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Student in the Department of Chemical and Environmental Engineering at the University of Arizona from which she also received her undergraduate degree in 2003. As an undergraduate at the U of A, she was involved in various research projects, including surface modifications to be used in separation techniques, as well as the encapsulation of waste in a polymer matrix. The latter project derived from her participation in the NSF/DOE Faculty and Student Team Program at Pacific Northwest National Laboratory. The research conducted resulted in a poster presentation at the AAAS National Conference in 2003, and a publication in the DOE Journal of Undergraduate Research, 2002.

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SEPARATION & FIXATION OF TOXIC COMPONENTS IN SALT BRINES USING A WATER-BASED PROCESS

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ABSTRACT

Efforts to implement new water quality standards, increase water reuse and reclamation, and minimize the cost of waste storage motivate the development of new processes for stabilizing wastewater residuals that minimize waste volume, water content and the long-term environmental risk from related by-products. This work explores the use of an aqueous-based emulsion process to create an epoxy/rubber matrix for separating and encapsulating waste components from salt laden, arsenic contaminated, amorphous iron hydrate sludges. Such sludges are generated from conventional water purification precipitation/adsorption processes, used to convert agueous brine streams to semi-solid waste streams, such as ion exchange/membrane separation, and from other precipitative heavy metal removal operations. In this study, epoxy and polystyrene butadiene (PSB) rubber emulsions are mixed together and then combined with a surrogate sludge. The surrogate sludge consists of amorphous iron hydrate with 1 part arsenic fixed to the surface of the hydrate per 10 parts iron mixed with sodium nitrate and chloride salts and water. The resulting emulsion is cured and dried at 80 °C to remove water. Microstructure characterization by electron microscopy confirms that the epoxy/PSB matrix surrounds and encapsulates the arsenic laden amorphous iron hydrate phase while allowing the salt to migrate to internal and external surfaces of the sample. Salt extraction studies indicate that the porous nature of the resulting matrix promotes the separation and removal of as much as 90% of the original salt content in only one hour. Long term leaching studies based on the use of the infinite slab diffusion model reveal no evidence of iron migration or, by inference, arsenic migration, and demonstrate that the diffusion coefficients of the unextracted salt yield leachability indices within regulations for non-hazardous landfill disposal. Because salt is the most mobile species, it is inferred that arsenic leaches from the host material at an even slower rate, making the waste forms amenable to unregulated land disposal options. These results indicate that the environmentally-benign, water-based emulsion processing of epoxy/PSB polymeric hosts show great promise as a separation and fixation technology for treating brine streams from wastewater treatment facilities.

INTRODUCTION

Processes to remove toxic heavy metals from drinking water and other aqueous streams are not new, but a number of recent trends have both greatly increased the frequency of their use and drawn critical attention to the nature of their by-products [1]. First, the Environmental Protection Agency (EPA) has recently passed or is formulating more strict standards for the allowable concentrations of many heavy metals in drinking water. The recently revised arsenic standard and the currently debated lead and copper rules are examples of this tightening of water quality standards [2,3]. Second, as the readily available sources of new potable water diminish, there is an increasing emphasis on reuse and reclamation of water to augment supplies. Finally, as the available space for solid waste disposal becomes scarcer and the potential negative impacts of such disposal are better realized, the scrutiny and cost of by-product disposal has greatly increased. As a consequence of these trends, there is a rapidly increasing volume of by-products requiring disposal at the same time as there is a need to minimize the volume, water content and long-term environmental risk of the by-products.

Since heavy metals are elemental, removal processes do not destroy them, but merely transfer them from the primary water stream to an alternative fluid or solid stream which constitutes the by-products (or as is often termed, residuals) of the process. Toxic heavy metal removal generates a metal-laden solid waste stream or, more commonly, a concentrated brine stream of the toxic inorganics mixed with various nontoxic salts. Large volume, troublesome examples include the ion exchange/membrane separation concentrate streams from

implementation of arsenic removal technologies by water treatment utilities and the aluminum and iron sludges emanating from precipitative, heavy metal removal operations utilized by semi-conductor, pulp and paper and metal plating industries. The emphasis of the research is on treating the sludges generated from conventional precipitation/adsorption technologies that are used to convert aqueous brine streams to semi-solid waste streams. Although precipitation/adsorption greatly reduces the waste volume, the sludges generated are still prone to toxics release unless disposed in specially constructed and handled hazardous waste sites. The only competing technologies to the proposed polyceram technology for stabilization of such wastes are grout/cement based, which are inapplicable to high salt wastes (as salt interferes with the calcination reactions necessary for cement curing) and cannot remove the added waste volume composed of benign salts [4,5]. To the knowledge of the authors, the combination of a stabilization technology for toxic waste and controlled release for nontoxic, highly soluble salts has not been successfully attempted before.

