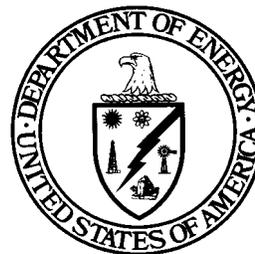# Stabilization of High Salt Waste Using a Cementitious Process

Salt Containing Mixed Waste Treatment

Mixed Waste Focus Area

Stabilization of High Salt Waste Using a Cementitious Process

Salt Containing Mixed Waste Treatment

OST/TMS ID 1683

Mixed Waste Focus Area

Demonstrated at
Oak Ridge National Laboratory (ORNL)
Oak Ridge, Tennessee

Purpose of this document

Innovative Technology Summary Reports are designed to provide potential users with the information they need to quickly determine whether a technology would apply to a particular environmental management problem. They are also designed for readers who may recommend that a technology be considered by prospective users.

Each report describes a technology, system, or process that has been developed and tested with funding from DOE's Office of Science and Technology (OST). A report presents the full range of problems that a technology, system, or process will address and its advantages to the DOE cleanup in terms of system performance, cost, and cleanup effectiveness. Most reports include comparisons to baseline technologies as well as other competing technologies. Information about commercial availability and technology readiness for implementation is also included. Innovative Technology Summary Reports are intended to provide summary information. References for more detailed information are provided in an appendix.

Efforts have been made to provide key data describing the performance, cost, and regulatory acceptance of the technology. If this information was not available at the time of publication, the omission is noted.

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SECTION 1

SUMMARY

Technology Summary

Throughout the Department of Energy (DOE) complex there are large inventories of homogeneous mixed waste solids, such as wastewater treatment residues, fly ashes, and sludges that contain relatively high concentrations (greater than 15% by weight) of salts. The inherent solubility of salts makes traditional treatment of these waste streams difficult, expensive, and challenging. Many of these materials are in a dry granular form and are the by-product of solidifying spent acidic and metal solutions used to recover and reformulate nuclear weapons materials over the past 50 years. At the Idaho National Engineering and Environmental Laboratory (INEEL) alone there is approximately 8,000 cubic meters of nitrate salts (potassium and sodium nitrate) stored aboveground with earthen cover. Current estimates indicate that over 200 million kg of contaminated salt wastes exist at various DOE sites. Continued primary treatment of wastewater coupled with the use of mixed waste incinerators may generate an additional 5 million-kg of salt-containing mixed waste residues each year.

One of the obvious treatment solutions for these wastes is to immobilize the hazardous components to meet Environmental Protection Agency/Resource Conservation and Recovery Act (EPA/RCRA) Land Disposal Restrictions (LDRs), thus rendering the mixed waste to a radioactive waste classification only, if the waste is not listed. One proposed solution is to use thermal treatment via vitrification to immobilize the hazardous component and thereby substantially reduce the volume, as well as provide exceptional durability. However, such melter systems involve expensive capital apparatus with complicated offgas systems. In addition, the vitrification of high salt wastes may cause foaming and usually requires extensive development to specify glass formulation recipes. As an alternative to thermal treatments, stabilization of these materials in basic cement grouts has also been widely employed. However, salts interfere with the basic hydration reactions of cement, leading to an inadequate set and/or deterioration of the waste form over time. Sufficient and compliant stabilization in cement can be achieved by lowering waste loadings, but this involves a large and costly increase in the volume of material requiring handling, transporting, and disposal. As a consequence of these stabilization deficiencies associated with salt-containing mixed wastes, the Mixed Waste Focus Area (MWFA), a DOE Environmental Management (EM-50) program, sponsored the development of five low-temperature stabilization methods as an alternative to simple Portland cement grouting. One alternative is stabilization with enhanced cements, which in some applications provides higher waste loadings, reduction of set problems, and a less leachable waste form than the baseline method of cement grouting.

Enhanced grouting involves the addition of cementitious additives to simple Portland cement to counter the effects of various salts existing in mixed waste. In particular, the additives of fly ash, blast furnace slag, and clays were evaluated at different waste loadings and at different concentrations of nitrate, chloride, and sulfate salts. Two levels of statistically designed experiments were performed in an attempt to quantitatively define the waste form performance response to the four binding constituents (i.e., Portland cement, fly ash, blast furnace slag, clays), as well as to different combinations and concentrations of the three salts. In the tests that are the subject of this report, salt loadings threefold greater than that obtainable with simple cement were achieved for waste forms made with specific amounts and types of the additives. The performance of these waste forms was determined through testing and was found to be acceptable for disposal in regard to compressive strength, free water, and hazardous metal leachability. However, no extensive testing was completed to determine if the use of the additives negated any of the long-term deteriorating effects that salt-containing mixed wastes can have on basic cement matrices.

Demonstration Summary

Under MWFA sponsorship, investigators employed by Lockheed Martin Energy Research Corporation at the DOE's Oak Ridge National Laboratory (ORNL) performed a series of development tests and treatability studies involving the stabilization of salt-containing mixed waste using enhanced cement methods. Specifically, the studies involved the stabilization of both surrogate and actual salt-containing mixed wastes with simple Portland cement and one or more of the three additives: fly ash, granulated blast furnace slag, and a clay plasticizer. The purpose of the testing was to determine if any of the additives could overcome the detrimental effects that salt wastes can have on basic cement waste form properties (e.g., % free water, compressive strength, RCRA hazardous metal leachability) under varying conditions of both salt types (i.e., nitrates, chlorides, sulfates) and salt concentrations.

The development effort involved two levels of statistical testing. The first level consisted of the formulation of over 50 different waste forms at varying levels of the four binder/additive materials (i.e., cement, fly ash, blast furnace slag, clays) and varying levels of the three salts (nitrates, chlorides, sulfates). Based on the waste form performance results of the first statistical test series, a second statistical test series of sequential design was performed. This test series involved the preparation and evaluation of over 30 different waste forms. Among this test population were cementitious waste forms made with surrogate salt waste compositions recommended by the MWFA, as well as with actual wastes from an Oak Ridge operation. The purpose of the sequential design was to allow focusing on waste forms with high salt loadings that were still expected to give acceptable waste form performance as indicated by the results from the original set of experiments. All fabricated waste forms were evaluated against established EPA, RCRA and Nuclear Regulatory Commission (NRC) criteria in regard to free water, compressive strength, and leachability of RCRA hazardous heavy metals, as determined by the toxicity characterization leaching procedure (TCLP).

