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March 1993

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US Army Corps of Engineers
Waterways Experiment Station

An Evaluation of Factors Affecting the Solidification/Stabilization of Heavy Metal Sludge

by R. Mark Bricka, Larry W. Jones
Environmental Laboratory

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Prepared for U. S. Environmental Protection Agency

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U.S. Army Corps of Engineers
Waterways Experiment Station
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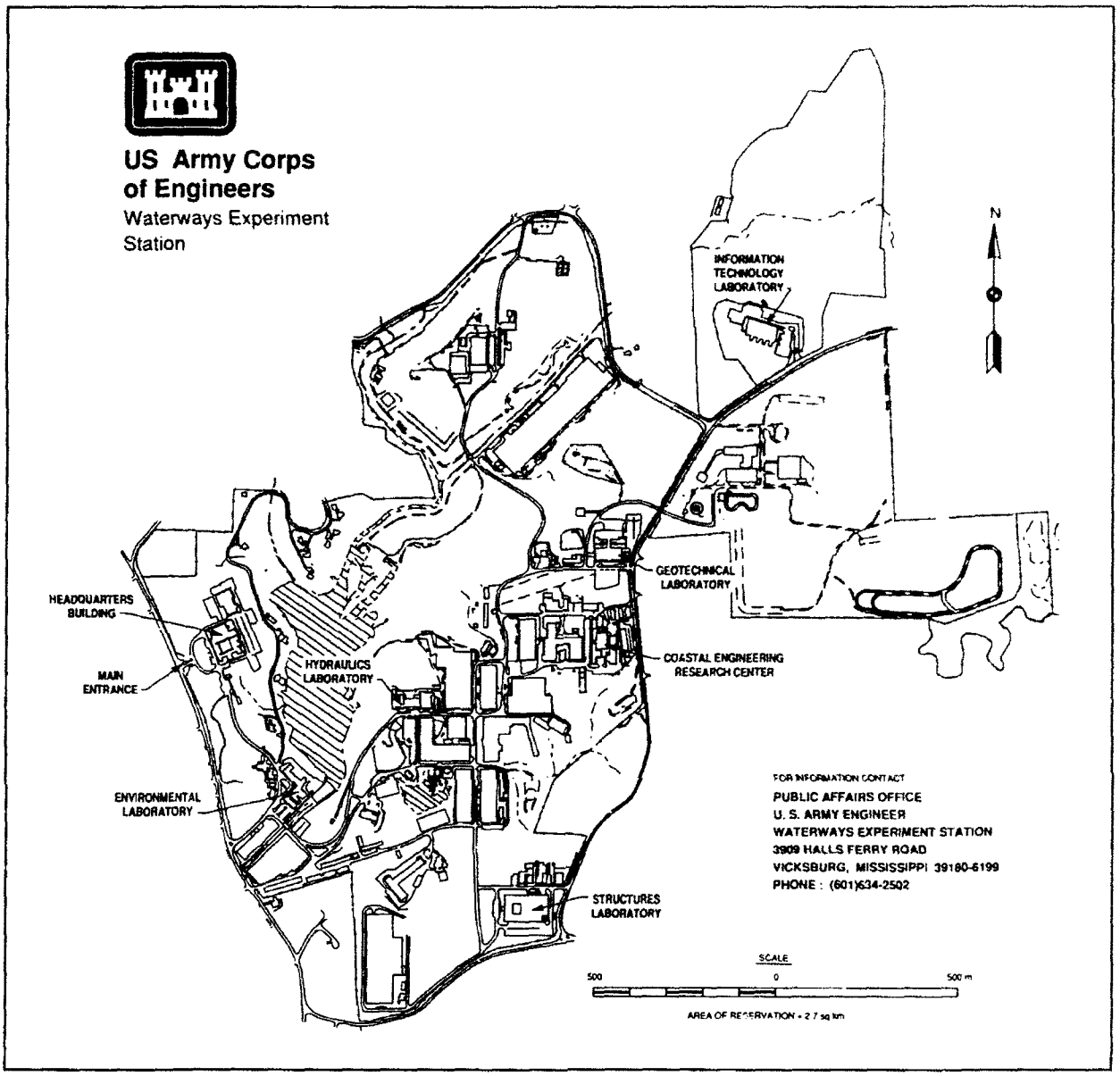

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PREFACE

The study reported herein was conducted by personnel of the Environmental Laboratory (EL) of the U.S. Army Engineer Waterways Experiment Station (WES). The research was sponsored by the U.S. Environmental Protection Agency (USEPA) Office of Research and Development under Interagency Agreement No. DW930146-01-01. The USEPA Project Officer was Mr. Carlton Wiles. Special assistance was provided by Dr. M. John Cullinane, Jr., of the WES.

The report was prepared by Mr. R. Mark Bricka and Dr. Larry W. Jones of the Water Supply and Waste Treatment Group (WSWTG), Environmental Engineering Division (EED), EL. Chemical analyses were performed by the Analytical Laboratory Group, EED. Permeability testing was performed by the Soil Testing Facility of the Soil and Rock Mechanics Division, Geotechnical Laboratory, WES, under the direct supervision of Mr. Jessie Oldham. Support for the unconfined compressive strength testing was provided by the Cement and Pozzolan Group, Concrete Technology Division, Structures Laboratory, WES, under the direct supervision of Dr. Toy Poole. Micrographic analysis of the solidified/stabilized specimens was performed at Louisiana State University under the direct supervision of Dr. Harval Eaton.

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At the time of publication of this report, Director of WES was Dr. Robert W. Whalin. Commander was COL Leonard G. Hassell, EN.

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LIST OF ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

BD	-- bulk density
BDAT	-- Best Demonstrated Available Technology
CEM	-- cement
CFA	-- cement/fly ash
CI	-- cone index
CoV	-- coefficient of variation
EDXRA	-- energy dispersion X-ray analysis
EP	-- extraction procedure
LFA	-- lime/fly ash
PERM	-- permeability
RMSE	-- root mean square error
SEM	-- scanning electron microscopy
S/S	-- solidification/stabilization
UCS	-- unconfined compressive strength
USEPA	-- U.S. Environmental Protection Agency
W/D	-- weight density
W/C	-- water-to-cement ratio
W/W	-- water-to-waste ratio
XRD	-- X-ray diffraction

SYMBOLS

Al	-- aluminum
Al_2O_3	-- alumina
Cd	-- cadmium
Cr	-- chromium
Cu	-- copper
HCB	-- hexachlorobenzene
Hg	-- mercury
Na	-- sodium
NaOH	-- sodium hydroxide
$Na_2(SO)_4$	-- sodium sulfate
Ni	-- nickel
Pb	-- lead
SO_4	-- sulfate
TCE	-- trichloroethylene
Zn	-- zinc

CONVERSION FACTORS, NON-SI TO SI UNITS OF MEASUREMENT

Non-SI units of measurement used in this report can be converted to SI units as follows:

Multiply	By	To Obtain
degrees (angle)	0.01745329	radians
gallons (US liquid)	3.785412	cubic decimeters
inches	2.54	centimeters
pounds (force) per square inch	6.894757	kilopascals
pounds (mass)	0.4535924	kilograms
quarts (US liquid)	0.9463529	cubic decimeters

SECTION 1

INTRODUCTION

BACKGROUND

As environmental regulations become more stringent, the need to improve hazardous waste treatment techniques increases. In an effort to protect our ground-water resources, the U.S. Environmental Protection Agency (USEPA) has imposed substantial new responsibilities on the handlers of hazardous waste. As recently as June 1989, the USEPA enacted requirements prohibiting the continued land disposal of untreated hazardous waste unless the waste was treated using Best Demonstrated Available Technology (BDAT). The BDAT concept requires the treatment of listed wastes to a level equivalent to that specified as BDAT by the Agency. Solidification/stabilization (S/S) has been identified as a BDAT for many listed wastes that contain heavy metals and/or other materials that do not lend themselves to incineration, or other treatment or destruction methods. As a precursor to standards development under the BDAT program, the USEPA recognized the importance of developing a better understanding of the long-term effectiveness of solidification/stabilization in immobilizing contaminants and, consequently, initiated studies to investigate such issues.

SOLIDIFICATION/STABILIZATION TECHNOLOGY

Solidification/stabilization is a process that involves the mixing of a hazardous waste with a binder material to enhance the physical and chemical properties of the waste and to chemically bind any free liquid (Cullinane, Jones, and Malone 1986). Solidification is generally conceptualized as enhancement of the physical characteristics of the waste material. This is accomplished by reducing exposed surface area, which in turn lowers the convective transport of contaminants from the treated waste. Solidification usually entails the incorporation of the waste into a solid matrix or monolith. In comparison, stabilization involves the reaction of the hazardous waste constituents with the S/S reagents to immobilize or otherwise contain them. This process may be as simple as the addition of lime or sulfide to a heavy metal slurry or may involve the development of special reagents specifically formulated to interact with waste components. Most commercial vendors have proprietary additives that are added for this purpose. Generally, commercial vendors use a combination of solidification and stabilization to maximize the immobilization capability of the treated waste.

Several binder systems are currently available and widely used for the S/S of hazardous wastes (Cullinane, Jones, and Malone 1986). Historically, S/S methodologies were developed primarily for inorganic wastes. More recently, as the technology and reagents improve, S/S techniques are being formulated for the containment of organic waste constituents.

Most common S/S techniques are designed with either Portland cement or some type of pozzolan as the basic reagent. Portland cement is widely available, relatively economical, and well known to the general public as producing a very durable product. Pozzolans are siliceous materials that, when added to a source of lime, will go through a cementitious process much like Portland cement but at a much lower rate. Fly ash and blast-furnace slags are common pozzolans that are generally considered as waste materials themselves.

Class F fly ash, made up primarily of silicates, iron oxides, and aluminates, requires the addition of lime for the pozzolanic setting reactions to occur. Class C fly ash, which is produced largely from coal mined in the western United States, contains enough lime to set with only the addition of water (self-cementing).

CHEMICAL INTERFERENCES WITH S/S MATERIALS

Wastes most amenable to S/S treatment are water-based sludges. Most wastes are a complex and variable mixture of many precipitated and dissolved materials, some of which might be expected to interfere with the S/S process and cause undesirable consequences. Effects that might be expected are a breakdown of the solidified matrix, flash or retarded set, or spalling and disintegration. Such reactions could cause contaminant loss to the environment.

Very few quantitative data are available concerning the effects of potential interfering compounds upon particular S/S processes. This study was undertaken to help fill this data void. An earlier report from this study reviewed the S/S literature and discussed theories and details of the effects of known interfering materials with cement, pozzolanic, and asphalt S/S systems and products (Jones 1989).

FACTORS AFFECTING PHYSICAL INTEGRITY

The prediction of long-term integrity of the solidified/stabilized waste form requires the consideration of all possible modes of failure (see listing below). For cementitious and pozzolanic S/S products, water is generally involved in all forms of deterioration. Also, as porous solids, the permeability of the solidified/stabilized material to water is often the primary factor affecting the rate of deterioration. Internal movement and/or changes in the structure of water may cause disruptive volume changes. Examples are freezing of water into ice, formation of an ordered structure of water inside fine pores, development of osmotic pressures due to different ionic concentrations, and hydrostatic pressure build-up by differential vapor pressures (Mehta 1987). All of these can lead to large internal stresses in moist solidified/stabilized solids and may result in ultimate failure.

Physical causes of deterioration of concrete products

Surface wear	Cracking
Abrasion	Volume changes
Erosion	Moisture gradients and humidity
Cavitation	Crystallization pressures of salts in pores
	Exposure to temperature extremes
	Freeze-thaw action
	Fire, pyrolysis
	Structural loading
	Overload or high impact
	Cyclic loading

In porous solids, water also acts as a vehicle for the transport of solutes through the material, both for transport of aggressive ions in, and waste materials out. Permeability is, therefore, of basic interest to the understanding of both durability and leachability. Permeability of concrete

products depends primarily upon the shape and size of the internal voids. The pattern of voids in the waste/concrete solid is a function of the original water/cement ratio (W/C). The W/C ratio determines the size, volume, and continuity of capillary voids, which are the primary cause of S/S product permeability.

The development of microcracks that occur between the cement paste and the surface of included solids (such as aggregates or waste solids) is also a major factor in determining the permeability of the solidified/stabilized waste. However, the suspended particulates in waste sludges (acting as aggregates) are typically very small. In general, the smaller the suspended particles, the fewer the microcracks at their surface, and the lower the overall permeability of the final product. Thus, the primary consideration regarding permeability of solidified/stabilized waste sludges of small particle size is the W/C ratio used in the original formulation.

Deterioration by Chemical Reactions

The effects of internal waste constituents and external aggressive agents on solidified/stabilized waste products must be known with some certainty before the long-term stability of the product can be ensured. The solid phase of the solidified/stabilized waste material exists in a stable equilibrium with the high-pH pore fluid. High concentrations of Na^+, K^+, and OH^- ions bring about a pH of 12.5 to 13.5 in the pore fluid.

The rate of chemical attack on the solidified/stabilized waste product is a function of the pH of the external fluid, its buffering capacity, and the permeability of the solidified/stabilized product. In general, pH levels above 6 bring about such a slow reaction that they can be neglected. However, natural CO_2, sulfates, and chlorides (common in ground- and rain-waters) and waste leachates, may bring about aggressive solutions below pH 6 that are detrimental to the integrity of the solidified/stabilized waste product.

Cation-exchange reactions can occur between the external solution and the cement or pozzolan binder. Anions in acidic solutions that form soluble calcium salts, such as calcium chloride, acetate, and bicarbonate, will cause the calcium in the solidified/stabilized product to be removed by leaching. This is particularly damaging because it increases the permeability of the concrete, which in turn increases the rate of further exchange reactions. Certain anions form nonexpansive, insoluble calcium salts such as calcium oxalate, tartrate, phosphate, or humic acid salts that do not cause much damage to the concrete. Magnesium salts may exchange with calcium in the binder, break down the fundamental cementitious matrix, and cause breakdown of the solidified/stabilized waste structure.

Sulfate attack on concrete products has been a serious and well known problem for many years and is a serious consideration in the S/S of sulfate-containing wastes in Portland cement. Concentrations of soluble sulfates greater than 0.1 percent in soil (or 150 mg/l in water) will endanger concrete products, and soils of over 0.5 soluble sulfates (or over 2,000 mg/l in water) can have serious effects.

