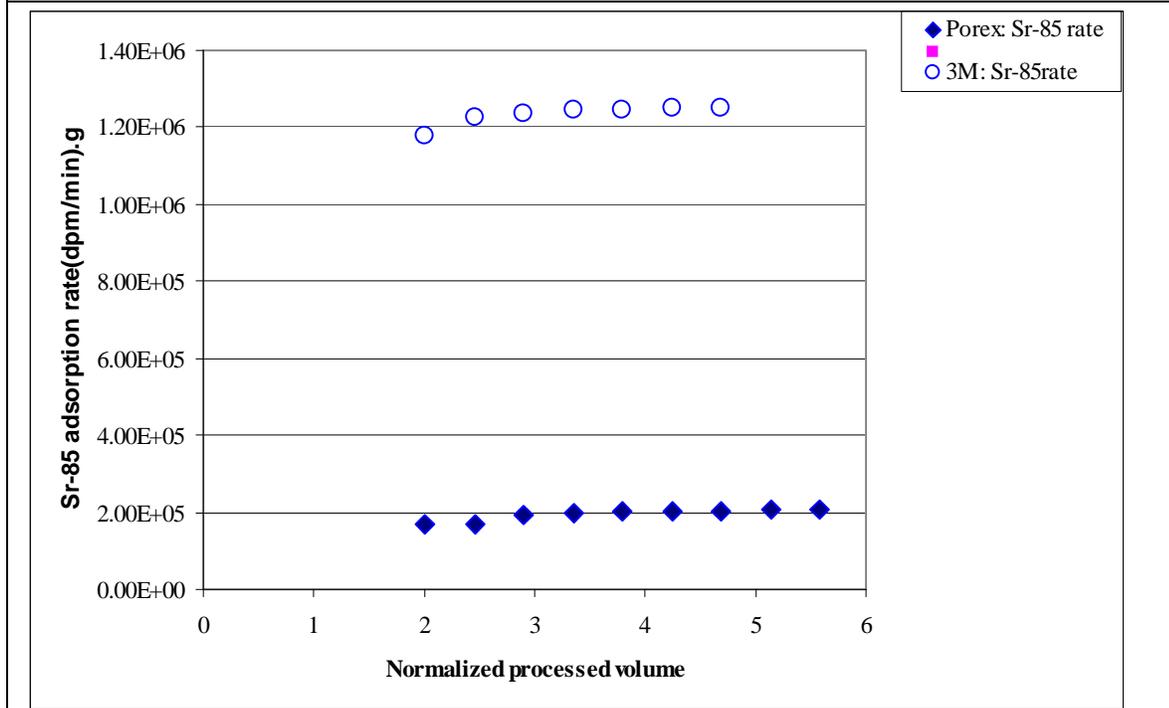
The cesium-137 permeate activity performance graph is shown in Figure 3 insert E for POREX[®]-produced CST in a low ionic strength salt solution. Tests with 3M produced CST cartridges for the sorption of CS-137 for the 0.29 and 5.6 M sodium simulant solutions are still in progress.

POREX[®]-based CST cartridge performance graphs in the uptake of Cs-137 indicate a steady decrease in Cs-137 activity in the simulant solution with increase in process volume. Figure 3 insert F shows the uptake rate of Cs-137 from the 0.29 M Na⁺ simulant with the CST cartridges prepared by POREX[®]. Overall, the POREX[®]-cartridge exhibited a high Cs-137 removal efficiency of 98% with instantaneous DF of 55. The Cs-137 uptake rate continued to increase steadily even after processing more than 7 liters of the simulant.

Previous testing had shown that MST does not adsorb cesium in strongly alkaline salt solutions. Analysis of samples taken immediately after the MST cartridges confirmed no measurable uptake of Cs-137 by the lead MST cartridges produced by POREX[®].