Results of waste form performance testing indicated that the use of both blast furnace slag and fly ash increased the allowable salt loading in cement, but the use of clays resulted in forms with unacceptably low compressive strength (i.e., less than 500 psi). Furthermore, the test results indicated that the use of blast furnace slag alone could accommodate higher salt waste loadings in disposal compliant waste forms. For example, a dry binder blend of only 90% blast furnace slag and 10 wt% cement was mixed with water and an MWFA recommended surrogate waste to obtain a waste form containing over 40 wt% nitrate salts (i.e., ~2.7 times greater than that obtainable with just Portland cement). The final waste form had no free water, a compressive strength of over 1,000 psi, and passed the RCRA hazardous metals TCLP test at the newer and more stringent Universal Treatment Standards (UTS) levels. In another test case, a cement/slag waste form containing 76 wt% of an actual Oak Ridge electrochemical machining (ECM) mixed waste residue spiked with heavy metals and salts was prepared and evaluated. The dry binder blends contained cement and slag in proportions of ~1:1 and the final waste form contained over 30-wt% of a combination of nitrate, chloride, and sulfate salts. The waste form contained no free liquids, barely passed the compressive strength criteria of 500 psi, but easily passed the RCRA heavy metal TCLP test at the more stringent UTS levels.

As a consequence of collecting performance data on over 80 different waste forms, an attempt to generate surface response models for predicting waste form performance as a function of both binder (cement, blast furnace slag, fly ash, and clay) and salt (nitrates, chlorides, sulfates) content was made. Based on a detailed statistical analysis of the data, linear model equations for predicting both compressive strength and percent of chromium metal extracted were determined. The response of the other performance properties (i.e., free water, TCLP performance of the other RCRA metals) was flat over the region of conditions tested. Therefore, these performance indicators did not appear to depend on the binder, salt types, or concentrations. However, the models are only applicable for wastes similar to the MWFA recommended surrogates or the Oak Ridge ECM mixed waste sludge.

The overall development effort as reported in this ITSR validates that the mature and inexpensive technology of cement grouting can be effectively used for salt-containing mixed wastes. Through the use of additives, especially blast furnace slags, the basic cement stabilization restrictions of low salt waste loadings and subsequent large final disposal volumes can be avoided.

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Other

All published Innovative Technology Summary Reports are available on the OST Web site at http://em-50.em.doe.gov under "Publications." The Technology Management System, also available through the OST Web site, provides information about OST programs, technologies, and problems. The OST Reference Number for the enhanced cement grout process is 1683.

SECTION 2

TECHNOLOGY DESCRIPTION

Overall Process Definition

The use of basic Portland cement as a waste solidification and stabilization medium has been in practice for decades. Since cement solidification occurs at low temperatures, generates no offgases, chemically bounds aqueous wastes to its matrix, results in no free water, and is relatively inexpensive, it is an excellent choice for the stabilization of many mixed waste types.

Basic dry Portland cement binder powder is formed by calcining natural limestone ($CaCO_3$) and clay materials at 1,400 to1, 500°C. The resultant clinker product usually consists of specific combinations of three basic oxides: silica (SiO_2), lime (CaO), and alumina (Al_2O_3). For most Portland cements, the specific oxide combination consists of at least the following three crystalline compounds:

Tricalcium silicate	$3CaO\text{-}SiO_2$
Dicalcium silicate	$2CaO\text{-}SiO_2$
Tricalcium Aluminate	$3CaO\text{-}Al_2O_3$

For most cements the three compounds above make up ~80% of the dry matrix, and their proportions determine which of the eight American Society of Testing and Materials (ASTM) standard types of Portland cements is formulated. Sulfur containing gypsum, $CaSO_4$, is also added to most Portland cements. The role of the gypsum is to slow down the cement setting during hydration (i.e., the addition of water). The tricalcium aluminates and gypsum react with water to form a mineral (i.e., ettringite) and gel that coats the remaining unhydrated cement clinkers. Setting is slowed since the water has to now diffuse through the mineral barrier to hydrate the remaining cement clinkers. This allows the cement waste mix to be workable before complete setting. Under normal conditions, the rate of hydration is such that after 28 days only two-thirds of the cement has reacted with water.

The hydration reactions between the dry basic cement clinker, the waste, and any added water can be complex, and even the most simple cement hydration mechanisms are not yet fully understood. In general, the bulk of the crystalline calcium silicates in the cement clinkers react with water to form amorphous hydrated calcium silicates (also known as tobermorite gels or $3CaO\text{-}2SiO_2 - 3H_2O$), and crystalline slaked lime [also known as calcium hydroxide ($Ca(OH)_2$) or Portlandite]. The tobermorite gel, which causes setting, is the main cementing component of the waste form and the presence of the soluble slaked lime in the pores causes the cement form to be basic (pH ~11.5). As a favorable consequence of the high pH, the acetic acid solution used as an extract in the TCLP test will be immediately neutralized and will thus lose its ability to leach any hazardous RCRA metals. The presence of the hydroxide will also result in the favorable conversion of any RCRA hazardous metals present in the waste to their less soluble hydroxide form. Unfortunately, the slaked lime is also soluble, and over time will diffuse through the pores and leach out of the cement waste form. Since the $Ca(OH)_2$ can constitute over 30% of the waste form, its removal from the waste monolith can seriously degrade it.

During cement hydration and the onset of setting, some of the tobermorite gel is formed from a sol (i.e., a homogeneous dispersion of fine solids in a liquid) that follows the principles of colloidal chemistry. According to these principles, the formed tobermorite sol coagulates into floccules or gel substances. The gel then precipitates to cementing solids once the static charge is lost. Because this precipitation or setting step is greatly affected by the ionic strength of the sol, the presence of salts can vastly impact the cement's setting rate in either direction (i.e., accelerate or retard the set). Once all the gel has settled it begins to dry and crystallize in what is designated as "the cement hardening or curing phase." During this curing phase, crystalline slabs and needles are created that decrease the porosity and increase the strength of the waste form.

In addition to the short-term effect of altering setting rates, salts can also have long–term impacts on the integrity of the cement waste form. Many different types of salts can react with the slaked lime and

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hydrated calcium aluminates existing in the pores of the cured cement waste form. The undesired product of these reactions is usually that of an expansive mineral that can cause pores to crack.

Based on an understanding of the fundamental mechanisms and products of simple cement hydration, additives to the cement-waste mix were chosen in an effort to negate, combat or counter the negative effects of stabilizing mixed wastes containing salts. The cementitious additives chosen, as described below, were ground granulated blast furnace slag, fly ash, and a clay plasticizer. These three additives are often used in specialty formulations for cementitious, glassy, and ceramic materials in industrial and commercial applications. Although they can contain RCRA toxic metals themselves, as of late 1999, blast furnace slag by-product from iron manufacturing and fly ash from coal combustion were specifically excluded from the RCRA hazardous waste category by 40 CFR sections 261.4(b)(7) and 261.4(b)(4), respectively.

