ANL/CMT/CP--88394

CONF-9606116--23

OVERVIEW OF MINERAL WASTE FORM DEVELOPMENT FOR THE ELECTROMETALLURGICAL TREATMENT OF SPENT NUCLEAR FUEL*

C. Pereira
Argonne National Laboratory
Chemical Technology Division
9700 South Cass Avenue
Argonne, IL 60439
(708) 252-9832

M. A. Lewis
Argonne National Laboratory
Chemical Technology Division
9700 South Cass Avenue
Argonne, IL 60439
(708) 252-9832

J. P. Ackerman
Argonne National Laboratory
Chemical Technology Division
9700 South Cass Avenue
Argonne, IL 60439
(708) 252-9832

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Submitted for Presentation at
DOE Spent Nuclear Fuel & Fissile Material Management
Embedded Topical Meeting
Spent Nuclear Fuel - Treatment Technologies
Reno, Nevada, June 16-20, 1996

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*Work supported by the U.S. Department of Energy, Nuclear Energy Research and Development Program, under Contract No. W-31-109-Eng. 38.

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J. P. Ackerman
Argonne National Laboratory
Chemical Technology Division
9700 South Cass Avenue
Argonne, IL 60439
(708) 252-9832

ABSTRACT

Argonne National Laboratory is developing a method to treat spent nuclear fuel in a molten salt electrorefiner. The wastes from this treatment will be converted into metal and mineral forms for geologic disposal. A glass-bonded zeolite is being developed to serve as the mineral waste form that will contain the fission products (rare earths, alkali and alkaline earth metals, halogens, and chalcogens) that accumulate in the electrorefiner salt. Fission products are ion exchanged from the electrorefiner salt into the zeolite A structure. The crystal structure of the zeolite after ion exchange is filled with salt ions.. The salt-loaded zeolite A is mixed with glass frit and hot pressed. During hot pressing, the zeolite A may be converted to sodalite, which also retains the waste salt. The glass-bonded zeolite is leach resistant. MCC-1 testing has shown that it has a release rate below 1 g/(m^2day) for all elements.

I. INTRODUCTION

Argonne National Laboratory is developing a method to treat spent nuclear fuel in a molten salt electrorefiner.[1] The wastes from this treatment will be converted into metal and mineral forms for geologic disposal.[2] Spent fuel elements are chopped, placed in steel baskets, and immersed in an electrolyte, where they are anodically dissolved from the cladding. Pure uranium is deposited on steel cathodes. The transuranic (TRU) elements in the fuel remain in the electrolyte and are removed in the mineral waste, along with the salt-borne fission products (rare earths, alkali and alkaline earth metals, halogens, and chalcogens.) Alternatively, the TRU elements can be removed with a liquid cadmium cathode[1,3,4] and put in the metal waste form. Noble metal fission products and cladding remain in the anode baskets; they are incorporated into a stainless steel--zirconium alloy as metal waste.

A glass-bonded zeolite is being developed as a mineral waste form to contain the fission products that accumulate in the electrorefiner salt.[5] The salt is trapped within the crystal structure of the zeolite. Fission products ion exchange into the zeolite. The fission-product cations are associated with the salt and with the anionic framework of the zeolite. This glass-bonded zeolite will encapsulate the waste salt in a molecular cage, with the zeolite particles embedded in the glass matrix.

II. PROCESS OVERVIEW

The overall process for producing the mineral waste form is shown in Figure 1. The fission product-loaded electrolyte is contacted in an ion-exchange process using a column filled with zeolite A, which sorbs the fission products and salt into its crystal structure. The column concentrates the fission products in the zeolite, producing a relatively clean effluent salt which may then be recycled. The zeolite column is pre-loaded with LiCl-KCl eutectic salt (~42.5 wt% LiCl) to maintain the overall chloride balance and to improve ion-exchange kinetics. The fission product-loaded zeolite from the column is the starting material for waste form production. The effluent salt is recycled to reduce the overall mineral waste volume.

The ion-exchange process may be by-passed by blending the salt directly with zeolite in a single high-temperature batch mixing process. In this case, the zeolite is blended with an appropriate quantity of anhydrous zeolite so that the chloride is fully occluded in the zeolite structure. "Fully occluded" implies that there are somewhat fewer than 12 chloride ions per unit cell of zeolite.[5,6] More waste is produced than in the column process because no salt is recycled. However, fewer processing steps are required before the salt is fully occluded in zeolite if the ion-exchange column is eliminated. The hot-blended zeolite contains very little free salt.

