Brine Encapsulation Laboratory Study

Ash Conditioning Compared to Engineered Encapsulation Strategies

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EPRI Project Manager

K. Ellison

ELECTRIC POWER RESEARCH INSTITUTE
3420 Hillview Avenue, Palo Alto, California 94304-1338 ▪ PO Box 10412, Palo Alto, California 94303-0813 ▪ USA
800.313.3774 ▪ 650.855.2121 ▪ askepri@epri.com ▪ www.epri.com

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Electric Power Research Institute
1300 West W.T. Harris Blvd.
Charlotte, NC 28262

Principal Investigator
K. Ellison

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ABSTRACT

This report describes results of a laboratory investigation of wastewater encapsulation. Five mix designs were created to compare moisture conditioning of fly ash with brine to engineered brine encapsulation approaches. Engineered approaches include changing the moisture content of the brine and ash mixture as well as using chemical additives (for example, quicklime, portland cement, and aluminate) to create a material with superior physical and chemical properties such that the leaching of salts and other constituents from the brine is minimized.

Data are provided for all ingredient materials used in the study. Physical property data, including hydraulic conductivity and unconfined compressive strength, are presented for each mix design over a range of curing intervals, out to 365 days. Mineralogical data, including X-ray diffraction as well as thermogravimetric analysis, are also presented. Environmental performance data, gathered via leaching tests as well as in-depth mineralogical investigations, are currently under way and will be presented in a follow-up report.

The study described in this report shows that for the brine and ash chemistry investigated, compared to a baseline moisture-conditioned mix containing only brine and ash, a flowable paste material containing fly ash, quicklime, and aluminate additives provides a less permeable and stronger material. Hydraulic conductivity (permeability) values lower than what is required for a landfill liner system were observed for the best performing mix design.

Keywords
Ash
Brine
Encapsulation
Landfill
Paste
Wastewater

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BACKGROUND AND OBJECTIVES

Background

The study described in this report was undertaken to understand the interactions between highly concentrated wastewater brines and fly ash such that long-term disposal considerations can be made. Specifically, this study compares brine–ash moisture-conditioning strategies to an engineered encapsulation approach. For this study, brine derived from flue gas desulfurization (FGD) wastewater and fly ash were obtained from a bituminous coal-fired power station located in the United States. Fly ash is collected via electrostatic precipitators and stored in silos, and FGD wastewater is processed in a falling film evaporator to produce a concentrated brine.

Brine disposal is achieved at the site by combining the brine with the ash in a weight-controlled pan-style batch mixer to create a "moisture-conditioned ash" (MCA) from where it is transferred into trucks for transportation to a nearby lined landfill facility. At the landfill site, the MCA is unloaded onto the surface of the landfill from where it is spread by bulldozer and compacted in place using rolling compactors. The weight-based ratio of brine to ash is controlled by the operator based on experience, and it typically varies between 12% and 15% in the current operations at the site. This approach to brine disposal is practiced at other locations in the power industry and was, therefore, selected as the basis for comparison in this study.

Conventional moisture conditioning of ash with brines is typically not considered an encapsulation process. Ash conditioning is typically used as a convenient way to dispose of wastewaters, and sequestering constituents of concern is usually not considered. Therefore, it is the goal of the study presented in this report to investigate ways in which brine may be more effectively encapsulated within an ash matrix.

Overall, wastewater encapsulation is a long-term goal for sequestering constituents of interest derived from wastewater sources into a solid matrix where they will be bound both physically and chemically. Ultimate success of this application will be defined by the amount of constituents released back into the environment, potentailly creating future environmental challenges.

When exploring ways in which conventional moisture conditioning can be improved to better achieve the goal of encapsulation, one possible approach is to use a higher moisture content. Water serves an important purpose in the geochemical reactions that result in the properties required to encapsulate contaminants in a fly-ash-based waste form. These reactions include hydration of various calcium-alumina-silicate phases and occur during the curing of the waste. The resultant waste is more lithified, retaining a monolithic nature and is therefore typically less permeable and stronger compared to a drier mixture in which these hydration reactions were not allowed to proceed to the same degree. Calcium silicate hydrate (CSH)[1] is one of the most abundant hydration reactions. Another common hydrate mineral is calcium aluminate hydrates (CAH); it forms concurrently with CSH in many systems. Additional hydrate minerals, are also known to form with salts such as sulfate and chloride.

However, higher moisture content materials, which have a flowable grout-like or “paste” consistency (see Figure 1-1), cannot be transported and deposited into landfills with trucks like conventional MCA can. As an alternative, a paste approach can be used whereby the material is transported by pumping and deposited passively into the landfill where it is then allowed to cure and harden into a monolith in place. Using this technology, which has precedent in the mining and grouting industries and is similar to standard concrete pumping practices, viscous material that has little to no evolved water can be pumped. Compared to MCA, the moisture content of a paste can be nearly doubled, allowing for more brine to be sequestered per unit mass of fly ash available.

Figure 1-1
Slump testing results showing a flowable paste material

A second but no less important modifier to achieve encapsulation in a wastewater and ash system is the use of additives or admixtures. Although highly dependent on brine and ash chemistry, typically, whether in a MCA or a flowable paste, additives are necessary for important hydration reactions to proceed in order to achieve encapsulation. This is especially true of bituminous Class F fly ashes, which are by definition pozzolanic, but they will not readily self-cement. Therefore, additives can be included in an encapsulation mix design to enhance the performance of the resultant waste form. Commonly used encapsulation additives include quicklime (CaO) and portland cement.

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[1] Cement notation is used to represent oxide moles. C = CaO, S = SiO_2, A = Al_2O_3, F = Fe_2O_3, M = MgO, N = Na_2O, K = K_2O, and H = H_20, **S** = SO_4, **C** = CO_3.

Objectives

The objective of this investigation was to carry out a laboratory test plan to compare the properties of a conventional MCA to several encapsulated mixed designs that used additives and higher moisture contents so that the potential release of salts and other constituents could be better understood. The hypothesis was that the higher moisture content and additives present in the paste method results in improved long-term physical and environmental properties in comparison to the MCA method by favoring a higher degree of chemical hydration. To test this hypothesis, this investigation compared the development of physical properties of the encapsulated material in a series of mix designs. Mineralogical and environmental (leaching) properties are currently being studied but are not published in this technical update.

Five mix designs were studied. Two of the mix designs represented the MCA method, and three additional mix designs represented variations of the flowable paste approach. In addition, the testing methodology for this study follows the methodology described in Electric Power Research Institute (EPRI) Technical Update Report 3002010779, *Wastewater Encapsulation Testing References: Evaluating Co-Management of Liquid Waste with Combustion Byproducts at Bench and Field Scale.*

Conversion Factors

Several English units (for example, inches) are used in this report. Table 1-1 presents conversion factors used to convert these values between English and International System of Units (SI).

Table 1-1
Conversion factors used in this report

Parameter	English to SI Units
Density	1 lb/ft^3 = 16018 g/m^3
Length	1 in. = 2.54 cm
Pressure (weight/area)	1 psi = 6.89 kPa 1 ton/ft^2 = 95.76 kPa
Temperature	°F = 1.8 (°C) + 32 °C = (°F - 32) × 5/9
Volume	1 gal = 3.78 L
Weight	1 lb = 0.45 kg

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TEST PLAN DESCRIPTION

Test Plan Methodology

Mix designs were formulated to replicate at the bench scale two different scenarios for the combined disposal of brine and fly ash. One set of mixes was designed to replicate the process of conditioning ash with brine and compacting it into a landfill. The next set of mix designs was formulated to replicate the proposed process of mixing the brine and ash at a much higher liquid-to-solid ratio, forming a paste that can be pumped to the disposal site for passive deposition. For both mix design concepts, additives were explored for their effectiveness in inducing hydration reactions to achieve encapsulation after a setting and hardening period.

The properties and rationale for each of the five mix designs were as follows:

Mix 1: For this mix design, fly ash was moisture conditioned with FGD brine from the site's thermal evaporator and compacted to a predetermined density and moisture content. This sample represents the baseline to simulate the site's current MCA operation.