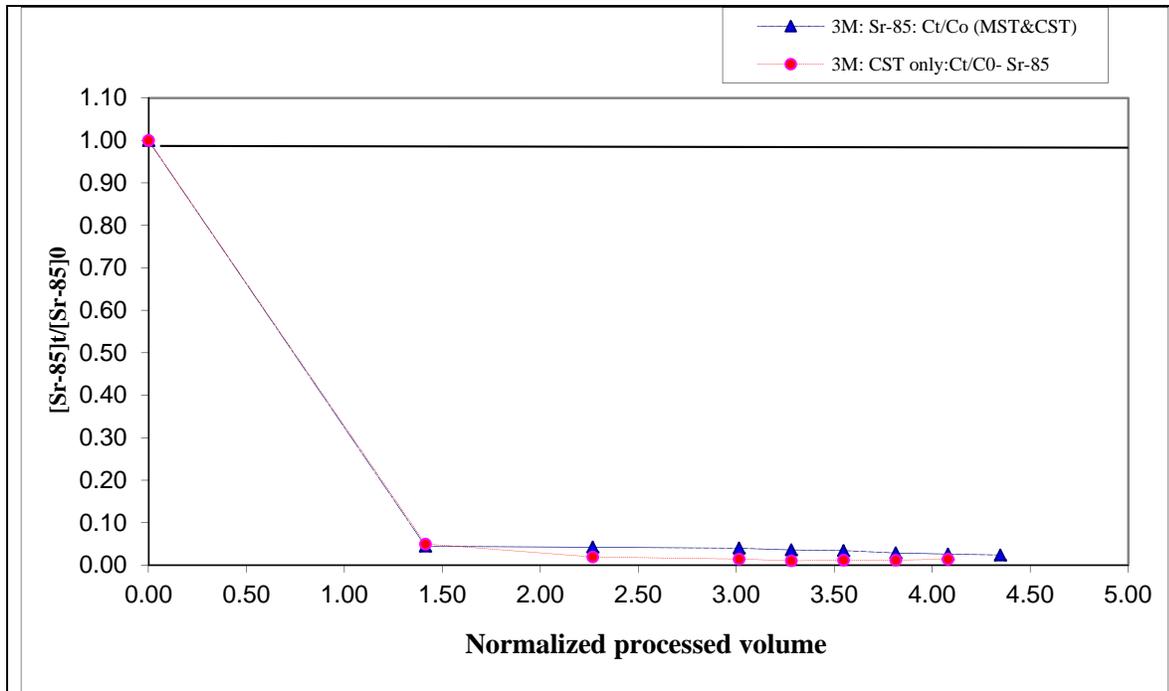


Insert A. Overlay plots for Sr-85 activity changes with respect to normalized volume of simulant processed (POREX® and 3M MST cartridge designs).

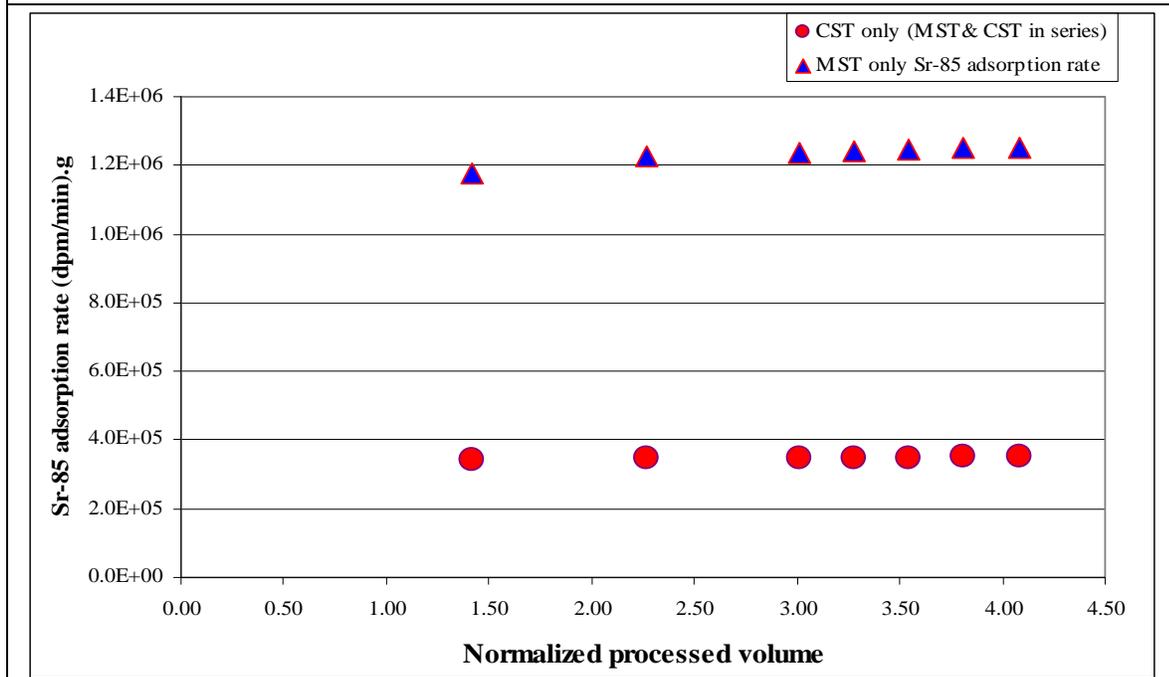


Insert B. Overlay sorption rate plots for strontium-85 removal with MST cartridges designed by 3M and POREX®.

