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DEVELOPMENT OF POLYMERIC WASTE FORMS FOR THE ENCAPSULATION OF TOXIC WASTES USING AN EMULSION-ENCAPSULATION BASED PROCESS

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ABSTRACT

Developed technologies in vitrification, cement, and polymeric materials manufactured using flammable organic solvents have been used to encapsulate solid wastes, including low-level radioactive materials, but are impractical for high salt-content waste streams (Maio, 1998). In this work, we investigate an emulsification process for producing an aqueous-based polymeric waste form as a preliminary step towards fabricating hybrid organic/inorganic polyceramic matrices. The material developed incorporates epoxy resin and polystyrene-butadiene (PSB) latex to produce a waste form that is non-flammable, light weight, of relatively low cost, and that can be loaded to a relatively high weight content of waste materials. Sodium nitrate was used as a model for the salt waste. Small-scale samples were manufactured and analyzed using leach tests designed to measure the diffusion coefficient and leachability index for the fastest diffusing species in the waste form, the salt ions. The microstructure and composition of the samples were probed using SEM/EDS techniques. The results show that some portion of the salt migrates towards the exterior surfaces of the waste forms during the curing process. A portion of the salt in the interior of the sample is contained in polymer corpuscles or sacs. These sacs are embedded in a polymer matrix phase that contains fine, well-dispersed salt crystals. The diffusion behavior observed in sections of the waste forms indicates that samples prepared using this emulsion process meet or exceed the leachability criteria suggested for low level radioactivity waste forms.

INTRODUCTION

Many industrial processes create wastes containing high concentrations of salts mixed with toxic metals such as cadmium, lead, and arsenic. These processes include semiconductor manufacturing, mining/mineral processing, agricultural desalination, glass manufacturing, and pulp and paper mills, among others. Water treatment utilities use ion removal processes such as packed-bed adsorption, membrane filtration and ion exchange to trap toxic metals and other contaminants in iron or aluminum hydroxide containing sludges or residuals that often contain large quantities of salt. Another source of salt waste includes the 200 million kilograms of chloride, sulfate, and nitrate salts, contaminated with toxic metals and low-level radioactive elements, which are distributed among the various Department of Energy sites around the nation (Maio et. al., 1998). Attempts to dispose

of this low-level radioactive waste generate an additional 5 million kilograms of salt wastes per year.

Disposal of high salt containing waste is problematic because the salt component is vulnerable to leaching and high loadings tend to destabilize most matrices currently used for waste encapsulation. Encapsulation is necessary because the contaminating toxic metals must be prevented from entering the environment in an uncontrolled manner in the unlikely event that the storage facility be compromised. Currently, various types of technologies exist to encapsulate waste. Vitrification requires firing the waste at high temperatures where some toxic components may volatilize. Encapsulation using polymer matrices or composite polymers usually relies on the use of volatile and flammable organic solvents (Conner, 1990). Salt loading in grout is limited because large amounts decrease durability and lead to poor leaching control (Krishnamoorthy et al., 1993, Maio et. al., 1998, Leist et al., 2000).

Organic-inorganic hybrid materials known as polyceramics (i.e., materials that are a mixture of polymer and ceramic components) possess good mechanical integrity, high chemical durability, low processing temperatures (below 100°C), and ease of fabrication, properties which qualify them as candidate materials for encapsulating and storing a wide range of salt wastes. Polyceramics using rubber (butadiene) as the organic component and silica as the ceramic component were first developed as waste forms for salt encapsulation through a joint collaboration between the Arizona Materials Laboratory of the University of Arizona and Pacific Northwest National Laboratory (Smith, Gary L, 1999). These materials greatly exceed the strength requirements specified by the Nuclear Regulatory Commission (NRC) Technical Position Paper for stabilized low-level radioactive waste forms (USNRC 1991). That paper also specifies that the leachability index (a measure of the difficulty of leaching toxic components out