Mix 2: Mix 2 was prepared in the same manner as Mix 1 but with the addition of quicklime. In practice, this would represent a minor change for the site requiring essentially only the addition of an extra quicklime silo with no further changes required to the current trucking and placement operations. This quicklime addition was done to test whether alkaline activation of pozzolanic reactivity results in better constituent encapsulation compared to the baseline Mix 1 MCA approach.

Mix 3: This mix design was intended to simulate a flowable paste using the site's brine along with fly ash and quicklime. It is, therefore, based on the same ingredients as Mix 2 but at higher moisture and quicklime content. The rationale was to explore the difference in material end-properties for a mix that can be pumped to the disposal site with concurrent alkaline activation. This approach allowed for more brine disposal per unit mass of ash and had the potential for better properties due to allowing hydration reactions to proceed more completely compared to the MCA approach.

Mix 4: Mix 4 started with the same ingredients as Mix 3 but included the addition of sodium aluminate. Aluminate availability, based on ongoing geochemical and mineralogical studies, is postulated to increase encapsulation performance by enhancing the formation of halide-sequestering hydrate minerals. Therefore, by adding additional aluminates, this mix attempted to isolate the effect of increased aluminate availability. More generally, this mix design explores the impact that a tertiary[2] additive could have on improved performance. The rationale was to explore the feasibility of further work that may be done to continue optimizing mix designs to drive properties to their optimized values.

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[2] For this report, *fly ash* is defined as the primary additive while *quicklime* and *cement* are defined as secondary additives. *Pure aluminates, silicates,* and *proprietary formulations* such as retarders or accelerants are defined as tertiary additives.

Mix 5: Mix 5 was prepared as an ash–brine paste using a portland cement (PC) additive as a direct comparison to the use of quicklime in Mix 3. PC is a common encapsulation additive and, therefore, a good reference as a performance benchmark.

Table 2-1 summarizes the mix design concepts explored along with the corresponding real-world scenarios they represent.

Table 2-1
Description of mix design concept

Mix Number	Description	Real-World Scenario
Mix 1	Moisture conditioned ash	Ash conditioned with FGD brine and compacted
Mix 2		Ash conditioned with brine and compacted, with quicklime added for additional encapsulation
Mix 3	Ash–brine paste	Ash–brine paste with quicklime added and pumped to a disposal site
Mix 4		Ash–brine paste with quicklime and aluminate added and pumped to a disposal site
Mix 5		Ash–brine paste with PC pumped to the disposal site

Ingredient Material Characterization

The first step in the bench scale testing program was to acquire the raw ingredient materials for testing. The objective was to initiate the test work with enough ingredient materials to produce all of the samples necessary to conduct all ingredient material characterizations and sample preparation tasks. The planning included making provision for excess sample preparation for each mix design to allow for additional or repeat testing that may be deemed useful during the course of the curing period. Although it is possible, if required, to prepare additional samples from retained ingredient materials at a later date in a testing program, this requires additional curing time for the material, significantly slowing down the overall study. In addition, using materials from the same initial batch of ingredient materials minimizes variations in subsequent testing results that could be attributed to differences in received ingredient materials used. Furthermore, it is often the case that as results are collected during the curing period, the investigator might identify the need for additional analytical work not planned for from the outset of the test work. It is, therefore, useful to have excess samples available for additional testing. Table 2-2 summarizes the ingredient materials and respective quantities that were acquired for this testing program.

Table 2-2
Summary of raw ingredient materials and quantities received for testing

Ingredient Materials	Total Quantity Received for the Project
Fly ash (Class F)	10 5-gal buckets, 376 lb
FGD brine (evaporator brine)	14 5-gal buckets, 62.6 gallons, 523 lb
Quicklime	4 5-gal buckets, 146 lb
Portland cement (Type I/II)	1 bag, 45 lb
Sodium aluminate (CAS 1302-42-7)	1 container, 2 lb

As-received ingredient materials were homogenized and then characterized to determine their physical, chemical, and mineralogical characteristics. This is an important step in this kind of study because characteristics of the ingredient materials can be used to benchmark against other studies or variability that may arise at a site. In addition, understanding the makeup of the ingredient materials can inform, and possibly explain, observations during sample preparation and trends that emerge during short-term and long-term physical and chemical analyses of the end products.

To facilitate characterization, the FGD brine slurry, which contained a significant precipitate fraction, was separated into its liquid and solid components by 0.45-micron filtration. The as-received slurry as well as the filtered liquid component were characterized for their chemical makeup. These results are presented in Tables 2-3 and 2-4. The brine solids along with the other solid ingredient materials were similarly characterized. Those results are presented in Table 2-5. The specific testing methods used for each individual component analysis are provided for both sets of results. The brine used in this study was constituted primarily of calcium and magnesium chloride along with a significant concentration of sulfate (both dissolved and precipitated). The brine had a total dissolved solids concentration of 101,000 mg/L. The observed chemistry of the precipitated brine solids was somewhat anomalous, containing a large fraction of calcium and boron whereas it was expected that the precipitated solids would be primarily calcium sulfate. The thermal evaporator used at this site uses a vapor scrubber for boron; therefore, it is likely that at the time of sampling, a slug of solids derived from this scrubbing system was collected.

The fly ash, derived from bituminous coal, met ASTM C618 Class F fly ash specifications with 89.06% of the ash consisting of $SiO_2 + Al_2O_3 + Fe_2O_3$, a CaO content of only 0.89%, and a loss on ignition of 2.56%. The quicklime used in this study was donated by a lime supplier and considered a finely ground quicklime with a <1/8-in. particle size and a free lime content of 82.84%. Standard off-the-shelf Type I/II PC was used.

Table 2-3
Brine slurry as-received properties

Test	Units	Method	Brine Slurry	Brine Liquid (filtered)
			Concentration	
pH	s.u.	SM 4500 H+B	7.34	NA
Alkalinity	mg $CaCO_3$/L	SM 2320	NA	1,760
Total suspended solids (TSS)	mg/L	SM 2540D	1,360	NA
Total dissolved solids (TDS)	mg/L	SM 2540C	101,000	NA

Table 2-4
Chemical characteristics of filtered brine liquid

Test	Units	Method	Brine Liquid (filtered) Concentration
Chloride (Cl)	mg/L	EPA 9056	56,200
Calcium (Ca)	mg/L	EPA 3005A	12,200
Magnesium (Mg)	mg/L	EPA 3005A	11,300
S as Sulfate (SO_4)	mg/L	EPA3005A	4,230
Sodium (Na)	mg/L	EPA 3005A	4,220
Sulfur (S)	mg/L	EPA 3005A	1,410
Potassium (K)	mg/L	EPA 3005A	1,060
Boron (B)	mg/L	EPA 3005A	838
Bromide (Br)	mg/L	EPA 9056	635
Fluoride (F)	mg/L	EPA 9056	<90.5
Silicon (Si)	mg/L	EPA 3005A	2.93
Barium (Ba)	mg/L	EPA 3005A	2.09
Selenium (Se)	mg/L	EPA 3005A	0.587
Mercury (Hg)	mg/L	EPA 7470A	<0.20
Phosphorous (P)	mg/L	EPA 3005A	0.110
Aluminum (Al)	mg/L	EPA 3005A	<0.10
Iron (Fe)	mg/L	EPA 3005A	<0.10
Titanium (Ti)	mg/L	EPA 3005A	<0.07
Chromium (Cr)	mg/L	EPA 3005A	0.059
Lead (Pb)	mg/L	EPA 3005A	<0.05
Arsenic (As)	mg/L	EPA 3005A	<0.05
Cadmium (Cd)	mg/L	EPA 3005A	0.032
Silver (Ag)	mg/L	EPA 3005A	<0.02
Cation sum	mg/L	—	1,982
Anion sum	mg/L	—	1,717
Anion/cation	Unitless	—	0.87
Measured TDS/ calculated TDS	Unitless	—	1.10

Table 2-5
Chemical characteristics of filtered brine solids and as-received solid ingredient materials