The effectiveness of the zeolite column system in treating the electrolyte salt is currently being tested. The use of a zeolite column will be determined by the effectiveness of the column in sorbing fission products, the fission-product heat load in the electrolyte, and the cost of the additional processing steps that are introduced. Heat load is critical because the zeolite structure begins to degrade at temperatures much above 900 K. A blending step is required to fully occlude residual chloride

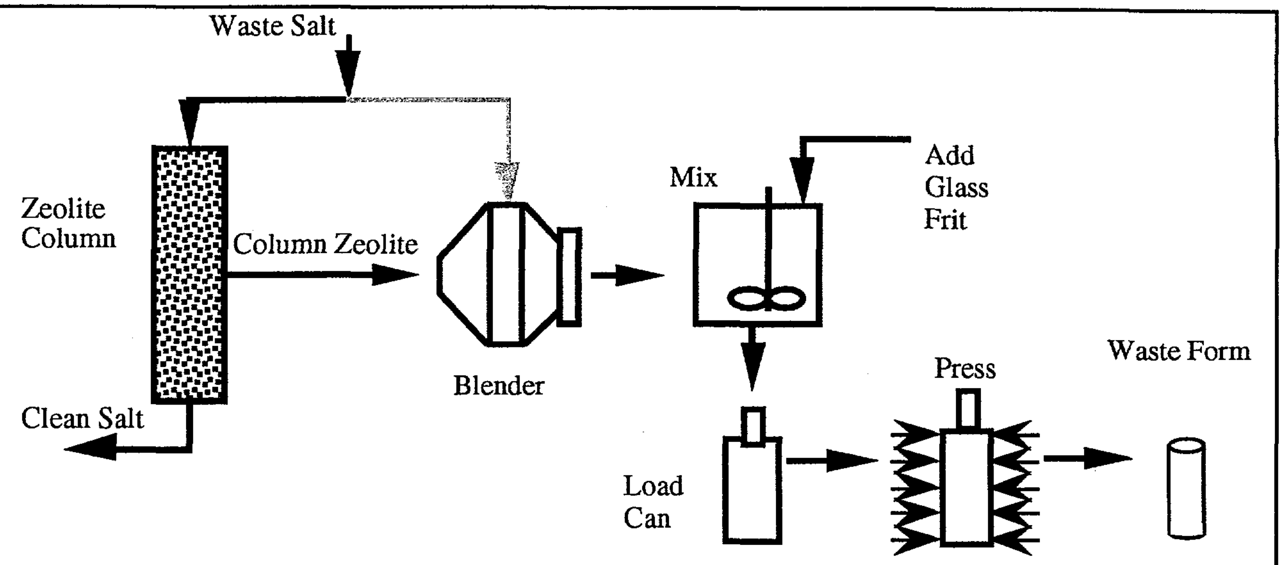

Figure 1: Overview of Mineral Waste Form Process

remaining in the interstices of the column packing. Because the column zeolite is pelletized, a grinding step may be required before hot pressing. Finally, the effluent salt from the column must be filtered and treated to remove any oxides or metals before it is reintroduced into the electrorefiner.

After hot blending, the fission product-loaded zeolite is mixed with glass frit and hot pressed. Mixing is carried out at ambient temperature under inert atmosphere. The powder mixture is pre-compacted at room temperature, dried at 773 K, and then hot pressed.

Two hot pressing methods have been tested. The products that have been produced by the two methods have similar physical properties. Hot uniaxial pressing was used to form all of the samples that have been leach tested to date. A hot isostatic press (HIP) is currently in operation and will be used in the eventual process. It can manufacture larger samples, and the final product is contained within a stainless steel canister. The processing conditions required with the HIP are currently being refined.

The glass-bonded zeolite product is a solid two-phase material. The zeolite (or sodalite) crystals have an average particle size of 3 to 4 μm and are dispersed within the glass phase, which serves as a binder. The zeolite contains all of the salt and fission products.

Several glass compositions are being tested; in general, the glasses contain 50 to 65 wt% silica with lesser amounts of alkali and alkaline earth oxides. The final product is a dense monolithic solid with low, open porosity and good leach resistance. Gross physical properties measured to date are comparable to published data for other borosilicate waste glasses.[7] The composition and manufacture of the final product is being optimized to provide the greatest leach resistance.