Sample ID	Waste Loading	Water Content
	(wt% NaNO ₃)	(wt. ratio of Water/Polymer)
8L	8	0.5
15L	15	0.5
20L	20	0.5
21H	21	1.5
27H	27	1.5
40I	40	0.75

Table 1. Designation and composition of samples from this study. In the designation, the number refers to the wt% of salt loading in the cured sample and the letter refers to the amount of water in the uncured sample, L: Low, I: Intermediate, H: High.

of the waste form) exceed a value of six. However, a major obstacle to their widespread implementation is the use of highly volatile and flammable organic precursors and solvents in the process flowsheet. To circumvent this problem, work was begun to replace the hazardous organic precursors and solvents with water-borne equivalents to develop environmentally-benign aqueous based processing routes for fabricating durable waste forms (Liang et al., 2002). To date, efforts have focused on fabricating polymer waste forms by cross-linking aqueous emulsions of epoxy and polystyrene-butadiene precursors in the presence of salt waste.

The objectives of this project were to characterize the microstructure of these water-based, epoxy/polystyrene-butadiene polymer waste forms with the intent of identifying how salt is incorporated into their structure and to quantify the rate of leaching of the salt from the waste forms. Most regulatory restrictions address the leaching of toxic metals and other contaminants. However, the salt is likely to be the most mobile species in these waste forms. Thus, quantification of its behavior will represent an upper limit on the amount of leaching to be expected from other slower, less mobile waste form contaminants.

METHODS

To fabricate the samples, polystyrene-butadiene (PSB) latex (BASF, Styronal ND 656), epoxy resin (Buehler, Epo-Kwick Resin), and the surfactant sorbitan monooleate (Span-80) were mixed for 30 minutes to create an emulsion. To this, the cross-linking agent diethylenetriamine (DETA, 99%, Aldrich) and sodium nitrate salt (NaNO_3) were added. This mixture was stirred to uniformity, cast into glass containers and placed in an oven to dry and cure at 80°C until weight loss ceased (typically 3 days). Cured samples were cylindrical and typically weighed 15 g, had diameters of about 2 cm, and lengths of about 3.5 cm. All samples were prepared using equal parts, by weight, of the PSB latex and epoxy resin. A variety of waste forms were prepared having nominal salt loadings in the range of 8-40 wt% of NaNO_3 in the cured samples. The amount of water in the uncured waste forms was also varied in the range of 0.5 to 1.5 wt ratio of water to polymer component (epoxy plus PSB). Table 1 lists the samples and their compositions.

Sample microstructures were characterized using carbon-coated sections using a JEOL 5900 LV SEM with a built-in Everhardt-Thronley (ET) secondary electron (SE) detector and a Robinson series VI scintillation-based backscattered electron (BSE) detector. Compositional data was obtained using an EDAX EDS detector with Genesis v1.0 software with integrated digital imaging and mapping capabilities.

To characterize the leaching behavior of the waste forms, thin sections were extracted from the middle of samples and immersed in a known volume of well-stirred water at 20°C to leach out the salt. The thickness of the thin sections was kept intentionally thin (2.25 to 5.30 mm) to minimize the

time required to characterize the behavior of species having low diffusivities. Measured variation in the conductivity of the liquid was used to determine the amount of salt leached into the solution as a function of time. To determine the total initial salt content samples were leached until variations in the conductivity of the leachate were no longer detected.

