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Stabilization of arsenic-bearing solid residuals in polymeric matrices

J.K. Shaw^a, S. Fathordoobadi^a, B.J. Zelinski^b, W.P. Ela^a, A.E. Sáez^{a,*}

 ^a Department of Chemical and Environmental Engineering, University of Arizona, Tucson, AZ 85721, United States
^b Department of Materials Science and Engineering, University of Arizona, Tucson, AZ 85721, United States

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Abstract

This research investigates the use of polymeric matrices to encapsulate solid sorbents used to remove arsenic from drinking water. Arseniccontaining granular ferric oxy/hydroxide and ferric hydroxide amended alumina residuals were encapsulated in a polymeric matrix using a novel aqueous-based manufacturing process. The polymer was a blend of poly(styrene butadiene) and an epoxy resin. The polymeric waste forms produced were capable of containing more than 60 wt% of sorbent (dry basis), while keeping good mechanical properties. Arsenic leaching from encapsulated and unencapsulated residuals was evaluated using the standard toxicity characteristic leaching procedure (TCLP) and the California Waste Extraction Test (CA-WET). The results show that waste forms of the polymer-encapsulated residuals crushed for testing retain good leaching resistance when evaluated with the more aggressive CA-WET test, yielding leachate arsenic concentrations below the toxicity characteristic (TC) standard of 5 mg/L. When residuals were preprocessed and encapsulated in a polymer form that avoided the size reduction required by leaching protocols, arsenic leached up to 700 times less than that from the unencapsulated residuals. Comparison of the waste form developed here with conventional cement matrices containing the same residuals show that the polymeric matrices were capable of encapsulating appreciably more material and leached arsenic at concentration levels that were more than an order of magnitude lower than cement. © 2007 Elsevier B.V. All rights reserved.

Keywords: Encapsulation; Solid residuals; Arsenic stabilization

1. Introduction

The recently reduced USEPA maximum contaminant level (MCL) for arsenic in drinking water affects over 4000 US utilities. Many facilities are either implementing new or modifying existing arsenic removal technologies to meet the 10 μ g/L MCL [1]. Of the impacted utilities, 92% are facilities serving populations less than 3300. For these smaller facilities, the favored EPA-identified treatment option is adsorption onto solid media. The most common sorbents identified for treatment utilize an alumina or iron oxy/hydroxide surface such as: granular ferric hydroxide (GFH), Bayoxide E-33 (E-33), iron-amended activated alumina (AAFS) and iron-impregnated sand [2]. All of

E-mail address: esaez@engr.arizona.edu (A.E. Sáez).

these sorbents become arsenic-bearing solid residuals (ABSRs) that are considered not suitable for regeneration, and will have to be disposed. At an estimated generation of six million pounds of ABSR every year [3], disposal becomes a significant concern.

Arsenic in potable water supplies is typically present as arsenite, As(III), or as arsenate, As(V). In near-neutral pH water, arsenite is primarily present fully protonated and uncharged as arsenous acid, whereas arsenate is predominately in an anionic form as $H_2AsO_4^-$ and $HAsO_4^{2-}$. When water contains a significant amount of arsenite, USEPA recommends pre-oxidation prior to the implementation of an arsenic removal technology [1].

The potential for arsenic remobilization from ABSR is assessed by the toxicity characteristic leaching procedure (TCLP), which is the current USEPA protocol for determining whether the waste is hazardous [4]. The waste is determined to be non-hazardous if its leached concentration is below the toxic-

^{*} Corresponding author. Tel.: +1 520 621 5369.

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ity characteristic (TC) regulatory limit (5 mg/L for arsenic). The residuals from most arsenic removal technologies currently pass the TCLP and are eligible to be disposed of in non-hazardous landfills. However, it has become well-known that the TCLP poorly predicts the leaching of oxyanions, especially arsenic species [3,5-7]. Because the TCLP has a tendency to underestimate landfill leaching under certain circumstances, a more aggressive leaching test, the California Waste Extraction Test (CA-WET) [8], is often used as an alternative, although it is known that this test may also underestimate arsenic leaching [7]. Faced with the lack of an appropriate leaching assessment protocol, utilities must either (1) accept the possibility of later being required to "clean-up" landfills in which they have disposed ABSR, (2) send their ABSR to much more expensive hazardous waste landfills, or (3) stabilize the ABSR to reduce their leaching potential to an acceptable level. The increased expense, as well as the potential negative environmental impact of disposal, motivates the study of stabilization processes for ABSR.