Test	Method	Units	Brine Solids	Quicklime	Cement	Fly Ash
			Concentration			
Reported as the Element						
Aluminum (Al)	ASTM D6349	µg/g	365	2,424	25,033	142,366
Arsenic (As)	ASTM D6349	µg/g	<24.7	<24.5	<24.3	58
Barium (Ba)	ASTM D6349	µg/g	35.5	511	159	696
Boron (B)	ASTM D6349	µg/g	103,720	<24.5	31.8	160
Cadmium (Cd)	ASTM D6349	µg/g	<4.9	<4.9	<4.9	<4.8
Calcium (Ca)	ASTM D6349	µg/g	142,939	681,818	464,551	12,293
Chromium (Cr)	ASTM D6349	µg/g	80.7	47.3	155	231
Iron (Fe)	ASTM D6349	µg/g	4,504	776	23,431	60,572
Lead (Pb)	ASTM D6349	µg/g	<24.7	<24.5	<24.3	67
Magnesium (Mg)	ASTM D6349	µg/g	10,491	4,655	15,014	5,366
Mercury (Hg)	ASTM D6414	µg/g	0	<0.031	<0.032	0
Phosphorous (P)	ASTM D6349	µg/g	<49.5	54.6	317	800
Potassium (K)	ASTM D6349	µg/g	477	272	4,458	20,339
Selenium (Se)	ASTM D6349	µg/g	<24.7	<24.5	<24.3	<24.2
Silicon (Si)	ASTM D6349	µg/g	4,768	4,548	93,493	250,093
Silver (Ag)	ASTM D6349	µg/g	<24.7	<24.5	<24.5	<24.2
Sodium (Na)	ASTM D6349	µg/g	1,758	<18.2	979	2,559
Titanium (TI)	ASTM D6349	µg/g	<24.7	125	1,816	7,853
Bromide (Br), water soluble	ASTM C1216	µg/g	241	<5	<5	<5
Chloride (Cl), water soluble	ASTM C1216	µg/g	19,100	9.6	37.2	134
Fluoride (F), water soluble	ASTM C1216	µg/g	<5	<5	7	32
Sulfur (S), total	ASTM D6349	µg/g	2,730	247	12,000	2,630
Total sulfur as sulfate (SO_4)	ASTM D6349	µg/g	8,190	741	36,000	7,890
Sulfate (SO_4), water soluble	ASTM C1216	µg/g	3,430	53	10,700	4,400
Reported as Oxides						
SiO_2	ASTM D6349	Wt%	1.02	0.97	20.00	53.50
Al_2O_3	ASTM D6349	Wt%	0.07	0.46	4.73	26.90
Fe_2O_3	ASTM D6349	Wt%	0.64	0.11	3.35	8.66
K_2O	ASTM D6349	Wt%	0.06	0.03	0.54	2.45
MgO	ASTM D6349	Wt%	1.74	0.77	2.49	1.72

Table 2-5 (continued)
Chemical characteristics of filtered brine solids and as-received solid ingredient materials

Test	Method	Units	Brine Solids	Quicklime	Cement	Fly Ash
			Concentration			
Reported as Oxides (continued)						
TiO_2	ASTM D6349	Wt%	<0.004	0.021	0.303	1.31
CaO	ASTM D6349	Wt%	20.00	95.40	65.00	0.89
SO_3	ASTM D6349	Wt%	0.68	0.062	3.00	0.66
Na_2O	ASTM D6349	Wt%	0.24	<0.00245	0.13	0.35
P_2O_5	ASTM D6349	Wt%	<0.011	0.013	0.073	0.18
Material balance of major ash oxides		Wt%	57.9	97.8	99.6	96.6
Ash at 650°C			58.6	97.5	97.5	97.4

Beyond the standard chemical analyses that were conducted on the liquid and solid ingredient materials, additional chemical and physical characterization of the solid ingredient materials was conducted to determine free lime content, density, loss on ignition, total sulfur, halogen content (total and water soluble), and total carbon content. These results and test methods are presented in Table 2-6.

Table 2-6
Additional chemical and physical characteristics of filtered brine solids and as-received solid ingredient materials

Test	Method	Brine Solids	Quicklime	Cement	Fly Ash
Free lime (wt%, AR)	ASTM C114 sec 30	NA	82.84	0.57	0.21
Density (g/cm^3), AR	ASTM D854	NA	4.959	3.545	2.219
Density, H_e Pycnometer (g/cm^3), AR	ASTM D5550	NA	3.297	3.232	2.346
Loss on ignition at 650ºC		41.44	2.52	2.52	2.56
Total sulfur (wt%, AD)	ASTM D6349	0.27	0.025	1.20	0.26
Total sulfur (wt%, AD)	ASTM D4239	<0.25	<0.1	1.04	0.22
Total halogens (µg/g, AD)	ASTM D6721	17300	<10	51.3	97.9
Total water-soluble halogens (µg/g, AD)	ASTM D1216 (Table 2b)	19341	9.6	43.7	165.8
Total carbon (wt%, AD)	ASTM D6316	<0.5	<0.5	0.24	1.94

Figure 2-1 shows particle size distribution curves (via laser diffraction) for the as-received brine slurry as well as the solid ingredient materials used for testing (quicklime, PC, and fly ash, respectively). As shown in Figure 2-1, the brine slurry had the finest particle size distribution, followed by the quicklime, PC, and fly ash, respectively. Median particle size (D_{50}) ranged from close to 6 microns in the brine slurry to just over 17 microns in the fly ash.

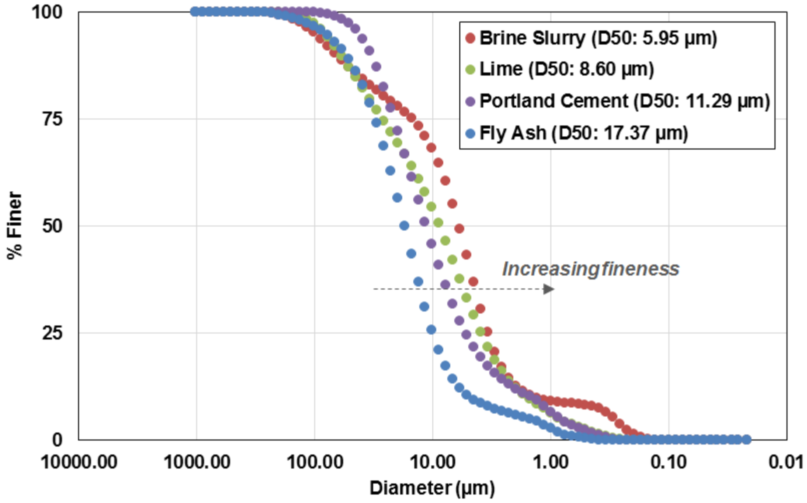

Figure 2-1
Ingredient material particle size distribution curves

These initial characterization results provide a basis for analyzing, comparing, and potentially explaining observations and results from the sample-mixing and specimen-testing phases of the project as well as providing data for benchmarking against other studies. Collecting as much of this type of data early in the project is highly recommended because working with such heterogeneous and complex materials often produces results and observations that may seem random and unexplainable in the beginning. However, over time, as trends emerge, one can begin developing hypotheses and theories that explain them with the help of a fundamental understanding of the ingredient materials with which the process began. Additional ingredient material data can be found in Appendix A.

Mix Formulation Design

Table 2-7 summarizes the desired mix proportions that were developed for this study. Mixes 1 and 2 are the conditioned-ash mixes, and Mixes 3, 4, and 5 are the paste mixes.