Figure 1. Overlay plots for Sr-85 activity and sorption rate changes in a 0.29M Na⁺ simulant with both POREX® and 3M designed MST cartridges (MST and CST cartridges in series).



Insert C. Overlay plots for strontium-85 removal with MST only and CST cartridge only (MST/CST cartridges in series-0.29 M Na⁺ simulant).

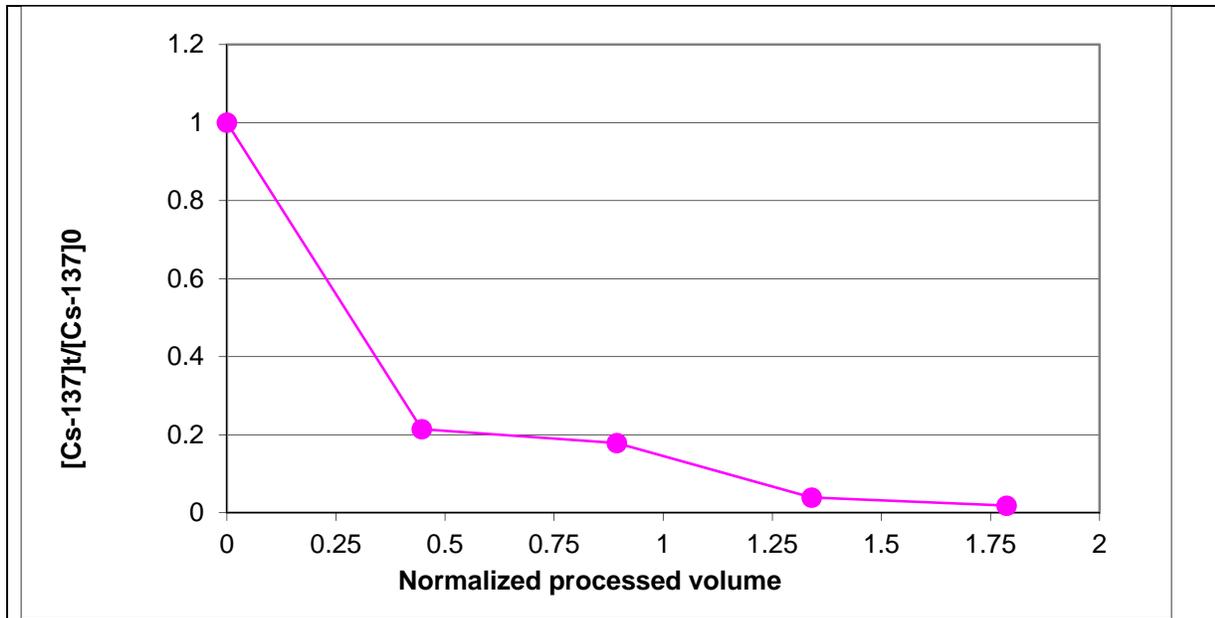


Insert D. Overlay sorption rate plots for strontium-85 removal with MST only and CST cartridge only (MST/CST cartridges in series-0.29 M Na⁺ simulant).