The most common reaction in sulfate attack is the reaction of alumina-containing hydrates with sulfate and calcium hydroxide to produce the mineral ettringite, which forms expansive crystals that break up the final

solidified/stabilized product. Use of Portland cement containing less than 5 percent tricalcium aluminate (ASTM type V) overcomes the effects of moderate sulfate exposure. Cements that contain little or no calcium hydroxide (e.g., high alumina cements, or Portland cement with more than 70 percent (w/w) blast-furnace slag or with at least 25 percent of a pozzolan such as fly ash--see below) can withstand very high sulfate conditions. Pozzolanic S/S systems are also useful for S/S of high sulfate wastes, since they do not contain free calcium hydroxide and thus are nonreactive to sulfate.

Crystallization of Salts in Voids

Solidified/stabilized waste products often contain substantial concentrations of salts and/or organic molecules with appreciable water solubilities. Concentration of these materials at or below the surface of the solid where evaporation of pore water is occurring can cause the development of supersaturated solutions and the formation of salt crystals in the pores of the solidified/stabilized waste product.

Crystallization occurs only when the concentration of the solute (C) exceeds the saturation concentration (Cs) at a given temperature. Generally, the higher the degree of supersaturation (the ratio of C/Cs), the greater the crystallization pressure exerted on the solid structure. Table 1 gives

TABLE 1. TYPICAL CRYSTALLIZATION PRESSURES FOR SALTS

Salt	Chemical formula	Pressure (atm) at C/Cs = 2	
		0° C	50° C
Anhydrite	$CaSO4$	335	398
Bischofite	$MgCl_2 \bullet 6H_2O$	119	142
Dodekahydrate	$MgSO_4 \bullet 12H_2O$	67	80
Epsomite	$MgSO_4 \bullet 7H_2O$	105	125
Gypsum	$CaSO_4 \bullet 2H_2O$	282	334
Halite	$NaCl$	554	654
Heptahydrite	$Na_2CO_3 \bullet 7H_2O$	100	119
Hexahydrite	$MgSO_4 \bullet 6H_2O$	118	141
Kieserite	$MgSO_4 \bullet H_2O$	272	324
Mirabilite	$Na_2SO_4 \bullet 10H_2O$	72	83
Natron	$Na_2CO_3 \bullet 10H_2O$	78	92
Tachhydrite	$2MgCl_2 \bullet CaCl_2.12H_2O$	50	59
Thenardite	Na_2SO_4	292	345
Thermonatrite	$Na_2CO_3 \bullet H_2O$	280	333

Source: Winkler 1975.

some examples of crystallization pressures for C/Cs = 2 of a series of salts that are common in solidified/stabilized waste materials. These values were calculated by Winkler (1975) in an effort to understand the rapid deterioration of stone and concrete monuments by smog and acid (high-sulfate) rain. For example, at a C/Cs = 2 and 25° C, halite (NaCl) produces 605 atm. (8,900 psi) of pressure and at a C/Cs = 10 , produces 2,020 atm. (29,690 psi). These pressures are strong enough to disrupt the structure of the S/S products and cause spalling and/or disintegration. Common salts have a wide range of crystallization pressures. Due to the wide variety of materials found in hazardous wastes, this effect can be appreciable; however, it is difficult to predict the conditions and concentrations under which these effects would be operative.

The powdering or spalling of the subsurface of the solidified/stabilized waste material progressively deepens into the material as its porosity increases. Damage to S/S products due to wet/dry cycles, as used in this study, may be due to the cyclic dissolution and recrystallization of contained salts.

Mineral Admixtures

The use of pozzolanic and cementitious by-products (such as fly ash, blast-furnace slag, or kiln dust) as admixtures is an important issue in waste S/S. When used in addition to, or as a partial replacement for Portland cement, the presence of the pozzolanic mineral generally retards the rate of strength gain. However, the reaction of the mineral admixture with the excess calcium hydroxide present in the hydrated Portland cement paste leads to a significant reduction in porosity and an increase in the ultimate strength of the final product (Marsh, Day, and Bonner 1985). Consequently, considerable improvement in the ultimate strength and impermeability (water-tightness) can be achieved by the incorporation of pozzolanic material. For this reason, one of the S/S formulations chosen for use in this study incorporates fly ash with Portland cement.

Wet-Dry Cycling

Other than cracking by salt dissolution and recrystallization, as described above, wet/dry cycling of normal concrete products does not usually produce significant damage to their structure. However, as the total proportion of cement or pozzolan is reduced, or the water/cement ratio is increased, as is common for economy in waste solidified/stabilized practices, wet/dry cycling may cause rapid deterioration of the solidified/stabilized waste product. Jones and Malone (1982), using ASTM standard test procedure D559-82 for compacted soil-cement mixtures (ASTM 1988c), reported rapid deterioration of solidified/stabilized inorganic waste products produced by vendors of commercial S/S vendors. Wet-dry cycle testing using this ASTM test method was incorporated into this study as a simple but effective method of testing the durability of the various solidified/stabilized waste mixtures.

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* A table of factors for converting non-SI units of measurement to SI (metric) units is presented on page xiv.

Air Entrainment

Additives causing stable air incorporation into the cement paste are universally deleterious to the ultimate strength and permeability of concrete due to the added large-pore space. Air entrainment is not a common practice in waste S/S. However, many of the common organic compounds may act as air-entraining agents. The entrained air greatly increases the resistance of the products to freezing damage.

Freeze-Thaw Damage

In a manner analogous to salt crystallization, formation of ice crystals at subfreezing temperatures can cause rapid deterioration of water-saturated solidified/stabilized products. In practice, the durability of concrete products to freeze-thaw cycles is provided by entraining small air bubbles into the cement mixture. Small amounts of certain air-entraining agents added to the cement paste (e.g., 0.05 weight percent of cement) will bring about the incorporation of stable, 0.05- to 1-mm bubbles in the final product. Depending upon the size and number of voids, and their spacing, the degree of protection against freezing damage can vary a great deal with a given amount of entrained air, as illustrated in Table 2. However, in medium- and high-strength concretes, every 1-percent increase in the air content reduces the ultimate strength of the final product by about 5 percent and increases its permeability by a comparable amount.

TABLE 2. DURABILITY TO FREEZE-THAW CYCLES OF NEAT CEMENT SAMPLES WITH DIFFERENT NUMBERS AND SIZES OF AIR BUBBLES

Air voids/cm^3	Total air volume (%)	Freeze-thaw cycles to show 0.1% expansion
24,000	5-6	29
49,000	5-6	39
55,000	5-6	82
170,000	5-6	100
800,000	5-6	550

Source: Woods 1968.

The degree of water saturation of the solidified/stabilized material is an important factor in determining the potential for freeze-thaw damage. Above the critical degree of saturation, usually between 80 and 90 percent, the solidified/stabilized product is likely to crack and spall when exposed to freezing temperatures. Below the critical level, freeze-thaw damage is not likely to occur. A solidified/stabilized waste product may fall below the critical degree of saturation after adequate curing; however, depending upon its permeability, it may reach the critical degree of saturation in a short time when exposed to a moist environment. The greater the porosity of the solidified/stabilized product, the greater the amount of freezable water that will be present at any given temperature and humidity.

Test Specimen Parameters

In addition to factors that can affect the physical integrity of the solidified/stabilized materials, sample geometry can also affect strength measurements. The standard test specimen for the uniaxial compression test in the United States (ASTM C 469-87) is a right cylinder, 6 in. in diameter and 12 in. high (ratio h/d = 2) (ASTM 1988b). Measured strengths vary indirectly with h/d ratio, a h/d ratio of 1 giving 15 to 20 percent higher strength, and a ratio of 4 about 10 percent less. Even specimens of different sizes with h/d = 2 have different measured strengths, a 3- by 6-in. cylinder having about 6 percent higher strength, and a 9- by 18-in. specimen about 10 percent lower strength than the standard 6- by 12-in. cylinder.

Strength tests based on the 6-in. standard test cube, which are common in European countries, generally indicate strengths 10 to 15 percent higher than tests of the same material using the 6- by 12-in. cylinder (Metha 1987). Similarly, the use of a 2-in. cube for compressive strength testing, as used in this study, would be expected to give 15 to 25 percent higher strength values for the same solidified/stabilized product evaluated using a 6- by 12-in. cylinder.

Sample preparation factors can also affect strength development characteristics of the final product. The most important factor in strength of concrete is the water/cement ratio-porosity relationship. This is usually explained as the natural consequence of the weakening of the cement matrix caused by increasing porosity with increasing water/cement ratios. Typical data illustrating this point are shown in Table 3, which also illustrates the increase in strength with increasing cement hydration (time of cure). Increasing strength also corresponds to a decrease in the total pore space.

TABLE 3. INFLUENCE OF WATER/CEMENT RATIO AND DEGREE OF HYDRATION (SET) ON CONCRETE STRENGTH

Water/cement ratios (by weight)	Unconfined compressive strength (psi) after different lengths of cure (days)*			
	1	2	3	28
0.35	1,300	2,900	4,450	6,300
0.45	900	2,200	3,500	5,250
0.55	550	1,650	2,750	4,350
0.65	300	1,200	2,000	3,450
0.75	200	950	1,550	2,800

* Based on 6- by 12-in. cylinders of neat type I cement undergoing moist cure.

INFORMATION FROM CONCRETE/POZZOLAN TECHNOLOGY

Experience from cement and pozzolanic concrete technologies, as well as data from waste S/S studies, has demonstrated that small amounts of specific compounds can significantly reduce the strength and durability and, hence, probably the contaminant containment characteristics of waste/binder mixtures.

It may be necessary to specify acceptable limits of interfering and complexing materials allowed in solidified/stabilized hazardous waste to ensure adequate waste containment.

Concerns about the effects of industrial wastes in the environment on concrete corrosion have been expressed for many years. For instance, Biczok (1967) listed the following industrial wastes that are largely harmless to concrete and mortar products:

(1) Brines containing bases but no sulfates.

(2) Potassium permanganate, occurring at fermenting and purification installations.

(3) Sodium carbonate (soda) and potassium carbonate (potash).

(4) Bases (caustic lyes of potash and soda, lime and ammonia), provided their concentration is not excessively high.

(5) Oxalic acid occurring at tanneries.

(6) Mineral oils and petroleum products (benzene, kerosene, cut-back oil, naphtha, paraffin, tar), as long as these contain no acids that can continue to remain in the products after chemical treatment.

Industrial wastes deemed to be aggressive and detrimental to concrete and mortar products are

(1) Water containing gypsum, e.g., such as used for quenching coal slag.

(2) Ammonia salts.

(3) Zinc and copper sulfate.

(4) Vegetable, animal, and mineral oils.

(5) Hydrochloric, nitric, and sulfuric acids.

(6) Chlorine and bromine.

(7) Acetic acid.

(8) Pure alcohol (in certain cases only, e.g. absolute alcohol, owing to the dehydrating effect).

(9) All sulfur and magnesium salts.

(10) Hydrogen sulfide and sulfur dioxide gas.

(11) Animal fats.

(12) Salts of strong acids, e.g., those formed with magnesium, zinc, copper, iron, aluminum, and other metals the hydroxides of which are poorly soluble in water.

MOBILITY OF WASTE CONSTITUENTS

The internal mobility of solidified/stabilized waste constituents, and the rate of loss of these waste constituents to contacting waters, is the single most important measure of the effectiveness of S/S technology. Predicting the effects of interfering materials on mobility of waste constituents is difficult. In fact, simply measuring the mobility of the waste constituents is a highly debated issue within the scientific and S/S communities. Many laboratory leaching (or extraction) methods have been designed in the attempt to predict the long-term mobility of waste constituents (Cote', Bridle, and Hamilton 1984; Cote' et al. 1988), although few studies and little data are available to verify the effectiveness of such tests in predicting long-term contaminant containment.

Because of the difficulties associated with measuring contaminant mobility, no information is available on the effects of interfering compounds on the mobility of waste constituents. One of the objectives of this study was to initiate development of such data.

PARAMETERS USED TO INVESTIGATE INTERFERENCE EFFECTS

The goal in evaluating the effects of the interference materials was to compare a solidified/stabilized waste without interferences to a solidified/stabilized waste containing interferences. Difficulty arose in selecting the parameters to be used in comparing these materials and the tests by which to measure them.

Cement and pozzolan materials are typically used in construction applications. The users of these products usually limit their concerns to the strength, porosity, and durability properties. Unwanted secondary materials contained in cements and pozzolans, incidental to the strength and durability properties of the final product, are not considered to present problems.

Users of S/S technologies for the treatment of wastes place less emphasis on the physical characteristics of the final product. The ability of a solidified/stabilized waste to contain the waste components is the primary objective of S/S treatment. In fact, if a S/S process generates a friable material with little structural integrity but the solidified/stabilized product has good long-term contaminant containment, it would considered to be a successful treatment process.

With solidified/stabilized wastes it is difficult to know if the primary waste containment mechanism is chemical stabilization on a molecular level or simply entrapment of the contaminant in the solidified matrix. If waste constituents are entrapped, the mobility of waste components will be highly dependent on the amount and kinds of porosity of the solidified/stabilized waste. (Water or some solvent must contact the waste constituents for convective transport to occur.) Specimen porosity is related to physical properties such as durability and strength. Thus, if the primary containment mechanism for the waste components is entrapment, results from physical testing may be

the best indicators of the ability of a solidified/stabilized waste to contain these contaminants over the long term.

If the primary containment mechanism is chemical stabilization, the issue of determining contaminant mobility is more difficult to resolve. Leaching tests are the most widely implemented tools for investigating contaminant mobility. Because of the many chemical interactions that may affect contaminant mobility, the leaching test used to evaluate the effects of interferences must be carefully selected.

In this study a strong emphasis is placed on the measurement of the physical properties of the materials. Several physical methods were selected to evaluate the effects of the interference material on the solidified/stabilized materials. These tests included unconfined compressive strength, cone index, bulk density, wet/dry durability, and permeability. The leaching properties were also investigated, but only the USEPA regulatory test (the extraction procedure, EP, toxicity test) was used. Test specimens were also evaluated for any microchemical/micromorphological changes resulting from the interference chemical addition.

EXPERIMENTAL DESIGN

The initial experimental design of this project was based upon the recommendations of a panel of experts composed of representatives from academia, regulatory agencies, landfill operators, and S/S product vendors (see Appendix A). The design and execution of this study adhered to the panel's recommendations as closely as possible. The recommendations of the panel are summarized below.

Sludge Production

The panel recognized the need to identify a heavy metal sludge for use in this study that was free of unknown and unwanted contaminants. Such contaminants might have significant but undetermined effects on the final product and test results. No known industrial wastes met this criteria. Thus, the panel's recommendation for the use of a laboratory-generated "reagent grade" waste offered the most practical solution.