Table 2-7
Mix design proportion summary

Mix Number	Intended Consistency	Fly Ash	FGD Brine	Quicklime	Portland Cement	Sodium Aluminate
		Mix Composition (mass %)				
Mix 1	Moisture conditioned to reduce dust and facilitate compaction	82	18	—	—	—
Mix 2		79.5	18	2.5	—	—
Mix 3	Pumpable paste (8.0–9.5 in. slump)	65.0	30	5.0	—	—
Mix 4		64.0	30	5.0	—	1.0
Mix 5		65.1	30	—	4.9	—

In practice, initial mix designs typically need to be adjusted as unique material properties are encountered in the lab. Standard proctor testing was performed to determine the brine content to be used for Mix 1. The results of the standard proctor test for Mix 1 (see Figure 2-2) showed a value of 89.1 ft^3 for maximum dry density achievable and 20.3% optimal moisture content. The conditioned-ash samples (Mixes 1 and 2) in the bench scale study were prepared based on these proctor test results. The Mixes 1 and 2 specimens were prepared by compacting the moisture (brine) conditioned-ash samples using standard proctor energy and storing the sample in standard 3 × 6 in. capped concrete molds.[3] It was decided to prepare the samples slightly dry of the optimal moisture (2% less than optimum). This 18% moisture content is still slightly higher than that which is practiced on average at the site (12–15%).

For Mixes 3, 4, and 5, initial trial mixes showed that the idealized design proportions shown in Table 2-7 may not result in a material with similar rheological (flow) properties due to different chemical reactions. For example, as shown in Table 2-9, initial mix designs for Mixes 3 and 4 resulted in a low slump, nonpumpable material. Therefore, these mix designs were adjusted in order to ensure that each mix had similar flow characteristics, regardless of the mix ingredients. A target slump between 8.0 and 9.5 in. was used to quantify this metric. An iterative approach was used to hone in on each liquid-to-solid ratio to achieve the target slumps. The final mix proportions for all five mixes are shown in Tables 2-8 and 2-9.

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[3] Further details on this procedure can be found in EPRI report 3002010779, *Wastewater Encapsulation Testing References: Evaluating Co-Management of Liquid Waste with Combustion Byproducts at Bench and Field Scale.*

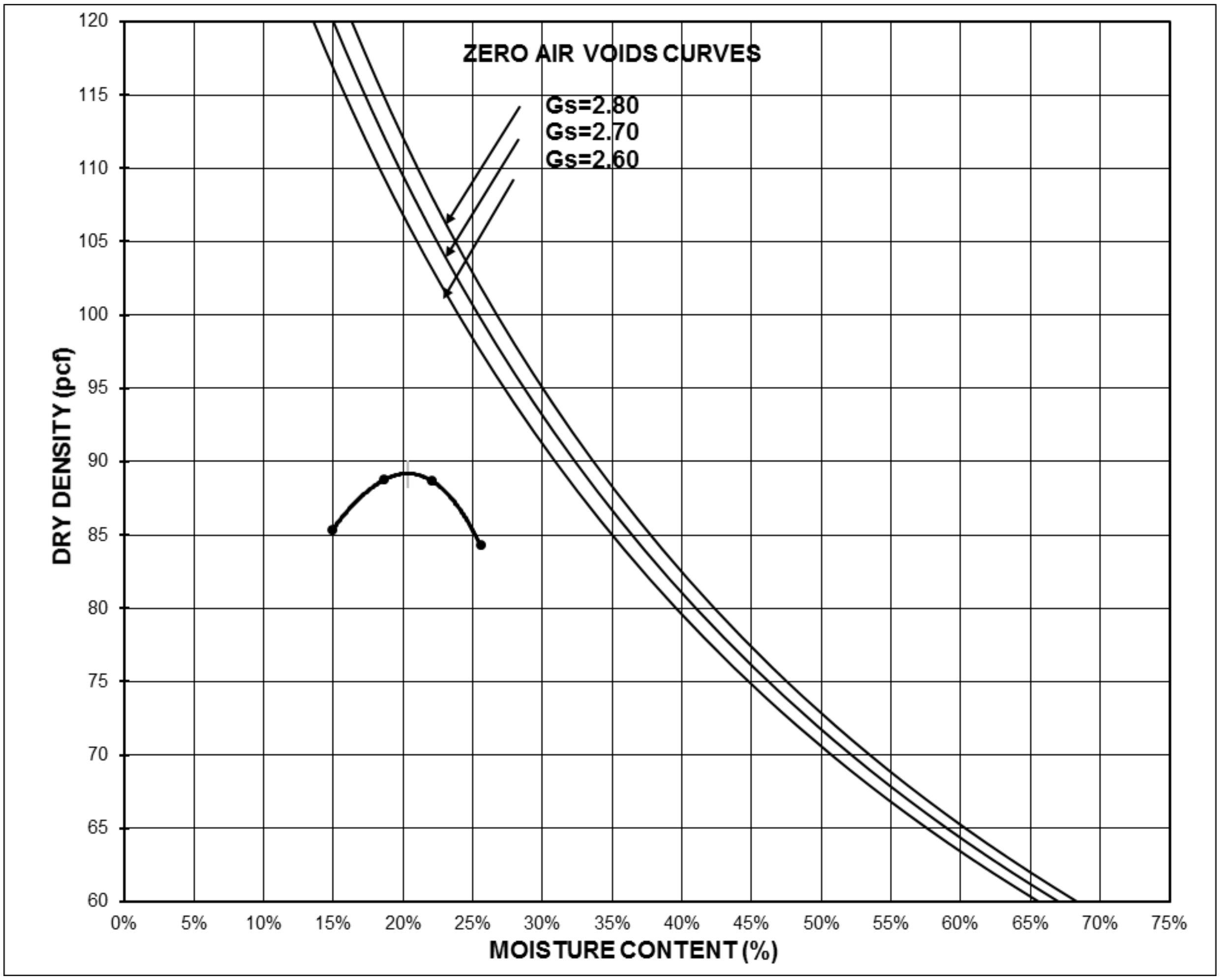


Figure 2-2
Standard proctor test results for Mix 1

Table 2-8
Summary of sample mixing and monolith cylinder preparation for Mixes 1 and 2 (conditioned ash)

Mix Number	Mix Composition	Fly Ash	FGD Brine	Quick lime	Portland Cement	Sodium Aluminate	Slump (inches)	Number of Sample Cylinders Prepared
Mix 1	Mass %	82.0	18.0	—	—	—	NA	16
	Mass (g)	13,472	2,948	—	—	—		
Mix 2	Mass %	79.5	18.0	2.5	—	—	NA	16
	Mass (g)	13,018	2,948	408	—	—		

Table 2-9
Summary of sample mixing and monolith cylinder preparation for Mixes 3, 4, and 5

Mix Number		Mix Composition	Fly Ash	FGD Brine	Quicklime	Portland Cement	Aluminate	Slump (inches)	Number of Sample Cylinders Prepared
Mix 3	Intended mix design	Mass %	65.0	30.0	5.0	—	—	<4	20
		Mass (g)	26,333	12,167	2,043	—	—		
	Final mix design	Mass %	64.0	31.0	5.0	—	—	9.25	
		Mass (g)	26,333	12,761	2,043	—	—		
Mix 4	Intended mix design	Mass %	64.0	30.0	5.0	—	1.0	<4	20
		Mass (g)	16,820	7,890	1,317	—	272		
	Final mix design	Mass %	62.2	32.0	4.9	—	1.0	8.5	
		Mass (g)	16,820	8,646	1,317		272		
Mix 5	Intended mix design	Mass %	65.1	30.0	—	4.9	—	>10	20
		Mass (g)	20,885	9,621		1,585			
	Final mix design	Mass %	68.1	27.4	—	4.5	—	9.25	
		Mass (g)	23,885	9,621	—	1,585	—		

Brine Encapsulated Monolith Evaluation Plan

The samples that were prepared as described previously in this section were capped and placed in a temperature-controlled (70°F) curing chest. Samples were subsequently retrieved at predetermined time intervals ranging from the short term (the first seven days) up to the long term (365 days). The testing methods described next are explained in greater detail in EPRI report 3002010779, *Wastewater Encapsulation Testing References: Evaluating Co-Management of Liquid Waste with Combustion Byproducts at Bench and Field Scale.*

Short-Term Properties

The following short-term tests were performed:

- A paint filter test following U.S. Environmental Protection Agency (EPA) method 9095B
- Early strength testing using a pocket penetrometer

Long-Term Physical Properties

The following long-term tests were performed:

- Hydraulic conductivity (permeability) following ASTM D5084
- Unconfined compressive strength (UCS) following ASTM D2166
- Cured unit weight following ASTM D7263

Data were collected after 7, 28, 60, 90, and 365 days of curing.