The effectiveness of a stabilization technology is judged in terms of mechanical strength and leach resistance of the final waste form. Mechanical strength is important due to the relatively high loads that the waste forms must endure during placement and permanent storage in a landfill. An unconfined compressive strength (UCS) of 0.35 MPa is considered satisfactory by EPA for disposal of stabilized materials in municipal landfills [9]. The UCS is the maximum applied stress that a sample experiences during compression until failure in an unconfined environment. In general, leach resistance is evaluated using the TCLP and CA-WET tests, but consideration must be given to the different conditions prevailing in non-hazardous waste landfills. Specifically, the pH of mature landfills is appreciably higher (pH 7–9, compared to the tests' pH of around 5). In addition, mature landfills possess anaerobic microbial activity and reducing conditions, which are not reflected by the standard tests. Both higher pHs and reducing environments are conducive to increased arsenic leaching [3,7,10].

Two widely used techniques for the stabilization of solid wastes are cementation and vitrification. The latter does not seem suitable for encapsulation of ABSR due to its relatively high cost and the use of high temperatures. Cement and lime encapsulation has been used successfully in a wide variety of applications, including stabilization of industrial waste containing relatively high amounts of arsenic [11]. Recently, Singh and Pant [12] studied encapsulation of an activated alumina sorbent loaded with arsenic in Portland cement, and mixtures of Portland cement with fly ash, calcium hydroxide, polystyrene and poly (methyl methacrylate). The leachability of arsenic from the encapsulated waste forms was evaluated using the TCLP test and a dynamic leaching test in distilled water. Leachability indices were considered adequate for all the samples generated, but by far the most stable matrix was a mixture of Portland cement, fly ash and calcium hydroxide. Singh and Pant determined that the stability of this waste form was in part due to the precipitation of calcium arsenite, which has low solubility at the leaching conditions employed. However, arsenic leaching under conditions typical of non-hazardous waste landfills was not assessed.

This work investigates the stabilization of ABSR (GFH, E-33 and AAFS) by means of an aqueous-based emulsion process that forms an epoxy/rubber matrix to encapsulate the waste. The process was developed in a previous work as a technique to stabilize soluble salts [13,14]. Preliminary results of its application to amorphous ferric hydroxide sludges containing sorbed arsenic were promising. Benefits of this method of stabilization include: mechanical and chemical durability, low processing temperature, and diminished flammability risk in the manufacturing process by use of an aqueous processing route [13].

2. Materials and methods

2.1. Sorbents

The sorbents GFH, E-33 and AAFS were obtained from the City of Mesa, Arizona, arsenic treatment pilot testing site. The GFH and E-33 had previously been loaded to 2.52 mg As/g GFH and 4.91 mg As/g E-33, respectively, by equilibrating them with aqueous solutions containing an arsenic concentration of 12 µg/L (arsenate). The AAFS was unused, as delivered from the manufacturer. To obtain an ABSR more challenging and representative of drinking water treatment, the arsenic loading of the three media was increased using a batch equilibration process with 1-1.5 L purified water (Milli-Q) containing 12-20 g sodium arsenate heptahydrate (Na₂HAsO₄·7H₂O, Aldrich). The mixtures were equilibrated at pH 7. After equilibration, the supernatant water was removed. The loaded sorbents were placed in glass jars and capped. The loading process was performed at room temperature (25 °C). The resulting water content, arsenic loading and equilibrium aqueous arsenic concentration are presented in Table 1. The total arsenic loading was determined through (inductively coupled plasma-optical emission spectrometry) ICP-OES analysis of microwave-digested sorbent samples, and is reported with respect to dry weight of sorbent. The equilibrium aqueous arsenic concentration is the resulting concentration of arsenic in water after the sorbent/water mixture was equilibrated at pH 7.