III. ION EXCHANGE INTO ZEOLITE A

A. Materials

Zeolite A was obtained in powder form and as clay-bound pellets from UOP. The pellets contained between 5 and 10 wt% binder and were formed into 12 x 25 mesh beads. The zeolite was dehydrated stepwise to 800 K under a nitrogen flow and transferred directly to an inert atmosphere facility. The X-ray diffraction patterns of the initial and dehydrated materials did not change.

The simulated waste salts were formed from reagent grade components (Johnson Matthey, Co.), which were physically mixed, melted, filtered, and coarsely ground. The LiCl and KCl were obtained at the eutectic composition (42.5 wt% LiCl, melting point ~615 K) from APL Engineered Materials, Inc. Typical salt compositions used in most of the tests are given in Table 1; most tests were done with salt 1 or salt 2. In addition, one salt that contained 14.4 wt% plutonium and 4.2 wt% cesium was also used in a single-batch test. Plutonium and uranium were added by oxidizing the metals into LiCl-KCl eutectic. Other components were added as chlorides.

Table 1: Salt Compositions (wt%)

Component	Salt 1	Salt 2	Pu Salt
KCl	45.3	49.7	54.4
LiCl	39.7	39.5	45.0
CsCl	3.7	2.3	*
NaCl	6.0	*	*
$BaCl_2$	1.4	1.05	*
$SrCl_2$	0.6	0.4	*
KI	0.3	0.3	*
$CeCl_3$	0.8	2.0	0.6
$LaCl_3$	1.1	0.6	0.6
$NdCl_3$	1.0	3.2	0.2
$PrCl_3$	*	0.8	*
YCl_3	0.1	0.1	0.02
UCl_3	*	*	0.3
$PuCl_3$	*	*	0.7

* Not present in salt.

B. Ion-Exchange Tests

1. Preliminary Batch Ion-Exchange Tests. In practice, fission products in the molten salt will be ion exchanged into salt-loaded zeolite A in a column operation. Laboratory-scale batch tests were done in order to determine design requirements for a pilot plant-scale column facility. The batch tests were carried out by contacting anhydrous zeolite A, either as powder or as pellets, with a large excess of waste salt. Zeolite pellets (in a basket) or powders (in a cup) were immersed in the molten salt from 10 min to 72 h at ~725 K. After immersion in the salt, the samples were washed with deionized water to remove residual salt that was not occluded in the zeolite structure. Plutonium ion-exchange experiments were conducted in a similar fashion, but only for 72-h contact times.

Figure 2 presents the results for a series of tests in which zeolite pellets are contacted with an excess of salt. The ion-exchange process that it describes is rather slow. The cesium concentration in the zeolite increased relatively rapidly with time, reaching a steady level after immersion in the salt for approximately 1 h. The alkaline earths behaved differently, reaching maximum loading after 4 to 6 h, but decreasing thereafter. Rare earths exchanged into zeolite continuously but very slowly. After the first 72 h, rare earth exchange continued, partly by displacing alkaline earths from the zeolite. Cesium is not displaced.

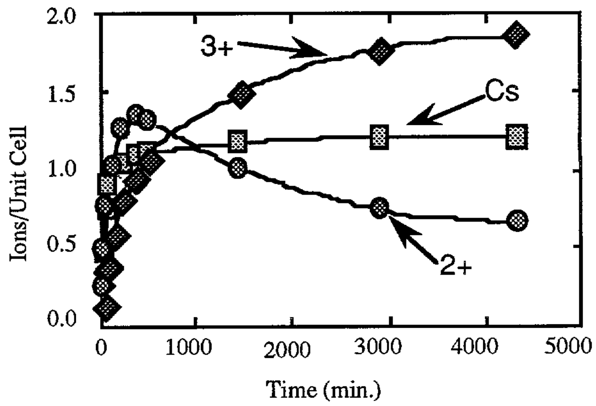

Figure 2: Ion-Exchange Kinetics; Cesium (▨), Alkaline Earth (●), and Rare earth (◆).