Ground Granulated Blast Furnace Slag

Blast furnace slag is produced as a by-product of the iron and steel production industries. Its earthly constituents come from iron ore processing and it consists of the same oxides as Portland cement, but in different proportions. Immediately after its production, slag is usually quenched for rapid cooling in a process known as granulation. The granulation results in a reactive amorphous glass and avoids any crystallization. Like Portland cement, blast furnace slag also reacts with water (i.e., is hydrated) to form specific hydrated calcium silicates known as tobermorite gels. However, unlike basic Portland cement, it forms this critical cementing agent (tobermorite gel) by consuming the slaked lime, $Ca(OH)_2$, provided by the hydration of the Portland cement. Removal of some of the slaked lime is advantageous since less of it in the waste form will lead to less dissolution of the lime over time, and thus consequently less long –term waste form degradation. In addition, there will be less slaked lime available to potentially react with salts, and thus produce undesired expansive and destructive minerals in the future.

Additionally, it has been postulated that since blast furnace slag is amorphous and not crystalline, it hydrates to tobermorite gel without forming the colloidal solutions common to the calcium silicates in basic Portland cement. Since sols are not formed, the ionic nature of the salts should not affect the tobermorite gel formation or set rate of the waste form.

Flyash

Fly ash has been used as a low cost cement substitute in cement formulations for several decades and it modifies the cement formulation by improving workability and lowering the heat of hydration. Similar to blast furnace slag, the fly ash generated from the combustion of coal is of the class of cement additives referred to as artificial pozzolans. In general, pozzolans are naturally occurring substances or are substances produced, as ore processing industry by products that also have cementlike characteristics. As such, the use of these additives is cost- effective since a special manufacturing step is not required, as in the case of Portland cement. The fly ash generated from coal combustion and other pozzolans contain a glassy silica phase that reacts with slaked lime in cements during hydration to form calcium silicate hydrate (CSH) gels, the main setting agent of cement, that also include tobermorite gel. Unlike Portland cement, this reaction does not seem to be affected by the presence of an ionic field generated by any existing salts in the mixed waste. Like blast furnace slag, fly ash is activated to hydrate to CSH by consuming slaked lime. As described above, the removal of lime increases the waste form's durability over time by reducing the risk of future salt initiated mineral expansions.

Clay Plasticizers

Examples of clay additives that have been used in basic Portland cement waste matrices include bentonite, attapulgite, naphthalene sulphonate, and melamine-formaldehyde. These clays have the property of being elastic and when added to the cement-waste mix will reduce its rigidity on complete curing. As such, their addition may compensate for any future salt initiated mineral expansions in the pores of the form's microstructure. A waste form more forgiving of future mineral expansions may be at less risk to deteriorate or crack, and thus provide no easy leach pathways for radiological or hazardous components.

System Operation

Operational systems for deploying enhanced cementitious mixed waste stabilization processes are identical to those for basic Portland cement, and involve simple, readily available equipment. Basic equipment components of any system will involve waste feed, cement, and additive hoppers, waste feed transfer pumps, a mixer, a mixing vessel, and/or disposal containers. Existing DOE Portland cement systems at both the INEEL and the West Valley Demonstration Facility could be easily modified to handle the addition of the blast furnace slag, fly ash, and /or clay platicizers needed for an enhanced process. These various chosen additives could be preblended with Portland cement and fed from a designated single feed hopper, or dedicated feed hoppers for each additive could be installed and employed. Hopper weighing probes are used in all systems to ensure the mixing of accurate amounts of waste and additives.

In-container or inline mixer systems are applicable. Many existing systems in the non-DOE hazardous waste industry are inline, where the cement, additives, waste, and water are added to a dedicated mixer and then transferred, upon the onset of hardening, to a final disposal container. Fifty-five gallon batch systems are most common, but continuous operations are possible for consistent and homogeneous mixed waste streams. To ensure disposal compliant waste forms and quality control, samples of the mix are usually taken when transferring from the mixer to the final disposal container.

In-container systems are those that mix the cement, additives, water, and mixed waste in the same drum to be used for disposal. They include drum tumblers, disposable mixers, or removable mixer systems. A popular removable mixer system involves a 55-gallon drum-scale planetary mixer, where hydraulics are used to lower and raise the mixing blades for drum handling, insertion, and removal. Regardless of the chosen system, high shear, high-speed mixers are recommended.

Operational protocol sometimes involves the addition of the premeasured and required amounts of blast furnace slag, cement, fly ash, and/or clays to the drum mixer followed by alternating additions of waste and water to control heat evolution. However, cement and additives are frequently added to precharged waste to control mixing and ensure the best waste loadings. Mix times are usually an hour followed by several days of setting and curing before disposal.

SECTION 3

PERFORMANCE

Demonstration Plan

Numerous surrogate and actual waste stabilization tests involving enhanced cement formulations were completed to identify the collective effect of blast furnace slag, fly ash, and clays in increasing salt waste loadings. Specifically, the waste form performance effects of salt loading were initially evaluated by conducting two multidimensional experiments. These tests were then followed by basic coordinatewise designed experiments where one component was varied, while the others were held constant. The variable range for this set of experiments was narrowed to a small region as defined from the initial two experiments.

The first set of multidimensional experiments involved the preparation of over 50 surrogate test waste forms. Waste loadings ranged from 20 to 60wt% with corresponding salt loadings of 6 to 42-wt%. The composition of each waste form was determined and varied based on a statistical method defined mathematically as a D-optimal experimental design. The design allows the obtaining of informative and correlating data without testing all possible combinations of the four binder additives and the three salts. As a result of the D-optimal design, the 51 waste form tests consisted of the following binder combinations: 10 with all four binders, nine with cement and fly ash binders only, nine with cement and clay only, seven with cement and blast furnace slag only, six with all binders except fly ash, four with all binders except slag, three with all binders except clay, and three with Portland cement only. As a consequence of the experimental design, each of the 8-binder test groups listed above also consisted of varying binder proportions, as well as different combinations and concentrations of the three salts.

