Web Technology in the Separation of Strontium and Cesium from INEL-ICPP Radioactive Acid Waste (WM-185)

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Summary

Strontium and cesium were successfully removed from radioactive acidic waste (WM-185) at the Idaho National Engineering Laboratory, Idaho Chemical Processing Plant (ICPP), with web technology from 3M and IBC Advanced Technologies, Inc. (IBC). A technical team from Pacific Northwest Laboratory, ICPP, 3M, and IBC conducted a very successful series of experiments from August 15 through 18, 1994. The ICPP, Remote Analytical Laboratory, Idaho Falls, Idaho, provided the hot cell facilities and staff to complete these milestone experiments. The actual waste experiments duplicated the initial "cold" simulated waste results and confirmed the selective removal provided by ligand-particle web technology.

Acknowledgments

The experimental work at the Idaho National Engineering Laboratory, Idaho Chemical Processing Plant (ICPP), Remote Analytical Laboratory (RAL) would not have been possible without the encouragement and resources provided by Dr. Teresa Fryberger, manager of the U.S. Department of Energy's Efficient Separations and Processing Integrated Program. In addition, Dr. Leroy Lewis of ICPP was most gracious in providing the hot cell facilities and excellent staff to complete these milestone experiments. The RAL experiments were conducted by chemist Carl W. Lundholm and by G. D. DeCoria, who were assisted by an analytical team including R. L. Cummins, M. L. Evans, I. D. Goodwin, J. P. Henscheid, L. R. Hinckley, B. R. Hunter, V. J. Johnson, K. F. Martin, B. J. Storms, W. D. Thompson, and T. J. Tranter.

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1.0 Introduction

The U.S. Department of Energy's (DOE) Office of Technology Development (OTD), Efficient Separations and Processing Integrated Program sponsored Pacific Northwest Laboratory (PNL)^(a) to develop high-capacity, selective solid extractants for the recovery of cesium (Cs), strontium (Sr), and technetium (Tc) from nuclear wastes. Emphasis is on developing and demonstrating ion exchangers with potential applications in nuclear waste management that will result in major cost reductions and environmental benefits. This approach entails the identification and testing of candidates for solid-based sequestering agents as well as the development and application of existing materials.

A solicitation of interest and capabilities was issued in FY 1992 through Commerce Business Daily to identify candidate industry and university teams to collaborate with PNL in the proposed studies. In November 1992, DOE-OTD awarded a contract to a team consisting of staff from 3M of St. Paul, Minnesota, and IBC Advanced Technologies, Inc., of Provo, Utah.

The objective of this study was to demonstrate the removal of radioactive strontium (^{90}Sr) and cesium (^{137}Cs) from Idaho National Engineering Laboratory (INEL), Idaho Chemical Processing Plant (ICPP) acidic waste with the use of 3M and IBC separation technology^(b) (Goken 1994). This was accomplished by successfully completing a small-scale Remote Analytical Laboratory (RAL) test at ICPP in August 1994. PNL, IBC, and 3M used the RAL, to first "cold" test the procedures and equipment, and then to complete a series of tests in the hot cells (Figure 1.1) with actual ICPP radioactive acid waste.

Cesium and strontium are major contributors to the thermal energy and penetrating radiation hazards of stored high-level waste. Removing these and other radionuclides greatly reduces the volume of high-level waste for disposal and decreases the hazard level of the balance of the waste, thus offering more disposal options.

At the Hanford Site near Richland, Washington, the contents of 177 underground storage tanks include a mixture of sludge, salt cake, and alkaline supernatant liquids. The majority of these wastes resulted from the dissolution of irradiated fuel elements and the extraction of these feeds to recover the plutonium and uranium. The resulting acid waste was neutralized with caustic and placed in underground storage tanks. The insoluble sludge fraction of the waste consists of metal oxides and hydroxides and contains the bulk of many radionuclides. The salt cake, generated by extensive evaporation of aqueous

(a) PNL is operated by Battelle Memorial Institute for the U.S. Department of Energy.

(b) IBC's patented SuperLig® particles supplied to 3M for production of webs.

Figure 1.1. Small-Scale Experiment in the Remote Analytical Laboratory Hot Cell

solutions, consists primarily of dried sodium salts. The supernates consist of concentrated aqueous solutions of sodium and potassium nitrate salts. The bulk of the water soluble radionuclides such as ^{137}Cs is contained in the water soluble salt cake and supernatant solutions.