The panel originally recommended the production of a synthetic sludge containing all of the toxic metals listed by the EP test (USEPA 1986), plus copper and zinc. The synthetic sludge was to be produced by dissolving salts of these metals at concentrations 1,000 times the EP limits in a slightly acetic solution and precipitating these metals from solution using calcium hydroxide. This process would produce a sludge containing either insoluble oxides or hydroxides of 10 metals. Sludge aging was also considered, as it could result in changes in the sludge properties and thus affect the study results. To avoid such problems, only "fresh" sludge was to be used in the study.

Initial laboratory studies revealed that, although each of the metals was soluble at the desired concentration independently, a synthetic metal waste containing all of the metals at these concentrations could not be generated due to solubility limitations. This phenomenon was believed to be attributed to the common ion effect for many of the metal compounds used in solution preparation. As a result, the panel recommended that the metals in

the synthetic waste solution be limited to mercury nitrate, chromium nitrate, cadmium nitrate, and nickel nitrate and that the concentration be reduced from 1,000 times to 600 times the EP limit.

Interference Chemical Selection

To investigate the effects of chemicals suspected of interfering with the contaminant containment capability of solidified/stabilized waste, the panel recommended that several general categories of interfering substances be evaluated. To avoid introducing uncontrolled variables, the panel recommended the use of laboratory reagents as interferences. These categories, the generating industries, and the recommended representative chemicals are listed in Table 4.

TABLE 4. RECOMMENDED INTERFERENCE SUBSTANCES

Category	Typical industry	Laboratory reagent recommended
Oil	Metal cleaning	Motor oil
Grease	Metal cleaning	Industrial grease
Phenol	Paint stripping	Phenol
Sulfates	Acid neutralization	Sodium sulfate
Strong base	Waste, caustic	Sodium hydroxide
Degreaser	Metal cleaning	Trichloroethylene
Pesticide	Pesticide contaminated waste	Not specified
Lead	Metal-contaminated wastes	Not specified
Copper	Metal-contaminated wastes	Not specified
Zinc	Metal-contaminated wastes	Not specified

The panel further recommended that interfering chemicals be added to the solidified/stabilized sludge at interference-to-solidified/stabilized sludge ratios of 0, 0.02, 0.05, and 0.08 by weight.

Binders Selected for Evaluation

At the time of study initiation, several S/S vendors were offering a variety of proprietary S/S processes for treatment of hazardous waste. The panel recommended for consideration only generic S/S systems that represented the basic technology of the majority of the proprietary S/S processes. Based on this criterion the panel recommended the evaluation of three generic binding agents: a Portland type I process, a lime/fly ash process, and a Portland type I/fly ash process).

The binder-to-sludge ratio for each binder system was to be established on the basis that the cured specimen would meet specific performance criteria. The three S/S processes were evaluated, at one binder-to-waste ratio. The binder-waste ratio selected for evaluation was based on the single performance criterion of an unconfined compressive strength greater than 100 psi.

OBJECTIVE

The principal objective of this study was to investigate the effects of interference chemicals on a solidified/stabilized heavy metal sludge. As part of this study, a number of issues were addressed, as are listed below.

(1) The testing and evaluation of materials with known or suspected interfering effects.

(2) The investigation of the relationship between the amount of interfering substance and the significance of any effect.

(3) The identification of appropriate parameters to evaluate the effects of the interferences.

(4) The development, modification, and/or adoption of new and existing test methods to evaluate the interfering effects.

(5) The investigation of the effects of the interference chemicals on the physical and contaminant containment properties of three generic binder systems and the comparison of these effects.

SECTION 2

MATERIALS AND METHODS

GENERAL APPROACH TO THE INVESTIGATION

The study was conducted in four phases, as summarized below and described in the subsections that follow:

- Phase I, Initial investigation--This investigation consisted of a literature survey to identify any existing data describing the effects on materials that interfere with cementation or pozzolanic setting reactions. Results of this survey were published separately (Jones 1989).

- Phase II, Synthetic metal sludge preparation--A synthetic sludge containing four heavy metal contaminants was prepared using lime precipitation. The lime sludge was dewatered to produce the sludge used in this study.

- Phase III, Sludge treatment and addition of interference agent--After the metal sludge was produced, binding agents were added to solidify/stabilize the waste and immobilize the contaminants. Prior to set, the sludge/binder mixture was divided into four portions. Selected interfering chemicals were mixed with three of the sludge/binder portions at three concentration levels. Molded specimens were prepared for the three interference/binder/sludge mixtures and for the binder/sludge mixture (control).

- Phase IV, Physical and chemical evaluations--The physical and contaminant release properties of the solidified/stabilized specimens were evaluated using an accepted testing regime.

INITIAL INVESTIGATION

As part of the study, a detailed literature survey was conducted to review information concerning Portland cement and pozzolan chemistry, the effects of added constituents on their setting characteristics, and the effects of typical organic waste components on the physical and contaminant containment properties of the treated waste product. The survey was conducted to assess the possible types of interference chemicals that might be encountered in typical waste/binder systems. The results were published in the report "Interference Mechanisms in Waste Stabilization/Solidification Processes" (Jones 1989).

SYNTHETIC METAL SLUDGE PREPARATION

Laboratory Investigation

Laboratory jar tests were performed on the synthetic metal waste solution to determine the settling properties of the sludge and the optimal calcium hydroxide dosage for maximum sludge formation. The synthetic metal solution was prepared by dissolving 23.1 g/l of $Cr(NO_3)_3 \bullet 9H_2O$, 14.9 g/l of $Ni(NO_3)_2 \bullet 6H_2O$, 1.6 g/l of $Cd(NO_3)_2 \bullet 4H_2O$, and 0.02 g/l of $Hg(NO_3)_2 \bullet H_2O$ in tap

water. Laboratory jar tests were performed using a Phipps and Bird six-paddle stirring apparatus and six 1-liter mixing vessels. Calculations revealed that a calcium hydroxide dosage of 10.6 g/l provided the stoichiometric amount of hydroxide required to precipitate the metals in the synthetic metal solution. Lime dosages of 10, 12, 14, 16, 18, and 20 g/l were evaluated in the jar tests.

A lime dosage of 20 g/l produced a sludge with optimal settling characteristics and a supernatant with the lowest turbidity. This lime dosage was twice the calculated stoichiometric amount required for metal precipitation. The additional lime required could be attributed to water hardness, the acidity of the metal solution, and impurities in the lime. A lime dosage of 20 g/l was used for sludge production throughout this study. The sludge contained approximately 9 percent solids and had a density of 1.08 g/ml. The supernatant had a final pH of 11.5.

Filtration tests, performed on the synthetic sludge, indicated that the sludge was relatively difficult to filter and that 35 g of filter cake with a solids content of 30 percent could be produced from 1,000 ml of synthetic metal solution. The maximum filter cake solids content that could be achieved using vacuum filtration was 30 to 35 percent. Based on these preliminary test results and the fact that filtrate could be used to dilute the sludge, a sludge containing 25 percent solids was selected for use in this study. By using a constant sludge solids content, the water-to-binder ratio for the solidified/stabilized products could be tightly controlled.

A curve for drum speed versus filtrate production rate, shown in Figure 1, was generated using an Eimco vacuum rotary drum filter. Data presented in this figure were used to set the rotational speed of the drum filter.

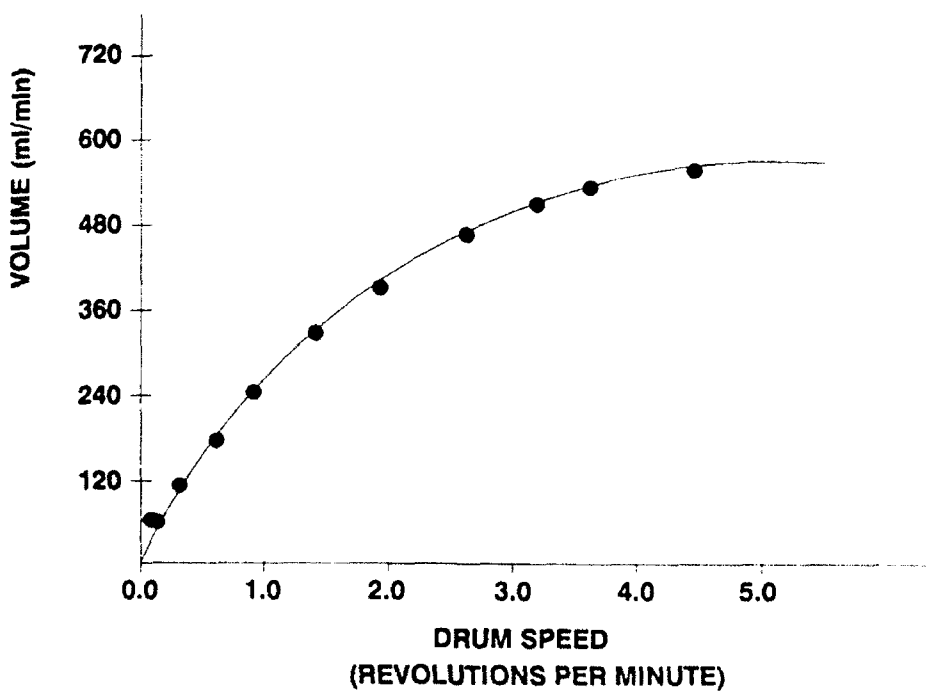

Figure 1. Drum speed versus filtrate production rate for the Eimco rotary drum vacuum filter.

Approximately one 8-hr workday was required to produce approximately 20 lb of filtered sludge containing 25 percent solids (w/w).

Pilot-Scale Sludge Production

Approximately 9,000 gal of synthetic metal solution was needed to provide the amount of sludge required for the preparation of all the specimens used in this study. Roughly 2 weeks were needed to filter the sludge produced by one 300-gal batch of metal solution. Sludge generated from one 300-gal batch provided enough sludge to evaluate one interference chemical. To complete this study, sludge was prepared in thirty 300-gal batches over a period of 18 months. Batch preparation water was recycled throughout this study to minimize the generation of wastewater. Details for the preparation of one 300-gal batch of synthetic metal waste solution and for filtration of the sludge are illustrated in the schematic diagram presented as Figure 2. Recycled batch water (270 gal) was metered and pumped into a 600-gal stainless steel tank. To this water, 4.1 lb of $Cd(NO_3)_2 \bullet 4H_2O$, 57.8 lb of $Cr(NO_3)_3 \bullet 9H_2O$, 37.2 lb of $Ni(NO_3)_2 \bullet 6H_2O$, and 0.51 lb of $Hg(NO_3)_2 \bullet H_2O$ were added and mixed for 60 min at 50 rpm using a Dayton model 4Z772 mixer and a Lightning A310 impeller. After mixing, no visual indication of undissolved solids was detected. The solution pH was 4.1.

After dissolution of the metal salts, the Lightning A310 impeller was replaced by a rectangular paddle-type impeller measuring 33 by 12 in. A calcium hydroxide (CaOH) slurry was prepared in a separate 35-gal tank by adding 58.5 lb of CaOH to 30 gal of fresh tap water. This CaOH slurry was added to the synthetic metal solution and was rapid-mixed with the paddle impeller for 10 min at 12 rpm. The slurry was mixed slowly with the same impeller for an additional 50 min at 5 rpm to maximize flocculation. At the completion of the mixing period, agitation was stopped and the sludge was allowed to settle for a minimum of 15 hr.

The clear supernatant was separated from the sludge by pumping from the top of the sludge using a Teel model 1P808 screw pump. The sludge contained about 8 percent solids and had a pH of approximately 11. A valve at the bottom of the tank was opened, and the sludge flowed by gravity into a 350-gal feed tank that supplied sludge to the filtration system. The sludge in the feed tank was periodically mixed to maintain the homogeneity of the sludge.

The sludge was dewatered using an Eimco model 2204 lab scale, drum filtration unit. A schematic of this unit is shown as Figure 3. The sludge was fed by gravity into the constant-level pan. Overflow from the constant-level pan was collected in a sludge overflow tank and was recycled as feed by pumping the overflow into the feed tank. The filter drum was covered with a 15-micron polyethylene filter cloth. The drum was rotated through the sludge, and a vacuum was applied to the reverse side of the cloth. The sludge was sucked from the pan, and the filtrate passed through the filter cloth into a vacuum/filtrate collection tank. The filtrate was pumped from the vacuum/filtrate collection tank into a series of recycle tanks where it was stored for later use. As the drum rotated, compressed air was used to puff the filter cloth from the drum's surface, and a knife edge scraped the dewatered cake from the cloth. Dewatered filter cake was discharged into a Stow 20E collection/mixing vessel.