Mineralogical and Environmental Properties

The following mineralogical tests were performed after 90 days of curing:

- Quantitative Rietveld X-ray diffraction (XRD)
- Thermogravimetric analysis (TGA)

Detailed mineralogical investigations and environmental performance testing are under way and will be presented in a follow-up report.

3

MONOLITH TESTING RESULTS AND CONCLUSIONS

Short-Term Physical Properties

For the flowable mixes, it was desired to investigate whether ingredient liquid would segregate out of the mix prior to chemically setting. For flowable mixes, this "bleed water" will begin to evolve as the liquid fraction of the mix increases. Bleed water evaluation is not applicable to dry mixes. Related to bleed water evaluation is the U.S. Environmental Protection Agency (EPA) Method 9095B, known as the paint filter test. This test is often imperfect for observing bleed water because often a fine particle-size material (such as paint itself or a flowable paste) will flow right through the filter, thus measuring not only bleed water. Although this test is not specifically designed, nor necessarily appropriate, to evaluate flowable wastewater-encapsulated mixes, it is a simple test to perform, and data were collected for this study. The results are shown in Table 3-1. Both of the flowable mixes containing quicklime (Mixes 3 and 4) were retained on the paint filter immediately after mixing due to fast hydration reactions in the material. For Mix 5, which contains cement and in which the hydration reaction kinetics are slower, the material flowed through the filter initially and required an additional 15 minutes of mixing before being thick enough to be retained on the filter. No visual observation of segregating bleed water was observed for Mixes 3, 4, and 5.

Table 3-1
Summary of paint filter test results

Mix Number	Paint Filter Test
	Pass/Fail
Mix 3	Pass
Mix 4	Pass
Mix 5	Fail initially, but passed after additional 15 minutes of mixing

For both flowable and dry compacted samples at the initial stages of curing, the material had not developed enough strength so that a break-type test (such as the UCS test) was suitable. This interim phase of early strength development is best monitored by using a soil pocket penetrometer, which measures the compressive strength of the material based on the penetration of a tip of known area. The results are reported as a force per unit area (typically, tons per square foot) that corresponds to a known preset penetration depth (usually 1/4 in.). Pocket penetrometer readings can normally be taken over the first one to two weeks of material curing by testing the top portion of cylinders that have been cast. The investigator can use a threshold pocket penetrometer value as the decision point for when it is appropriate to begin measuring long-term properties such as UCS.

For this study, unfortunately, not enough pocket penetrometer data were collected to provide meaningful details. Although the compacted mixes (Mixes 1 and 2) immediately registered significant strength with the pocket penetrometer (as expected), the flowable mixes required more time to gain strength. Although it is known that the materials gained enough strength for UCS testing to be performed after 28 days of curing, penetrometer testing was terminated after seven days; therefore, the curing time at which significant strength was gained was not determined. Pocket penetrometer results are summarized in Table 3-2.

Table 3-2
Summary of pocket penetrometer test results

Mix Number	Pocket Penetrometer tons per ft^2 (tsf)
Mix 1	>3.0
Mix 2	>3.0
Mix 3	Samples were too soft to test during first seven days.
Mix 4	
Mix 5	

Long-Term Physical Properties

Saturated Hydraulic Conductivity

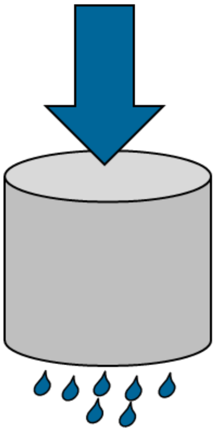
The saturated hydraulic conductivity (HC) of a material describes its ability to allow fluids to flow through its pores. For soils, this flow is described by Darcy's law. This law relates the volumetric flow rate of water through a soil to its permeability coefficient (k), the hydraulic gradient driving the flow, and the cross-sectional area of soil mass perpendicular to the flow direction. HC is performed in the laboratory or in the field on a specimen under saturated conditions to estimate the ease with which fluids flow through interconnected pores in the material. The HC of a chemically active material in which hydrate minerals are forming evolves with hydration and aging of the material. As hydration progresses and cementation of the material improves, the HC of an intact/unfractured material decreases. The measured values of HC of cemented materials are influenced by the curing period, the quantity and type of alkaline chemical additives/binder used, the reactivity of the pozzolans, and the phase formation of minerals in the matrix. To provide perspective on analyzing HC data, it is useful to note that a typical landfill liner specification is an HC of 1 × 10^{-7} cm/sec. For additional perspective, HC is related to flow via Darcy's law (see Equation 3-1).

$$Q = -KA\frac{dh}{dl} \qquad \textbf{Eq. 3-1}$$

Where:

Q represents volumetric flow
K represents hydraulic conductivity
A represents cross-sectional area.
$\frac{dh}{dl}$ represents the hydraulic gradient

Therefore, as a simplistic example, for saturated flow conditions, example flow rates are shown as a function of different HC in Figure 3-1. As HC decreases, a material becomes less permeable and allows less water to leach through a material as a function of time.



Example

Hydraulic Conductivity (cm/s)	Flowrate (unitless)
1e-4	1,000
1e-5	100
1e-6	10
1e-7	1

Figure 3-1
Simplistic example of how hydraulic conductivity affects observed flow rates under Darcy's law

The HC test results for this study are presented in Figure 3-2 and Table 3-3. Data were collected after 7, 28, 60, 90, and 365 days of curing. When comparing the mix designs, significant variability was observed both as a function of time and with each mix design.

- **Mix 1:** HC did not change significantly with time, indicating that chemical hydration reactions were not occurring to any significant degree in the material. In addition, this HC ($>1 \times 10^{-4}$ cm/sec) was similar to reported values[4] for traditional fly ash landfill disposal. Data were not collected at 365 days of curing.
- **Mix 2:** Due to the addition of quicklime, it appears that limited chemical hydration reactions were occurring. After a small rise in HC at 28 days of curing, the HC decreased slightly with curing time. The quicklime appears to be making the material slightly less permeable compared to Mix 1.
- **Mix 3:** This mix took more time to initially gain strength because of the higher moisture content. However, when HC was measured for the first time after 28 days of curing, it was observed to be an order of magnitude lower than the baseline case of Mix 1. As time progressed, HC continued to decrease as hydration reaction proceeded, continually making the material less permeable.
- **Mix 4:** Similar to Mix 3, a strong decrease in HC occurred as a function of curing time. It is hypothesized that the addition of aluminates enables the formation of more hydrate minerals, which are constituted chiefly of calcium, silicon, and aluminum. In addition, after 365 days of curing, Mix 4 has a HC lower than typical landfill liner requirements.
- **Mix 5:** Like the other two paste mixes, it was observed that the initial HC of this mix was significantly less than the baseline Mix 1. However, it appears that this mix, which used PC instead of quicklime, did not undergo continued hydration reactions as a function of time; no significant loss in HC was observed over the curing time measured.