Granular ferric hydroxide (GFH) is a weakly crystalline β -FeOOH produced by conditioning previously compacted iron hydroxide slurry into irregular grains of 0.32–2 mm in diameter [15]. It is made from akaganeite (Fe³⁺O(OH, Cl)) and goethite (FeO(OH)) and has a wet density of 1.26 g/cm³ [16]. To preserve its activity, the material must not be dried. The GFH particles specifically adsorb As(V). This sorbent is manufactured by GEH

Table 1

Water content, arsenic loading and aqueous equilibrium arsenic concentration for sorbents employed in this work

Sorbent	Water content (wt%)	As loading (mg As/g sorbent)	Nominal As equilibrium concentration (µg/L)
GFH	45.6	3.8	35
E-33	60.4	8.0	48
AAFS	46.5	2.7	21

and distributed in the United States by U.S. Filter. Bayoxide SORB 33 (E-33) is a synthetic iron oxide hydroxide containing about 90% α -FeOOH. It is manufactured by Bayer for Severn Trent with a dry density of 0.45 g/cm³ and particle sizes of 0.5–2 mm. The sorbent AAFS is amended aluminum oxide which contains 83% Al₂O₃ and a proprietary iron hydroxide addition. It has a dry density of 0.91 g/cm³ and particle size in the range 0.3–0.6 mm.

2.2. Polymeric waste forms

All waste forms were formulated to contain a 1:1 ratio by weight of polystyrene–butadiene (PSB) rubber to epoxy resin, a 1:10 ratio by weight of surfactant to epoxy resin and a 1:10 ratio by weight of cross-linker to epoxy resin. The waste loading was calculated as a mass fraction of dry sorbent to total dry components. This represents the waste loading of the polymer after complete water loss and curing.

To generate the waste forms, a PSB latex (Styronal ND 656, BASF) and the surfactant sorbitan monooleate (Span-80, Aldrich) were mixed at 500 rpm, using an overhead mixer provided with a one in three point propeller, for 5 min in a 100 mL beaker to create an emulsion. Epoxy resin was then added dropwise using a syringe, while the mixing speed was simultaneously increased to 2000 rpm, and then mixed for an additional 10 min. The cross-linking agent, diethylenetriamine (DETA, Aldrich), was then added and allowed to mix for five more minutes. Lastly, the ABSR was added slowly and allowed to mix for 10 min. The resulting mixture was cast into 2.5 cm diameter glass vials to a height of approximately 3.5 cm to form cylindrical monoliths. The vials were placed in an 80 °C oven to dry and let the polymerization process proceed (curing) until no further weight loss was detected (3–7 days) [13].

As an alternative to the cylindrical monoliths, rod-shaped waste forms (higher length-to-diameter ratio) were cast. This was done by two methods: casting or extrusion. Casting involved packing the uncured suspension into a 6 mm diameter glass pipette by applying vacuum to one end. Extrusion consisted in passing the polymer/waste mixture through a syringe with a 6 mm diameter opening. For both methods the final rod waste forms were approximately 5 mm in diameter and 3-5 cm in length. Post-processing of the rod waste forms was considered and some of the samples were modified by dipping the cured rod into a blank polymer emulsion, allowing the excess emulsion to drip off and curing the sample for an additional 24 h. An increase of less than 1 mm in diameter resulted from the dipping process. The reason for the production of dipped samples was to apply an extra coating of polymer to the outer surface and analyze its effect on arsenic leachability.

Processing of the waste prior to encapsulation was also considered. This included drying the ABSR in an 80 $^{\circ}$ C oven and grinding the dry sorbent to pass a 200-mesh sieve. The grinding was done by pulsing the dry sorbent in a coffee grinder. The procedure to encapsulate this dry and ground sorbent sample was similar to that explained above, except that ultrapure water was added as needed to achieve uniform mixing of the sorbent with the polymer emulsion.

2.3. Cement waste forms

For comparison purposes, cement waste forms were prepared using a 1:3 weight ratio of Portland cement to sand, and a 0.42–0.8 weight ratio of water to dry ingredients, cement plus sand. Portland cement and sand were pre-mixed in glass vials 2.5 cm in diameter. The wet ABSR and water were then added to the glass vial and mixed manually with a stir rod. Various amounts of sorbent were added to the cement waste forms with loadings being reported as weight ratios of dry sorbent to total dry components. The vials were capped and allowed to cure for at least 30 days. After curing was complete, any excess water was removed by decanting.