The compressive strength, free water, and leachability of all waste forms produced from the above experimental design were determined. Based on the performance of these properties relative to the established EPA/RCRA and NRC criteria, 32 additional waste forms were prepared under varying conditions as based on a simple coordinatewise, D-optimal sequential design. For this set of experiments, 16 waste forms were prepared incorporating the recommended MWFA salt surrogates and the remaining 16 were prepared with actual mixed waste residues from an Oak Ridge ECM operation. Waste loadings ranged from 39 to 60-wt% (i.e., the upper end of the multidimensional experiments), with corresponding salt loadings of 27 to 42-wt%. The compositions of the two recommended MWFA surrogates are provided in Table 1. One of the surrogates waste streams contained a high quantity of nitrate salts and represented a waste stream that had previously been unsuccessfully stabilized with simple Portland cement. Such waste streams are not uncommon in the DOE complex. The other surrogate waste stream contained salts of chloride and sulfate at levels representative of a dried, but not concentrated, spent incinerator offgas scrub solution (i.e., blowdown). Both of the surrogates were free of moisture, contained oxide forms of RCRA heavy metals in the 1,000-ppm range, and contained trichloroethylene as a trace organic contaminant. To obtain comparable waste form performance data, the same surrogate formulations were provided to other investigators involved in parallel efforts to also develop salt waste stabilization alternatives. Hazardous RCRA metal concentrations in the ECM sludge are provided in Table 2. On the average, the ECM sludge contained ~63-wt% water and ~14-wt% nitrate salts.

Based on the waste form performance (i.e., free water, compressive strength, leachability) results of the sequential experimental design involving the 32 waste forms, a final test series of waste forms were prepared involving only the MWFA recommended surrogate salt wastes. This series resulted in preparing a total of 10 waste forms, five involving the nitrate salt surrogate and five involving the chloride/sulfate surrogate. As depicted in Figure 1, all waste forms were prepared with a dry binder consisting of 90-wt % blast furnace slag and 10-wt% simple cement. As indicated by Table 3, salt waste loading fractions ranging between ~0.43 to ~0.77 were tested. This corresponded to salt loadings as high as 46-wt%. All 10 waste forms prepared were then subjected to free water, compressive strength, and leachability testing.

Table 1. Composition of standard Mixed Waste Focus Area (MWFA) recommended surrogate salt sludges

Constituent	High-chloride sludge (wt %)	High-nitrate sludge (wt %)
$Fe(OH)_3$ as Fe_2O_3	12.75	6.00
$Al(OH)_3$	8.50	4.00
Na_3PO_4	4.25	2.00
$Mg(OH)_2$	8.50	4.00
MicroCel E	17.00	8.00
Portland Cement (Type II)	4.25	2.00
H_2O	29.75	14.00
Salts		
NaCl	10.00	0.00
$CaSO_4$	5.00	0.00
$NaNO_3$	0.00	60.00
Total	100.00	100.00
Contaminants (mg/kg)		
Toxic Metal Oxide		
PbO	1,000	1,000
CrO_3	1,000	1,000
HgO	1,000	1,000
CdO	1,000	1,000
NiO	1,000	1,000
Trichloroethylene	1,000	1,000

Table 2. Average, standard deviation, and percentage Relative Standard Deviation (RSD) of the significant total concentrations measured in the electrochemical machining (ECM) sludge samples and toxicity characterization leaching procedure (TCLP) extract concentrations of bucket composite samples

Bucket	Sample	Moisture (wt %)	Total sludge concentration (mg/kg)			TCLP extract concentration (mg/L)	
			Chromium	Nickel	Nitrate	Chromium	Nickel
1	Average	63.62[a]	113	6.80	143,200	0.09	0.26
	Std. Dev.	0.75	2	0.36	2,482		
	% RSD	1.19%	1.58%	5.34%	1.73%		
2	Average	61.86[b]	135	7.92	143,600	0.32	0.26
	Std. Dev.	0.24	2	1.64	2,728		
	% RSD	0.39%	1.24%	20.68%	1.90%		
3	Average	63.64[c]	94	6.47	137,600	<0.03	0.25
	Std. Dev.	2.24	3	0.65	3,382		
	% RSD	3.52%	2.82%	10.05%	2.46%		
4	Average	64.26[d]	97	7.35	140,400	0.31	0.24
	Std. Dev.	0.46	4	0.43	2,332		
	% RSD	0.72%	4.28%	5.82%	1.66%		

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Table 3. Compositions of grouts made from the standard Mixed Waste Focus Area (MWFA) surrogate sludges using a dry blend of 90:10 slag cement

Test	Nominal standard sludge loading (fraction)	Composition in the final grout (mass fraction)							
		Actual Standard Sludge Loading (fraction)	Dry Blend	Added Water	Total Wet Sludge Loading Standard + Added Water	Salts			
						NaCl	CaSO$_4$	NaNO$_3$	Subtotal
		**Standard high chloride MWFA surrogate sludge**							
1	0.90	0.6766	0.0744	0.2489	0.9256	0.07	0.03		0.10
2	0.80	0.5493	0.1373	0.3134	0.8627	0.05	0.03		0.08
3	0.70	0.4904	0.2102	0.2995	0.7898	0.05	0.02		0.07
4	0.60	0.4199	0.2799	0.3002	0.7201	0.04	0.02		0.06
5	0.50	0.3581	0.3581	0.2838	0.6419	0.04	0.02		0.06
		**Standard high nitrate MWFA surrogate sludge**							
6	0.90	0.7673	0.0847	0.1480	0.9153			0.46	0.46
7	0.80	0.6892	0.1723	0.1385	0.8277			0.41	0.41
8	0.70	0.6076	0.2604	0.1319	0.7396			0.36	0.36
9	0.60	0.5167	0.3445	0.1388	0.6555			0.31	0.31
10	0.50	0.4276	0.4276	0.1447	0.5724			0.26	0.26

Results

Results of the multidimensional experiment involving the preparation of 51 different waste forms gave insight into the specific effects that each of the binder additives had on waste form properties and performance. Nine of the waste form specimens failed to obtain the minimum NRC compressive strength criteria of 500 psi and five more marginally passed. All of these waste forms notably contained clay as an additive binder. To the contrary, those waste forms that contained exclusively fly ash as an additive had compressive strengths in excess of 4,000 psi. Waste forms containing blast furnace slag provided adequate compressive strength as long as the waste loading was not excessive. Nineteen of the 51 waste form test specimens failed the TCLP leachability test for the hazardous heavy metal cadmium. In most of these cases the waste form lacked the presence of blast furnace slag.

As a consequence of the above results, the sequential experimental design involving the formulation of 32 waste forms (of either ECM sludge or the MWFA recommended salt surrogates) used neither fly ash nor clays. The test not only involved varying waste loadings at the upper end, but also the fraction of blast furnace slag mixed with the basic cement binder. Slag content was varied between 20 and 85-wt%.

Only six of the 32 waste form test specimens passed both the compressive strength and leachability criteria that have been established by the EPA/RCRA and NRC. In the majority of these test cases, the fraction of blast furnace slag in the dry binder mix exceeded 50-wt%.