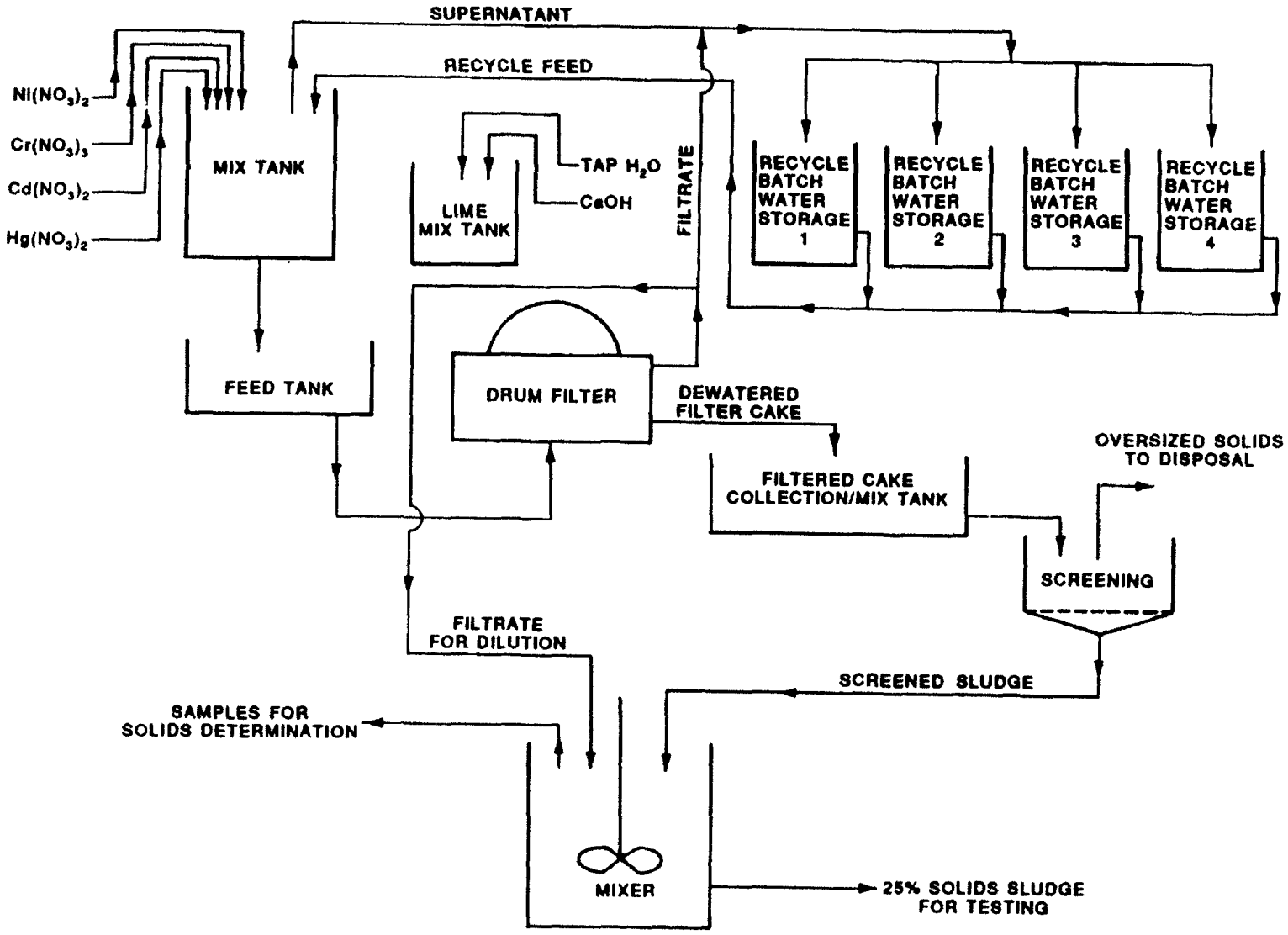

Figure 2. Schematic diagram of the sludge generation process.

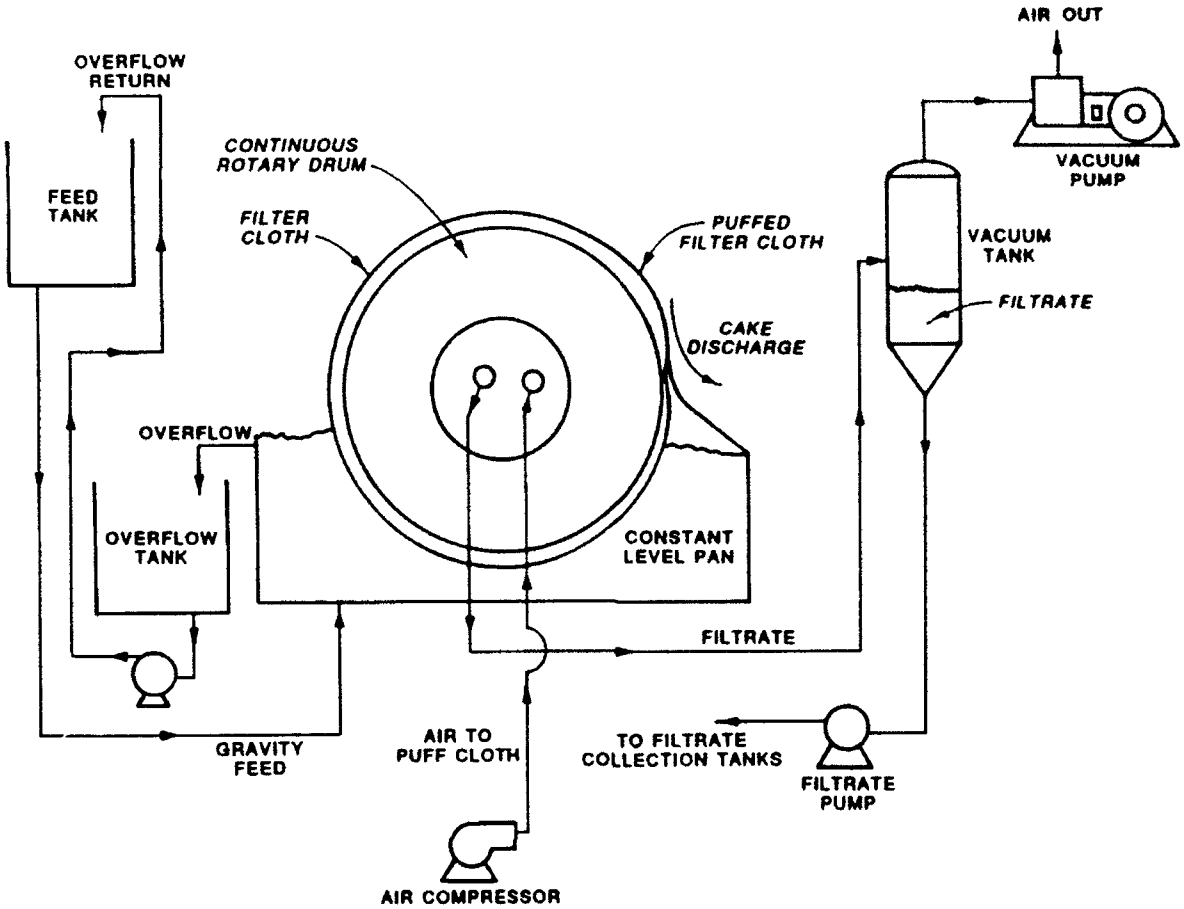

Figure 3. Schematic diagram of the laboratory drum filter.

About 2 weeks after the sludge was initially precipitated, all the sludge had been dewatered and discharged into the collection/mixing vessel. Immediately prior to specimen preparation, the dewatered sludge was homogenized in the collection/mixing vessel using paddle-type agitation. The sludge was mixed for a minimum of 1 hr. After mixing, the sludge was fluid. The fluid sludge was poured through a 1/16-in. mesh nylon screen to ensure the removal of any large particles. The screened sludge was stored in a 60-qt stainless steel mixing bowl. Three 10-g samples of the sludge were collected from the mixing bowl. The sludge was remixed by hand for 1 min between each sampling. These samples were used to determine the solids content of the sludge, as described below.

To maintain a constant sludge solids concentration and to efficiently conduct the remainder of the sample preparation phase of this study, it was necessary to quickly determine the solids concentration of the screened sludge. A Kenmore model 747.9957821 microwave oven was used to facilitate drying of the 10-g sludge specimens. Methods development activities conducted prior to sample preparation revealed that this oven, on its highest setting could dry to constant weight a sludge sample with a solids content ranging between 25 and 35 percent solids in 12.5 min with only minimal solids loss.

Thus, each sample of the sludge was separately dried in this microwave oven for 12.5 min. The solids content of the sample was determined by comparing the sample's wet and dry weights.

Periodically throughout the study, sludge samples were collected in addition to the three samples for the solids content determination discussed above. Concurrent moisture determinations were preformed on these samples according to ASTM method D 2216-80 (ASTM 1988c). These results were used to ensure the quality of the microwave method.

After the solids content of the sludge was measured, calculations were preformed to determine a dilution factor for the sludge. Using this dilution factor, filtrate was added and mixed using a Hobart C-600 mixer for 10 min, producing a sludge with a solids content of approximately 25 percent. At the completion of the mixing period, an additional solids determination was performed to ensure that the solids content of the sludge was 25 ±0.5 percent. Analysis of this sludge indicated that it contained 18.1 mg/g Cd, 81.3 mg/g Cr, 1.39 mg/g Hg, and 81.1 mg/g Ni on a dry weight basis.

SLUDGE TREATMENT AND SPECIMEN PREPARATION

Criteria Testing

Unconfined compressive strength (UCS) testing was performed on the binder/sludge mixture to select the binder/sludge ratio that would be evaluated. Binder/sludge ratios were formulated to produce a solidified/stabilized product that had a 28-day UCS of at least 100 psi.

For the three recommended binder systems (Portland type I cement, Portland type I cement and Class F fly ash, and lime and Class C fly ash) various sludge/binder ratios were prepared. At 28 days of cure, the UCS of these specimens are determined. The results of this test are presented in Figure 4 for the cement binder (CEM), in Figure 5 for the cement/fly ash (CFA) binder, and in Figure 6 for the lime/fly ash binder (LFA). Based on these results the binder ratios shown in Table 5 were selected for use in this study.

Specimen Preparation

One interference chemical was evaluated for each batch of dewatered sludge prepared. It was determined that in addition to the interference ratios of 0.02, 0.05, and 0.08 selected for evaluation, a binder/sludge without interference chemical addition would be prepared for each batch of sludge to serve as a control. Thus, for each batch of specimens prepared, one binding agent in combination with one interference chemical at interference ratios of 0, 0.02, 0.05, and 0.08 were evaluated. These specimens were subjected to physical and chemical evaluations after curing. Figure 7 presents a flowchart illustrating the specimen preparation scheme.

Typically, 120 lb of 25 percent solids sludge was generated for each batch of sludge prepared. Although the 120 lb of sludge could be contained in a single 60-qt mixing bowl, binder addition and mixing caused excessive splashing, resulting in material losses. To prevent splashing, the 25 percent sludge was divided into two portions. Each portion was placed in a separate 60-qt mixing bowl. The required amount of binding agent was added at the correct ratio to each sludge portion. Each sludge/binder mixture was mixed for

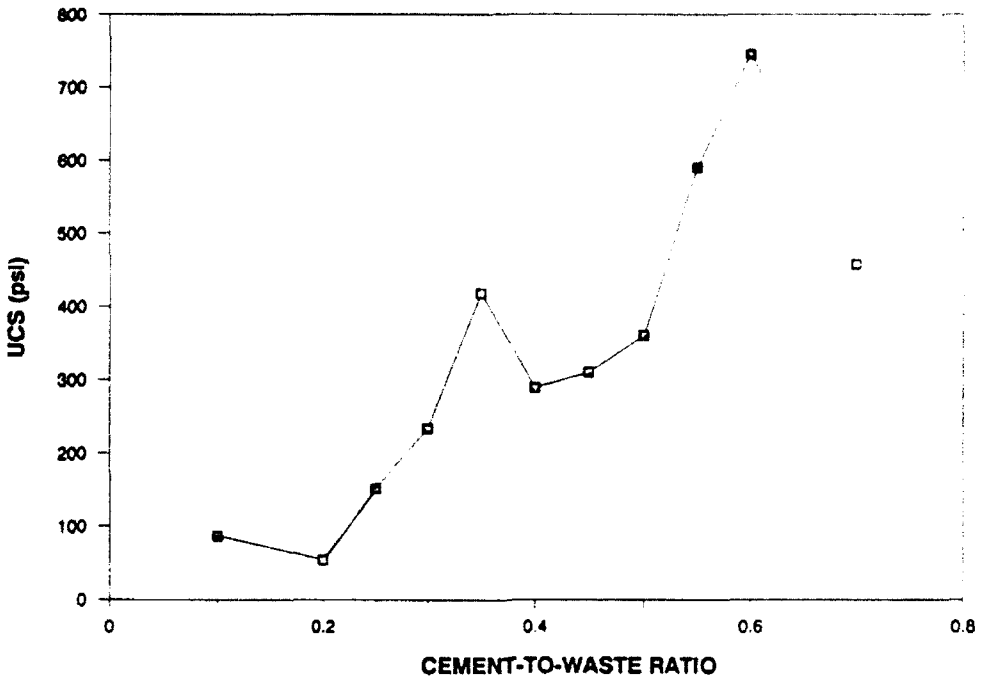

Figure 4. Initial screening results for the cement binder where the cement ratio is plotted versus the 28-day UCS results.

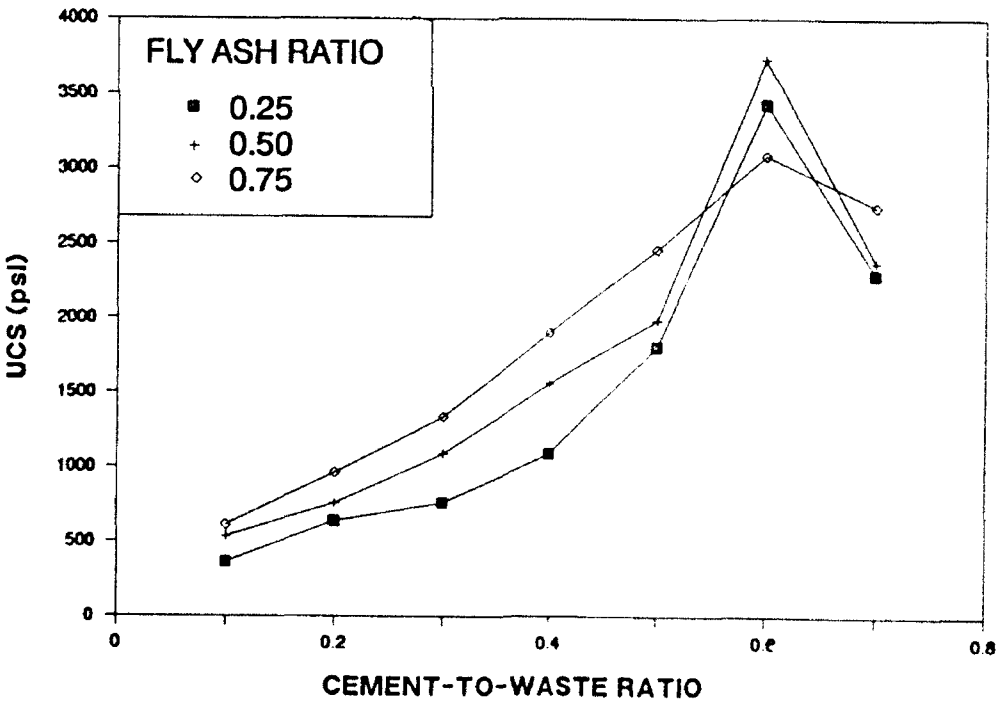

Figure 5. Initial screening results for the cement/fly ash binder where the cement ratio is plotted versus the 28-day UCS results for varying fly ash ratios.