The leaching data from the conductivity measurements is interpreted by assuming that the sample is an infinitely long and wide slab with a thickness of $2L$. It is assumed that the sample contains a crust on the exterior of the thin section that dissolves very rapidly in the first moments of leaching. Because the solution is well-stirred, there is a negligible boundary layer in the solution near the surface of the sample, therefore the mass transfer from the sample surface to the liquid is instantaneous, and the salt concentration of the liquid is so small in comparison to that in the sample that it is assumed to be zero. The concentration – time data from the conductivity measurements is interpreted using the infinite slab solution to Fick's 2nd law, adapted to give the fraction of salt still present in the sample after the leaching time t :

(1)

where \bar{c} is the average salt concentration in the sample, c_0 is the initial concentration in the sample, D is the diffusion coefficient, and t is leaching time. The percent of salt retained in the sample, S , does not include the initial fraction of salt in the crust, characterized by the percent P_c :

$$S = \frac{\bar{c}}{c_0} (100 - P_c) \quad (2)$$

The diffusivity and P_c can be fit to experimentally-determined curves of S vs. t . The diffusivity is then used to calculate the leachability index, L_i , defined as:

$$L_i = \log \left(\frac{\beta}{D} \right) \quad (3)$$

where Equation (3) is an appropriate expression for the leachability index as defined by the American National Standards Institute (ANSI/ANS-16.1-1986) assuming that the diffusion coefficient is independent of time during the course of leaching.

RESULTS

Each of the samples listed in Table 1 appear to be homogeneous on a macroscopic scale when visually inspected. Despite this visual homogeneity, SEM analysis revealed that these samples are inhomogeneous at the microscopic scale. Additionally, each sample contains a crust of salt on the external surfaces of the monolith. The characteristics of this crustal layer are shown in Figure 1. In backscattered SEM micrographs, elements with a high atomic number give a stronger signal than elements with low atomic numbers because they contain more electrons. Thus the higher atomic numbered elements show up brighter, or whiter. In this study, the dark regions are polymer and the light regions are salt. As seen in Figure 1, the surface crust may contain a combi-

nation of dendritic crystals which have grown on the exterior of the monolith, large salt crystals in voids or bubbles just below the surface, a continuous layer or coating of salt on the exterior surface, and/or large salt regions just below the surface of the monolith.

To characterize further the microscopic homogeneity of the waste forms, the quantity of salt in various portions of a monolith of sample 40I was determined by sectioning and leaching. Prior to sectioning, the surface salt that was not incorporated into the sample was removed. Table 2 shows the average composition in wt% NaNO_3 for the different sections identified in Figure 2. The top, crustal section of the sample contains the highest concentration of salt at 31.3 wt%. The disk taken from the top but below the crustal disk contains 23.6 wt% salt. Two samples were taken from the middle of the monolith: one was a disk containing the peri-