The exchange reaction can be represented by the following equation:

$$A_{(z)} + 1/nM^{n+}_{(s)} \rightarrow 1/nM^{n+}_{(z)} + A_{(s)} \qquad (1)$$

Here A represents an alkali metal, (Li, K, or Na), z represents the zeolite phase, M is a fission product with charge n, and s the salt phase. The exchange process can be modeled by ion-exchange factors (EF) relative to sodium (or another alkali), similar to equilibrium separation factors in solution:

$$EF = [M]_z^{1/n} x[Na]_s/[M]_s^{1/n} x[Na]_z \qquad (2)$$

Potassium and sodium are held weakly and have exchange factors of ~1. Lithium and the alkaline earths show intermediate adsorption. Cesium and the rare earths are held most strongly by the zeolite and have the highest exchange factors. The exchange factors vary with the fission product concentration in the salt and temperature as shown in Figures 3 and 4. Temperature has little effect on the exchange factors. Exchange factors of alkaline earths and samarium, which is divalent cation in this system, may decrease slightly with increasing temperature. The exchange factors of the rare earths, however, particularly lanthanum, appear to increase with temperature. The other alkali metals are constant. The alkaline earths and samarium show a slight decrease with higher temperatures.

The fission-product concentration in the salt has a more marked effect on the exchange factors in Figure 4.

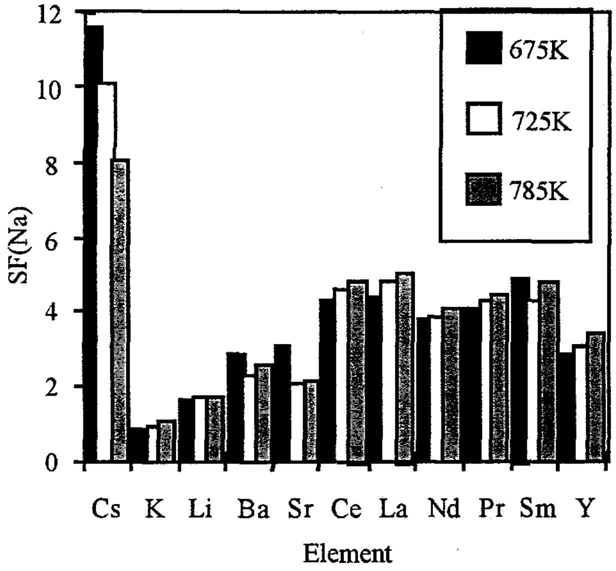

Figure 3: Effect of Temperature on Exchange Factors: 675K (■), 725K (□), and 785K(▨).

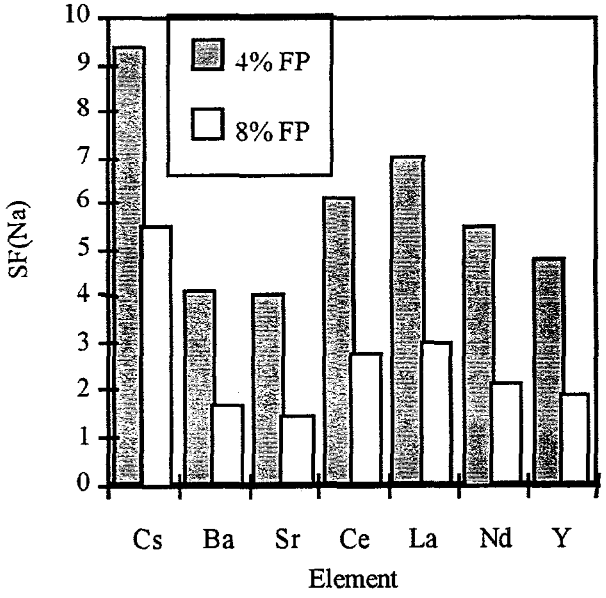

Figure 4: Effect of Salt Composition on Exchange Factors: 4 wt% Fission Products (FP) (▨) and 8 wt% Fission Products (□) in Salt.

These tests were conducted with two salts of differing compositions: salt 2 (containing 8 wt% fission products) and a salt formed by adding enough LiCl-KCl eutectic salt to salt 2 to lower the overall fission-product concentration by half to 4 wt% fission products. Little variation exists in the relative values of the exchange factors, but the magnitude of the individual factors varies inversely with the concentration in the salt. The exchange factors are higher when the fission product concentration in the salt is low.

2. Ion Exchange of Plutonium into Zeolite A. Batch ion-exchange tests were also used to determine the extent to which plutonium ion exchanges into zeolite A. Tests with plutonium-bearing chloride salt indicate that plutonium ion exchange is similar to that measured for the rare earths. Zeolite A has a strong affinity for plutonium in the molten salt, although the final zeolite composition is a strong function of the plutonium concentration in the electrolyte.