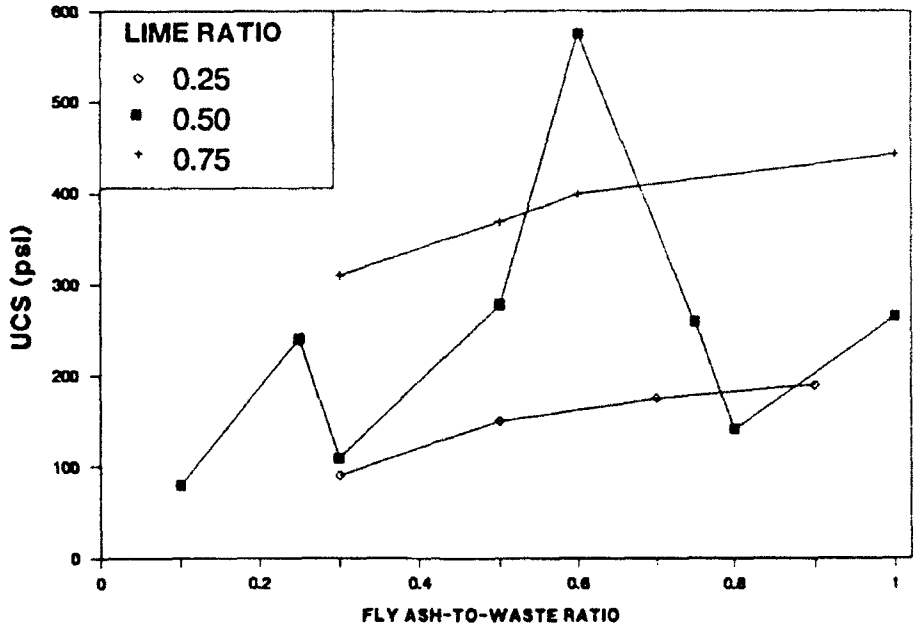

Figure 6. Initial screening results for the lime/fly ash binder where the fly ash ratio is plotted versus the 28-day UCS results for varying lime ratios.

TABLE 5. BINDER-TO-SLUDGE RATIOS SELECTED FOR EVALUATION

Binder	Binder/sludge ratio	Dilution factor*
Portland cement, type I	0.3:1 cement:sludge	5.2
Lime/fly ash, type C	0.3:1 lime:sludge 0.5:1 fly ash:sludge	7.2
Fly ash, type F/Portland cement, type I	0.2:1 cement:sludge 0.5:1 fly ash:sludge	6.8

* Weight of final mixture/weight of dry sludge.

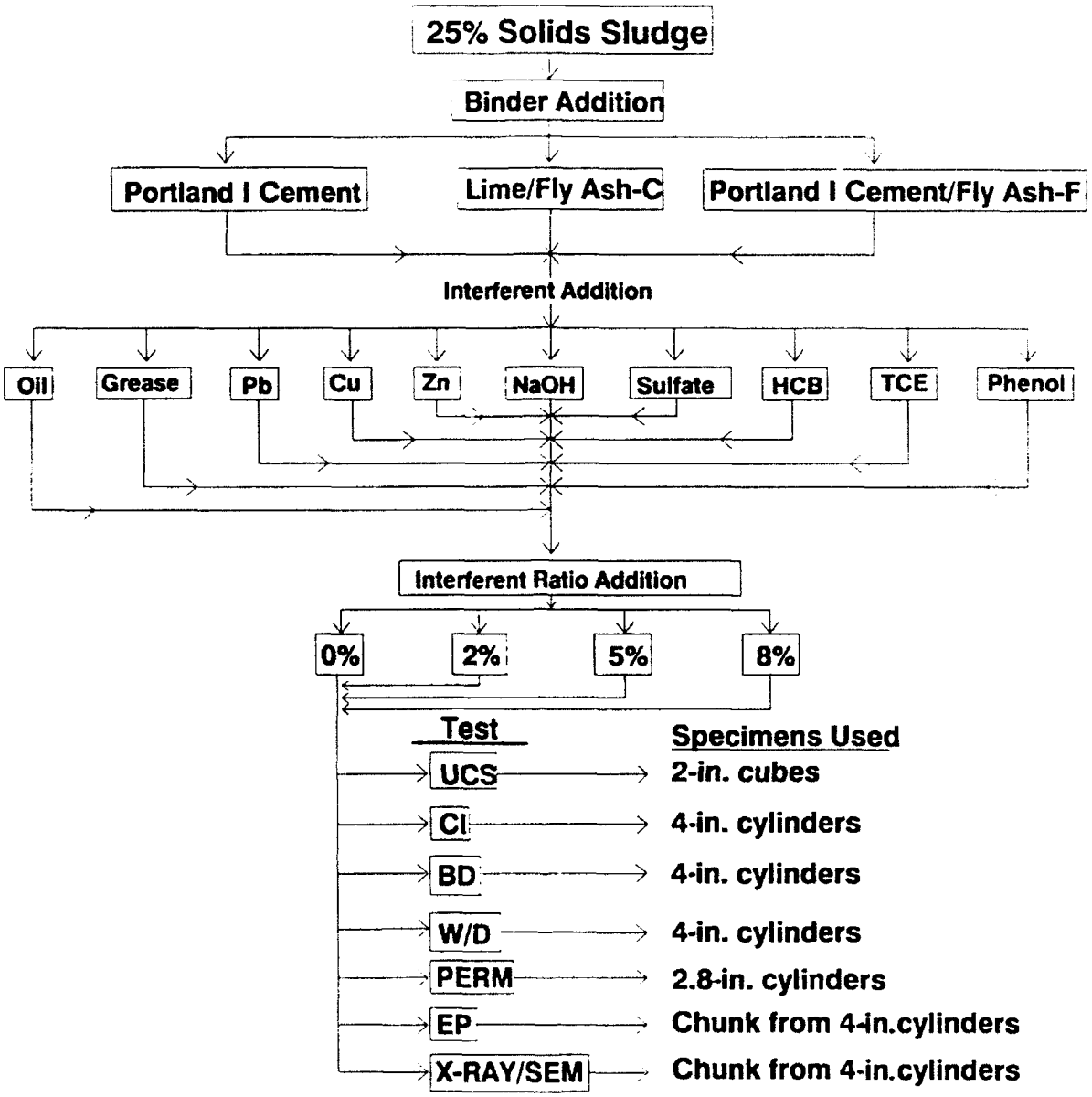

Figure 7. Flow diagram of specimen preparation.

10 min using a Hobart C-600 mixer. After mixing, the separate sludge/binder portions were recombined into a single bowl. To ensure that the sludge/binder was well mixed, the material was poured from the full bowl to the empty bowl a minimum of three times. The binder/sludge mixture was then divided into four 40-lb portions. The desired interfering chemical was added to three of the portions at interference-to-binder/sludge weight ratios of 0.02, 0.05, and 0.08, respectively. These ratios are referred to as percentage levels throughout the remainder of this report. Thus, a 0 ratio = 0 percent, a 0.02 ratio = 2 percent, a 0.05 ratio = 5 percent, and a 0.08 ratio = 8 percent by definition. The fourth portion had no interfering chemical added, and served as the control for each batch.

Chemical reagents were used to introduce the desired interference into the sludge/binder system. Many of the reagents used contained inert substances (such as waters of hydration), which added to the reagent's weight. Because it was desired to investigate the effects of the interference chemical at specific ratios, weight adjustment factors were applied to compensate for any inert materials contained in the reagents. For example, for every pound of lead interference desired, 1.6 lb of lead nitrate was actually added to the sludge/binder mixture. The desired interference, the interference reagent used, and the weight correction factors are listed in Table 6.

TABLE 6. INTERFERENCE REAGENTS

Desired interference	Interference reagent	Weight correction factor
Oil	30 weight motor oil	1.00
Grease	Axle grease	1.00
Phenol	Phenol	1.00
Sulfates	Sodium sulfate [Na_2SO_4]	1.48
Strong base	Sodium hydroxide [NaOH]	2.35
Degreaser	Trichloroethylene [TCE]	1.00
Pesticide	Hexachlorobenzene [HCB]	1.00
Lead	Lead nitrate [$Pb(NO_3)_2$]	1.60
Copper	Copper nitrate [$Cu(NO_3)_2 \bullet 3H_2O$]	3.80
Zinc	Zinc nitrate [$Zn(NO_3)_2 \bullet 6H_2O$]	4.55

The three portions containing interfering agent were placed in nine 4- by 4.5-in. cylindrical, carbon-steel molds (4-in. cylinder); one 2.8- by 2.8-in. cylindrical, carbon-steel mold (2.8-in. cylinder), and twelve 2-in. brass cube molds (2-in. cube). The remaining portion, containing no interfering chemical, was placed in eight 4-in. cylinders, one 2.8-in. cylinder, and fifteen 2-in. cubes. Immediately after the interference/binder/sludge mixture was placed in the molds, they were vibrated on a Sentron model VP61D1 vibration table to remove voids.

All specimens were cured in the molds at 23° C and 98 percent relative humidity for a minimum of 24 hr. Specimens were removed from the molds when they developed sufficient strength to be free standing and were cured under the same temperature and relative humidity conditions until required for further testing.

PHYSICAL TESTING

Five physical tests were performed on all solidified/stabilized samples: unconfined compressive strength, cone index, bulk density, wet/dry cycling-durability, and permeability. The specifics of these tests are outlined below.

Unconfined Compressive Strength

The UCS test was used to determine the strength development characteristics of the various solidified/stabilized wastes. The UCS of the treated wastes was determined using ASTM method C 109-86, Compressive Strength of Hydraulic Cement Mortars (using 2-in. or 50-mm cube specimens) (ASTM 1988a). As previously discussed, the only deviation from this method was specimen vibration. The surface area of each cube was determined using a Flower Max-Cal caliper, and each cube was fractured using a Tinius Olsen Super L compression apparatus. The UCS was reported as the force per unit surface area (pounds per square inch) required to fracture the cube.

As shown in Table 7, either four or five replicate specimens were used to determine the UCS during the first week of cure, during the second week of cure, and at the 28th day of cure. Flexibility in the testing schedule during the initial curing period was required because some of the interfering chemicals significantly retarded initial strength development, and the materials could not be removed from the molds until late in the first week of cure.

TABLE 7. UCS SAMPLES EVALUATED

Interference ratio	Number of samples tested		
	Sample age 1-7	Sample age 8-14	Sample age 28
0.0	5	5	5
0.02	4	4	4
0.05	4	4	4
0.08	4	4	4

The USC determination during the second week of cure was performed 1 week after the first UCS determination.

Cone Index

The cone index (CI) was used to evaluate the strength development characteristics of the solidified/stabilized materials during the initial strength development period. The CI measures the resistance of a material to the penetration of a 30-deg right circular cone. The CI is measured using a penetrometer following the method specified in Technical Manual 5-530 (US Army 1971). The CI values are reported as force per unit area of the cone base required to push the cone through a test material at a rate of 72 in./min. Two penetrometers are available for this test. The standard US Army Engineer Waterways Experiment Station (WES) penetrometer has a cone with an area of 0.5 in.; the airfield penetrometer has a cone with an area of 0.2 in. It was convenient to use the standard WES cone on materials with a CI less than or equal to 100 psi and to use the airfield cone on materials with a CI greater than 100 psi.

The 4-in. cylinders were used for CI determination. The CI was determined by placing demolded specimens back into the molds and subjecting the specimens to testing with the penetrometer. A minimum of four specimens were tested for each interference ratio each time the CI measures were taken. Up to a total of nine CI measurements were taken per specimen. The CI measurements were collected until the CI value exceeded 750 psi, the maximum reading of the airfield penetrometer, or until 28 days of curing, whichever occurred first.

Bulk Density

Bulk density (BD) measurements were performed to evaluate density changes resulting from the added interfering chemicals. While a standard method was not followed, the method used in this study was tailored after ASTM method D-558-82 (ASTM 1988c). The method selected for this study used 4-in. cylindrical specimens. Each specimen was weighed, and the dimensions of the sample were determined. Small metal tags were placed in each specimen for identification purposes. This tag also helped in aligning the specimen on the measuring jig (Figure 8). The height and diameter of each specimen were determined using a Flower Max-Cal caliper. The specimens were placed on the measuring jig, and the metal tag was aligned with marks 2 and 4 on the jig (Figure 9). The height for each specimen was determined at marks 1 through 4 on the jig. The diameter was determined at marks 1 and 3, and at 2 and 4 for both the top and bottom surfaces for each sample. Thus, the height and diameter were determined in four distinct areas for each specimen.

The bulk density was calculated using the equations shown below.

$$Bulk\ density = \frac{SM}{SV} \tag{1}$$

where SM = mass of the specimen, g
SV = volume of the specimen, mm^3 (calculated using Eq. 2)

$$SV = AH \times \frac{AD^2}{4} \times \pi \tag{2}$$

where AH = average height of the specimen, mm (as defined by Eq. 3)
AD = average diameter of the specimen, mm (as defined by Eq. 4)

$$AH = \frac{H_1 + H_2 + H_3 + H_4}{4} \tag{3}$$

where H_1 = height determined at jig mark 1
H_2 = height determined at jig mark 2
H_3 = height determined at jig mark 3
H_4 = height determined at jig mark 4