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[4] EPRI report 3002001138, *Geotechnical Properties of Fly Ash and Potential for Static Liquefaction: Volume 2 – Data Summary.*

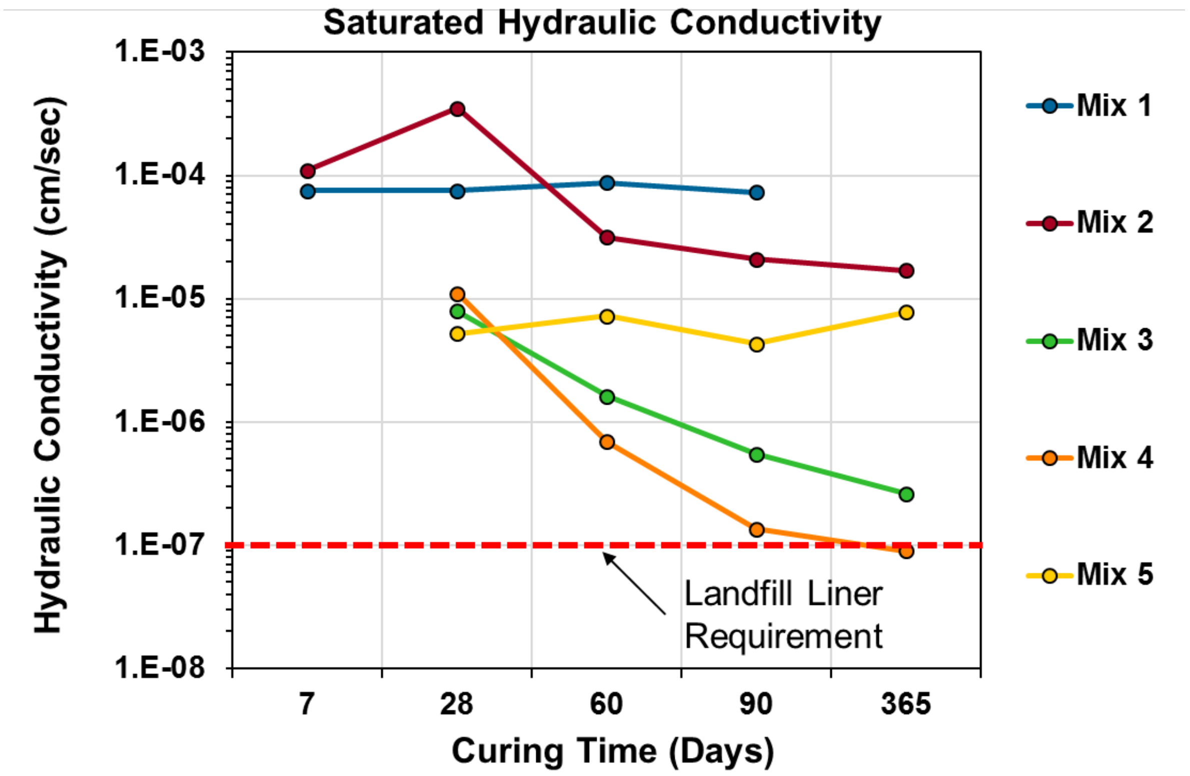

Figure 3-2
Hydraulic conductivity of cured monolith cylinders versus time

Table 3-3
Hydraulic conductivity values versus time

Mix #	7 Day	28 Day	60 Day			90 Day			365 Day		
	HC (cm/s)	HC (cm/s)	HC (cm/s)			HC (cm/s)			HC (cm/s)		
	S1	S1	S1	S2	Avg	S1	S2	Avg	S1	S2	Avg
Mix 1	**7.50E-05**	**7.50E-05**	8.7E-05	-	**8.7E-05**	7.3E-05	-	**7.3E-05**	-	-	**-**
Mix 2	**1.10E-04**	**3.50E-04**	2.6E-05	3.7E-05	**3.2E-05**	2.1E-05	-	**2.1E-05**	1.7E-05	-	**1.7E-05**
Mix 3	*Sample too soft to test*	**7.90E-06**	2.8E-06	4.2E-07	**1.6E-06**	5.6E-07	5.4E-07	**5.5E-07**	4.5E-07	7.0E-08	**2.6E-07**
Mix 4		**1.10E-05**	4.7E-07	9.1E-07	**6.9E-07**	1.6E-07	1.1E-07	**1.4E-07**	9.1E-08	8.8E-08	**9.0E-08**
Mix 5		**5.20E-06**	5.6E-06	8.9E-06	**7.3E-06**	3.7E-06	4.9E-06	**4.3E-06**	1.1E-05	4.6E-06	**7.8E-06**

Unconfined Compressive Strength

This test gives a measure of the compressive and shear strength of cohesive or cementitious specimen by applying axial load in increments until the sample fails. Typically, a material gaining strength is an indicator that hydration reactions are proceeding and lithifying a material together. Therefore, by comparing the strength between samples and as a function of curing time, conclusions on the degree of hydration reactions that have proceeded in a material can be drawn. UCS results are shown in Figure 3-3 and Table 3-4. Data were collected at the same curing intervals as HC. When comparing the mix designs, it was observed that there were significant differences between mix designs and as a function of time.

- **Mix 1:** While the material has sufficient strength to be structurally stable and be driven on with equipment, no significant gain in strength was observed with time. UCS was not measured at 365 days of curing.
- **Mix 2:** As curing time progressed, strength increased, which agrees with the observed drop in HC. UCS was not measured at 365 days of curing.
- **Mix 3:** This mix exhibited the strongest gain in strength as a function of curing time.
- **Mix 4:** This mix gained strength earlier than all the other mixes. However, the increase in strength over time is not as dramatic as in Mix 3.
- **Mix 5:** Similar to the HC observations, only minimal continued hydration reactions appear to be taking place within this material as a function of curing time.

When comparing HC to UCS, there was general agreement between samples, but no direct correlation. For example, comparing Mix 3 to Mix 4, while both show a gain in strength with time, Mix 4 developed less strength, but had the lowest HC.

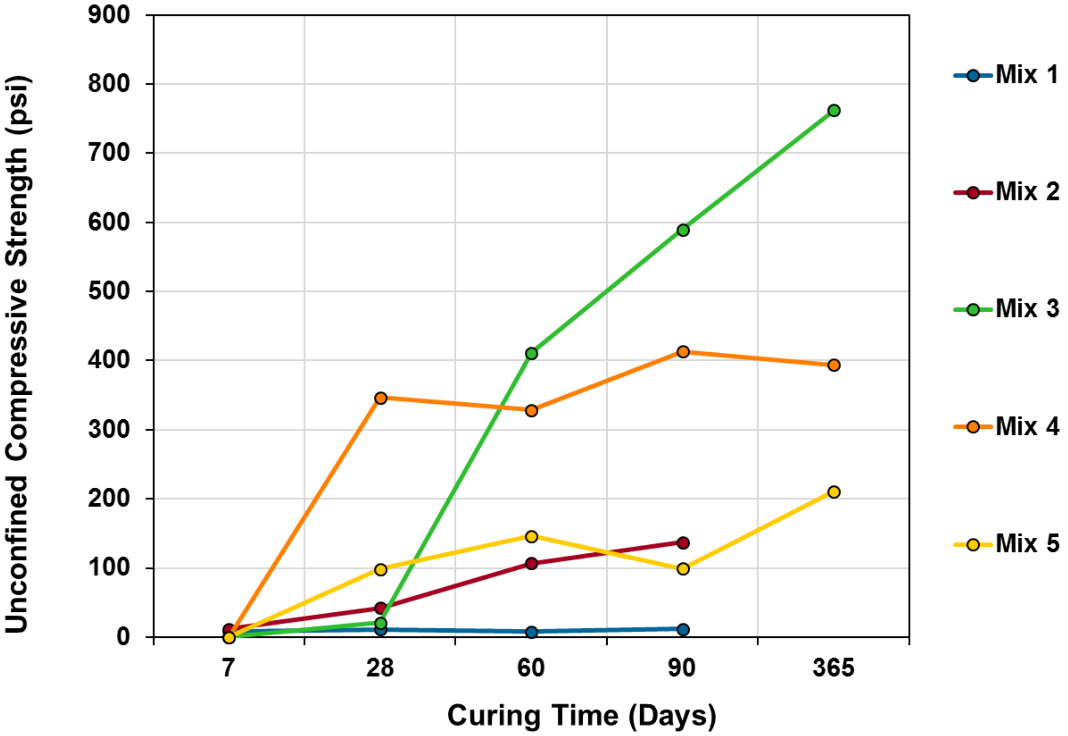

Figure 3-3
Unconfined compressive strength of cured monolith cylinders with time

Table 3-4
Unconfined compressive strength values versus time

Mix #	7 Day	28 Day	60 Day			90 Day			365 Day		
	UCS (psi)	UCS (psi)	UCS (psi)			UCS (psi)			UCS (psi)		
	S1	S1	S1	S2	Avg	S1	S2	Avg	S1	S2	Avg
Mix 1	**8.5**	**11.0**	8.8	6.9	**7.9**	12.1	-	**12.1**	-	-	**-**
Mix 2	**11.8**	**42.4**	102.0	111.4	**106.7**	137.0	-	**137.0**	-	-	**-**
Mix 3	*Sample too soft to test*	**20.8**	362.2	460.8	**411.5**	581.1	598.4	**589.8**	673.4	851.2	**762.3**
Mix 4		**346.9**	290.6	366.6	**328.6**	380.4	446.0	**413.2**	350.4	437.4	**393.9**
Mix 5		**98.5**	127.0	166.0	**146.5**	84.3	113.6	**99.0**	174.7	246.6	**210.7**