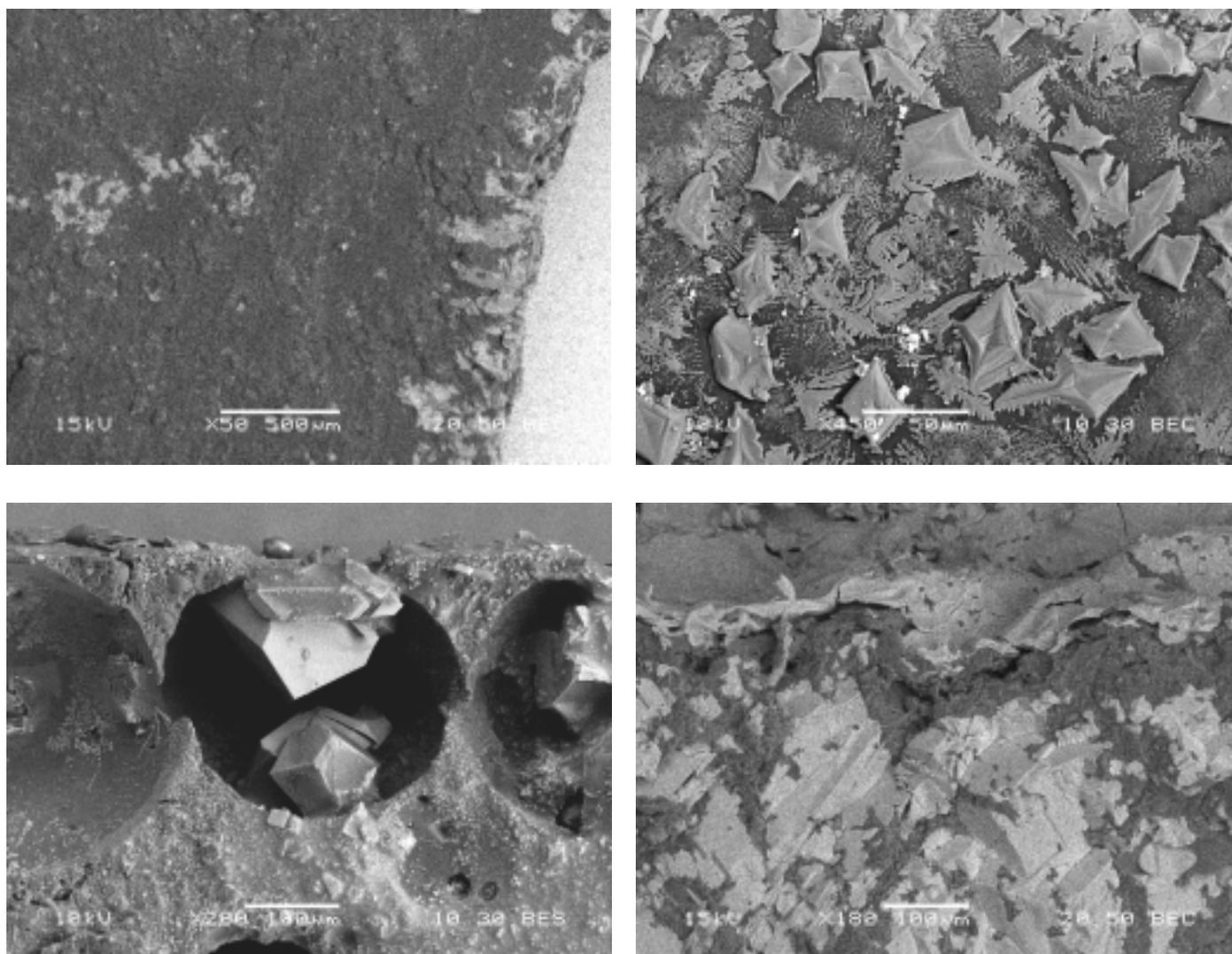


Figure 1. Backscattered SEM images of typical surface crust features: a) cross section containing the external surface of a monolith (vertically oriented at the right) shows a crustal region several hundreds of microns in thickness; b) dendritic crystals growing on external surface; c) large, well-formed salt crystals in near-surface voids; d) continuous layer of salt on external surface with large salt regions just below surface.

meter of the monolith (Section 3), while the other was just the core of the monolith (Section 4). The middle disk has a composition of 17.2 wt% salt while the core contains 9.4 wt% salt. These results confirm that the salt distribution in these waste forms is inhomogeneous, with higher salt concentrations near the external surfaces and lower concentrations in the middle of the waste form.

As seen in Figure 3, the microstructure of the interior or central portion of sample 15L consists of corpuscles or sacs of polymer that are mostly filled with salt. These sacs are embedded in a continuous matrix phase. EDS analysis over large areas (> 100 mm x 100 mm) reveals that the interior of this sample contains about 9.2 wt% salt. A similar result is obtained when the size of the scanned area is reduced so as to sample only the matrix phase between polymer sacs, indicating that the matrix phase contains small, finely dispersed salt crystals. Figure 4 shows the corpuscles or sacs observed in sample 8L. Here the salt crystals inside the sacs are relatively large and possess a well-defined morphology. The interior characteristics of other samples of this study contained similar features, namely corpuscles or sacs of salt embedded in a polymer matrix containing small, well-dispersed salt crystals.

