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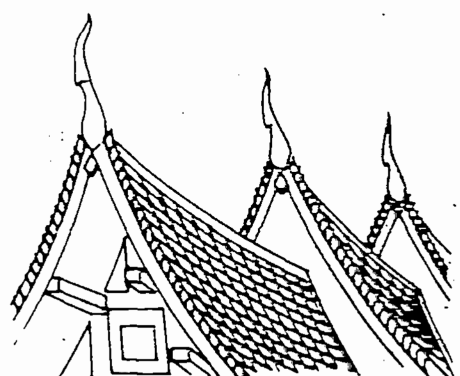

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TECHNICAL ISSUES ON LONG-TERM PERFORMANCE OF SOLIDIFIED/STABILIZED WASTE FORMS

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ABSTRACT

The success of solidification and stabilization treatment of hazardous wastes depends on the ability of the treated waste to endure physical and chemical weathering without releasing trapped contaminants. The expected durability of treated wastes has been estimated from the results of laboratory tests, but has never been confirmed by field tests and application monitoring. This paper describes the status of estimating waste form durability and a proposed research program to improve prediction of the success of solidification/stabilization treatment. Current research projects are also summarized.

INTRODUCTION

Stabilization/Solidification Treatment Processes

Solidification/Stabilization (S/S) is a process used to physically and/or chemically immobilize hazardous constituents in wastes, soils, and sludges. While solidification does not necessarily involve a chemical interaction with the solidifying agent, the process can restrict contaminant mobility by encapsulating the contaminant in a material of reduced surface area, lower permeability and better handling characteristics. Stabilization converts the contaminants into less soluble or less toxic forms, without necessarily achieving solidification.

The U. S. Environmental Protection Agency (EPA) has developed standards for the management of both hazardous and nonhazardous wastes in the U.S. for the protection of human health and the environment through the Resource Conservation and Recovery Act of 1976 (RCRA) and 1984's Hazardous and Solid Waste Amendments (HSWA). Other wastes and contaminated materials are covered under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA, Superfund) as amended by the Superfund Amendment and Reauthorization Act (SARA). These laws and regulations have significantly influenced the use of S/S technology in the United States: provisions in them require that land disposal of hazardous waste be preceded by treatment with the best demonstrated available technology (BDAT). Land disposal is the least preferred waste disposal method, but is allowed with S/S treatment for hazardous wastes that cannot be destroyed by chemical, thermal, or biological means. Thus, S/S treatment as a BDAT is particularly pertinent for metals, since they are impossible to destroy otherwise; it is also used for residuals remaining from other treatments. In the cleanup of waste sites, on-site S/S or in-situ S/S treatment is often the total remedy or part of a treatment train.

A stabilization/solidification process is accomplished by mixing a waste or contaminated soil with treatment agents. The process selection and design for any given waste is usually determined by screening tests and treatability studies (Wiles 1990). Selection of the optimum waste-to-binder ratio is usually based on chemical, physical, and leach test results. The most common binders are inorganic systems based on cement and pozzolanic materials. Organic stabilization/solidification with thermoplastic binders, synthetic polymers, and organophilic clays have been applied to selected wastes but at a much smaller scale than inorganic S/S.

S/S treatment is considered the best-demonstrated available technology for several metals in many waste codes affected by the land disposal restrictions of the Hazardous Solid Waste Amendments of RCRA. The process involves mixing binders with the waste materials and water, sometimes incorporating additives such as fly ash, sodium silicate, bentonite or proprietary materials. The ratios of waste, binder, additives, and water must be tested to optimize the final product integrity and performance (contaminant leachability, strength, curing rate, contaminant binding, pore size, or waste dispersion).