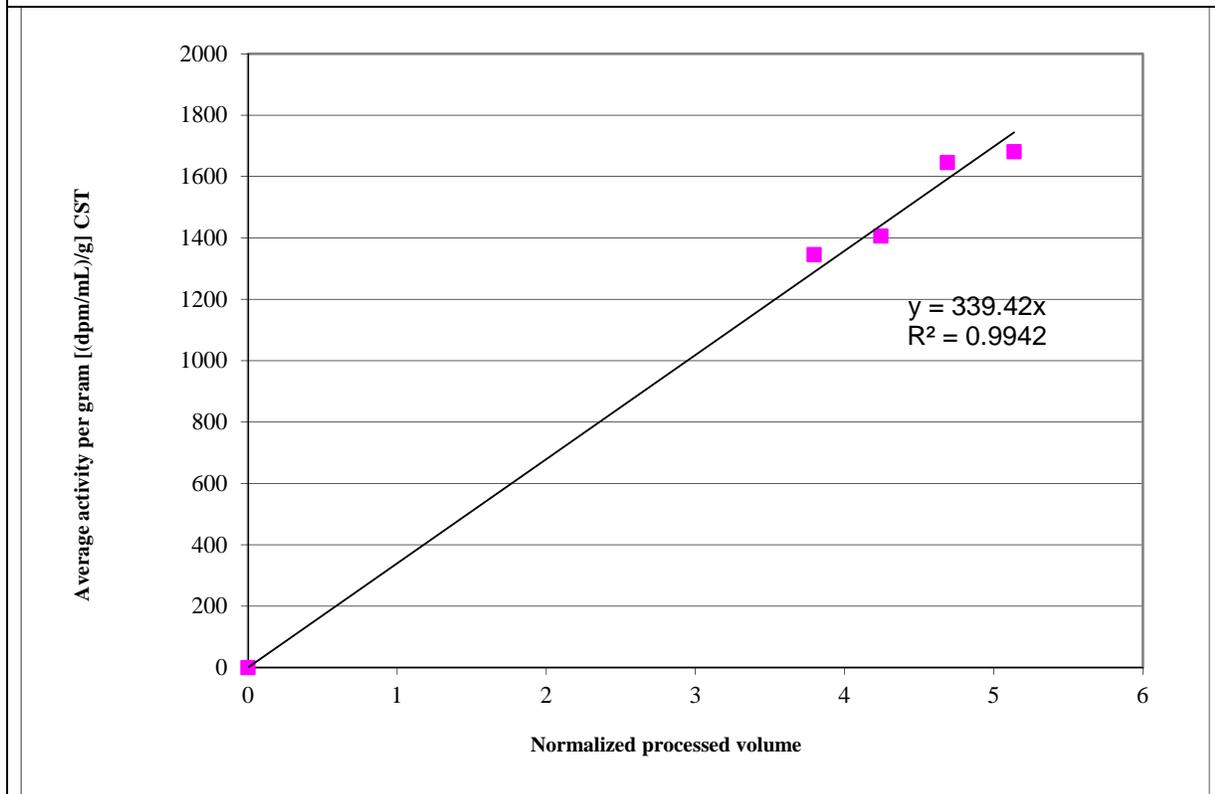
Figure 2. Overlay plots for Sr-85 activity and sorption rate changes in a 0.29M Na⁺ simulant with separate MST and CST cartridges only (POREX[®] and 3M designs)

The removal of Sr-85 from the 5.6 M Na⁺ salt simulant solution (higher ionic strength) was also tested. All experiments using the 5.6 M Na⁺ solution were performed only with the 3M-cartridges. Figure 4, insert G, provides a plot of the uptake rate of Sr-85 with the MST-cartridge only and with the MST (lead) and CST (lag) cartridges in series. The Sr-85 removal efficiency with the MST cartridge only measured 89% with an instantaneous DF of 8.3. Again, as observed with previous experiments, the lagging CST cartridge also removed Sr-85. This Sr-85 uptake with the lagging CST cartridge remained fairly constant with increase in volume of simulant processed as shown in Figure 4 (insert H). The Sr-85 removal efficiency with 3M MST and CST cartridges operated in series (3M design) measured 89% with an instantaneous DF of only 9.0.

To ascertain if changes in particle morphology of the CST material occurred as a result of grinding a 24-hour batch contact test with the “as-received” and ground CST materials was conducted. The results, as shown in Table 3, indicate no difference in the removal of Cs-137 between the “as-received” and ground CST. Thus, it is concluded that grinding did not have any influence on the uptake of Cs-137. Note that the ground CST exhibited a slightly higher uptake of Sr-85, but lower uptake of Np-237 compared to the as-received MST.