For a salt with an initial plutonium concentration of 0.5 wt%, the zeolite contained 2.1 wt% plutonium, while the fission products and uranium comprised 7.3 wt% of the final zeolite. For the more concentrated salt having an initial plutonium concentration of 15 wt% and a cesium concentration of 4.7 wt%, the zeolite contained 22.5 wt% plutonium while cesium uptake was 3.9 wt%. Cesium was not sorbed by the zeolite as effectively as plutonium. There was some evidence for contamination of the zeolite samples with cadmium metal for this latter case. The plutonium loading in the zeolite may have been higher than reported here.

The final concentration of plutonium in the zeolite is much higher than in the salt in both tests. The relative increase in plutonium concentration is greater at low concentrations, even though there were rare earths present in the salt at substantial levels (~0.7 wt% of the initial salt). Rare earth uptake in the zeolite was high (5.9 wt%). Rare earths would be expected to compete with plutonium to a greater extent than cesium.

Plutonium oxides were not detected in the zeolite powders by X-ray diffraction, which indicates the following: (1) the plutonium is contained within the zeolite structure, and (2) a second crystalline phase that is independent of the zeolite was not formed by reaction with zeolite or during washing.

3. Pilot Plant-Scale Ion-Exchange Column System. The ion-exchange column test system is a small pilot plant-scale facility. Zeolite pellets are placed in a stainless steel tube, forming the "column." Columns up to 12 in. long and 1 in. in diameter have been tested. The system contains two salt tanks, the column feed, and the effluent receiver; they are housed in a large oven located in a glovebox well. When in position, the steel tube containing the zeolite is immersed in the salt with the bottom end of the zeolite column lying just above the salt surface. The entire system is maintained above 400°C. Argon gas is slowly added to the feed tank, forcing the salt up through the column to the receiver tank as the pressure increases. Salt samples are collected as the salt is transferred. The column zeolite is examined after testing.

Initial tests with the column indicate that it effectively sorbs all of the fission products present in the salt, particularly the rare earths. Flow rates are maintained below 2 cm/min. To date, the best performance has been obtained for a flow rate of 1 cm/min and a temperature of

815 K. Under these conditions, 1 g of dehydrated zeolite was found to remove over 90% of the fission products from ~5 g of waste salt. Figure 5 shows the change in concentration in the column effluent (C/Co) with the amount of salt that has passed through the column per gram of zeolite in the column. The salt-loaded column zeolite contained 12.5 wt% fission products on average.

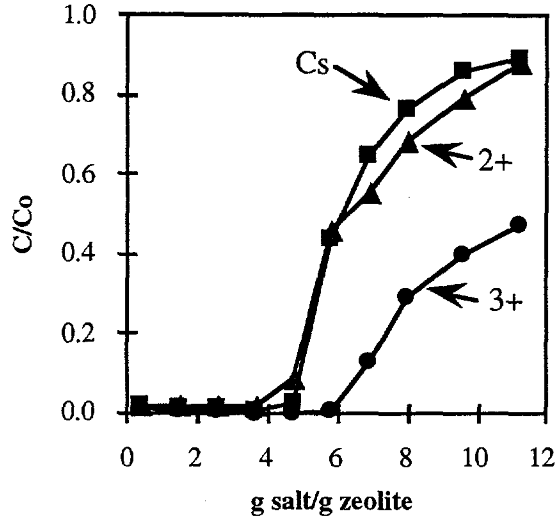

Figure 5: Ion-Exchange Column Test Results.

A mass transfer-based computer model incorporating the ion-exchange kinetics is being developed. This model will be refined as additional data from column experiments is obtained. The model will eventually be used to predict performance of large columns particularly under off-normal conditions.

Improvements in column performance will be made as the operating conditions are optimized. The system temperature and salt flow rate both strongly affect the performance of the column system. However, the column has only been operated at 775 and 815 K, near the extreme process temperatures, and intermediate temperatures must be tested. Lower salt flow rates, if attainable, will improve ion exchange between the salt and the zeolite.