Several cement- and pozzolan-based S/S processes for inorganic wastes have been commercially available for a number of years, and are routinely applied to RCRA and CERCLA wastes. S/S technology is not routinely applied to wastes dominated by organic contaminants, although in some instances it could be effective (Wiles and Barth 1992). Destruction processes such as incineration, biodegradation, chemical oxidation, and dechlorination are preferred for organic contaminants, but are sometimes ineffective and expensive in treating small amounts of toxic organics. The application of S/S treatment to organic-containing wastes has some potential problems: (1) organic compounds are unreactive with many of the reagents used in S/S processes and interfere with the physical and chemical processes which take place in S/S (i.e. set/cure time, cement hydration, and unconfined compressive strength), (2) possible volatilization of semi-volatile and volatile organics during the exothermic mixing process, and (3) lack of chemical binding between binder and contaminant during cement S/S due to the high alkalinity in the interior of the cement-based waste form repelling hydrophobic organic compounds. One formulation potentially applicable to mixed organic-inorganic contaminants uses a combination of cement and organophilic clay.

The Long-Term Durability Issue

Even though S/S technology has been used for over 30 years, there still remain some questions about the efficacy and durability of S/S treatment. A number of physical and chemical tests are performed on S/S wastes to determine structural strength, resistance to chemical and biological attack, and potential to release materials intended to be retained in the S/S matrix. Generally, these tests are applicable only to a short time period after treatment. Many ideas about optimum S/S techniques have originated from

1990 and Federal Register 1986) and the Waste Extraction Test (WET; California Code). Other tests have been developed to reflect contaminant mobility in a particular disposal setting (Monofill Waste Extraction Procedure, U.S. EPA 1986); response to acid contact (Acid Neutralization Capacity, Environment Canada and Alberta Environmental Center 1986; Multiple Extraction Procedure, U.S. EPA 1986a); or dynamic response to continuing leaching (ANS 16.1, American Nuclear Society 1986). The leachability of a particular waste form varies widely with test procedure. Without correlation between test results and field performance, the regulatory and regulated sectors will continue to dispute the over- and under-estimation of contaminant mobility.

Tests actually designed to simulate the long-term performance of treated waste forms are typified by the ANS 16.1 procedure. This test is designed to meet most of the conditions necessary to monitor diffusion-controlled leaching of contaminants. Thus, the results can be used to calculate an effective diffusion coefficient, which can be used in turn to predict long-term leaching by mathematical modelling. Alternatively, the effective diffusion coefficient can be converted to a leachability index for comparison of the relative immobilization achieved by different treatment formulations. The most obvious drawbacks of such a test/model evaluation scheme are that the essential conditions used to conduct the test and the assumptions used in modelling may not prevail in the field. For example, the ANS 16.1 test procedure will not readily dissolve contaminants precipitated as sulfides whereas disposal in an oxygenated environment will. In addition, modelling equations for waste forms usually simulate a leached rim/shrinking core system, but soil-like treatment products might be better simulated by an eroding particle system.

To date, the true long-term performance of solidified/stabilized waste forms largely has been inferred from laboratory tests without verification, or has been measured indirectly by applying the same tests to materials that have been stored after field trials for various lengths of time. The U.S. EPA's Superfund Innovative Technology Evaluation (SITE) Program incorporates periodic re-evaluation of material treated during demonstration tests of new technologies, including solidification/stabilization processes. For example, a soil contaminated with lead was treated and lab-scale samples were analyzed at 28 days, 6 months and 1 year (Barth 1991). Leachable lead, as measured by TCLP, increased from 2.5, to 14, to 24 mg/L over this time span; untreated soil yielded 880 mg/L leachable lead. In contrast, the leachability index calculated from ANS 16.1 data was essentially constant, with values of 13.2, 13.7 and 13.8 over the time period. These results illustrate the problem with durability evaluation: one commonly-employed method suggests degradation of immobilization performance while another indicates no change in performance.

RESEARCH NEEDS

Research is needed to improve interpretation of existing tests and, probably, to gain insight for the design of new tests that will accurately predict the long-term performance of solidified/stabilized wastes. The research goal is to achieve an understanding of the mechanisms and rates of contaminant mobilization from raw and treated wastes as the actual laboratory investigations and use of physical and chemical tests, but there has never been a clear correlation between field leachate quality and laboratory leachate quality. While many test procedures have been developed and interpretation schemes have been developed to predict field leachate quality, none of these methods has been verified. It is essential that the reliability of these data and interpretations be evaluated, to develop a valid prediction model that can be used for assessing the risk reduction achieved and maintained by S/S treatment.