2.4. Leaching tests

The TCLP and CA-WET tests were used to evaluate the comparative leaching potential of arsenic among the samples. Following leaching, the leachate from all samples was filtered through a 0.45 μ m glass fiber filter before analysis. Details of the leaching protocols are given below. It is important to stress that these tests are used in this work to compare performance of various stabilization options. As discussed before, the performance of the various waste forms under landfill conditions could be appreciably different. Unless otherwise noted, leaching tests were performed in triplicate for most of the original sorbents and encapsulated sorbents, and standard deviations are reported.

2.4.1. TCLP

The extraction solution was prepared according to EPA SW-846, Method 1311 [4] by adding 5.7 mL of glacial acetic acid to 64.3 mL of 1.0N NaOH and bringing the mixture volume up to 1 L with deionized water. Prior to addition of waste, the pH of this stock leaching solution was 4.93 ± 0.05 . To run a leaching test, 5.129 g of size-reduced (<1.4 mm) waste form was added to a glass bottle with 100 mL of solution and sealed. The sample was then rotated end-over-end for 18 ± 2 h to complete the protocol.

2.4.2. CA-WET

According to CA-WET protocol classification [8], the ABSR of this project are classified as Type (ii) substances. As per the protocol, the waste form sample is size-reduced by crushing to <1.4 mm. The testing solution for the CA-WET test consists of 0.2 M sodium citrate at pH 5.0 ± 0.1 , prepared by titrating analytical grade citric acid in Milli-Q water with 4N NaOH solution. To run a test, 5.0 g of the crushed waste form was placed in a glass bottle with 50 mL extraction solution, purged with nitrogen gas and sealed. The sample was then rotated, end-over-end for 48 ± 1 h.

2.5. Compressive tests

Polymer waste forms made using the procedure described above were cut down to parallelepipeds measuring $1.3 \text{ cm} \times 1.3 \text{ cm} \times 2.54 \text{ cm}$. For the same purpose, cylindrical cement waste forms were made with 3 cm diameter and lengths varying between 2.5 and 3 cm. These samples were tested without cutting to preserve their mechanical integrity. Compressive strength and modulus of elasticity measurements were measured in an Instrom 1101 stress tester.

2.6. Analytical

Arsenic, iron and aluminum were measured using a Perkin-Elmer Optima 5300 DV inductively coupled plasma optical emission spectrometer (ICP-OES). The gas flow to the nebulizer used in the measurements was 0.60 L/min, with a sample flow rate of 1.5 mL/min. Arsenic measurements are taken at a wavelength of 193.7 nm. Scanning electron microscopy (SEM, JEOL 5900 LV) was used to analyze the microstructure of the waste forms. Microwave digestion (CEM Inc.) was performed on samples of the unencapsulated ABSR prior to ICP-OES analysis.

3. Results and discussion

An initial analysis of the leachability of arsenic from the unencapsulated sorbents was conducted using the TCLP and CA-WET tests. The results are shown in Fig. 1. Note that E-33 has the highest leachate concentrations among the sorbents, but a contributing reason for this is its higher loading (Table 1). The CA-WET test yields arsenic concentrations that are almost two orders of magnitude higher than the TCLP. As mentioned before, the TCLP test has been noted to underestimate the potential for arsenic leaching upon exposure to actual landfill leachate. Since the TCLP is not sufficiently aggressive to predict accurately arsenic leaching, emphasis in this discussion is placed on results from the CA-WET test, although this test may also somewhat underestimate leaching when compared to actual landfill conditions.

Cement waste forms were prepared to characterize the adequacy of cement for the stabilization of ABSR. Their mechanical stability was found to be dependent on the type of sorbent added to the waste form. Cement waste forms containing 25 wt% loadings of GFH and E-33 crumbled immediately upon removal from their vials. Waste forms loaded with 15–20 wt% of the same sorbents could be extracted from their vials without breaking. However, in some cases, they easily crumbled upon handling, suggesting that good mechanical integrity may be retained only



Fig. 1. TCLP and CA-WET leaching results for unencapsulated GFH, E-33 and AAFS. The line marked TC corresponds to 5 mg/L (arsenic toxicity characteristic).