Physical changes to the system should also affect performance. Only one form of pelletized zeolite has been tested to date; tests may be conducted with zeolites that are not type A or that have other binder compositions. Changing the size or the shape of the pellets may improve ion exchange in the column. Pellets with other binder compositions will also be tested; use of a zeolite other than type A is also possible. More salt would be treated if the aspect ratio of the small test columns were increased. Then more zeolite would be contacted by the waste salt before it exits the column. The fission-product loading in the zeolite at the head of the column should increase as more salt is passed through the column during a test. Based on batch ion-exchange results, it may be possible to increase fission product loading in the column zeolite to as high as 15 to 20 wt%, increasing the amount of salt that is treated to between 6 and 8 per grams of zeolite.

C. Mineral Waste Form Production

1. Incorporation of Salt into Zeolite A. Free salt on the surfaces of pellets or powders was incorporated into the zeolite structure in a hot blending operation by tumbling salt-covered zeolite with added anhydrous zeolite in an axially-rotating double-cone blender. Baffles within the blender lifted the salt to induce vertical mixing, while the steeply angled cones promoted mixing along the horizontal axis of rotation. The blender, which had a capacity of about 500 g, was heated by an external furnace to 725, 775, or 825 K to evaluate temperature effects. The blended product was washed with deionized water; the chloride content of the wash water measured to evaluate blending effectiveness.

The fission-product salt must be fully occluded in the zeolite by hot blending. Figure 6 shows the change in free chloride as the total chloride level is changed based on a water wash. Free chloride can be reduced to below 0.1 wt% of the final product (0.5% of total chloride). Although zeolite has been found to occlude 12 chloride ions per unit cell in batch contacting and in column operation, it appears wise to limit chloride in the blending process the chloride level must be less than 10 ions per unit cell to avoid excessive free salt.[3]

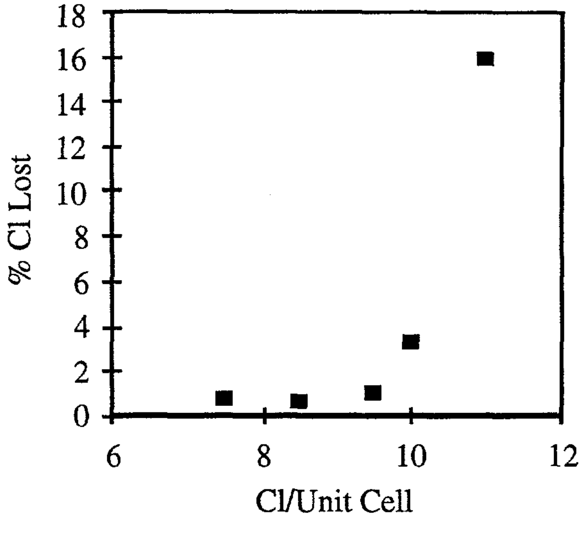

Figure 6: Free Chloride Lost with Washing.

2. Consolidation of Powder Materials. Glass frit is added to the blended zeolite to serve as a binder for consolidating of the fission product-loaded zeolite powders. Several compositions of commercial borosilicate glass have been studied in order to optimize leach performance of the hot-pressed product. Though the study

yielded no clear relationships between glass properties and the effectiveness of the glass-bonded zeolite, the composition of the glass was important. The glasses yielding the most leach-resistant products contained between 54 and 61 wt% silica. Glasses with high contents of alkaline earth oxides performed better than those with high contents of alkali oxides.

Hot uniaxial pressing was done under vacuum. The powder samples were loaded into a graphite die with a 1-in. bore. Once in place, the sample was cold pressed up to 40 MPa. Hot pressing was then conducted at pressures between 20 and 100 MPa and temperatures up to 1025 K. The sample formed was typically a 1-in.-diameter 0.15-in.-thick disk. These samples were polished and sectioned for leach testing.

Sample powders for the HIP are prepared similarly. The powders are cold pressed into 1-in. diameter stainless steel cans. The cans are weld sealed and evacuated. Samples have been prepared at pressures up to 165 MPa and temperatures to 1075 K. Samples typically undergo a 25% reduction in diameter after hot pressing.

Both uniaxial hot pressing and hot isostatic pressing have been used to consolidate the powders. Theoretical pellet densities between 90 and 100% are routinely achieved. Fission products were not volatilized during the hot-pressing. Table 2 shows the calculated composition of the starting powder mixture and the measured composition of the final pelletized product.