The primary concerns and issues to be considered for long-term durability consist of a number of questions that need to be addressed. Do the available tests generate qualitative or quantitative long-term predictions in the field? If appropriate predictions cannot be made with existing tests, what parameters should be investigated in order to better predict long-term durability? How should a test be designed to simulate at an accelerated rate the degradation processes experienced by treated waste disposed in the field? Long-term monitoring of S/S wastes, both in the field and the laboratory, is needed to verify existing tests and any new tests that are developed.

CURRENT METHODS OF ESTIMATING LONG-TERM PERFORMANCE

The long-term performance of solidified/stabilized waste forms depends on both physical durability and resistance to chemical alteration. While numerous test procedures are available to evaluate these properties, none of them have been fully correlated with field performance.

Physical durability of monolithic waste forms is often assessed by wet-dry or freeze-thaw tests, in which a sample specimen is cycled through alternating conditions and subsequently analyzed for weight loss or leaching resistance. The American Society for Testing and Materials has published standard methods for these physical durability tests (ASTM). Conner (1990) points out that testing methods originally developed for cement-based construction materials may have little relevance to landfill-disposed treated wastes, since the latter will rarely be subjected to the full climatic conditions and mechanical stresses affecting surface structures. EPA's recommendation that treated waste forms have a minimum unconfined compressive strength of 50 psi is more important to landfill construction and closure than to contaminant mobility (U.S. EPA 1989), since the latter is estimated on samples reflective of no structural integrity. Tests that measure resistance to overall erosion become more relevant when leaching behavior is measured on intact monoliths.

The longevity of contaminant immobilization is generally not measured directly by laboratory tests in common use (U.S. EPA 1989). Instead, many test procedures are designed to simulate harsh environmental conditions: complete physical disintegration of the monolith, various strength attacks by acids or other chemicals, and flushing with successively more aggressive leachants. Such tests are considered to reflect worst-case conditions, often for regulatory determination of hazard. Examples of these tests include the Toxicity Characteristic Leaching Procedure (TCLP; Code of Federal Regulations wastes and host matrices change in response to environmental exposure. Given the number of contaminants of concern, the variety of contaminated materials, the range of binders and additives used for S/S treatment, and the wide range of disposal settings, the research program needs to focus on key areas that will elucidate controlling factors for common waste problems.

Contaminant Speciation

The mobility of a contaminant, its interaction with the host material, and its interaction with treatment agents are all dependent on the chemical form of the contaminant. Speciation is critical for many contaminant metals because a single metal can exhibit large differences in electronic charge and bonding. Yet very few Superfund or RCRA investigations include determining the speciation of contaminants, because of cost and time constraints as well as technical difficulties. The oxidation state will sometimes be determined, as for Cr(III)/Cr(VI) or As(III)/As(V), but the soluble, sorbed, and solid forms of each oxidation state are almost never determined. Changes in the binding state of the metal contaminant can provide direct evidence of stabilization. Analytical tools, such as x-ray photoelectron spectroscopy, silicon nuclear magnetic resonance spectroscopy and fourier-transform infrared spectroscopy, will allow detailed examination of waste form structure and contaminant speciation not possible with conventional techniques like scanning electron microscopy and x-ray diffraction analysis.

Contaminant Species Migration

The mobility of contaminants in raw and treated wastes must be characterized. Both the mechanism of dissolution and the rate dependence on solution- and solid-phase chemistry must be known for accurate prediction of long-term immobilization performance.

Once the dominant contaminant species are identified from examination of many field materials, synthetically-contaminated host matrices and treatment agents can be used to systematically study the species behavior. However, the list of variables that could affect contaminant mobility is almost endless, and complete examination for each contaminant of concern could take decades. Instead, field studies can be used to assess apparent controlling factors and rule out those parameters that appear to have only minor effects on contaminant mobility. Laboratory work can then focus on a subset of parameters.

Contaminant Matrix-Treatment Agent Interactions

The mobility of contaminant species in existing contaminated matrices and fresh treatment binders will be characterized by studies described above. But the long-term durability of treated waste forms depends on the stability of all components in the disposal setting. Further studies will be needed to identify changes in the host materials that occur over time and have effects on contaminant speciation and microenvironment. While such studies use laboratory microcosms, it is essential that complementary field studies are conducted so that the simulated disposal studies induce the kinds of changes that occur in the environment.