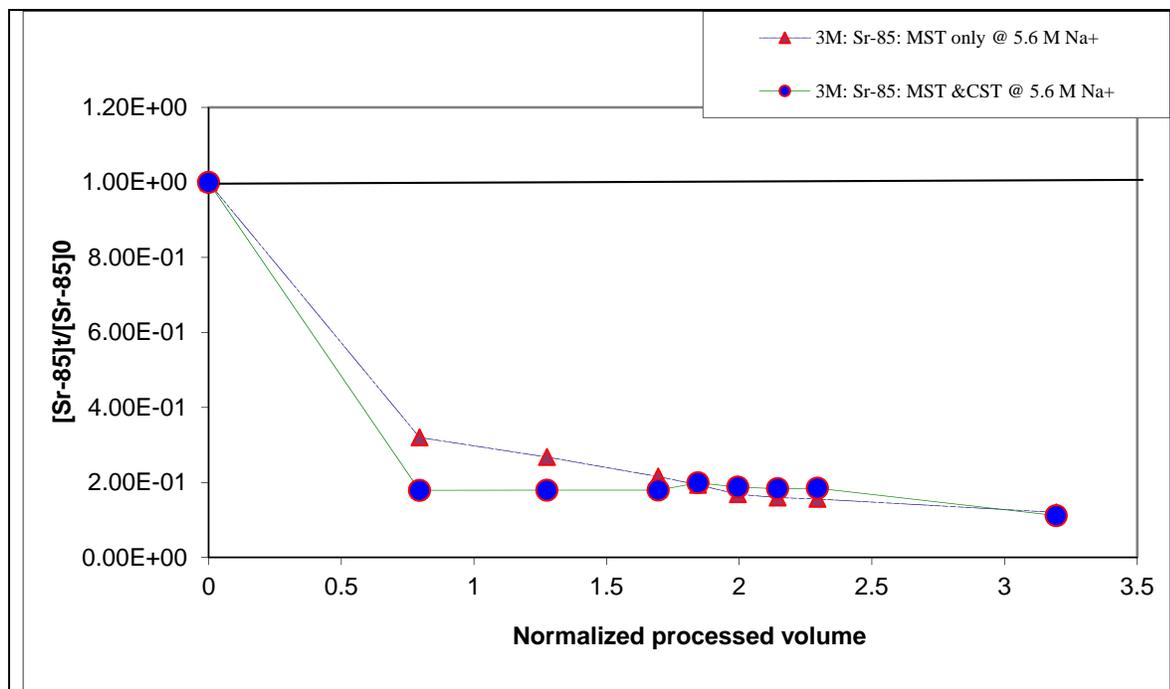


Insert E. Cs-137 activity changes with respect to normalized volume of simulant processed (POREX[®] cartridge designs).

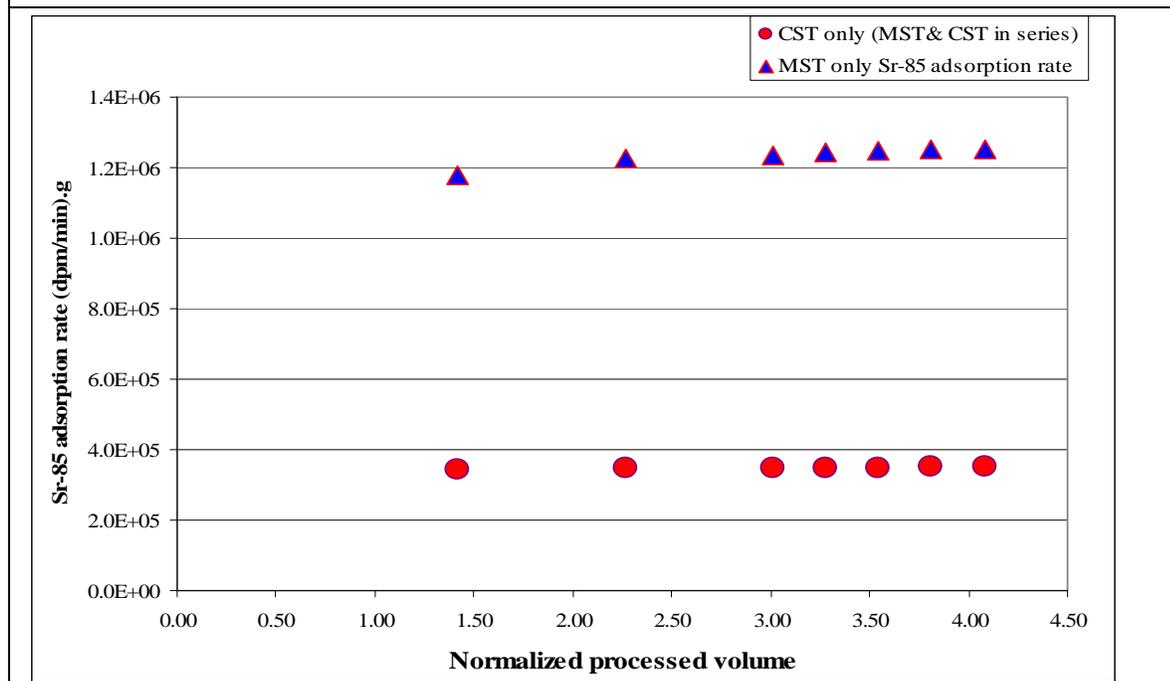


Insert F. Sorption rate plots for cesium-137 removal with CST cartridge POREX[®] designs.