Table 2: Retention of Fission Products after Hot Pressing

Pellet	Ba	Cs	K	Li	Sr	Y
1 (calc)	0.38	0.93	3.81	1.14	2.67	0.07
1 (meas)	0.38	0.92	3.86	1.15	2.67	0.07
2 (calc)	0.48	0.84	3.38	0.91	3.85	0.08
2 (meas.)	0.48	0.83	3.34	0.87	3.90	0.08

Scanning electron micrography shows a two-phase system in which 3- to 4-µm zeolite crystals are embedded in a glass phase. Energy dispersive spectrometry indicates that the chloride is associated with the zeolite phase; little or no chloride was detected in the glass phase.

D. Leach Testing of Glass-Bonded Zeolite.

A modified MCC-1 leach test procedure was conducted on monolithic samples. Tests have been conducted with both deionized water and brine at 363 K for up to 56 days. The ratio of leachant to pellet mass was maintained at 100 mL/g. Teflon® vessels were used in all tests. The results are reported as normalized release rates (NRR), calculated as follows:

$$NRR = C_i V / f_i A d \qquad (3)$$

The NRR values are below 1 $g/m^2 \cdot day$ for 28-day tests, during which several glass-bonded zeolite samples were immersed in deionized water at 363 K. The NRRs are highest for the alkali cations, ranging from 0.3 to 0.8 $g/(m^2 \cdot day)$. The rare earths show very low release rates, <0.05 $g/m^2 \cdot day$. The alkaline earths show intermediate release rates.

Figure 7 shows the effect of leachant on the release rates. Glass-bonded zeolite was immersed in deionized water and in brine at 363 K. If ion exchange were the primary release mechanism for zeolite A, much higher release rates would be expected for tests with brine. However, there was little difference in the release rates between the tests with brine solutions and those with deionized water. The greatest change was observed for elements which were present in only the glass (calcium and boron) or in both the glass and zeolite (barium and strontium). The elements present solely in the zeolite (cesium and lithium) show a lower release rate in brine. These results indicate that ion exchange with the glass binder may be more significant than for the zeolite.

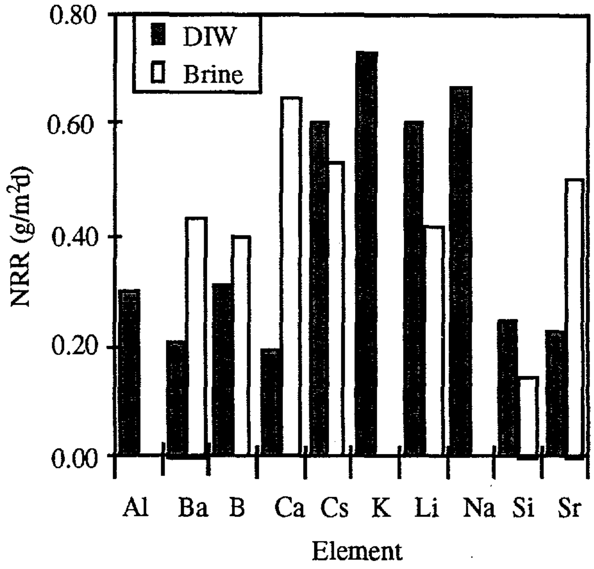

Figure 7: Effect of Leachant on Normalized Release Rates for Deionized Water (■) and Brine (□) in 28-d Tests at 363 K.

Figure 8 shows the decrease in release rates with time for 28- and 58-day tests with de-ionized water. Release rates for 56-day tests were approximately half those observed for 28-day tests, indicating a high initial release followed by a longer period with relatively low fission product release.

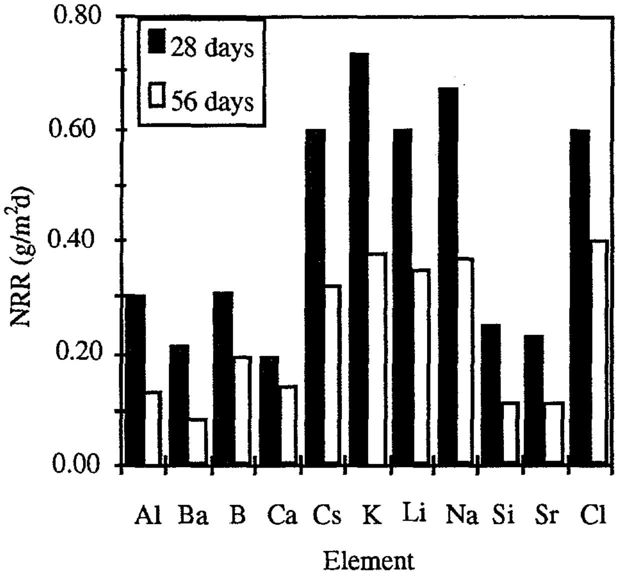

Figure 8: Change in Normalized Release Rates with Time for 28- (■) and 56-day (☐) Tests with Deionized Water at 363 K.