Co-Contaminant Effects

The long-term durability of solidified/stabilized waste forms is likely to be affected by non-target contaminants present in the waste. Normal cement products used in road-building and construction are susceptible to attack by mild acids and sulfate salts, for example; these contaminants are present in many wastes. In waste mixtures, oily components can sequester target contaminants from contact with binding agents; other organic and inorganic components can complex contaminants and change their solubility. Laboratory research is needed to identify common co-contaminants that either directly interact with target contaminants or interact with matrix materials.

Evaluation of Laboratory Estimates of Durability

The ability of existing tests to predict the absolute long-term performance of treated waste is unknown. Field studies, of at least 10 years' duration, must be started to measure real contaminant mobilities. The same materials used in the field can be subjected to existing tests, and the predictions of treatment performance compared with actual contaminant immobilization as field data are acquired. At the same time, the results of existing tests can be compared with each other to identify effects of test conditions (particle size, waste form size/geometry, temperature, etc.) and develop correlations between existing short- and long-term test data. The outcome of combined field and laboratory tests may be validation of test procedures that partially or fully predict long-term contaminant immobilization. More likely, the outcome will be design and validation of new test procedures based on improved understanding of waste form behavior.

An archive of raw and treated wastes needs to be established to support this research. Despite a 30-year history of S/S application, there is no repository of samples and treatability data that would allow comparison among raw material characteristics, immediate post-treatment performance, and the continuing stability of the field application; for older applications, there is usually no record to review in the event of a treatment failure that results in off-site contamination. The archive would consist of raw waste, treatment materials, and treated castings collected at the time of treatability testing and field implementation. It would also contain treatability data and field monitoring data as they became available. In addition to retrospective review in light of field data, the archive would provide a material and data base for validation of proposed test methods and performance prediction schemes.

Research Program Products

The goal of this research is to be able to predict, in advance of treatment, the immobilization of target contaminants by solidification/stabilization treatment and the longevity of the treatment effect. In each of the program areas outlined, mathematical modelling will be used to express and synthesize the growing understanding of contaminant-waste-binder systems. The products of this research will include test procedures of known predictive capability and models that provide contaminant source terms that can be used in contaminant fate modelling and overall risk assessments.

CURRENT EPA RESEARCH

Several current projects conducted or cosponsored by EPA address some of the research needs described in the preceding section. EPA anticipates that portions of the necessary research will be conducted by other research organizations in several countries. Specific active projects are described below.

Long-Term Durability of Solidified/Stabilized Wastes (Laboratory Evaluation)

The main objective of this project is to better predict long-term fate of S/S hazardous waste forms from Superfund sites around the country. It is necessary to assess or reevaluate selected techniques to predict long-term durability and provide better techniques for making predictions. The approach to this research consists of using existing tests (TCLP, ANS 16.1, and a sequential leaching procedure) to determine the degree of change in a S/S waste sample before and after accelerated aging and weathering is applied (Bishop 1992). The research will also determine and assess the factors important to degradation (i.e., permeability, porosity, and internal and surface cracking). The intent is to use the data obtained from this research to select the best method of predicting long-term durability and make any modifications if necessary.

Stabilized/solidified waste samples were prepared from sludges comprised of lead nitrate, sodium arsenite and cadmium nitrate at high pH. The sludge was solidified with portland cement, lime/flyash or kiln dust, then cast into molds and cured. Final metal concentrations and sludge-binder ratios varied for different experiments. Some samples were subjected to acid-induced chemical weathering, while others were placed in high-temperature environments to accelerate the aging process. Control samples (not aged or weathered) and the test samples (aged or weathered) were then tested for porosity, permeability, and response to chemical leaching and physical weathering tests.