The work described here uses an arsenic-laden sludge as the residual of interest. Arsenic sludges were chosen because in 2001 drinking standards were enacted that reduced the allowable levels of arsenic from 50 ppb to 10 ppb [2]. These new standards will affect thousands of small utilities and cause production of an estimated six million pounds of solid residuals every year [2,3]. Among the options expected to be used for arsenic removal from drinking water-modified lime softening, modified coagulation/filtration and ion exchange will produce arsenic bearing brine streams, which in turn will be treated to generate a final, arsenic-laden residual of amorphous ferric hydroxide (AFH) sludge. These residuals typically will not

pass the Toxicity Characteristic Leaching Procedure (TCLP) specified by EPA to discriminate between hazardous and non-hazardous wastes and, consequently, must be disposed of in controlled, hazardous waste landfills [6]. Hazardous waste disposal costs are a factor of 5 to 10 higher than non-hazardous disposal costs and disposal of non-hazardous arsenic residuals is estimated to constitute 5-10% of total arsenic treatment costs. Thus, considering implementation of the new arsenic rule will cost an estimated \$180-206 million per year [2], it will cost water providers approximately \$9-19/lb more to dispose of a hazardous as opposed to non-hazardous treatment residual. In order to place the sludge in a non-hazardous landfill, toxic components have to be stabilized so they do not leach significantly into the environment. At the same time, reducing the total volume of residuals will minimize landfill costs; hence there is an incentive to separate benign components from wastes prior to disposal. Consequently, this work focuses on an overall program objective: to develop a separation/fixation technology for arsenic-laden AFH sludges that is simple, cost effective, safe, efficient, and durable. The development effort is based on application of polyceram waste form technology as a means of meeting the objectives.

The sludge used in this study was modeled after an AFH sludge that represents the solid residuals expected from an ion exchange (IX) operation used for removal of arsenic from drinking water. To treat water by IX, contaminated water containing arsenic passes through an IX column filled with an anion exchange resin containing free chloride ions. As the water passes through, the chloride ions in the resin are exchanged with the arsenic ions in the water and the treated arsenic-free water exits the system. After about one thousand bed volumes of contaminated water has been passed through the column, the resin is exhausted and must then be regenerated. At this point, four bed volumes of a regenerating solution containing 3% sodium chloride (NaCl) are passed through the column to return chloride ions to the resin and remove the arsenic ions. This process concentrates the contaminated water down to four bed volumes as opposed to one thousand bed volumes. The resulting brine, containing 2% NaCl and arsenic, then exits the column and enters a settling tank. Sodium hydroxide (NaOH) is added to the brine to precipitate ferrihydrite (Fe(OH)₃) to to which the arsenic adsorbs. Enough ferric chloride (FeCl₂) is added to bring the iron to arsenic mass ratio to 10 to reduce the arsenic concentration below the drinking water standard of 10 ppb. The supernatant liquid in the settling tank contains a toxic-free brine solution of 2% NaCl which is extracted and recycled. The sludge that is deposited on the bottom of the settling tank consists of solid Fe(OH), arsenic (both sorbed/coprecipitated with the AFH and as free species in the sludge associated water), NaCl, and water. It is this sludge that requires the fixation of the iron and arsenic components while simultaneously separating the salt in order to reduce the volume to minimize the cost of disposal.

Polycerams, a hybrid of organic and inorganic components, have been known to be mechanically stable, chemically durable, to have low processing temperatures (under 100° C),

and are easily fabricated, which make them feasible materials for fixating Fe(OH)₂. However, one disadvantage of polycerams involved the use of flammable and volatile solvents [7]. This disadvantage was mitigated by the application of an aqueous processing route in which an emulsion was created by mixing two polymers throughout a continuous water phase [8]. When cured, the water evaporates and the emulsion undergoes a phase inversion causing the polymer to become the continuous phase with the salt and solid residuals distributed throughout. It was found that soluble salts were successfully encapsulated in the polymer matrix using this water-based route [9], and the salt diffused at a slow rate yielding leachability indices well within the minimum requirements for land disposal. Microstructural characterization revealed that some portion of the dissolved salt was carried to the outer regions of the waste form as water evaporated during the curing process. This migration of salt can be further exploited as a separation and fixation technology that would separate the salt from the toxic components in a brine stream and fix those toxic components in a durable waste matrix.