Table 3 shows the effect of the glass/zeolite ratio on leach resistance. Because high glass content reduces the fission-product loading and increases the waste form volume, it is desirable to minimize the amount of glass added. Leach resistance was not significantly affected when the amount of blended zeolite was increased from 50 to 67 wt%. The leach test results obtained to date show indicate that glass bonded zeolite is a promising waste form for waste streams that contain high levels of chloride.

Table 3: Average Normalized Release Rate For Pellets at Three Zeolite:Glass Ratios

	NRR (g/m^2day)		
Element	50:50*	60:40	67:33
Al	0.22	0.25	0.29
Ba	0.1	0.11	0.075
B	0.35	0.29	0.29
Ca	0.31	0.21	0.16
Cs	0.4	0.55	0.59
K	0.31	0.58	0.66
Li	0.39	0.62	0.63
Na	0.35	0.71	0.73
Si	0.22	0.26	0.25
Sr	0.2	0.17	0.13

* Zeolite/glass by weight.

IV. FUTURE WORK

Optimization of all aspects of the mineral waste form process is continuing. The ion -xchange column has been effective in removing fission products from the electrorefiner salt. Operating conditions including salt flow rate, temperature, and the fission-product distribution are being optimized. The pellet geometry, as well as the compositions of the initial zeolite and the binder, also significantly affects column performance and ultimately the properties of the waste form.

Hot blending to occlude salt in zeolite A has been shown effective. Nearly all of the salt is occluded in the zeolite at loadings as high as 10 chloride ions/unit cell. Blending at 775 or 825 K does not have a detrimental affect on zeolite structure; both are equally effective, thus providing a large operating window. Examining the effectiveness of a blending operation with plutonium-bearing salt is planned.

Hot pressing has proven effective in producing solid monolithic samples. Scale up is underway as processing has shifted from hot uniaxial pressing to hot isostatic pressing. The latter process allows multiple samples to be produced in a single run under identical conditions. Larger samples, up to 3.5-in. in diameter and 4-in. long can be produced in the installed facility. A hot uniaxial press specifically designed for manufacture of plutonium-bearing waste forms has been built and will be used to produce samples for leach testing.

The leach-resistant properties of glass-bonded zeolite will undergo further optimization. Variables to be determined include the effect of HIP pressure and temperature on the waste form properties. The effect of pressing conditions on the zeolite structure are currently being evaluated. The effect of fission product and chloride loading on the leach resistance of the zeolite is also under study. A study of the radiation effects on the waste form is planned. An optimal glass composition window must be determined.

The individual steps of the process have been shown to be viable. Significant improvements in performance should be achievable in most areas.

V. CONCLUSION

In the spent fuel treatment process, the fuel components that remain in the electrolyte must be contained in the mineral waste form. The processing of the electrorefiner salt can only be as effective as the final waste form. In addition to a varied mixture of fission products (rare earths, alkali and alkaline earth metals, halogens, and chalcogens), the waste form must contain a large quantity of chloride. Glass-bonded zeolite can serve as a mineral waste form since it has been shown to effectively contain the fission products that accumulate in electrorefiner salt.

The product cations are readily sorbed by the zeolite. TRU elements, if they are left in the electrolyte, are also sorbed by the zeolite. Free salt can be essentially fully incorporated and retained within the crystal structure of the blended zeolite. A monolithic leach-resistant ceramic composite is formed in which a glass serves as a matrix for the zeolite. Glass-bonded zeolite remains a viable waste form for radioactive fission products.

NOMENCLATURE

EF, EF(Na)	Exchange Factor (sodium basis)
$[M]_Z$	Concentration of species M in zeolite
$[M]_S$	Concentration of species M in salt
$[Na]_S$	Concentration of sodium in zeolite
$[Na]_Z$	Concentration of sodium in salt
n	Valence
C	Concentration in salt
C_0	Initial concentration in salt
NRR	Normalized Release Rate
C_i	Concentration of species i in leachate, g/mL
V	Volume, mL
f_i	Atom fraction of species i in solid
A	Geometric surface area of leach test samples, m^2
d	Length of leach test, days

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