Samples 20L and 27H were prepared in an attempt to increase the salt content in the interior of the waste form. Sample 20L contains sacs of salt similar to those observed in previous samples. However, it also contains a family of pores with sizes in excess of 100 mm (Figure 5). EDS analysis of the interior portions of this sample show that increasing the nominal loading of this waste form does not lead to a corresponding increase in salt content in the interior of the sample. The average composition in the interior of sample 20L was 9.2 wt% salt. Even increasing the target salt loading to 27 wt% did not significantly increase the concentration of salt measured in the center of the waste form monolith. EDS measurements at the center of sample 27H show a composition of 9.8 wt% NaNO₃. Notice that these values agree well with the value of 9.4 wt% measured in the interior or core region of sample 40I as determined by leaching (see Table 2).

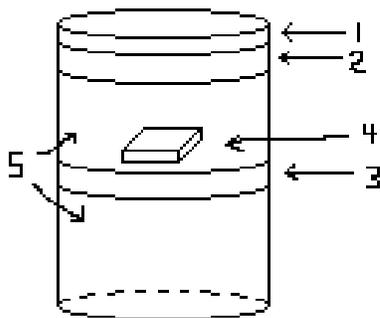


Figure 2. Schematic showing the sections of sample 40I leached to determine average salt content.

Section	Description	% Salt
1	Crust	31.3
2	Top disk	23.6
3	Middle disk	17.2
4	Middle core	9.4
5	Rest of monolith	18.8

Table 2. Salt content (wt% NaNO₃) of various sections of Sample 40I as identified in Figure 2.

The microstructural characteristics of fully leached samples are shown in Figure 6. In both samples 15L and 27H, the polymer corpuscles or sacs appear to be devoid of salt after leaching. EDS analysis indicates that almost all of the salt leaches out of the thin sections used for the leach tests. This indicates that the fine salt crystals distributed in the matrix phase surrounding the sacs leach out as well. After leaching, sample 15L contains approximately 1.3 wt% NaNO₃ and sample 27H contains about 0.7 wt%. Consequently, it seems that at long times, almost all of the salt leaches from these thin sections of the waste forms. Table 3 summarizes the composition analysis obtained by EDS for the interior portions of both the leached and unleached samples of this study.

Figure 7 shows the leaching data, plotted as S vs. t. The model fit is obtained by substituting equation (1) into equation (2), and then fitting D and P_c. Note that the experimental data starts at S=100% at t=0, whereas the model starts at S=100-P_c (i.e., the crustal salt is assumed to be removed instantaneously for modeling purposes).

Table 4 contains the leaching results for samples 15L, 21H and 27H. As seen in the table, the crustal salt of

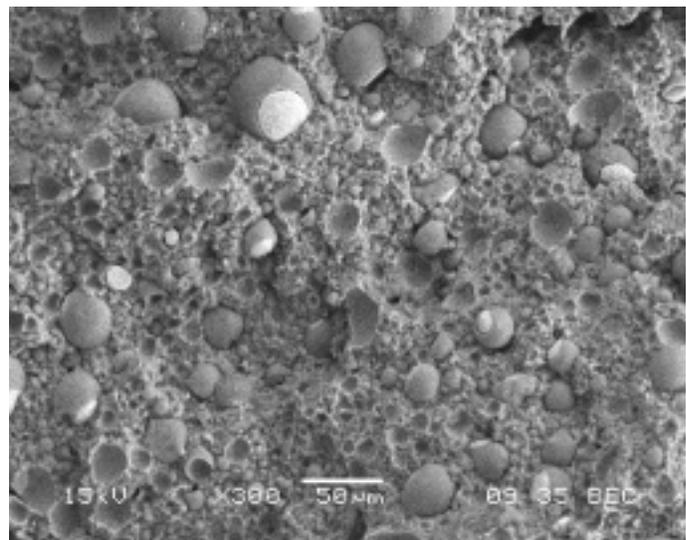


Figure 3. SEM backscattered image of the interior of sample 15L showing corpuscles or sacs of polymer containing salt imbedded in a continuous matrix.