Based on the results of the sequential design experiments, a final series of waste forms were prepared using a slag/cement binder mix of 9:1. The tests involved only the two types of MWFA recommended salt surrogates and resulted in the preparation of waste forms with waste loadings between ~43-wt% and ~77-wt%. As indicated by Table 4, eight of the 10 waste forms prepared passed the compressive strength test criteria level of 500 psig. Only those at the higher waste loadings failed. In regard to leachability, only the waste form prepared with a high loading of the chloride/sulfate surrogate failed for chromium. All other nine waste form test specimens passed at the stringent UTS levels.

Table 4. Waste form performance test results for the standard Mixed Waste Focus Area (MWFA) surrogate sludges

Test	Grout density g/ml	Volume ratio grout/sludge		28-d free water (vol. %)	28-d unconfined compressive strength (psi)				
		Standard surrogate sludge	Total wet sludge standard + added water		1	2	3	Average	Standard deviation
Standard high chloride MWFA surrogate sludge									
1	1.53	0.50	1.03	0.00	578	479	550	535	42
2	1.50	0.63	1.06	0.00	641	664	647	651	10
3	1.56	0.68	1.10	0.00	1,074	1,072	1,108	1,085	16
4	1.56	0.80	1.19	0.00	1,599	1,430	1,536	1,522	70
5	1.63	0.89	1.26	0.00	2,215	2,118	2,185	2,173	41
Standard High Nitrate MWFA Surrogate Sludge									
6	1.76	0.39	1.06	0.00	210	260	218	230	22
7	1.80	0.42	1.13	0.00	995	1,065	1,036	1,032	29
8	1.86	0.46	1.22	0.00	1,973	2,075	1,945	1,998	56
9	1.99	0.51	1.25	0.00	2,973	3,068	3,120	3,053	61
10	1.60	0.76	1.72	0.00	3,858	3,513	3,253	3,541	248

Figure 1. Stages of the enhanced cement solidification process for the Mixed Waste Focus Area (MWFA) recommended nitrate salt waste surrogate.

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TECHNOLOGY APPLICABILITY AND ALTERNATIVES

Competing Technologies

Over the last 50 years of DOE operations, large quantities of mixed low-level wastes (MLLW) were generated from past nuclear weapons based industries and more recently from waste treatment processes. Stabilization has been and still is an effective, inexpensive, and simple treatment alternative for many types of these mixed wastes. This alternative is also accepted as safe and environmentally sound by both the regulators and the concerned public. A review of the technical literature and of past DOE operating experience shows that simple low-temperature hydraulic cement, polymer, and bitumen based stabilization methods produce waste forms that meet or exceed final disposal requirements. The success level of the waste form is dependent on the original waste medium and the type and amount of hazardous and/or troublesome components in the untreated waste. Unfortunately, these current stabilization techniques have had limited success in accommodating homogeneous solid and sludge wastes containing relatively high concentrations of salts, in addition to RCRA hazardous heavy metals.

Salts (defined as the reaction product generated when a metal ion replaces the hydrogen ion of an acid) are highly soluble, and reactive. As a consequence of these characteristics, low- temperature stabilized forms of MLLW containing salts do not adequately set or are susceptible to deterioration over time due to the salt mineral expansions in the macropores of the concrete microstructure. This deterioration may lower the durability and strength of the stabilized waste form and create pathways for the hazardous and radiological constituents to be released from the immobilized waste.

In some cases, leach resistant salt waste forms of sufficient durability are possible with simple cement stabilization. However, these techniques usually result in forms with excessive increases in waste volume due to low waste loadings. Basic Portland cement formulations can only accommodate ~15 wt% of salts. These process inefficiencies and subsequent high disposal costs offset any benefits. The limitations of these current methods are of immediate concern since future volumes of salt wastes are anticipated as other MLLW treatment processes are implemented. Future effluents from MLLW wastewater treatment systems and scrubber blowdown from future and present MLLW thermal systems (i.e., incinerators and melters) will significantly add to the MLLW salt inventory.

There are many mixed waste stabilization/solidification technologies at various stages of development that could be considered as competing with the enhanced cement grout process described in this ITSR. Numerous tests with low-temperature stabilization techniques involving ceramics and polymers indicate that greater waste loadings (than those achievable with conventional Portland cement) are possible with even the troublesome salt-containing wastes. In addition, alternatives involving thermal-sintering techniques also may lead to acceptable waste forms with considerably more volume reduction compared to that achievable with basic or enhanced grouts. Mixed waste stabilization methods currently in the later stages of development include phosphate-bonded ceramics, and several methods provided by commercial vendors. Low-temperature methods like phosphate bonded ceramics not only provide a low porosity barrier, but render the RCRA metal less hazardous by converting it to the low solubility phosphate salt. Microencapsulation techniques involving polyesters, polyethylene, polycerams sol gel systems, and polysiloxane have been demonstrated on surrogate and/or actual wastes.

Sintering differs from vitrification in that only melting at grain phase boundaries occur without the complete amorphous restructuring that takes place in glass formation. Like vitrification, sintering occurs at temperatures over $1,000^0$C and can emit volatile hazardous metal vapors. Even though densification is possible for some additional volume reduction, slight volume increases usually occur. However, waste loadings as high as 80% are possible. The equipment for sintering is less complex than vitrification, but more complex than grouting. For a typical sintering process, grinding, mixing, and extruding equipment are required, as well as ovens, calciners, and offgas treatment systems. For most waste streams, sintering methods will require an extensive process development effort involving statistically designed experiments.
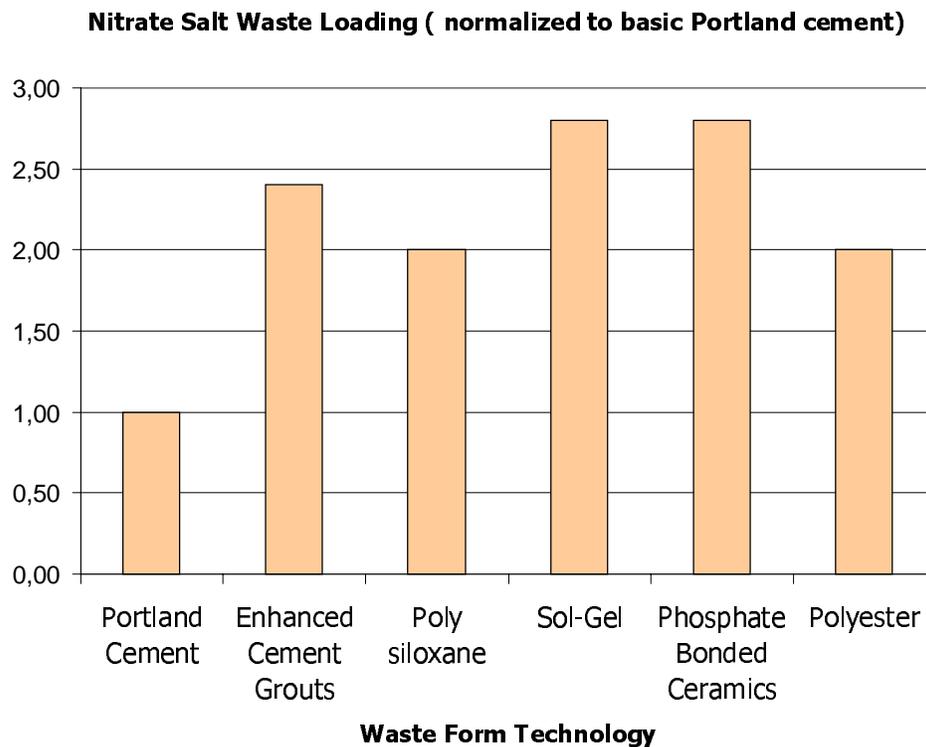
Recently developed polymeric methods using batch mixers or extruder systems are currently available. These low-temperature microencapsulation techniques do not chemically incorporate the waste, but create an impermeable barrier between the hazardous components in the waste and the environment. Waste loadings in these organic media are usually on the order of 50% for many troublesome wastes, such as incinerator fly ash or those containing appreciable salts. This value is nearly twice that achievable with conventional cement grout methods.