Cured Unit Weight

The cured unit weight of a specimen is used to provide information on the sample's density and provide quality control between samples. Cured unit weight is determined by simple measurements based on volume (calculated based on length and diameter measurements) and mass following ASTM D7263. Typically, cylinders are measured and then weighed prior to being tested for HC or UCS. Cured unit weight data are shown in Figure 3-4 and Table 3-5. Each of the flowable paste mixes was denser than either of the compacted mixes, despite having a higher moisture content. In addition, for all mixes, no significant weigh loss was observed with time, verifying that significant moisture was not lost to the atmosphere from the samples through evaporation during the curing process.

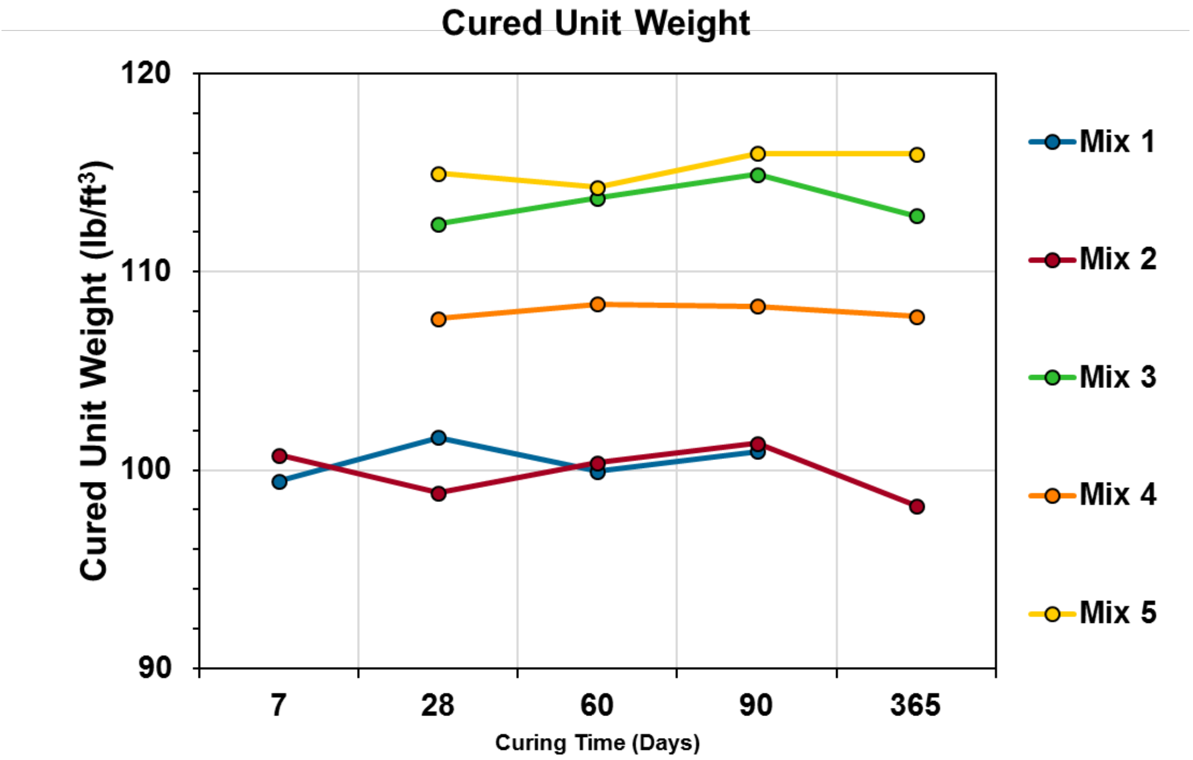

Figure 3-4
Unit weight of cured monolith cylinders versus time

Table 3-5
Unit weight values versus time

Mix #	7 Day weight (lb/ft^3)	28 Day weight (lb/ft^3)	60 Day weight (lb/ft^3)	90 Day weight (lb/ft^3)	365 Day weight (lb/ft^3)
	Avg	Avg	Avg	Avg	Avg
Mix 1	99.5	101.7	99.9	101.0	-
Mix 2	100.8	98.9	100.4	101.4	98.2
Mix 3	-	112.4	113.7	114.9	112.8
Mix 4	-	107.7	108.4	108.3	107.8
Mix 5	-	115.0	114.3	116.0	115.9

Mineralogical Properties

To assist in evaluating the effectiveness of encapsulation, in addition to physical properties, mineralogical properties help to evaluate mix designs that perform well by obtaining a deeper understanding of the chemical reactions taking place in the material.

X-Ray Diffraction

Initial mineralogical characterization of the monolith was carried out by conducting XRD spectroscopy analysis to identify crystalline mineral phases. Rietveld refinement was used to quantify mineral phases and amorphous materials.

As shown in Table 3-6, XRD results showed high concentrations (>72 wt%) of amorphous material in each sample. The composition of the amorphous material cannot be identified or quantified by XRD. The major crystalline species, mullite, comprises only 13–17 wt% of each monolith sample. Quartz, calcite, gypsum, calcite, iron oxides, and pyroaurite are each present at concentrations less than 5%.

Despite significant physical property differences observed for each mix design, little can be discerned from this XRD data to assist in determining what may be happening chemically in each sample. Due to the high amorphous content in this material, it is likely that the majority of chemical interactions that are occurring within these materials are happening as amorphous–noncrystalline mineral phases.

Table 3-6
Quantitative X-ray diffraction analysis for each mix design after 90 days of curing

Mineral Phase	Approximate Composition	Concentration (Weight %)				
		Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Mullite	$Al_6Si_2O_{13}$	17	15	15	13	15
Quartz	SiO_2	4	3	4	3	3
Gypsum	$CaSO_4 \cdot 2H_2O$	1	1	2	-	1
Calcite	$CaCO_3$	-	2	3	1	2
Pyroaurite	$Mg_6Fe_2(CO_3)(OH)_{16} \cdot H_2O$	-	< 1	1	1	-
Hematite	Fe_2O_3	1	1	1	1	1
Magnetite	Fe_3O_4	2	2	1	1	1
Halite	$NaCl$	< 1	-	-	-	< 1
Amorphous	**-**	**75**	**72**	**74**	**79**	**76**

Thermogravimetric Analysis: Non-Evaporable Water Content

TGA is a semi-quantitative technique that shows the mineralogical decomposition of the material at certain temperatures by continuously measuring the loss in weight of a sample as temperature is increased under a nitrogen (N_2) atmosphere. TGA is widely used to measure the bound water content in cementitious materials and to assess the hydration products of PC and the reactivity of fly ash and other pozzolans. It is particularly useful to identify temperatures at which minerals and hydrates undergo thermal reactions that are typically associated with weight changes. For this study, TGA data were compared between each mix design to assess the degree to which the water contained in the brine was retained chemically within each mix design.

As shown in Table 3-7, the loss of weight up to 70°C is attributed to loss of residual moisture that is present in the capillaries of a material and not chemically bound. Additional weight loss up to 350°C is attributed to dehydration of cementitious hydrate mineral phases. Additional weight loss at higher temperatures is attributed to the liberation of carbon.