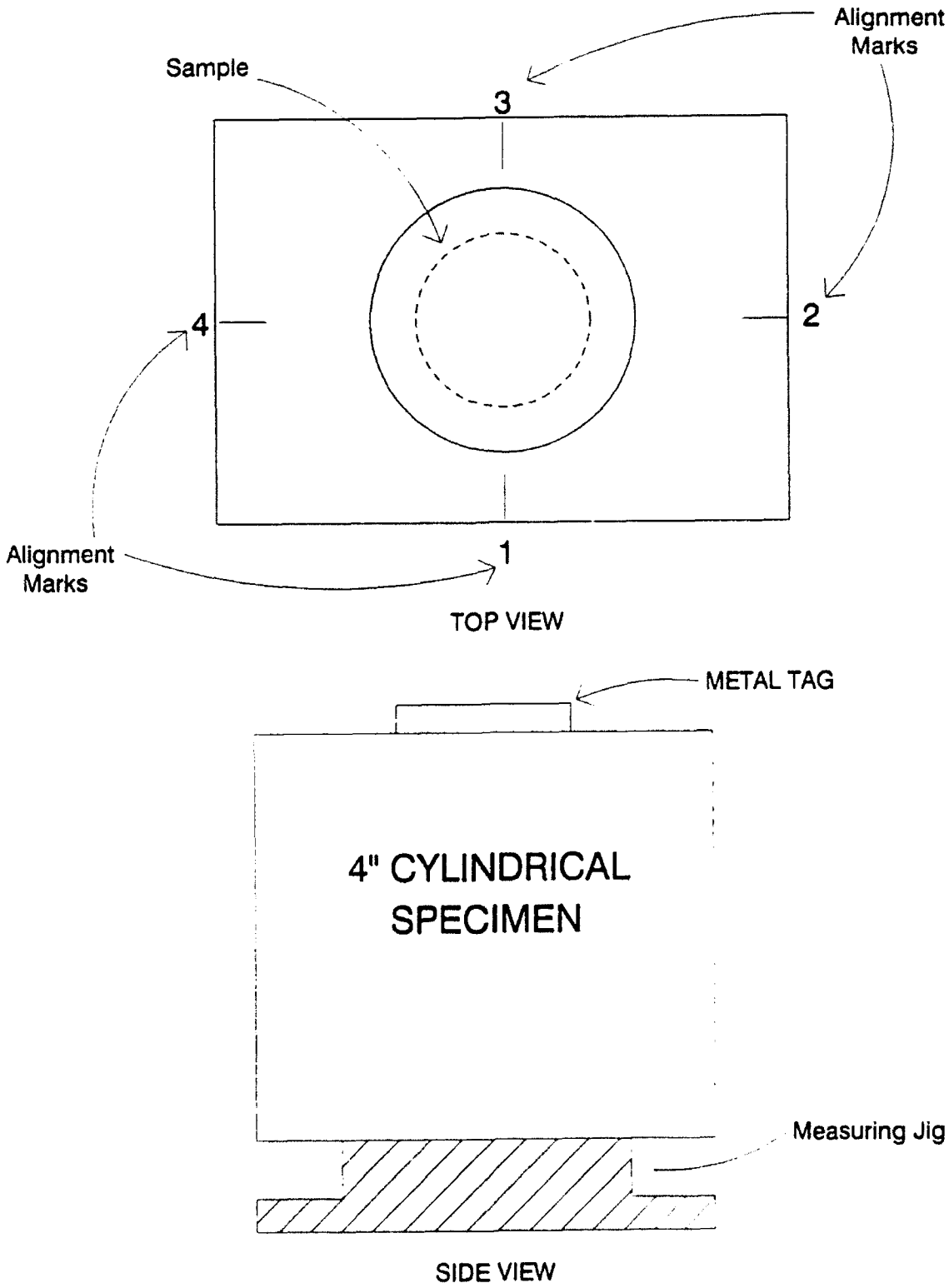

Figure 8. Measuring jig used in the bulk density determinations.

Figure 9. Measurement of a bulk density sample.

$$AH = \frac{D_{t1} + D_{t2} + D_{b1} + D_{b2}}{4} \tag{4}$$

where D_{t1} = top diameter determined at jig marks 1 and 3
D_{t2} = top diameter determined at jig marks 2 and 4
D_{b1} = bottom diameter determined at jig marks 1 and 3
D_{b2} = bottom diameter determined at jig marks 2 and 4

To determine if any large changes in volume or weight occurred as the specimens cured, the bulk density was determined once during the first week of curing and again after 28 days of curing. Bulk densities for a minimum of four specimens were determined at each interference level.

Wet/Dry Cycling-Durability (W/D)

This testing was performed to measure the specimens' durability to cyclic weathering conditions and the effect of the interference chemicals on sample durability. A modified ASTM 599-82 (ASTM 1988c) was conducted on specimens after they had cured for 28 days. This method consisted of placing a minimum of two 4-in. cylinders in a forced air oven at 60° C. These specimens were designated as samples A and B. To limit sample handling, both samples were placed on preweighed sample holders (Figure 10). Once placed on the sample holders, the samples were not removed until W/D testing was completed.

Samples A and B were dried in the oven at 60° C for 48 hr. At the end of this drying period both samples were removed from the oven, cooled, and weighed on a Mettler model PE-6000 balance. Sample A was placed back in the 60° C oven, and sample B was immersed in ASTM type III water (ASTM 1988d) for a 5-hr period. At the end of this immersion period, sample B was placed in the oven and dried for 42 hr. After the drying period specimens A and B were removed from the oven and allowed to cool. The specimens were then brushed on all sides with a soft-haired paint brush to remove any loose material. The base of the sample holders was also brushed to removed all fallen material from the base. Then, specimens A and B were reweighed to determine the weight of material lost. One complete cycle consisted of a 5-hr immersion period and a 42-hr drying period (for sample B). A total of 11 cycles comprise one wet/dry test. Disintegration of over 70 percent of the original sample was recorded as failure of a product.

Sample A was carried through this test to serve as a W/D control. Any material loss resulting from heating the sample or from sample aging appeared as a weight reduction for sample A.

Wet/dry testing on sample B was run in duplicate, except for the 0-percent interference level, for which a single sample was run.

Permeability

Permeability (PERM) was determined using the 2.8-in. specimens. A falling head permeability test using a triaxial cylinder was used in all permeability measurements. Specimens were saturated using a back-pressure saturation technique following the method outlined in Appendix VII of Engineer Manual 1110-2-1906 (U.S. Army Corps of Engineers 1970). Triplicate permeability determinations were performed on a single individual specimen that had cured a minimum of 28 days.

CHEMICAL EVALUATION OF CONTAMINANT RELEASE

Extraction Procedure (EP) Toxicity Test

USEPA Method 1310 (USEPA 1986) was the method followed with the exception that all materials leached were ground to pass a 9.5-mm sieve. The solid materials used in the extraction were composited from the 4-in. cylinders. This extraction consisted of contacting dilute acetic acid with approximately 100 g of ground waste using a 20:1 liquid-to-solid ratio. The duration of the test varied from 24 to 28 hr, depending upon the waste's alkalinity. After the samples were extracted, the liquids were separated from the solids using a Millipore (HAWP 142-50) membrane pressure filter.

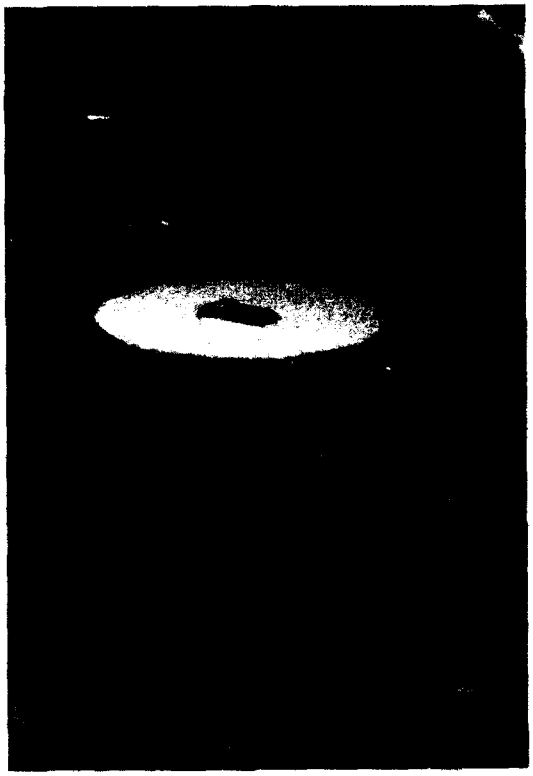

Figure 10. Wet/dry sample holders and samples.

Each interference/binder/sludge mixture at each interference concentration was extracted in duplicate after the solidified/stabilized materials had cured for 28 days. Duplicates were prepared by compositing ground samples and dividing this sample into two portions. Each time a series of EP extractions was executed, nine individual extractions were performed, including a duplicate extraction for the four interference concentrations (eight extractions) and a method blank. The EP extracts were analyzed for Cd, Cr, Hg, Ni, and the interference contaminant of interest according to the analytical methods prescribed by USEPA (1986) and outlined in Table 8.

TABLE 8. ANALYTICAL CHEMICAL METHODS

Contaminant of interest	USEPA analytical method	Detection limit mg/ℓ (ppm)
	Sludge Contaminants	
Cadmium	7131	0.0001
Chromium	7191	0.001
Nickel	7521	0.001
Mercury	7470	0.0002
	Interference Components	
Oil and grease	413.1	5
Phenol	420.2	0.002
TCE	624	0.0019
HCB	625	0.0019
NaOH (as Na)	6010	0.100
Na_2SO_4 (as SO_4)	375.2	3
Lead	7421	0.001
Zinc	6010	0.03
Copper	7211	0.001

Microchemical/Micromorphological Characterization

The microchemical/micromorphological characteristics of the solidified/stabilized materials were investigated to characterize the effects of the interference chemicals on microscopic properties of the samples. All microchemical/micromorphological analyses were performed by Louisiana State University's (LSU) Hazardous Waste Research Center. Three analytical techniques were employed by LSU to study the solidified/stabilized samples--scanning electron microscopy (SEM), SEM in conjunction with energy dispersive X-ray analysis (EDXRA), and X-ray diffraction (XRD).

Sample Preparation--

Large pieces of samples were collected from the 4-in. cylindrical specimens. Specimens were collected after the 28-day cure time and sealed in 500-ml polyethylene bottles. The specimens were shipped to LSU for analysis shortly after collection. Pure sludge samples (sludge controls) were also sent to LSU to be used for comparison purposes, along with binder control samples. Binder control samples were prepared by mixing binder with clean sand. The sand was added to simulate the sludge solids, and the amount added corresponded to the binder/sludge ratios used in the study.

Scanning Electron Microscopy and Energy Dispersive X-Ray Microanalysis--

The SEM technique allows visual observations of the micromorphology of the sample surface at magnifications ranging from 25× to 15,000×. From these observations, macroscopic properties, such as the nature and degree of sample

porosity (Timur 1971 and Welton 1984) can be inferred. In some cases, micromorphology can be used to identify chemical phases by comparing observed phases to known phases.

The EDXRA technique is used in conjunction with SEM. EDXRA provides local elemental analysis of the specimen viewed at a point on the viewing screen or over the entire area visible on the screen. SEM coupled with EDXRA is a useful tool to subjectively characterize specific phases contained in the sample. Although quantitative elemental analysis is possible with EDXRA, this requires an extremely smooth, flat surface. The samples analyzed using EDXRA for this study were not conducive to proper preparation for quantitative analysis; thus, only qualitative analysis was provided by the EDXRA.

Both SEM and EDXRA were performed using an ISI 60A scanning electron microscope equipped with an EDAX 9100 energy dispersive spectrometer. Two separate specimens from each sample shipped to LSU were prepared for SEM and EDXRA. Specimens were prepared by immersing them in liquid nitrogen for 1 hr and fracturing the specimen into small centimeter-sized pieces. The small pieces were coated with gold for SEM analysis and with carbon for EDXRA analysis. Carbon was used for EDXRA because the spectrometric peaks of gold overlapped with the contaminant peaks of the waste. The EDXRA spectrum was collected for 200 live seconds, with a count rate of approximately 3,000 counts per second.

X-Ray Diffraction--

The XRD analysis is used to identify the crystalline phases of the bulk sample. Spectra of an unknown sample are compared to known standard spectra for peak identification. XRD spectra may also indicate the crystallinity of a material, which may relate to the strength properties of the specimen.

The XRD analyses for this study were performed with a Rigaku DMAX automated X-ray diffractometer. The intensities were digitally recorded. For these runs, Mo K_a and Cu K_a radiation was used. The slit widths were 4 and 1 mm, respectively. The voltage for the Cu K_a radiation was 40 kilovolts; the current was 30 milliamperes. The samples were scanned from 3 to 70 deg two-theta at a scan rate of 0.02 deg two-theta per second for the Cu K_a runs. The Mo K_a runs used a two-theta value that ranged from 3 to 36 deg at a step width of 0.01 deg two-theta.

Aluminum (Al) or alumina (Al_2O_3) powder was typically added to the samples as an internal standard for calibration purposes. Quartz, a component of fly ash, was also used as an internal standard. By using a component of the sample for calibration, problems with sample dilution were avoided.

Attempts were made to identify the phases using the XRD patterns by matching them to Joint Committee on Powder Diffraction Standards (1984, 1986) Powdered Diffraction File data. This was difficult, and very few identical matches were observed. To identify changes in the interfered sludge, samples of pure sludge, pure binder, and solidified/stabilized sludge without interference addition were used as references.

QUALITY ASSURANCE AND QUALITY CONTROL

A detailed Quality Assurance Project Plan (QAPP) was prepared for this study (Bricka 1986). As stated in the QAPP, the overall objective of the quality assurance and quality control (QA/QC) measures was to provide information to support the quality of data collected in association with this study. In an effort to meet the QA/QC objectives, the procedures outlined in the QAPP were followed as closely as possible.

The QA/QC measures associated with this study are mainly limited to those affiliated with the chemical analysis of the EP extracts. The QA/QC data are presented in Appendix B.

The EP extractions were performed in groups of nine samples. These nine samples included the extraction of duplicate samples of the 0, 2, 5, and 8 percent interference samples for a single interference chemical and a single binder, and a method blank. Intermittently, an additional sample of known concentration was submitted to the lab for analysis with this set of nine samples. This sample is referred to as an external standard. Internal QA/QC procedures were those preformed by the analytical laboratory.

The QA/QC procedures affiliated with the chemical analysis of the EP extracts include the following:

- External method blank samples--These are blank samples that underwent all the procedures that were performed on the actual samples. A contaminated blank could reflect contamination from the reagents, sample container contamination, and/or contamination associated with the digestion and filtration steps. One external method blank was submitted with every set of eight samples.

- External standards--USEPA-certified or National Bureau of Standards (NBS) traceable standards are submitted to the analytical laboratory as part of the sample set (blind standards). External standards were submitted intermittently through this study.

- External duplicate samples--An external duplicate sample consists of the extraction of separate solid specimens generated from the same batch of material. Duplicate samples provide a measure of sample variability. Duplicate sample analysis was performed for each EP extraction.

- Internal split samples--The analytical laboratory divided a sample into two portions. Each portion was analyzed independently. One internal split sample was run for every eight samples analyzed.

- Internal spiked samples--Samples were spiked with known concentrations of a reference material and carried through the analyses. Results of the spike samples were reported as the percent of the reference material recovered, and are indicative of analytical variability. One internal spiked sample was run for every eight samples analyzed.

- Internal standards--These are USEPA or NBS traceable standards used for calibration verification by the analytical laboratory. Internal standards were performed intermittently.

SECTION 3

RESULTS AND DISCUSSION

VISUAL OBSERVATIONS

Prior to testing, visual observations of the samples were recorded. While visual observations are subjective, they may indicate potential problems that may be reflected by the physical and chemical tests. These observations are presented below.