Table 5 and Figure 2 give 'head-to-head' comparisons of the waste form performance between that of enhanced cement stabilization and some of the other more recently developed salt-containing mixed waste stabilization methods as described above. Table 5 provides waste loading, strength, and leachability results for five low-temperature technologies stabilizing the same two types of salt-waste surrogates. The pass criteria for TCLP leachability in Table 5 are the less stringent toxicity characteristic levels given in 40 CFR 261 for determining whether a waste bears an EPA hazardous waste code for cadmium, chromium, mercury or lead. Figure 2 graphically represents the waste loading benefit achieved over simple Portland cement grouting when using enhanced cement stabilization or any of the other recently developed methods. The values have been normalized to the approximate salt-waste loading limit (i.e., 15-wt%) of basic Portland cement. Therefore, the value of 2.4 for the enhanced cement process represents a 36-wt% (i.e. 2.4 x 15-wt%) salt loading (e.g., a 60-wt% loading of a surrogate waste containing 60-wt% nitrate salts).

Table 5. Comparison of waste loading and waste form performance for stabilization of Mixed Waste Focus Area (MWFA) NO_3 and Cl salt waste surrogates using enhanced concrete and three other stabilization technologies

Waste Form Performance Measure	Waste Loading wt%	Salt Loading wt%	Compressive Strength Psi	Results of TCLP Leachability test for Cd, Cr, Hg, and Pb Toxicity Characteristic	Leachability ANSI 16.1 index
Surrogate Waste Type	NO_3/Cl	NO_3/Cl	NO_3/Cl	NO_3/Cl	NO_3/Cl
Technology					
Enhanced Concrete	**60/55**	**36.3/8.2**	**1,032/535**	**Passed for Cd, Cr, Ni, Hg and Pb**	**NA**
Polyester (vinyl ester resin only)	50/50	30/7.5	6,200/5,100	Passed for Cr, Pb, Hg	7.7/7.6 for Na ions
Polysiloxane	50/50	30/7.5	420/>637	Passed for Cd, Cr, Hg	NA
Phosphate Bonded Ceramics	70/58	42/9.0	3,500/1,800	Passed for Cd, Cr, Pb, Hg,	7.1 for Cl 9.0 for NO_3
Sol-gel	70/60	43.5/11.5	1,513/1,050	Passed for Cd, Cr, and Hg	7.6/7.5 for Na ions

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Nitrate Salt Waste Loading (normalized to basic Portland cement)

Waste Form Technology

Alternatives and Portland Cement Grouting

As indicated by the comparison data in Table 5 and Figure 2, no alternative, low-temperature stabilization technology for salt waste clearly outperforms the others, although the alternatives clearly outperformed simple Portland cement. Potential end users may need to consider other factors outside of waste form performance in choosing an alternative. These factors include, but are not limited to, the issue of 'stabilization versus encapsulation' technology, the availability of equipment, previous operating experience, the applicability of the technology to other types of waste media, and issues involving both safety and stakeholder concerns.

Technology Applicability

Cementitious stabilization processes involving the additives of blast furnace slag, fly ash, and/or clay platicizers are applicable to a variety of mixed waste streams, including salt sludges, spent metal plating sludges, soils, wastewater treatment residues, fly ash from mixed waste incinerators, and spent offgas scrub solutions. Enhanced cementitious methods are also potentially applicable to crushed debris waste, paint chips, and spent activated carbon beds, as long as there are not excessive amounts of organics present. The method is not applicable to large debris, oily sludges, organic liquids, reactives, and /or explosives.

For over 40 years, cement and cement admixture combinations have been used to stabilize low-level wastes generated by the nuclear power industry. Specific wastes include ion exchange resins, precoat filters, and evaporator condensates. Cement and cement –pozzolan stabilization methods employing fly ash, blast furnace slags, and clays have also been widely used in the hazardous waste industry. Their successful application to wastewater treatment residues containing hazardous heavy metals and trace organics is well documented.

Patents/Commercialization/Sponsor

Basic Portland cement stabilization operations for various waste materials has been in practice for over 50 years and as a consequence is not protected by any patents. However the application of mixing blast furnace slag, fly ash, and/or clays to cement specifically for the stabilization of salt containing mixed waste may be unique. As a consequence, the ORNL principal investigator has recently sought some protection by submitting a disclosure on the enhanced cement method to the United States Patent Office.

Similar cement –admixture stabilization technologies may be available through commercial vendors. One potential supplier is Adtech, a division of the Japanese Gasoline Corporation (JGC). Their trade literature refers to a "super cement," which consists of blast furnace slag, cement, and proprietary ingredients.

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SECTION 5

COST

Methodology

Cost estimates for deploying an operational-scale system for the enhanced cement stabilization of mixed wastes are based on previously established systems for just basic Portland cement methods, particularly, if these existing operational-scale systems include planetary type mixers. Capital, labor, and material cost estimates assumes a batch 55-gallon drum capacity system with a waste form production rate of three barrels per shift, --- or nine barrels a day for an 'around the clock' operation. This rate corresponds to a waste throughput of ~1.70 cubic meter per day, assuming an average waste-to-waste form volume expansion of ~10%. The mixed waste throughput is based on waste form performance data collected during the development of the enhanced cement process as reported in this ITSR. These data indicate that the enhanced cement waste forms can consistently and reliably maintain a 60 wt-% loading of dry mixed waste containing a 60 wt% salt component (i.e., the final waste form contains 36 wt% of the salt). This loading is approximately 2.4 times that achievable with the basic Portland cement process.