Table 3-7
Thermogravimetric analysis results showing loss in weight due to mineral thermal decomposition

Temperature (N_2 atmosphere)	Probable Cause of Weight Loss	Loss in Weight (Weight %)				
		Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Ambient- 70°C	**Residual Moisture**	**2.10**	**1.80**	**2.31**	**1.05**	**2.74**
70°C-110°C	Water of Hydration	0.85	1.16	2.34	2.58	1.96
110°C-240°C		0.61	0.70	1.40	1.36	0.79
240°C-350°C		0.35	0.70	1.40	1.36	0.79
Sum of Hydration Water Lost		**1.81**	**2.56**	**5.14**	**5.30**	**3.54**
350°C-500°C	Volatile Carbon	2.07	2.13	2.61	2.38	1.98
500°C-900°C	CO_2 from Carbonate	0.41	0.93	1.47	1.60	1.03
Mass Balance		98.40	99.20	98.20	95.30	97.90

For all mixes, only a small amount of residual capillary moisture (ambient to 70°C) was released, despite the paste mixes containing ~40% more liquid than Mix 1 and Mix 2. This suggests that the paste mixes did not contain excess water that could not chemically react with the solid ingredient materials and that nearly all of the water from the brine was chemically bound. Mix 4 released the smallest amount of residual moisture, suggesting an increased amount of chemical hydration within the material.

Comparing the loss in weight (due to water release) from 70 to 350°C assists in quantifying the amount of hydrate mineral phases present in each mix. Comparing these data for each mix provides good agreement to observed HC data, suggesting that the formation of hydrate minerals are the primary mechanism by which the HC or permeability of a material is reduced. The largest portion of hydrate mineral formation appears to have occurred in Mixes 3 and 4, corresponding to the lowest observed HC values. The paste sample created using PC, Mix 5, did not react chemically as well as the quicklime-based materials. Both moisture-conditioned materials contain less hydrate minerals, suggesting that they were not allowed to fully hydrate because of a lack of water in the mix.

Conclusions

This study set out to compare the impacts of moisture-conditioning fly ash with brine to engineered encapsulation approaches on the retention of salts and other constituents from the brine. Both physical property and mineralogical data were collected.

The study results show that for the specific chemistry of the brine and ash used in this study, flowable paste materials using lime (Mixes 3 and 4) have the potential to be the best performers. Because of an observed lowering of HC compared to the baseline moisture conditioned Mix 1, rainfall is less likely to infiltrate the material, allowing for the formation of leachate that could liberate salts and other constituents from the material.

Mix 4, which includes the addition of tertiary aluminates, shows the opportunity for further optimizing mix designs. Most likely, additional mineral phases, such as CAH, formed in this mix (as observed in TGA results), lithified the material, and created a lower HC.

Results from this study should be considered specific to the materials evaluated. Similar studies on materials with different chemical compositions are needed to evaluate the effectiveness of encapsulation for those systems.

Future Research

The next phase of this study is to collect data on environmental performance of each mix design by conducting laboratory leaching studies. This work is ongoing and will be presented in a follow-up report. In addition, detailed mineralogical investigation are under way to better understand the amorphous phases quantified with XRD analysis. By understanding mineral assemblages, the underlying chemical reactions can be better understood. In addition to this environmental and mineralogical testing, geochemical modeling is under way to better interpret the data.

Beyond additional lab studies, a field study is commencing to observe mix designs similar to those presented in this study at a larger scale. Field lysimeters will be created and left for long-term onsite monitoring of runoff and leachate water quantity and quality. In addition, a weather station and moisture sensors embedded in the lysimeters will be used.

A

ADDITIONAL INGREDIENT MATERIAL PROPERTIES

Tables A-1 and A-2 summarize the results from thermogravimetric analysis of the filtered brine solids, quicklime, portland cement (PC) and fly ash. The samples were gradually heated from ambient room temperature to 900°C and sample weight loss was monitored throughout the heating regime. Heating intervals with negative weight loss for quicklime in Table A-1 indicate weight increases in the sample most likely as a result of hydration reactions between ambient and 250°C, and then carbonation reactions between 425 and 500°C. Over all, the brine solids showed the most significant total weight loss compared to the as-received solid ingredient materials. The majority of this mass loss was from dehydration of chemically bound water in the brine particles below 250°C. Quicklime, PC, and fly ash each showed similar total mass loss up to 900°C.

Table A-1
Brine solids and quicklime thermogravimetric analysis

Temperature	Measured Weight Loss (%)	Calculated Weight Loss (%)	Possible Cause
Brine Solids			
Wt% loss at ambient to 70°C in N_2	3.68	39.81 (stoichiometric calculations)	Residual moisture, multi-step dehydration of $CaCl_2*2H_2O$, $MgCl_2*6H_20.CaSO_4*2H_20$ Partial dehydration or dihydroxylation of $CaB_3O_3(OH)_5*4H_2O$
Wt% loss at 70–110°C in N_2	17.89		
Wt% loss at 110–250°C in N_2	13.15		
Wt% loss at 250–350°C in N_2	2.63		Dehydration or dihydroxylation of $CaB_3O_3(OH)_5*4H_2O$
Wt% loss at 350–425°C in O_2	1.98		
Wt% loss at 350–500°C in O_2	NA		
Wt% loss at 425–500°C in O_2	1.45		
Wt% loss at 500–650°C in O_2	0.66		Possible decomposition of $CaCO_3$
Wt% loss at 650–900°C in air	0.92		
Total wt loss	42.36		

Table A-1 (continued)
Brine solids and quicklime thermogravimetric analysis

Temperature	Measured Weight Loss (%)	Calculated Weight Loss (%)	Possible Cause
Quicklime			
Wt% loss at ambient to 70°C in N_2		4.4 (7% X-ray diffraction [XRD] + 11.1%)	Hydration of CaO causes weight gain (formation of an additional 11.1 wt% of $Ca(OH)_2$)
Wt% loss at 70–110°C in N_2			
Wt% loss at 110–250°C in N_2	-2.70		
Wt% loss at 250–350°C in N_2	0.1		Dehydration of $Ca(OH)_2$
Wt% loss at 350–425°C in O_2	3.83		
Wt% loss at 350–500°C in O_2	NA		
Wt% loss at 425–500°C in O_2	-0.25		Possible carbonation of CaO (formation of 0.57 wt% $CaCO_3$ from CaO)
Wt% loss at 500–650°C in O_2	1.54	0.69 (1% XRD + 0.57%)	Decomposition of $CaCO_3$
Wt% loss at 650–900°C in air	0.07		
Total Wt loss	**2.59**		

Table A-2
Portland cement and fly ash thermogravimetric analysis

Temperature	Measured Weight Loss (%)	Calculated Weight Loss (%)	Possible Cause
Cement			
Wt% loss at ambient to 70°C in N_2	0.17	0.79 (XRD)	Residual moisture or dehydration
Wt% loss at 70–110°C in N_2	0.33		Dehydration of gypsum
Wt% loss at 110–250°C in N_2	0.25		
Wt% loss at 250–350°C in N_2	0.05		Dehydration of calcium-silicate-hydrate
Wt% loss at 350–500°C in O_2	0.19		
Wt% loss at 500–650°C in O_2	1.53		
Wt% loss at 650–900°C in air	0.08		Decomposition of carbonate
Total Wt loss	**2.60**		

Table A-2 (continued)
Portland cement and fly ash thermogravimetric analysis

Temperature	Measured Weight Loss (%)	Calculated Weight Loss (%)	Possible Cause
Fly Ash			
Wt% loss at ambient to 70°C in N_2	0.23		Residual moisture or dehydration
Wt% loss at 70–110°C in N_2	0.05	0.26 (XRD)	Dehydration of gypsum
Wt% loss at 110–250°C in N_2	0.11		
Wt% loss at 250–350°C in N_2	0.07		
Wt% loss at 350–500°C in O_2	1.27	1.94	Oxidation of unburned carbon
Wt% loss at 500–650°C in O_2	0.83		
Wt% loss at 650–900°C in air	0.04		
Total Wt loss	**2.60**		

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Electric Power Research Institute
3420 Hillview Avenue, Palo Alto, California 94304-1338 • PO Box 10412, Palo Alto, California 94303-0813 • USA
800.313.3774 • 650.855.2121 • askepri@epri.com • www.epri.com