MATERIALS AND METHODS

A representative sludge was created to mimic the end residual of an IX operation for arsenic removal. All the chemicals utilized to make the sludge were reagent grade or better. Sodium arsenate heptahydrate was first added to de-ionized water. Ferric chloride, was then added and completely dissolved in solution. While the solution was being mixed, NaOH was added to precipitate Fe(OH)₃. Once the mixture was stirred to homogeneity, the batch was allowed to settle for 24 hours, during which time the arsenate adsorbed to Fe(OH)₃. After the 24-hour period, free liquid was decanted from the settled sludge layer and more NaCl was added to bring the chloride concentration up to that typical of an AFH sludge from IX. Sodium nitrate (NaNO₃) was also added because it typically coexists in the brine stream with the arsenate. The pH was adjusted

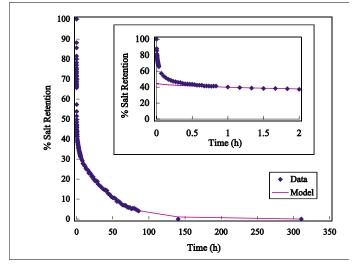


Figure 1: Example of experimental data fit to diffusion model [9] with an inset of the first 2 hours of leaching for sample 3.08 (Table I)

to neutral with NaOH, the batch was mixed, and then allowed to settle to facilitate liquid decanting when needed.

Sludge of varying water content was used in sample fabrication to cover the range of sludge produced by industries. Changing the liquid content in the wet sludge affects the salt content of the dry sludge. At low liquid content (~70% w/w), the sludge is mud like and, when dried, contains 33 wt% salt. At high liquid content (~95% w/w), the sludge is a fine particulate slurry and dries to contain 75 wt% (remainder is the AFH phase.) X-ray powder diffraction reveals that the AFH in the

Sample	Dry wt%	Dry wt%	Dry wt%	Dry wt%
ID	Fe(OH)	HAsO.	ŇaCl	NaNO.
3.08	12.5 ´°	0.8 4	35.7	2.3 °
3.12	16.3	1.0	22.3	1.5
3.13	12.5	8.0	35.9	2.3
3.17	10.0	0.6	4.6	0.3
3.18	10.0	0.6	4.6	0.3

Table I: Sample numbers and overall compositions based on dry weight.

sludge is structurally amorphous. The final sludge composition (dry basis by weight) is NaCl: 29.6%; NaNO₃: 1.9%; Fe(OH)₃: 64.5%; HAsO₄: 4.0 %.

To fabricate the waste form, polystyrene-butadiene (PSB) latex (BASF, Styronal ND 656) and the surfactant sorbitan monooleate (Span-80) were mixed for 10 minutes in a narrow, rounded-bottom mixing vessel to create an emulsion. While continually mixing, the sludge containing arsenic was added. Then epoxy resin (Buehler, Epo-Kwick Resin) was

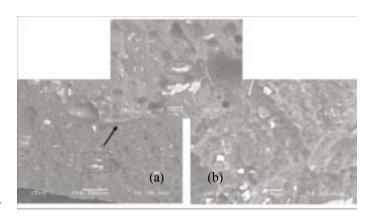


Figure 2: SEM micrographs of a sample with a low salt loading (sample 3.17, Appendix I) showing (a) a cross-section of cookie where the upper boundary of the image indicates the top of the cross-section and the lower boundary is the bottom; (b) the matrix of cookie at high magnification; the image directed by the arrow is a magnified image of the region shown in the rectangular box in figure (a). Dry salt content of sample is 4.9%.

added drop-wise with a syringe while stirring. Next, a cross-linking agent, diethylenetriamine (DETA), was added to the mixture. The emulsion was mixed for 5 additional minutes to ensure uniformity and then cast in shallow aluminum dishes and placed in an oven to dry and cure at 80° C until no further weight loss was detected (approximately 3 days). Cured samples were flat "cookie" shapes with diameters of about 4 cm and ranging from 0.15-0.4 cm in thickness. In some cases, cylindrical monoliths 2 cm in diameter and 3.5 cm long were

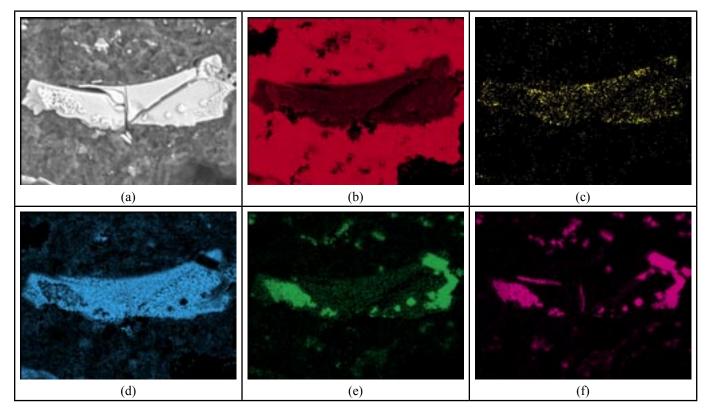


Figure 3: (a) SEM micrograph \Box chlorine.

cast. Samples with various iron loading, salt loading, and initial water content were made.