The accelerated aging procedure being developed and tested in this study is based on the Arrhenius model. Using Arrhenius activation energy, time at elevated temperature can be related to a much longer time at ambient temperature according to:

$$\frac{t_s}{t_a} = \exp\left[\left(\frac{E_a}{k}\right)\left(\frac{1}{T_s}-\frac{1}{T_a}\right)\right]$$

where:
- t_s = service time
- t_a = accelerated time
- E_a = activation energy
- k = Boltzman's constant
- T_s = service temperature
- T_a = accelerated temperature

Accelerated aging by heating is commonly used to estimate physical properties for cured concrete, although the time-temperature correlation has not been quantified (American Society for Testing and Materials 1989). The service time-accelerated time formula allows rapid simulation of various exposure-duration times by exposing samples to various elevated temperatures. Thermogravimetric Analysis (TGA) was used to measure the weight changes in a sample as a function of temperature. The resulting mass change versus temperature curve provided information concerning the thermal stability and composition of the sample. This information was then used to determine kinetic parameters of the material, specifically the activation energy.

Accelerated leaching by acid attack was also studied. Results showed that the amount and extent of leaching is essentially the same for samples exposed to a weak acidic leachant over a long time period and for a strong acidic leachant over a short time interval. Thus, if long-term leaching proceeds by a similar, constant mechanism of acid attack, rapid simulations of long time periods can be made by short exposure to acid equivalent to the acidic precipitation that would contact the waste form over the long time period.

Samples used in acid leaching experiments were examined for changes induced by chemical weathering. The cement-based waste forms were split and pH color indicators applied along the leaching penetration front. The pH in the surface-altered layer was found to vary from 5 to 6, which is very close to the pH in the bulk leachate. A reacting zone, where the pH abruptly changed from 6 to 12, sharply divided the altered surface layer from the remaining unleached waste form or "kernel". The reacting zone was white in color and was about 100 microns in width for the samples with 0.6 water-cement ratio leached in 0.4 N acetic acid solution for a total period of 29 days (Figure 1). Scanning electron microscopy/energy-dispersive x-ray analysis indicated that in the surface layer, most of the calcium and the stabilized metals were removed by the leachants. The metal contents of the kernel, however, were very close to those of the original material. It is believed that the leaching boundary was formed by the inward diffusion and reprecipitation of calcium hydroxide crystals in front of the acid. The porosity of the sample core was essentially the same as that of unleached controls, but the porosity of the leached layer increased significantly. These results suggest that any successful durability test or predictive model will have to account for significant chemical and structural changes over time that influence leaching rate. Even with an incomplete understanding of the processes, studies such as this can indicate the relative durability of alternative formulations.

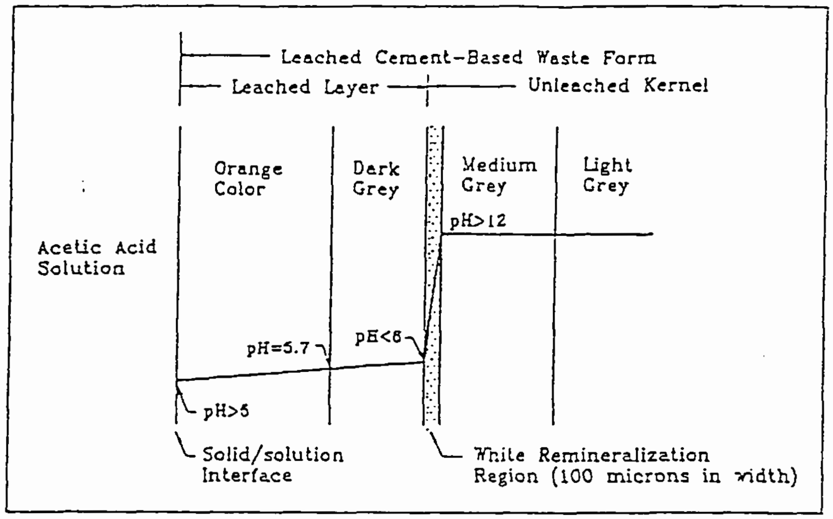

Figure 1. Schematic Profile of Leached Waste Form.

This research is continuing with accelerated aging tests being applied to mixed hazardous/radioactive waste. The application of leach tests in conjunction with weathering and aging techniques on field waste samples will provide valuable data on existing laboratory tests. This will aid in devising better techniques for making predictions on the long-term durability of S/S waste forms.