Labor costs assume four Full-Time Equivalents (FTEs) at the fully loaded rate of $70.00 per hour. Labor accounted for by the four FTEs include two full-time operational technicians, as well as part-time management, engineering, safety, maintenance, health physics, manifesting, and transportation personnel. The cost for the enhanced cement additive of blast furnace slag is assumed at the supplier bulk rate of ~ $0.02/lb. In comparison, the cost of basic Portland cement is approximately $0.05/lb. Therefore, assuming a dry blend mix of cement and blast furnace slag in the mass proportions of 10: 90, the total binder cost is estimated at ~ $0.03/lb. New standard DOT 7A barrels are estimated at $100.00 each and the assumed operation consists of using the same drum for the mixing, curing, and final disposal of each 55-gallon batch.

Disposal costs vary depending on which available low-level waste only, Subtitle D disposal site is used. At the INEEL's Radioactive Waste Management Complex (RWMC), a relatively low cost of $20 per cubic feet of waste form is available, whereas at the Barnwell Site in South Carolina costs can run as high as $1,500 per cubic foot of waste form. Commercial sites, such as Envirocare of Utah, charge ~ $35 per cubic foot following a one time waste fee of $20,000. For this particular analysis, a value of $60 per cubic foot of waste form was chosen, since it represents an average, as well as a number frequently used in cost and system engineering studies on mixed waste management throughout the DOE complex. Therefore, assuming that the densities of the enhanced cement and cementitious waste forms are 1,800 kg/m^3 and 2,650 kg/m^3 respectively, the cost of disposing an equal mass of waste in an enhanced cement matrix is 65% that of just basic Portland cement.

Cost Analysis

Available cost documentation for existing or planned stabilization systems at Argonne National Laboratory-East (ANL-E) and INEEL indicate capital costs, including equipment design and development, total ~$2,000k. The capital cost estimate assumes a 55-gallon batch system and also assumes available infrastructure and facility housing to support the system. Operating costs, which include labor and material costs, are estimated at ~ $3,643 per cubic meter of enhanced cement waste form based on the data provided in the above methodology. A corresponding cost for the baseline simple Portland cement process is ~$4,300 per cubic meter of waste form because of its slightly higher binder costs. The disposal cost of the enhanced cement waste form (~$2,335 per cubic meter of waste) is also less than that for cement (~$3,700) as a result of its accommodation of higher waste loadings. All estimates are based on immobilizing a sodium nitrate sludge waste with a stabilized specific gravity (Sp. Gr.) of 2.0 and a dry bulk waste Sp.Gr. of 0.7.

Cost Conclusions

The cost-effectiveness of an enhanced cement stabilization process is best determined by comparing it to the baseline process, simple Portland cement stabilization. A summary of the costs and associated data calculated for the two waste forms are provided in Table 6. The assumption that labor and capital costs for the two processes are the same is a valid one considering that for a given waste throughput, the processing times, batch capacities, and types and sizes of equipment are similar if not identical. Therefore, the cost benefit of enhanced cement stabilization over that of the baseline is based exclusively on material, development, and disposal costs. The benefits of using blast furnace slag over cement are two-fold. The use of blast furnace slag allows more waste to be treated on a weight basis for every drum of waste form produced. In addition, the unit cost and amount of blast furnace slag, for most applications, will be less in comparison to cement stabilizations. Cost estimates for treating waste volumes over 25 cubic meters indicate that with the high waste loadings achievable with enhanced cements, the process more than recovers the higher initial material and development costs. For small waste volumes, higher waste loading becomes less of a factor and simple cement stabilization may be just as cost-effective.

Table 6. Calculated costs, material, and performance data estimates for comparing enhanced cement and cement waste forms

	Enhanced Cement Waste Form	Cement Waste Form
Waste Form Production Rate bbls/day	9	9
Gals/day	495	495
Cubic meters/day	1.874	1.874
Kg/day	3,279	4,994
Waste-to-waste form volume expansion	10%	75%
Waste Throughput cubic meters / day	1.70	1.07
Kg/day	1,147	749
Waste Loading	35-wt%	15-wt%
Blast Furnace Slag Use kg/day	1,918	0
Cubic meters/ day	1.28	0
Blast Furnace Slag: waste weight ratio	1.67	0
Cement : waste weight ratio	0.186	5.66
Blast Furnace Slag: waste form weight ratio	.585	0
Cement :waste form weight ratio	.065	0.85
Labor $/day	6720	6720
$/ kg of waste	5.85	8.97
Barrels $/day	900	900
$/kg of waste	0.78	1.20
Materials-Blast Furnace Slag- $/day	85	0
$/kg of waste	0.08	0
$/kg of waste form	0.03	0
Materials-Cement $/day	21.30	425
$/kg of waste	0.02	0.57
$/kg of waste form	0.01	.09
Disposal $/M^3 of waste form	2118	2118
$/kg of waste	3.46	5.29
$/kg of waste form	0.34	0.80
Total (Labor+Material+Disposal[1])		
$/day	11,696	12,015
$/kg of waste	10.20	16.04

1. Disposal costs on a dollar per day basis is $3,970 for both the enhanced cement and cement methods.

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SECTION 6

REGULATORY AND POLICY ISSUES

Regulatory Considerations

The regulatory goal of any end user deploying the enhanced cement grouting process is to produce cement/slag waste forms that meet applicable RCRA LDR 40 CFR 268.40 treatment standards for land burial.   As for any stabilization treatment of mixed waste, it is important to know the characterization of the waste and stabilization media, research the currently applicable treatment standards, and perform treatability studies as needed to ensure that all applicable treatment standards will be met. The following demonstrate the importance of this precept. The TCLP based treatment standards used in this study for cadmium, chromium, lead, and mercury have decreased significantly since the study and may change again in the future. The mixed wastes to be treated may also contain additional toxic metals not tested in this study, but subject to RCRA LDR treatment standards, either as toxicity characteristics or as underlying hazardous constituents. The slag, fly ash, or clay used in the enhanced stabilization may differ in chemical analysis from that used here, particularly in the toxic metals content. In addition, NRC 10 CFR 61 waste form testing will be necessary if disposal is to be in an NRC licensed facility.

To treat mixed waste, any full-scale, enhanced cement treatment facility will require a Part B RCRA permit or a modification to an existing permit. An air emissions review should be performed to determine the applicability of requirements for emissions monitoring and air emissions permits under the Clean Air Act, including applicable National Environmental Standards for Hazardous Air Pollutants (NESHAPS) and Prevention of Significant Deterioration requirements for ambient air quality. For commercial facilities, NRC or Agreement State licensing, or possibly license modification will be required for radioactive material handling. Additional requirements for applying the enhanced cement grouting process at a federal facility include a National Environmental Policy Act (NEPA) review (a categorical exclusion is most likely to be applied).