At INEL, the solvent extraction separations plant dissolved spent fuel elements and separated uranium as its main product. However, the acidic wastes from the solvent extraction systems were not neutralized but stored in stainless steel tanks before calcination. Many of these acidic wastes contain solvent extraction wastes, as well as laboratory and decontamination wastes.

Although the pretreatment and disposal requirements are still being defined, one of the first steps in most pretreatment scenarios at Hanford will be a salt cake dissolution and sludge wash step followed by a solids and liquid separation. Most of the cesium is expected to be in the aqueous liquids, and it is these solutions which are the focus of the cesium ion exchange removal process. This process is being designed to remove enough cesium so that the resulting low-level waste (LLW) will meet the requirements of U.S. Nuclear Regulatory Commission 10 CFR (*U.S. Code of Federal Regulations*) 61 Class A limits for ^{137}Cs (1 Ci/m^3). The separated cesium may be concentrated and vitrified for disposal in the high-level waste repository. The decontaminated aqueous liquid could be solidified as a LLW glass for near-surface disposal.

A second scenario includes a washed sludge dissolution step with nitric acid followed by ^{137}Cs and ^{90}Sr recovery from acid waste. Therefore, both at INEL and Hanford, acid wastes need to be treated. Thus, there is a need for adsorbents that are selective for cesium and strontium in the presence of competing ions and which function in both highly alkaline and highly acidic solutions.

Ion exchange columns have been used to separate cesium (Kurath, Orth, and Brooks 1994) and strontium (Kurath and Orth 1994) radionuclides from nuclear wastes for some time. The 3M technology with membrane filters differs from the use of columns: Solid phase extraction membranes contain adsorbents with particle sizes in the range of 2 to 80 micrometers and are operated in the range of 300 bed volumes (bv) per hour. Ion exchange columns are usually packed with particles of 200 to 800 micrometers and are operated at 0.6 to 10 bv/hr. The most important advantage in the use of membranes is that the smaller particles permit much faster separation with flow rates that are 30 to 100 times greater than column operation.

This report contains five sections. Section 2.0 describes the experiment, Section 3.0 presents the results, Section 4.0 summarizes the conclusions, and Section 5.0 lists the references.

2.0 Experimental

As part of the ongoing testing program to demonstrate new ion exchange materials for the recovery of strontium and cesium, Dr. Leroy Lewis, ICPP, indicated that a quantity of high-level acidic waste was available at the RAL hot cells. However, because ICPP would be cleaning out the cells in the fall of 1994, the window of opportunity was limited. Waste from tank WM-185 was selected for use based on the availability of 0.5 to 1 liter of feed. The original feed analysis was provided to PNL, 3M, and IBC by the Analytical Chemistry Section (ACS) at ICPP. Table 2.1 lists the characterization analysis of ICPP Tank WM-185 used as radioactive feed for the ion exchange experiments. With this data, a simulated waste was developed at PNL and shipped to each site (Provo, Utah, St. Paul, Minn., and Idaho Falls, Idaho) to provide enough simulant for development of the adsorbents and test procedures. Table 2.2 lists the synthetic ICPP tank farm acid waste, WM-185.

Table 2.1. Characterization Analysis of ICPP Tank WM-185 Used as Radioactive Feed for the Ion Exchange Experiments

Species, M		Radionuclides	
H ⁺	1.63	Gross α	865 μ Ci/L
Al	0.75	²⁴¹ Am	79 μ Ci/L
B	0.03	²³⁷ Np	3.9 μ Ci/L
Cd38	<0.00	²³⁸ Pu	540 μ Ci/L
Ca	0.065	²³⁹ Pu	65 μ Ci/L
Cr	0.005	¹⁵⁴ Eu	0.4 mCi/L
Fe	0.021	⁶⁰ Co	2.57E-3 mCi/L
Hg	0.004	¹³⁴ Cs	0.4 mCi/L
Pb	0.00145 (300 ppm)	¹³⁷ Cs	114 mCi/L
K	0.19	⁹⁰ Sr	128 mCi/L
Na	1.44		
Zr	0.01		
Total Sr	~6 ppm		
Total Cs	~5 ppm		
Zr	0.01		
Ni	0.0017		
Mo	0.0005		
Cd	0.0037		
Hg	0.0036		
U	110 mg/L		
Sp.G	1.26		