Figure 3. Cs-137 activity and sorption rate changes in a 0.29M Na⁺ simulant with POREX[®] designed CST cartridges.



Insert G. Overlay plots for Sr-85 activity changes with respect to normalized volume of simulant processed (POREX[®] and 3M MST cartridge designs. 5.6 M Na⁺ simulant).



Insert H. Overlay sorption rate plots for strontium-85 removal with MST only and MST-CST cartridges in series (3M design) at 5.6 M Na⁺ concentration).

Figure 4. Overlay plots for Sr-85 activity and sorption rate changes in a 5.6 M Na⁺ simulant with both POREX[®] and 3M designed MST and CST cartridges.

Table 3. Batch Contact Experimental Results using the "As-Received" and Ground CST.

Section A- Experimental Results

	Concentration in simulant	Concentration after passing through "As-Received" CST	Concentration after passing through Ground CST
	dpm/mL	dpm/mL	dpm/mL
Cs-137	1.97E+05 ± 9.85E+03	1.12E+04 ± 5.60E+02	9.59E+03 ± 4.80E+02
Sr-85	1.24E+04 ± 6.20E+02	5.32E+02 ± 2.66E+01	1.29E+02 ± 8.37E+00
Np-237	1.79E+03 ± 9.76E+01	4.32E+02 ± 2.81E+01	6.07E+02 ± 4.30E+01

Section B-Calculated Df

		"As-Received" CST	Ground CST
Cs-137 D_F values	NA	17.6 ± 0.92	20.5 ± 1.08
Sr-85 D_F values	NA	23.3 ± 1.2	96.1 ± 5.2
Np-237 D_F values	NA	4.14 ± 0.24	2.95 ± 0.18
% Cs-137 Removed	NA	94.3%	95.1%
% Sr-85 Removed	NA	95.7%	99.0%
% Np-237 Removed	NA	75.9%	66.1%

Section C-Calculated K_d

		"As-Received" CST	Ground CST
Cs-137 K_d values, mL/g	NA	1.65E+03 ± 8.67E+01	1.92E+03 ± 1.01E+02
Sr-85 K_d values, mL/g	NA	2.22E+03 ± 1.17E+02	9.34E+03 ± 5.07E+02
Np-237 K_d values, mL/g	NA	3.13E+02 ± 1.84E+01	1.91E+02 ± 1.14E+01
Batch contact time, h.	24	24	24

CONCLUSIONS

Performance testing indicated that MST and CST sorbents embedded into inert PE and PTFE matrices and fabricated into filter cartridges effectively removed strontium and cesium from alkaline simulant salt solution. The POREX[®] filter cartridges exhibited excellent removal of strontium and cesium from the lower ionic strength simulant salt solution. Due to funding constraints, no testing was conducted with the POREX[®] cartridges with the higher ionic strength solution. The 3M filter cartridges exhibited excellent removal of strontium from both ionic strength solutions.

In general, the POREX[®] cartridges showed more resistance to flow of the salt solution through the cartridges than that observed for the 3M cartridges. Consequently, the test system operated at a higher pressure differential across the cartridges with the POREX[®] cartridges compared to that of the 3M cartridges to achieve flow rates of similar magnitude (67 mL/min for POREX[®] vs. 80 mL/min for 3M). Although POREX[®] cartridge designs involved the co-sintering of MST or

CST with polyethylene particles at a temperature range of 240-340 °F (212-171°C), the performance of these ion exchange materials were not significantly degraded, if at all.

Based on these findings we conclude that incorporating MST and CST sorbents into filter membranes represent a promising method for the semi-continuous removal of radioisotopes of strontium and cesium from waste solutions. Although not demonstrated fully in these tests, the strontium-removal performance of the MST cartridges suggests that these cartridges would also be very effective for alpha-emitting radionuclides (e.g., Pu, Np, Am and U) since previous tests indicate that MST alone is an excellent sorbent for these sorbates. Given these findings, the plan is to recommend additional studies to continue the development of this technology for treating radioactive waste solutions. Furthermore, given that MST effectively removed a variety of metal ions in weakly acidic and neutral solutions^{iii,iv} filter cartridges containing MST may also find utility for the removal of metal contaminants from a wide variety of aqueous waste streams^{v,vi}

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