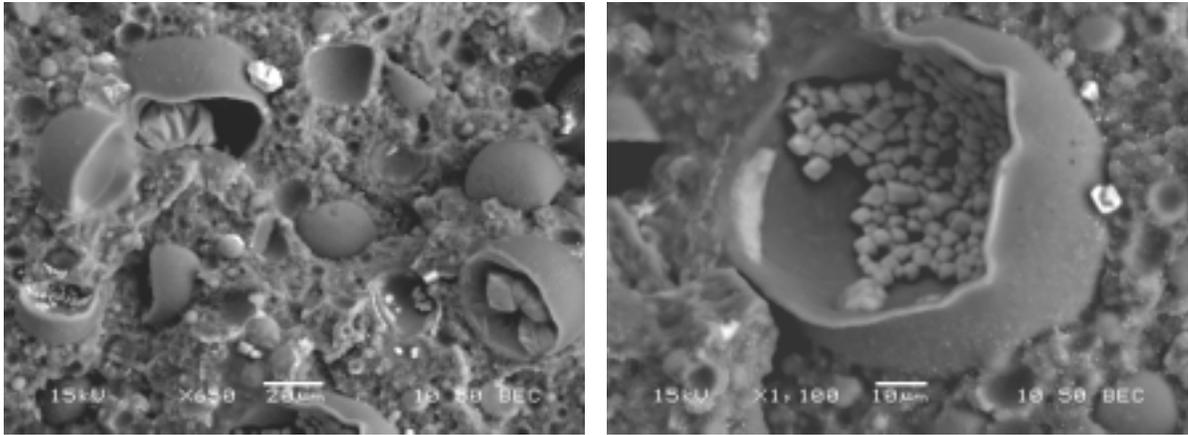


Figure 4. SEM backscattered images of the interior of sample 8L. Compared to 15L, the polymer sacs contain fewer, larger, and better defined salt crystals.

sample 15L made up 1.1% of the original weight of the sample. This quantity of salt leached out of the thin section very quickly and was calculated from the diffusion curve by extrapolation of the long time leach behavior to zero time. The quantity of salt leached from the interior of the sample in a manner consistent with the infinite slab model was calculated to be 9.9 % of the pre-leached sample weight. Taken together, 11 wt% of the sample was extracted during the leaching test. Modeling of the leaching data showed that the interior salt leached from the thin section of sample 15L with a diffusion coefficient of $2.5 \times 10^{-8} \text{ cm}^2/\text{s}$ to give a leachability index of 7.6 as per Equation (3). Analysis of the leaching data for samples 21H and 27H show that slightly more salt was extracted from the crust and interior regions of the sample during leaching than in sample 15L. Although the nominal salt loadings were increased, the amount extracted from the interior of the sample remains at approximately 10 – 12 wt%, a result consistent with EDS measurements of the salt composition at the very core of samples prior to leaching. Comparison of results from the three samples shows that the

diffusion coefficient increases and the leachability index decreases as the nominal salt loading in the waste form increases. However, the leachability index for all samples measured in this study exceeded the value of 6, the minimum value for acceptance as a low level waste form.

CONCLUSIONS

The key to making this material a viable waste form is believed to be the occurrence of a phase inversion during the curing process. Substantial evaporation must occur prior to curing so that the water phase becomes discontinuous and the polymer phase becomes continuous, thus encapsulating the water-soluble salt in the polymeric matrix. Since the water evaporates from the exterior surfaces of the waste form, it migrates from the interior and carries the highly soluble sodium nitrate with it. This migration produces the heterogeneous distribution of salt in the waste form as detected by EDS and measured in the different sections of sample 40I (see Table 2). Evaporation of the concentrated salt solution that migrates towards the surfaces of the waste form causes the formation of the dendritic crystals, cubic crystals, and large regions of embedded salt on or near the surfaces of the waste form as shown in Figure 1.