Table 2.2. Synthetic ICPP Tank Farm Acid Waste, WM-185

Component	FW, g	M/L	Species, M	
HNO ₃	15.7M	1.63	H ⁺	1.71
Al(NO ₃) ₃ ·9H ₂ O	375.15	0.75	Al	0.75
Ca(NO ₃) ₂ ·9H ₂ O	236.16	0.065	Ca	0.065
NaCl	58.45	0.03	Cl ⁻	0.03
NaF	42.0	0.16	F ⁻	0.16
Fe(NO ₃) ₃ ·9H ₂ O	404.02	0.021	Fe	0.021
Pb(NO ₃) ₂	331.23	0.00082	Pb	0.00082
Ni(NO ₃) ₂ ·6H ₂ O	290.80	0.0017	Ni	0.0017
KNO ₃	101.11	0.19	K	0.19
NaNO ₃	85.0	1.25	Na	1.44
H ₂ SO ₄	18M	0.04	SO ₄	0.04
Sr(NO ₃) ₂	211.65	6.62E-5	Sr	6.6E-5 (5.8 ppm)
CsNO ₃	195.0	3.9E-5	Cs	3.9E-5 (5.2 ppm)
			NO ₃ ⁻	5.52

IBC's patented SuperLig® particles used for strontium and lead (Pb) removal from the ICPP tank waste were supplied to 3M for production of webs. These particles involve non-ion exchange adsorbents that consist of highly specific ligands such as macrocycles covalently bonded to hydrophilic solid supports which are effective even for dilute-targeted elements in difficult matrices. Such particles exist in column or membrane forms for a large portion of the periodic table.

An IBC ligand for the recovery of cesium from acid solution was not fully developed in time for the RAL tests. PhTiA, a crystalline titanium phosphate ion exchange material (Jones 1966), was selected, ground to a suitable size, and placed into a web. ABEDem TiA (hereafter abbreviated PhTiA) was purchased under the registered trade name ABEDem TiA®, Societe d'Etudes, de Recherches et s'Applications pour l'Industrie (SERAI, Brussels, Belgium). PhTiA provided an opportunity to begin testing a wide variety of available small particle (2 to 80 mμ) ion exchangers in a web configuration.

"Cold" tests were completed at the St. Paul site by the PNL, 3M, and IBC team using synthetic waste to prepare for the RAL "hot" test. The team then went to the Idaho Falls site and repeated the simulant tests with RAL staff, before hot cell testing with actual waste.

A series of tests with either IBC ligand-silica webs, and/or the PhTiA webs was completed with 25-mm-diameter polypropylene filter holders (Cole-Parmer catalog, H-06623-32) containing one to two webs. The webs, 20 to 40 mils thick, were cut with a 25-mm punch, placed in the filter holders, and retained with an O-ring. The holder was assembled by screwing the collar onto its base. Because the filter holders tended to leak during "cold" testing, they were epoxied at the joints.

Synthetic or actual WM-185 feed was pumped through a series of filter holders with a MasterflexTM pump drive (Cole-Parmer catalog, H-07553-80) and a MasterflexTM pump head (Cole-Parmer catalog, H-07518-10) and controller. The filter holders were attached to the pump with size #13 C-flexTM tubing (Cole-Parmer catalog, H-06424-13). The volume of the feed pumped through the series of filter holders (mL/min) was determined by weighing the effluent and dividing by the feed-specific gravity. A series of weighed samples was taken as a function of time during the test and sent to the ICPP-ACS for analysis. The synthetic feed sample values (Sr, Cs) were determined by inductively coupled plasma-mass spectrometry (ICP-MS) or atomic absorption (AA), while the actual waste samples were determined by gamma (¹³⁷Cs) or beta (⁹⁰Sr) energy analysis. The column volumes (cv) or bv of feed that passed through the filter holders were calculated based on the volume of the web, that is, the diameter and the thickness of the web.