Table 2			
Modulus of elasticity (measured at 5% strain) of cement and	polymer samples

Sample	Modulus of elasticity (MPa)	
Cement	54	
Cement + 20% solid	3–13	
Polymer	102	
Polymer + 10% solid	42	

The solid added to cement samples was GFH, while the solid added to the polymer sample was sodium chloride.

for waste loadings of GFH and E-33 somewhat lower than 15 wt%. Good mechanical integrity was retained for waste forms loaded with both 15 and 25 wt% AAFS. These samples retained their shape with very little fracturing during removal from their vials. Mechanical testing of cement samples (Table 2) shows large changes in the modulus of elasticity of cement samples upon loading with GFH. The 20% GFH cement samples tested often fractured at strains between 5 and 10%, and their moduli varied considerably from sample to sample.

Results of leaching tests for sorbents encapsulated in cement are shown in Table 3. The comparisons shown here correspond to the maximum sorbent loading that can be achieved without compromising the mechanical integrity of the sample (15-18%)in the case of cement). At this level of loading, the results show that encapsulation with cement is ineffective to reduce arsenic leaching. Arsenic concentrations for waste forms containing AAFS were largely unchanged by encapsulation with cement. However, the use of cement for encapsulating GFH and E-33 drastically increased measured arsenic levels, indicating that cement-based waste forms have a tendency to promote an environment prone to increased arsenic leaching. The presence of crushed cement in the leaching solution increases the pH to levels that accelerate degradation of the waste forms and significantly enhance arsenic mobility. Nearly all of the cement samples showed an increase of the CA-WET solution pH from 5.0 to about 11.8, regardless of the type of sorbent encapsulated. This likely contributes to the measurement of higher arsenic leaching levels from the cement waste forms than from the unencapsulated sorbents themselves, as was the case for samples containing GFH and E-33.

To have a more independent evaluation of the effect of sample microstructure on leaching, dynamic leaching tests were conducted on waste forms containing a soluble salt (sodium nitrate) as model waste, instead of the sorbents. The samples were placed in well-stirred water, and the concentration of salt in the liquid was measured as a function of time. Results of these tests performed on the polymer matrix employed in this work have been reported elsewhere [13]. The main objective of the test was to determine the effective diffusivity of the salt in the waste form. Results for polymer samples yielded effective diffusivities of the order of 10^{-8} cm²/s [13], while measurements in cement samples were around 10^{-5} cm²/s (results not shown). These results indicate that the cement samples have a highly porous structure, which represents an additional disadvantage in the use of this technique.

The polymer encapsulation process was capable of producing mechanically stable waste forms at high sorbent J.K. Shaw et al. / Journal of Hazardous Materials 152 (2008) 1115-1121

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Sample	Waste loading (wt%)	TCLP leachate As concentration (mg/L)	CA-WET leachate As concentration (mg/L)	
GFH	Unencapsulated	0.065 ± 0.02	2.94 ± 0.2	
E-33	Unencapsulated	0.075 ± 0.01	6.96 ± 0.8	
AAFS	Unencapsulated	0.037 ± 0.01	5.03 ± 0.5	
Cement + GFH	18	-	9.84	
Cement + E-33	18	_	38.7	
Cement + AAFS	15	-	6.03	
Polymer + GFH	60	0.013 ± 0.01	1.17 ± 0.07	
Polymer + E-33	60	0.075 ± 0.05	5.05 ± 0.34	
Polymer + AAFS	60	0.449 ± 0.20	0.77 ± 0.20	

TCLP and CA-WET tests results for unencapsulated, cement-encapsulated and polymer-encapsulated (monolith) GFH, E-33 and AAFS

Cement encapsulation results were performed only once for comparison purposes.

Table 3

loadings. Polymer monoliths had higher mechanical strength and higher moduli of elasticity than cement samples (e.g., Table 2). Loadings of up to 80% sorbent were achieved in some cases, without loss of mechanical integrity. For all comparison evaluations, polymer samples were made at 60% loading. The results of leaching tests of these samples are shown in Table 3. Note that, even with the substantial sorbent loading, the polymer-encapsulated waste forms perform appreciably better than the original sorbent (except for two TCLP results). From a practical standpoint, however, the benefits of polymer encapsulation at this level of ABSR loading and under the assessment given by standard tests, do not seem to be dramatically favorable, although improvements over cement encapsulation are evident. Lower loadings of ABSR led to appreciably lower As concentrations in the leachate (see Fig. 4 and discussion below).