Scanning Electron Microscope (SEM) was used to analyze sample microstructure. Energy-Dispersive X-Ray Spectrometry (EDS) was used to obtain qualitative compositional analysis. Salt leaching and extraction were conducted to calculate the diffusivity of salt. Iron leaching was also performed. Arsenic leaching is assumed to be identical to the behavior of iron; however, leaching experiments will be conducted in the future to confirm this hypothesis.

To study the leaching behavior, portions of each sample were cut and suspended in a beaker of well-stirred water of known volume. An electrical conductivity probe was placed in the beaker to measure the changes in liquid conductivity as the salt leached out of the sample. Using known standards, the measurements were transformed to salt concentrations and a mathematical model based on Fick's law of diffusion was fit to the data. The model equations are presented elsewhere [9]. Assuming the samples leach to completion, the percent salt retention of the material is calculated using the following equation:

% Salt Retention =(Total salt in sample – Salt Concentration) / Total salt in sample \times 100%

Sample	Dry wt%	Dry wt%	% salt
Gampic	Fe(OH)。	salt in sample	extracted
3.12C	16.3	23.8	86.1
3.13A	13.9	31.4	91.3
3.13D	13.9	31.4	91.3
3.17A	10.0	4.9	59.2
3.17E	10.0	4.9	59.2

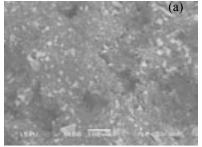
Table II: Amount of salt dissolved after one hour of initial salt

The model yields the effective diffusivity of the salt in the waste form, which is then used to calculate the leachability index for each sample [9]. Figure 1 shows an example of the model fit to actual experimental data. The model only starts correlating with the data after approximately 1 hour of initial extraction of salt from the outside of the sample. During the first part of the leaching experiment, solid salt close to the surface of the sample dissolves rapidly without participating in diffusion-limited transport. After this initial hour, all of the unincorporated salt has dissolved, and the remainder of the curve represents the diffusive mechanism of the leaching process.

RESULTS AND DISCUSSION

Table I lists the compositions of the waste form samples investigated in this study. Microstructural analysis by SEM reveals that AFH incorporation is similar in all waste forms but that salt features in the microstructure vary with the relative salt loading. For example, Figure 2a shows the microstructure of the waste form containing a low salt loading, Sample 3.17 (Table I). While Figure 2a shows only a portion of the

sample, it is representative of the entire cross section. Because the elements in the salt and AFH phase scatter electrons more strongly than the carbon and hydrogen atoms in the polymer, these phases appear light in the micrographs. The dark region is the polymer matrix. As seen in the figure, the AFH phase forms dense, elongated particles with aspect ratios around 1/5 and lengths along the largest dimension in the range of 20-150 um. The imaging done at a higher magnification and shown in Figure 2b shows that, at this low level of salt loading, the salt appears as cubic crystals about 5 µm in size embedded in the polymer matrix. EDS analysis was conducted on the region containing the ferrihydrite particle circled in red in the magnified image of Figure 2a and shown in more detail in Figure 3a. Figures 3b-e show the elemental maps that correspond to this region. The dark area surrounding the AFH particle is composed mainly of carbon as shown by the red in the carbon map (Figure 3b). Most of the light regions in the micrograph are the iron-containing phase, as shown by the light blue in Figure 3d. The arsenic map (Figure 3c) shows that arsenic is strongly associated with the AFH phase. Finally, the maps indicate that the groups of crystals seen on either end of the AFH particle are composed of both sodium (Figure 3e) and chlorine (Figure 3f).



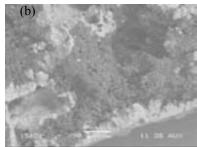


Figure 4: SEM micrographs showing microstructure of a sample with a high salt loading (sample 3.08, Table I) at (a) middle of monolith, and (b) bottom of monolith. Dry salt content of sample is 38%.

While the matrix phase does contain small salt crystals, EDS analyses of several samples indicate that the salt also concentrates as crystals in the cracks and interfacial regions of the AFH phase. The salt crystals seen on the ferrihydrite surface in Figure 3a originally lay inside cracks that ran parallel to the plane of EDS analysis. They were exposed for analysis when the sample was fractured for viewing.