Long-Term Durability of Solidified/Stabilized Wastes (Field Evaluation)

Laboratory studies have shown the chemical and physical changes that S/S waste forms undergo when subjected to real-time or accelerated laboratory simulation of weathering. The purpose of this project is to identify physical and chemical changes in field-disposed treated wastes. Identification of such changes in the field will provide support to the laboratory procedures that best mimic true weathering effects. Even though solidification/stabilization has been used throughout the country as a pre-disposal treatment to immobilize contaminants, there is a lack of scientific information on its durability.

Potential field sites have been identified from EPA records of S/S treatment applied at Superfund and other sites. The sites are being classified according to age, and data on the site, waste characteristics, and treatment processes is being gathered. At the same time, literature is being reviewed for sampling and monitoring methods that will allow detailed examination of treated waste surfaces at the boundary with native soil or landfill liner materials. A sampling plan will be devised and implemented; samples will be analyzed for physical and chemical properties as well as response to laboratory leach testing. Samples selected for the project will represent a range of waste materials, S/S treatment formulations and disposal ages. Retrieved samples will be archived for use in development and validation of new durability tests and models.

The Nature of Lead, Cadmium and Other Elements in Incinerator Residues and Their Stabilized Products

The University of New Hampshire is conducting this project under a cooperative agreement with EPA. Raw and treated residues from incineration of municipal and hazardous waste are being examined for metal speciation, and for the effects of speciation on both laboratory leaching behavior and projected field performance. Analytical techniques include scanning electron microscopy/energy-dispersive x-ray analysis, electron energy loss spectroscopy and auger spectroscopy. Several separation techniques are being used to isolate contaminant-rich fractions for detailed analysis of metal distribution, oxidation state, and bonding.

Evaluation of Solidification/Stabilization of RCRA/CERCLA Wastes

This new project combines the efforts of EPA and University of Cincinnati personnel to assess the success of commonly-employed immobilization techniques. The methodology is to apply existing laboratory tests to lead-contaminated wastes or soils, and then to monitor those same materials in the field to obtain actual treatment performance data. Field work will be conducted using lysimeters (instrumented containers) for in-situ measurement during exposure to site conditions. Laboratory work will include TCLP, acid neutralization capacity (ANC) tests, and ANS 16.1 tests applied to waste forms of three sizes and geometries. Samples of all tested materials will be archived for analysis by other test methods as they are developed. Correlations will be tested between laboratory results and field observations.

Related Research

In addition to the ongoing and new projects described above, EPA is cosponsoring several other research projects that will increase our understanding of contaminant immobilization.

Work at Lamar University is aimed at interactions between simple metal salts and cement matrices (McWhinney et al. 1990, Cocke 1990). Research includes detailed bulk and surface analysis to identify the speciation of metals in the treated waste form. Contaminant metals being studied include lead, chromium, zinc, cadmium, and barium. Significant differences in binding mode have been detected, with some metals forming insoluble compounds in the alkaline environment of the cement and others being incorporated into the cement structure. Another Lamar University project (Ortego 1991) is studying synthetic mixed-metal cement formulations with and without additives intended to stabilize a particular metal. Results of laboratory leaching showed a different time-dependence for dissolution of matrix metals and contaminant metals. With increasing cumulative acid contact, the leachability of some target metals increased. Researchers at Texas A&M University are evaluating and developing models that relate waste form properties to contaminant mobility. One significant finding is that a thermodynamic equilibrium model generally predicted pore water concentration of matrix elements, but generally did not predict contaminant concentrations accurately (Batchelor and Wu 1991).

Louisiana State University researchers are examining metal leachability as a function of cure time using synthetically-contaminated wastes and pure metal solutions in cement binders (Cartledge and Tittlebaum 1991). Lead, arsenic (V) and chromium (VI) were more extractable by TCLP after 1 year of curing time than after 1 month. ^{29}Si-nuclear magnetic resonance spectroscopy is being used to account for changes in metal leachability as a function of silicate structure development.

SUMMARY

Despite the number and variety of available test, there is no procedure of verified capability for predicting long-term performance of solidification/stabilization treatments. Laboratory studies to date have provided some insight into modes of immobilization and effects of physical and chemical weathering. However, a complete research program as proposed in this paper, is needed to improve the technology and ensure its long-term efficiency.

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16. ABSTRACT

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