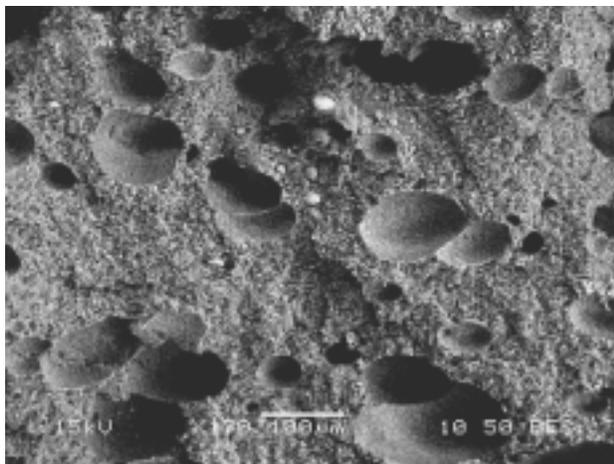


Figure 5. SEM backscattered image of the interior of sample 20L showing large pores.

	Composition (wt% NaNO ₃)	
Sample	Unleached	Leached
15L	9.2	1.3
20L	9.2	-
27H	9.8	0.7

Table 3. Composition of the interior portions of leached and unleached samples as determined by EDS.

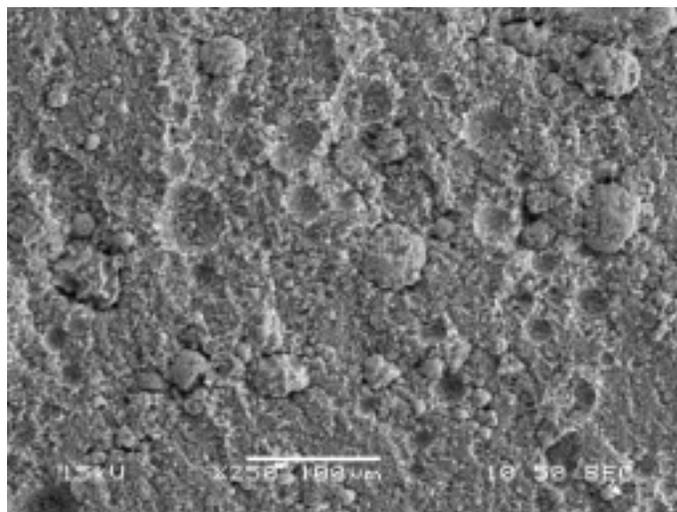
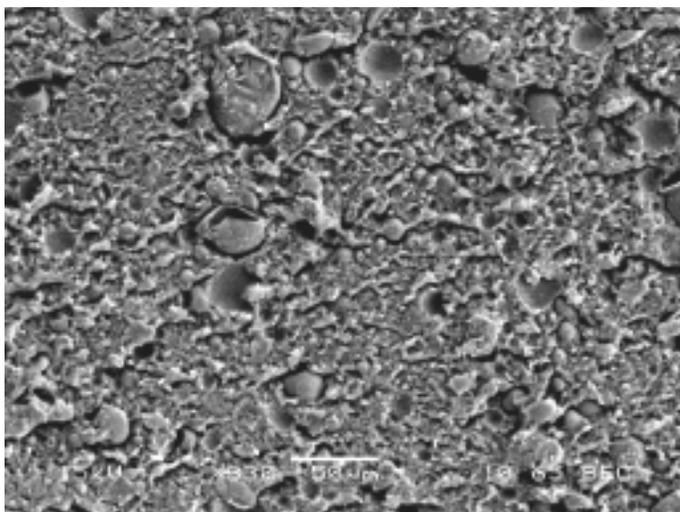


Figure 6. SEM backscattered images of leached samples. Polymer sacs appear devoid of salt and EDS indicates that the matrix phase contains little or no salt, as well: a) sample 15L, b) sample 27H.