If future development of the enhanced cement process requires testing with actual waste streams at a federal facility, a NEPA approval through a categorical treatability study exclusion must be obtained. In addition, the state cognizant environmental agency in which the treatability study is to be performed must be notified 45 days before receiving archived samples for testing. In addition, the regional EPA must be notified.

Safety, Risks, Benefits, and Community Reaction

Enhanced cement/slag stabilization is a relatively simple and safe process. Excessively high temperatures are not achieved (e.g., less than ~65°C), pressures are ambient, and with the exception of the mixing impeller, there are few complex or moving parts.

The process generates no potentially hazardous offgases or secondary wastes, and therefore should bring little or no resistance from community stakeholder groups. These groups may even favor the technology, since it will generate less disposal volume when compared to the baseline simple Portland cement method.

There may be some stakeholder sensitivity in regard to the long-term behavior of the enhanced cement waste form in its disposed state. Efforts should be made to educate and inform the concerned stakeholders that the enhancement additives (e.g., blast furnace slag, fly ash) have reduced the waste form's degradation potential by reacting away a portion of the soluble slaked lime. Leachability data should also be presented to reinforce the fact that hazardous metal constituents are not only bound in the matrix, but have been chemically altered to reduce their solubility and mobility.

Although the use of the additives may negate the later salt reactions that form expanding minerals, no archiving of waste forms has occurred to validate this behavior through long-term waste form performance testing. This may also raise stakeholder concerns, since the expansions could cause cracks and subsequent radiological component migration.

LESSONS LEARNED

Implementation Considerations

Given its operational similarity to that of basic Portland cement stabilization, implementation of enhanced cement technology into full-scale deployment for mixed wastes should be fairly straightforward. Designers of enhanced cement systems will need to choose among the numerous mixer and system options. Planetary mixer systems have been deployed in the DOE complex, but commercial entities are deploying alternative systems involving tumblers and subsequent pouring into disposal drums before setting. Decisions regarding the use of one blend hopper for all binders or separate hoppers for each binder will be required and will depend on the variety in the composition and types of mixed wastes to be treated by the system. Potential end users will also need to provide analytical and development support in order to qualify wastes for disposal and verify the operating parameters for new waste streams. Potential technology deployers will also have to give consideration to the need for any up-front waste pretreatment or sizing processes.

Technology Limitations and Needs for Future Development

Based on the results of the enhanced cement/ salt waste form testing reported in this ITSR, the following development on enhanced cement waste forms needs to be continued:

1. Experiments involving a wide range of salt types and concentrations. Only nitrates, sulfates and chloride salts have been extensively studied and it is well documented that inorganic salts affect the set rate either through acceleration or retardation. However, end users need to know precisely how different salts individually and collectively affect basic Portland cement stabilization so that the proper additive can be used in the dry binder mix.

2. Long –term performance testing (i.e., compressive strength, leachability) of archived waste forms made with the enhanced cement stabilization method. Testing is required to validate that the use of slag and fly ash reduces the potential for salts to react and produce expanding minerals in the form's macropores.

Technology Selection Considerations

Obviously, DOE complexwide end users with the responsibility of mixed waste management need to consider multiple factors when selecting a low-temperature stabilization technology like the enhanced cement stabilization method. The most important factors are usually total waste volume, technology simplicity, and stakeholder concerns.

Waste Volume

The greater the volume of a relatively homogeneous salt, mixed waste inventory, the greater the benefits of a cement grout alternative, like the enhanced cement stabilization method. The more waste treated, the greater the savings in handling, transportation, and disposal costs as a result of the greater waste loadings enhanced cement waste forms can provide. If there is sufficient waste volume, these greater savings can more than recover the higher up-front development and material costs.

Technology Simplicity

Very limited engineering development would be required to build a prototype enhanced cement stabilization facility, which could eventually be expanded into a full production operation capable of handling a variety of waste streams. No major investments would be required to perform engineering design studies; rather, the pilot plant could be designed using system integration of off-the-shelf

equipment. In most, if not in all cases, enhanced cement stabilization systems could be retrofitted from basic Portland cement systems.

Stakeholder Concerns

In general, stakeholders desire low–temperature, nonoffgas producing stabilization technologies that generate no secondary wastes, minimize disposal volumes, and ensure long- term durability. Enhanced cement stabilization meets the first four criteria, but very little data exist to support its long-term effectiveness in maintaining durability over its indefinite disposal life. Critics of the technology question an enhanced cement waste form's ability to remain durable over time, especially after stabilizing wastes containing high levels of salts and RCRA hazardous metals.

APPENDIX A

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APPENDIX B

TMS DATA ELEMENTS

Funding Source

This section provides cross-reference information in regards to the EM-50 Mixed Waste Focus Area contract established for development of the enhanced cement salt-containing mixed waste stabilization process. The Department of Energy- Headquarters (DOE-HQ) Technology Management System (TMS) title and tracking number is provided, as well as that of the specific Technical Task Plan (TTP).

> TMS # 1683 Stabilization of High Salt Waste using a Cementitious Process
> TTP # OR16MW41/42 Cementitious Stabilization with High Salt Wastes

APPENDIX C

ACRONYMS

ANL-E	Argonne National Laboratory-East
ASTM	American Society of Testing and Materials
CFR	Code of Federal Regulations
CSH	Calcium Silicate Hydrates
DOE	Department of Energy
DOE-HQ	Department of Energy Headquarters
ECM	electrochemical machining
EM	Environmental Management
EPA	Environmental Protection Agency
FTE	Full-Time Equivalent
INEEL	Idaho National Engineering and Environmental Laboratory
ITSR	Innovative Technology Summary Report
JGC	Japanese Gasoline Corporation
LDR	Land Disposal Restriction
MLLW	mixed low-level waste
MWFA	Mixed Waste Focus Area
NEPA	National Environmental Policy Act
NESHAPS	National Environmental Standards for Hazardous Air Pollutants
NRC	Nuclear Regulatory Commission
OST	Office of Science and Technology
ORNL	Oak Ridge National Laboratory
psi	pounds of force per square inch (pressure)
RCRA	Resource Conservation and Recovery Act
RSD	Relative Standard Deviation
RWMC	Radioactive Waste Management Complex
TCE	Trichloroethylene
TCLP	toxicity characterization leaching procedure
TMS	Technology Management System
TTP	Technical Task Plan
UTS	Universal Treatment Standard