3.0 Results and Discussion

Recently, the use of membranes for the separation of ^{137}Cs and ^{90}Sr from a sample of actual acidic tank waste was demonstrated at ICPP. Results for several tests show that both Sr and Cs can be removed from solution to very low concentration values. A summary of the test results is shown in Table 3.1. Removal of ^{137}Cs from the acid waste was accomplished with PhTiA, a crystalline titanium phosphate adsorbent, Figure 3.1. Good agreement was observed between actual (CSP, CSW, and CSZ) and simulated waste (2322-20 and -23) tests. The adsorbent had a high capacity despite the presence of sodium and potassium at high concentrations relative to cesium. In addition, the results for cesium capacity were similar to those previously reported with ion exchange columns and Hanford acidic radioactive wastes (Jones 1966).

Similar tests were completed for the removal of ^{90}Sr with three IBC proprietary adsorbents, SuperLig[®]-614, -320, and -620. Agreement for Sr recovery was observed between actual (SRX and SRY) and simulated waste (2322-18) tests, Figure 3. Normally only the SuperLig[®] 620 would have been used for the recovery of strontium. However, it was known that lead had been used to elute strontium from the SuperLig[®] 620 exchanger. Therefore, two IBC lead SuperLig[®] exchangers were placed in the system ahead of the SuperLig[®]620 to intercept the Pb before Sr recovery. Figure 3.2 shows the test in which six webs were used in series, the first four [SuperLig[®]-614(2),-320(2)] to capture Pb and the last two webs (-620) to retain the Sr. After 10 cv of feed passed through the system, the capacity of the Pb retaining webs was exceeded, and Pb began eluting the loaded strontium from the SuperLig[®]620 webs. This is indicated by noting that the effluent > 30 cv contains Sr in excess of $C/\text{Co} = 1$.

Agreement between simulated and actual wastes in the above tests indicates that the analysis of the waste was reliable and that tests completed with radionuclides can be duplicated with stable elements. Agreement between actual and simulated waste is important during scale-up of a membrane or cartridge process with less-expensive non-radioactive solutions.

Table 3.1. Summary of Test Results

Test No.	Adsorbent Web, ID	⁹⁰ Sr	¹³⁷ Cs	⁹⁰ Sr	¹³⁷ Cs
		# of cv @ C/Co = 0.5	# of cv @ C/Co = 0.5	Volume of 1 cv, mL	Volume of 1 cv, mL
CPS	PhTiA SuperLig® 614,320,620	Approx. 10	100	0.193	0.386
CSW	PhTiA	—	100	—	0.386
CSZ	PhTiA	—	100	—	0.386
2322-20●	PhTiA	—	130	—	0.386
2322-23●	PhTiA	—	95	—	0.386
SRX	SuperLig® 614,320,620	18	—	0.386	—
SRY	SuperLig® 614,320,620	25	—	0.772	—
2322-18●	SuperLig® 614,320,620	22	—	0.772	—

Note: Flow rates for the above tests were 1 to 2.5 mL/min, flow diameter of the web = 22 mm, and 0.51 mm (20 mils) web thickness; PhTiA was purchased under the registered trade name ABEDEM TiA®, Societe d'Etudes, de Recherches et s'Applications pour l'Industrie (SERAI, Brussels, Belgium); and (●) = synthetic waste test.

Figure 3.1. Removal of ^{137}Cs from ICPP Tank WM-185 Acid Waste with the Use of PhTiA Ion Exchange Web Technology

Figure 3.2. Removal of ^{90}Sr from ICPP Tank WM-185 Acid Waste with the Use of SuperLig[®] Ligand-Web Technology

4.0 Conclusions

Strontium and cesium were successfully removed from one of the ICPP radioactive acidic wastes with a new and innovative technology. The acidic waste experiments duplicated the initial simulated waste results and confirmed the selected removal provided by IBC ligand-particle (Sr, Pb) and the ion exchange (Cs) web technology. The tests confirm the efficiency of fine particle-web technology. This same technology can be applied to many other adsorbents that have been prepared as particles, too fine for use in standard ion exchange columns because they would create too much back pressure. The technique used to incorporate particles into membranes is flexible. It can be used with almost any material with an average particle size of 2 to 80 micrometers. Therefore, many adsorbents that could only be made as fine particles can now be used. Other adsorbents that are large enough for column use can be ground to a finer size and incorporated into membranes. Such materials can then be used at much higher flow rates to achieve more rapid separations.

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