While some salt migrates from the interior of the waste form to the surface, the EDS results from Table 3 and the leaching results from Table 4 confirm that at least 10 wt% of salt is encapsulated by the waste form. The micrographs of Figures 3-5 show that the salt is encapsulated as crystals in sacs or corpuscles of polymer embedded in a polymer matrix phase that also contains small salt crystals. These corpuscles may be generated during the mixing process, as the epoxy-PSB emulsion is forming. They become trapped in the polymer phase after the phase inversion occurs. The results of Figure 6 and Table 3 show that the salt is extracted from both the corpuscles and matrix during the leaching of thin sections for long times.

The leaching data of Table 4 confirm that polymer waste forms fabricated using this water-based emulsion flowsheet have good chemical durability. The leachability index measured for each of the samples was in the range of 7.6 to 6.1

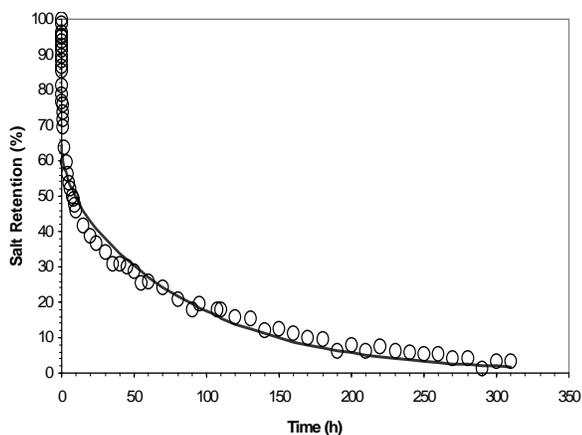


Figure 7. Leaching data for a section of a waste form. The solid line represents a fit of the data with the slab model. The initial fast drop in salt content corresponds to the rapid dissolution of crust salt present on the sample's external surface.

and exceeded the required value of 6. The corresponding diffusion coefficient was in the range of 2.5×10^{-8} to $75 \times 10^{-8} \text{ cm}^2/\text{s}$. This range is so low that measurements could only be accomplished by leaching very thin sections of the waste form for long times.

An ideal waste form encapsulates a large amount of waste and prevents the waste from leaching out of the matrix. Attempts to increase the waste loading in the interior of the samples were not successful using the procedures of the current flowsheet. As seen in Tables 2 and 4, samples 20L, 21H, and 27H contain between 10-12 wt% salt in their interior, despite their higher nominal salt loading. In sample 20L, it is likely that large pores form (see Figure 5) because the low liquid content of the sample produces an emulsion that is rather stiff prior to curing. This prevents the emulsion from shrinking as water evaporates during curing, leading to the formation of internal porosity. It is likely that the amount of salt encapsulated by the matrix is strongly influenced by the parameters that control the emulsification and curing processes. More work should be done to determine the influence of the cross-linking agent, surfactant, and curing temperature on waste form properties.

This work has shown that the water-based emulsion process creates waste forms that successfully encapsulate at least 10 wt% highly-soluble salt. When sections of the waste form are subjected to stringent leaching conditions, the measured leachability index for salt meets or exceeds criteria for low-level radioactive waste forms, suggesting that the less mobile toxic metals will meet or exceed the leachability criteria as well. Curing of the waste forms produces a higher concentration of salt in the near surface region than in the interior of the waste form. This analysis indicates that higher salt contents might be achieved if the migration of the solutions towards the sample surface during curing can be controlled.

Nominal wt % Salt	wt % Leached as Salt		Diffusivity (cm ² /s)	Leachability Index, L ₁
	Crust	Interior		
15	1.11	9.89	2.5 x 10 ⁻⁸	7.6
21	3.1	12.4	30 x 10 ⁻⁸	6.5
27	3.2	12.8	75 x 10 ⁻⁸	6.1

Table 4. Diffusivity and leachability index for NaNO₃ in the waste forms. The diffusivities were obtained by fitting the model developed in this work to experimental data. The leachability indices were calculated from equation (8).

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