The microstructural features of a waste form containing a high salt loading are shown in Figure 4. The image on the left illustrates sample 3.08 (Table I) at the middle of the monolith, which is representative of the majority of the sample. In contrast to sample 3.17, the polymer matrix of sample 3.08 contains large amounts of salt in the form of agglomerated regions distributed uniformly throughout the waste form and high concentrations of salt at the outer edges (Figure 4b). In particular, the bottom of sample 3.08 includes a well-defined salt crust approximately $100~\mu m$ thick. Additionally, the matrix near the bottom contains a number of pores around $400\mu m$ in diameter whose inner surfaces are coated with a thick layer of salt.

The high salt loading of Sample 3.08 causes it to be somewhat crumbly, which may limit its applicability as a viable waste form. However, the fact that the waste form retains a large amount of salt in regions that are accessible by a very open network of pores suggests that the underlying process has great potential as a technology for extracting or separating the salt to minimize the quantity of waste for disposal. A significant reduction in the quantity of waste would be achieved if this salt were separated or extracted from the waste form before storage.

To test the capabilities of the manufacturing processes for salt separation, salt extraction experiments were performed on a variety of samples containing varying amounts of AFH and salt. As seen in Table II, most of the salt (60 to 90%) in the sample was removed in the initial 1 hour of extraction. These results demonstrate that for a higher initial salt loading, a larger fraction of the salt is successfully removed.

To be an effective separation medium, the waste form must allow the rapid removal of salt while still retaining the arsenic containing AFH phase. Separate iron leach tests were performed, which indicated that no appreciable iron existed in the leachates after 300 hours of leaching. It is inferred from this knowledge that iron is therefore not extracted in 1 hour, and because arsenic adsorbs to the iron species in the waste form, it is also assumed that no arsenic is removed. This shows that the waste form can be used to separate the salts from toxic components from water treatment sludge. Upon contact with water, an important fraction of the salt will leach out of the waste form while keeping the AFH and associated arsenic encapsulated.

The salt extraction experiments measure the behavior of the salt that is easily removed by immersion in water. When the salt leaching experiments are continued beyond the initial 1 hour extraction time the rate of leaching of the salt that does not have ready access to internal or external surfaces of the waste form (e.g. the salt that is embedded in the polymer matrix) can be determined. The diffusion rate of the salt in this period should be representative in magnitude of the rate at which arsenic or iron will diffuse from the waste form. It is important to recall that driving forces for diffusion of iron or arsenic species are drastically lower than those of salt because of their low solubility (iron) and their strong adsorption to AFH (arsenic). Figure 5 shows the diffusion curve for the aforementioned sample 3.17 of comparably low salt loading. The diffusion model yields a diffusion coefficient of 3.50×10^{-8} cm²/s, which corresponds to a leachability index of 7.46. The minimum required leachability index for land disposal is 6.

Next, the model was fit to data for a higher salt intermediate waste form (Figure 6). The resulting diffusion coefficient for this sample was 6.00×10^{-8} cm²/s and a leachability index of 7.22. Table II showed that at various salt loadings, the majority of the salt can be separated using this waste form, while these leaching data show that the remaining salt (and, consequently, the arsenic species) can be encapsulated within land disposal regulations.

CONCLUSIONS

The important step in making a waste form for the encapsulation of toxic residuals occurs at the phase inversion during the curing process. The evaporation of water causes the material to transform from a continuous water phase to a continuous polymer phase. The evaporation of the water also results in carrying the salt to the outer surface of the polymer, while the arsenic laden AFH particles remain engulfed in the host material. Subsequent water immersion successfully extracts a high percentage of salt from the host material by dissolution and transport through interconnected porosity, which thereby promotes reductions in waste volume and ultimately reduced disposal costs. Most importantly, based on leaching studies of the unextracted salt, the diffusion coefficients of both a high and low salt loaded sample yielded leachability indices within regulations for non-hazardous landfill disposal. Because salt is the most mobile species, it is inferred that arsenic leaches from the host material at an even slower rate, making the waste forms amenable to unregulated land disposal options.

The environmentally-benign, water-based emulsion processing of epoxy/PSB polymeric hosts show great promise as a separation and fixation technology for treating brine streams from wastewater treatment facilities. Future work should address the explicit measurement of arsenic leaching rates, the increase in residual loading, and the waste form volume consolidation by post cure heat treatment of extracted host material. A sensitivity study determining the impact of processing variations should be performed before the waste form fabrication is scaled-up for marketing.

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