No significant visual differences were noted between the controls and the samples with added organic interferences (phenol, TCE, HCB, oil, and grease), even at the 8-percent interference level. Very little change was noted in the sample to which Pb was added. In contrast, significant changes were observed for the NaOH, Zn, Cu, and sulfate interferences. For all the binders studied, the samples with added NaOH appeared to be "wetter" or adsorb more water than the other samples. The CEM and CFA binders with as little as 5 percent Zn produced samples that were very soft and difficult to remove from the molds, even after 11 days of curing. This was not observed for Zn samples where LFA was used as a binder. The CEM and CFA samples with 5- and 8-percent Cu additions were noted to have developed about a 1/4-in. "crust" layer on the surface of the sample. This layer was very hard, but beneath the crust the sample appeared to be soft. No observations of the crust were recorded for the LFA binder; however, for the 8-percent Cu samples, it was observed that the samples were not hard but were "elastic" even after 28 days of cure. Surface cracks were observed in the CFA samples with 5- and 8-percent sulfate addition as early as 11 days of curing. No cracks were observed in 8-percent sulfate samples for the CFA binder, although these samples were "elastic" (like the LFA-Cu sample) early in the curing process. Only slight surface scaling was observed for the CEM-sulfate samples.

BULK DENSITY

Bulk densities of all samples were determined at 1 and 28 days of curing. The bulk density of the S/S products varied between binders and with the addition of different interferents.

Bulk Densities of Samples Without Interferent

The average bulk density of the control samples for the different binders is summarized in Table 9. The waste products solidified/stabilized with

TABLE 9. SUMMARY OF BULK DENSITY PARAMETERS FOR CONTROL SAMPLES

Binder	Bulk density (g/cc)			Coefficient of variation (%)
	Average	Lowest	Highest	
Cement	1.268	1.093	1.393	7.8
Cement/fly ash	1.407	1.240	1.501	6.1
Lime/fly ash	1.523	1.488	1.574	7.8

the CEM had bulk densities 15 to 20 percent lower than those solidified/stabilized with CFA or LFA, with LFA producing the highest bulk densities. The amount of variation seen in bulk density was consistently between 6 and 8 percent coefficient of variation (CoV) for all binder systems.

Effects of Interferent Additions

The bulk densities and percent of control values for the samples with interferents at 1 and 28 days of cure are arranged by interferent in Tables 10 and 11. Bulk density comparisons are also shown in Figures 11-13. The bulk densities of the samples containing metal interferent increased proportionally with the amount of metal added. Wastes solidified/stabilized with the CEM and CFA binders were affected to the greatest extent. Copper increased the density to the greatest extent (to about 135 percent of control), followed by zinc (to about 128 percent) and by lead (at 120 percent). The metals had a smaller effect on the bulk density of the more dense LFA solidified/stabilized samples, increasing them only 6 to 8 percent. Sodium hydroxide and sodium sulfate also increased the density of the final product by about 10 percent at the highest addition level (8 percent w/w).

Grease and oil interferents decreased the density of the final product, as would be expected from their lower densities. Densities of samples with 8-percent addition averaged about 90 percent of the control densities. The other organic interferents had smaller effects upon the densities of the solidified/stabilized products.

Changes in Bulk Density and Volume with Curing Time

Changes in sample densities between day 1 and day 28 of curing are summarized in Table 12. No appreciable differences in density were seen as the specimens cured, with most samples retaining between 90 and 100 percent of their original densities. Changes in density would be brought about by loss of water through evaporation or by changes in the volume of the final product during curing.

Changes in overall volume of the solidified/stabilized samples are also summarized in Table 13. Only sulfate addition seemed to have an appreciable effect on the measured volume of the curing products. The 8-percent sulfate addition appears to cause shrinking of all binder systems with volume changes observed from about 8 to 10 percent. Phenol may also cause slight shrinkage. Samples from all three binders with 8-percent phenol addition are 2 to 3 percent smaller at 28 days than at 1 day. Copper interference produces significant shrinkage in the LFA samples, up to 30 percent at 8 percent addition of copper. Zinc added to the cement solidified/stabilized samples may cause some swelling, but the data are not significantly different at the 0.05 significance level. All other products are within 1 to 2 percent of their original volume after the 28-day curing period. The measurements made here are not precise enough to detect changes in volume smaller than about 1 percent.

TABLE 10. BULK DENSITY AND PERCENT OF CONTROL OF S/S PRODUCTS WITH AND WITHOUT INTERFERENT AFTER 1 DAY OF CURE

		Bulk-density (g/cm^3)				Percent of control		
Binder	Interferent	0%	2%	5%	8%	2%	5%	8%
CEM	Copper	1.357	1.390	1.467	1.563	102	108	115
CFA	Copper	ND	ND	ND	ND	--	--	--
LFA	Copper	1.550	1.592	1.590	1.598	102	102	103
CEM	Lead	1.385	1.431	1.479	1.549	103	106	111
CFA	Lead	1.431	1.467	1.571	1.574	102	109	110
LFA	Lead	ND	ND	ND	ND	--	--	--
CEM	Zinc	1.391	1.442	1.464	1.476	103	105	106
CFA	Zinc	ND	ND	ND	ND	--	--	--
LFA	Zinc	1.548	1.576	1.560	1.615	101	100	104
AVG	Metals	1.443	1.483	1.522	1.562	102	105	108
CEM	NaOH	1.366	1.383	1.506	1.510	101	110	110
CFA	NaOH	1.521	1.523	1.547	1.601	100	101	105
LFA	NaOH	1.573	1.576	1.603	1.633	100	101	103
CEM	Sulfate	1.388	1.422	1.461	1.494	102	105	107
CFA	Sulfate	1.568	1.558	1.572	1.678	99	100	107
LFA	Sulfate	1.556	1.579	1.585	1.634	101	101	105
AVG	Na salts	1.495	1.507	1.546	1.592	100	103	106
CEM	Grease	1.400	1.386	1.360	1.306	99	97	93
CFA	Grease	1.541	1.519	1.495	1.459	98	97	94
LFA	Grease	1.555	1.533	1.481	1.444	98	95	92
CEM	Oil	1.396	1.341	1.308	1.242	96	93	88
CFA	Oil	1.538	1.519	1.458	1.337	98	94	86
LFA	Oil	1.556	1.486	1.438	1.424	95	92	91
CEM	TCE	1.402	1.388	1.376	1.376	99	98	98
CFA	TCE	1.538	1.536	1.533	1.530	99	99	99
LFA	TCE	1.537	1.572	1.544	1.552	102	100	101
CEM	HCB	1.382	1.370	1.358	1.346	99	98	97
CFA	HCB	1.509	1.515	1.504	1.510	100	99	100
LFA	HCB	1.515	1.524	1.531	1.527	100	101	100
CEM	Phenol	1.386	1.395	1.389	1.406	100	100	101
CFA	Phenol	1.492	1.494	1.497	1.498	100	100	100
LFA	Phenol	1.561	1.561	1.543	1.533	100	98	98
Avg	Organics	1.480	1.484	1.475	1.475	100	99	99

Note: ND = no data.

TABLE 11. BULK DENSITY AND PERCENT OF CONTROL OF S/S PRODUCTS WITH AND WITHOUT INTERFERENT AFTER 28 DAYS OF CURE

		Bulk density (g/cm^3)				Percent of control		
Binder	Interferent	0%	2%	5%	8%	2%	5%	8%
CEM	Copper	1.093	1.169	1.498	1.525	107	137	140
CFA	Copper	1.266	1.344	1.589	1.655	106	125	131
LFA	Copper	ND	ND	ND	ND	--	--	--
CEM	Lead	1.279	1.346	1.407	1.513	105	110	118
CFA	Lead	1.330	1.374	1.476	1.603	103	111	121
LFA	Lead	1.521	1.543	1.560	1.646	101	103	108
CEM	Zinc	1.111	1.263	1.519	1.475	114	137	133
CFA	Zinc	1.240	1.453	1.546	1.551	117	125	125
LFA	Zinc	1.505	1.568	1.590	1.599	104	106	106
AVG	Metals	1.314	1.398	1.526	1.562	107	118	120
CEM	NaOH	1.360	1.406	1.439	1.494	103	106	110
CFA	NaOH	1.441	1.508	1.549	1.591	105	108	110
LFA	NaOH	1.536	1.591	1.605	1.634	104	104	106
CEM	Sulfate	1.199	1.327	1.387	1.427	111	116	119
CFA	Sulfate	1.389	1.436	1.486	1.527	103	107	110
LFA	Sulfate	1.516	1.598	1.599	1.598	105	105	105
CEM	Grease	1.321	1.300	1.266	1.204	98	96	91
CFA	Grease	1.501	1.506	1.445	1.399	100	96	93
LFA	Grease	1.520	1.485	1.494	1.448	98	98	95
CEM	Oil	1.271	1.244	1.201	1.094	98	94	86
CFA	Oil	1.451	1.421	1.354	1.211	98	93	83
LFA	Oil	1.546	1.458	1.389	1.368	94	90	89
AVG	Grease/oil	1.435	1.402	1.358	1.287	98	95	90
CEM	TCE	1.368	1.325	1.373	1.381	97	100	101
CFA	TCE	1.470	1.500	1.525	1.397	102	104	95
LFA	TCE	1.506	1.537	1.494	1.510	102	99	100
CEM	HCB	1.350	1.336	1.322	1.333	99	98	99
CFA	HCB	1.458	1.454	1.441	1.430	100	99	98
LFA	HCB	1.488	1.482	1.510	1.513	100	101	102
CEM	Phenol	1.393	1.404	1.385	1.389	101	99	100
CFA	Phenol	1.455	1.497	1.491	1.491	103	102	103
LFA	Phenol	1.574	1.572	1.538	1.513	100	98	96
Avg	Organic	1.427	1.433	1.436	1.424	100	101	100

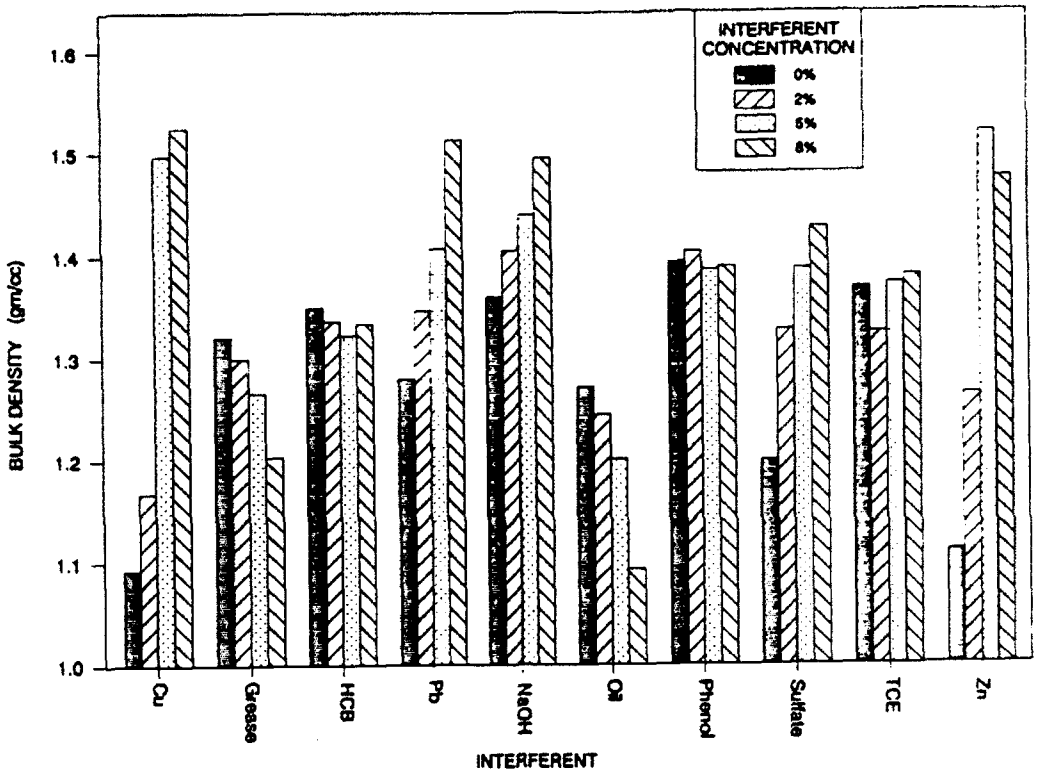

Figure 11. Bulk density of the CEM solidified/stabilized samples after 28 days of cure.

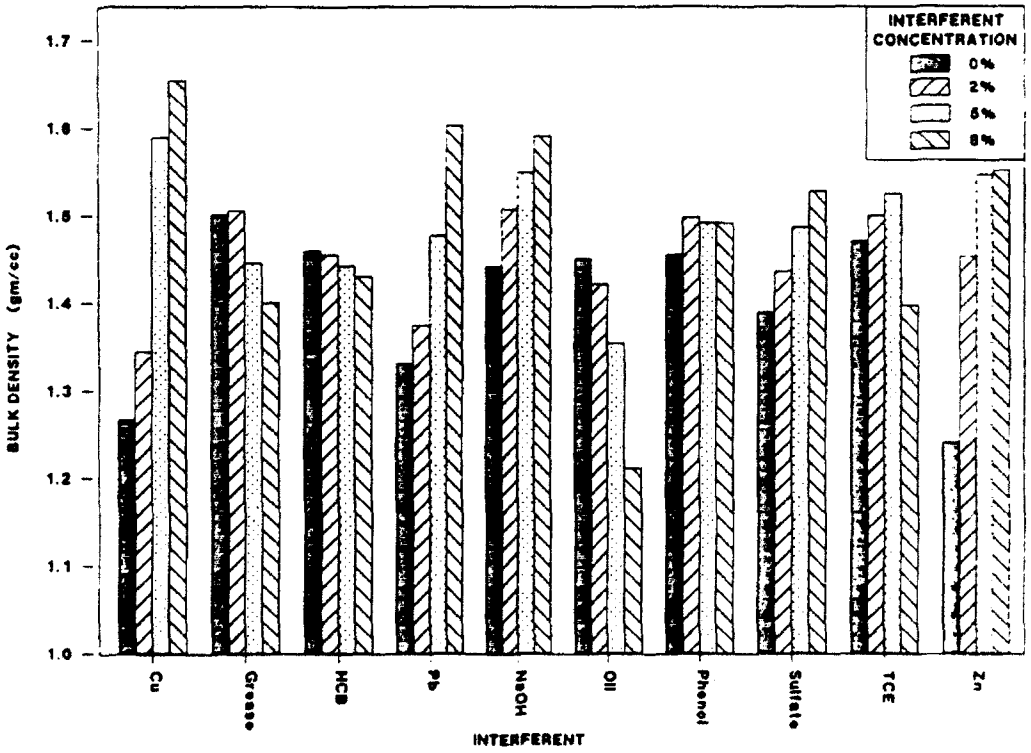

Figure 12. Bulk density of the CFA solidified/stabilized samples after 28 days of cure.