Observable differences in sample evolution were noted during the processing of the polymer waste forms. When added to the polymer solution, E-33 tended to disperse well and stay suspended during the mixing and casting process. However, GFH and AAFS immediately settled to the bottom of the mixing vessel when mixing stopped, leaving a polymer rich liquid phase present in the top portion of the vessel. It is important to note that due to the settling of these two sorbents in the polymer mixture, the waste loading of the lower portion of the waste form is, in fact, much greater than the target loading of 60%, indicating the potential for even higher efficiencies through further increases in waste loading. When size reduction (by grinding) of the media was performed prior to encapsulation, the settling effect in the GFH and AAFS samples was eliminated and the samples were homogenous.

Analysis by SEM was performed on an unloaded polymer waste form and a GFH-loaded waste form with 60% loading. The SEM micrograph of a polymer sample (Fig. 2) reveals spheroids about 5–10 μ m in size. Previous analysis using osmium tetroxide staining indicates that these spheroids are polymerized epoxy resin droplets dispersed in a continuous rubber matrix [13]. Fig. 3 shows the micrograph of a polished slice of the GFH-loaded polymer waste form taken from the center of the monolith. The sorbent particles are packed closely together with the polymer material occupying the interstitial space and forming films over the particles. In such a highly loaded waste form, the necessary crushing for size reduction required by the standard tests exposes sorbent directly to the leaching fluid



Fig. 2. SEM image of unloaded polymer waste form matrix.

(see below), which leads to arsenic losses that are higher than expected.

Arsenic leaching from the polymeric waste forms is greatly dependent on sample size. Both TCLP and CA-WET tests require size reduction of the encapsulated samples prior to leaching. The extent of size reduction performed on the sample changed the leaching values significantly. During the size



Fig. 3. SEM image of polished center slice of waste form with 60% loading of GFH.



Fig. 4. CA-WET leaching results for polymer encapsulated E-33, 5–100% waste loading, crushed to different particle size ranges. Results for each loading correspond to a single sample.

reduction process, samples fracture and expose the sorbent to the leaching solution. This is illustrated in Fig. 4, where E-33 monolith samples were crushed to two different particle size ranges. The categories included samples crushed to pass a 1.4 mm sieve, and samples crushed to pass between at 2.36 mm sieve and a 1.4 mm sieve. Samples crushed to less than 1.4 mm resulted in CA-WET arsenic leaching two to three times greater than that of samples crushed between 1.4 and 2.36 mm. This indicates that results of leaching tests performed on monolith samples that were size-reduced may not represent accurately the effectiveness of the encapsulation method. This motivated casting samples directly in the form of thin rods that, once cured, met the size requirement of the leaching protocols. However, the rod samples failed to display the full leaching reduction potential of polymer encapsulation because of a flawed process in their generation: it was found that during the manufacture of the rods, fractures and cracks were introduced. This is partly due to the high loadings employed, which make it difficult not to have the sorbent exposed at the surface of the waste form.

To compensate for the exposed sorbent surface in the manufacture of the rods, cured rods were dipped into a blank polymer emulsion to create a thin layer of polymer blend coating the outside of the waste form. This polymer coating did not significantly increase the diameter of the rod (coating films were less than 1 mm thick) and eliminated the exposure of media through cracks or fractures that were previously introduced. These samples were cured for an additional 24 h. Results of CA-WET



Fig. 5. CA-WET leaching results for 60% loaded GFH, E-33 and AAFS polymer samples in the form of monoliths, rods and dipped rods.

tests performed on dipped rods are shown in Fig. 5. The dipping process reduced arsenic leaching by more than an order of magnitude in all cases.

4. Concluding remarks

The proposed polymer encapsulation process is a viable option for the reduction of arsenic leaching from solid residuals. The waste forms are capable of containing high loadings (60% of more) of residuals with good leaching characteristics and excellent mechanical properties. Maximum benefits with regards to arsenic stabilization are obtained when the waste form is extruded and coated by a thin polymer film, in which case reduction of more than two orders of magnitude in leachate arsenic concentrations is obtained, as determined by the CA-WET test. What remains to be established is the durability and performance of the polymeric waste forms under landfill conditions. Current research is being performed in which polymeric waste forms (the dipped rods described here) containing ABSR are tested inside a continuous-flow system that simulates landfill environments, including microbial activity, following the procedures used in previous works [6,7]. Preliminary results show that the encapsulation process drastically reduces the leaching of arsenic with respect to the unencapsulated sorbent.

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