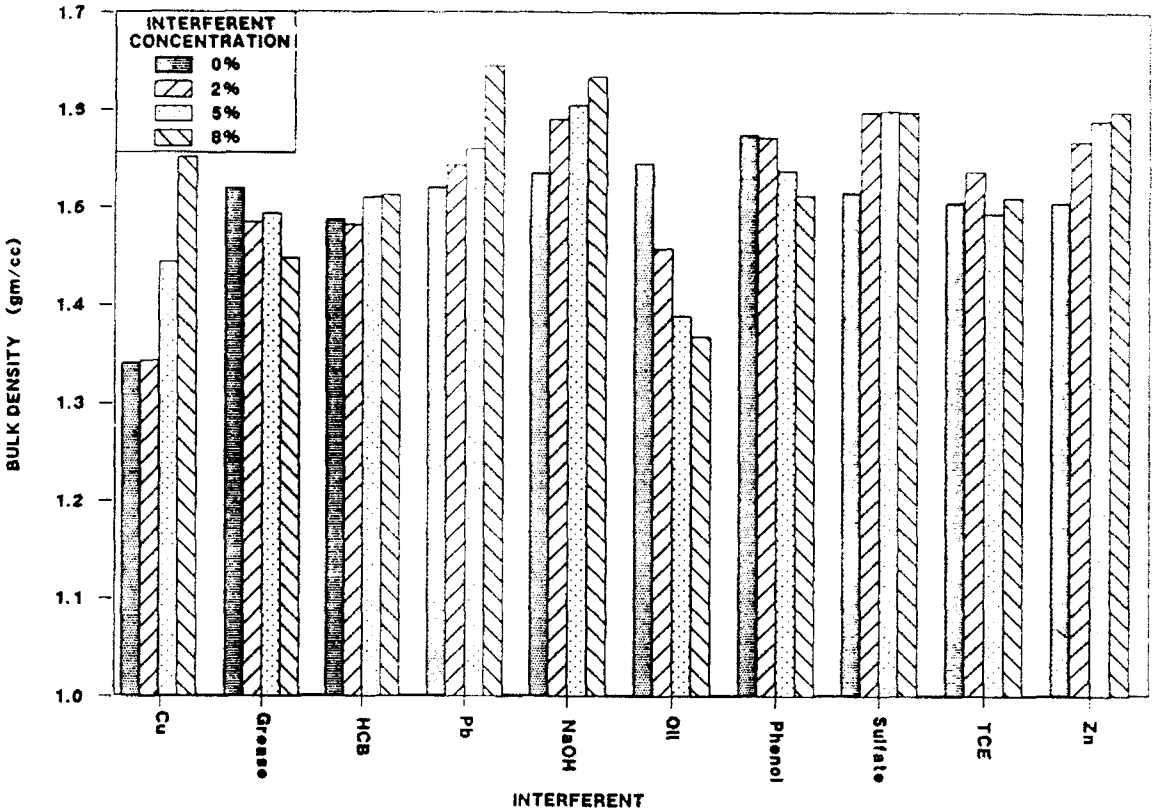

Figure 13. Bulk density of the LFA solidified/stabilized samples after 28 days of cure.

UNCONFINED COMPRESSIVE STRENGTH

The UCS of samples of all mixes was analyzed during the first week (day 1 to 4), the second week (day 7 to 11), and the fourth week (after 28 days of cure). The UCS of samples containing three levels of interferent was compared to samples without interferent (controls).

Samples Without Added Interferent

The average UCS in the second and fourth weeks for samples without interferent (controls) are summarized in Table 14. The CEM mixes were designed to give at least 100 psi UCS for the 2-in. cubes after 28 days of curing. UCS of control mixes after 28 days of curing varied from a low of 42 to a high of 240 psi with an average of 112 psi and a CoV of 58 percent. The range of UCS, by time of cure, for the CEM controls is shown in Figure 14. The variation in strength for the control samples, which were prepared identically, is clearly illustrated. The cement mixes had about 75 percent of their 28-day UCS after 7 to 11 days of cure. To produce samples with UCS values in this low range, the proportions of the reagents and mixing parameters of the batch are quite critical. Although great care was taken to reduce variability associated with the batch preparation step, and reagent addition was carefully controlled, substantial variability was observed in the control specimens. The variability within a specific batch or mix was low; however, variability

TABLE 12. BULK DENSITY AFTER 28 DAYS OF CURE AS A PERCENTAGE OF THAT AT DAY 1

		Percentage $((BD_{28}/BD_1)*100)$			
Binder	Interferent	0%	2%	5%	8%
CEM	Copper	80	84	102	97
CFA	Copper	ND	ND	ND	ND
LFA	Copper	ND	ND	ND	88
CEM	Lead	92	94	95	97
LFA	Lead	ND	ND	ND	ND
CFA	Lead	92	93	94	101
CEM	Zinc	79	87	103	99
CFA	Zinc	ND	ND	ND	ND
LFA	Zinc	97	99	101	99
CEM	Grease	94	93	93	92
CFA	Grease	97	99	96	95
LFA	Grease	97	96	100	100
CEM	Oil	91	92	91	88
CFA	Oil	94	93	92	90
LFA	Oil	99	98	96	96
CEM	TCE	97	95	99	100
CFA	TCE	95	97	99	91
LFA	TCE	98	97	96	97
CEM	HCB	97	97	97	99
CFA	HCB	96	96	95	94
LFA	HCB	98	97	98	99
CFA	Phenol	97	100	99	99
LFA	Phenol	100	100	99	98
CEM	Phenol	100	100	99	98
CEM	Sulfate	86	93	94	95
CFA	Sulfate	88	92	94	91
LFA	Sulfate	97	101	100	97
CEM	NaOH	99	101	95	99
CFA	NaOH	94	99	100	99
LFA	NaOH	97	100	100	100
Averages:					
All metals		88	91	99	97
All organics		97	97	97	96
Base + sulfate		94	98	97	97

TABLE 13. VOLUME AFTER 28 DAYS OF CURE AS A PERCENTAGE OF THAT ON DAY 1

Binder	Interferent	Interferent level 0%	2%	5%	8%
CEM	Copper	100	100	106	98
CFA	Copper	ND	ND	ND	ND
LFA	Copper	99	97	84	69
CEM	Lead	99	100	100	100
LFA	Lead	100	100	100	99
CFA	Lead	ND	ND	ND	ND
CEM	Zinc	101	101	113	109
CFA	Zinc	ND	ND	ND	ND
LFA	Zinc	100	99	101	97
CEM	Grease	101	101	101	101
CFA	Grease	97	98	99	100
LFA	Grease	101	101	100	100
CEM	Oil	101	100	101	98
CFA	Oil	100	100	100	99
LFA	Oil	101	100	100	100
CEM	TCE	98	99	99	100
CFA	TCE	98	99	98	99
LFA	TCE	101	99	100	101
CEM	HCB	99	99	99	99
CFA	HCB	99	99	99	99
LFA	HCB	101	101	100	99
CFA	Phenol	99	99	98	97
LFA	Phenol	100	99	97	96
CEM	Phenol	99	99	98	97
CEM	Sulfate	98	98	95	93
CFA	Sulfate	96	97	96	89
LFA	Sulfate	100	99	99	91
CEM	NaOH	100	99	95	99
CFA	NaOH	99	98	99	99
LFA	NaOH	100	99	99	99

TABLE 14. AVERAGE UNCONFINED COMPRESSIVE STRENGTH OF CONTROL MIXES AFTER 7 to 11 OR 28 DAYS OF CURING

	UCS (psi) after days of cure			
	7-11 days		28 days	
Binder	Avg	CoV*	Avg	CoV
Cement	84	58%	112	58%
Cement/fly ash	194	39%	624	23%
Lime/fly ash	249	14%	591	16%

* Coefficient of variation (standard deviation/treatment mean) times 100.

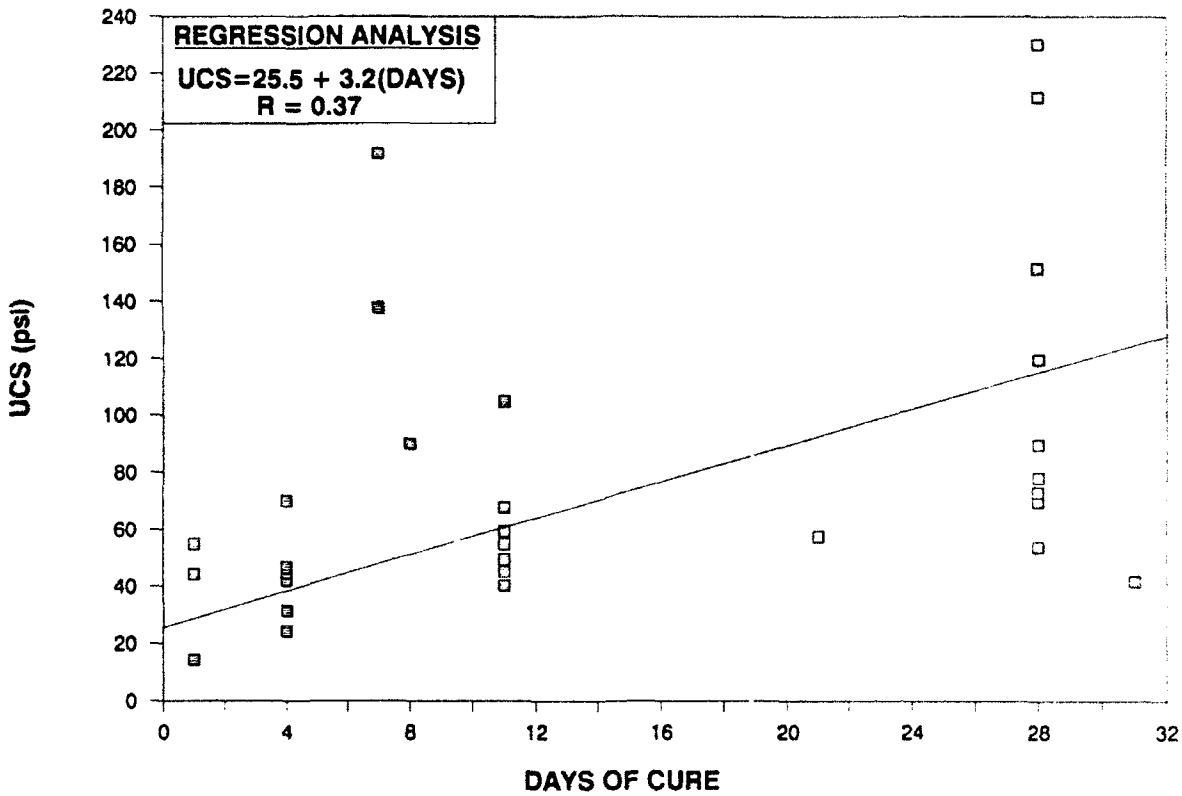

Figure 14. UCS for the CEM solidified/stabilized control samples.

between different mixes was appreciable. In all cases in this report, the properties of interferent samples have been compared only with their corresponding control samples.

The cement/fly ash and lime/fly ash mixes produced samples with higher compressive strengths, both exhibiting around 600 psi after 28 days of cure. As shown in Figures 15 and 16, the CFA and LFA control mixes also had less

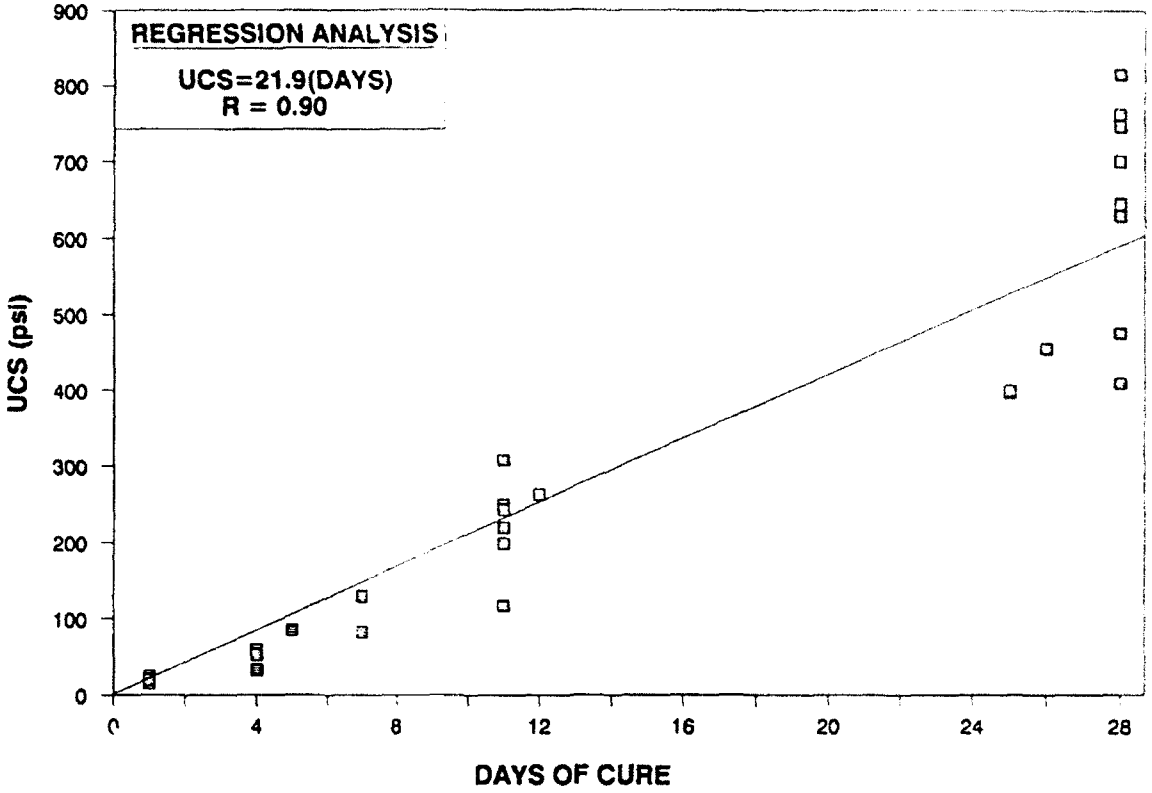

Figure 15. UCS for the CFA solidified/stabilized control samples.

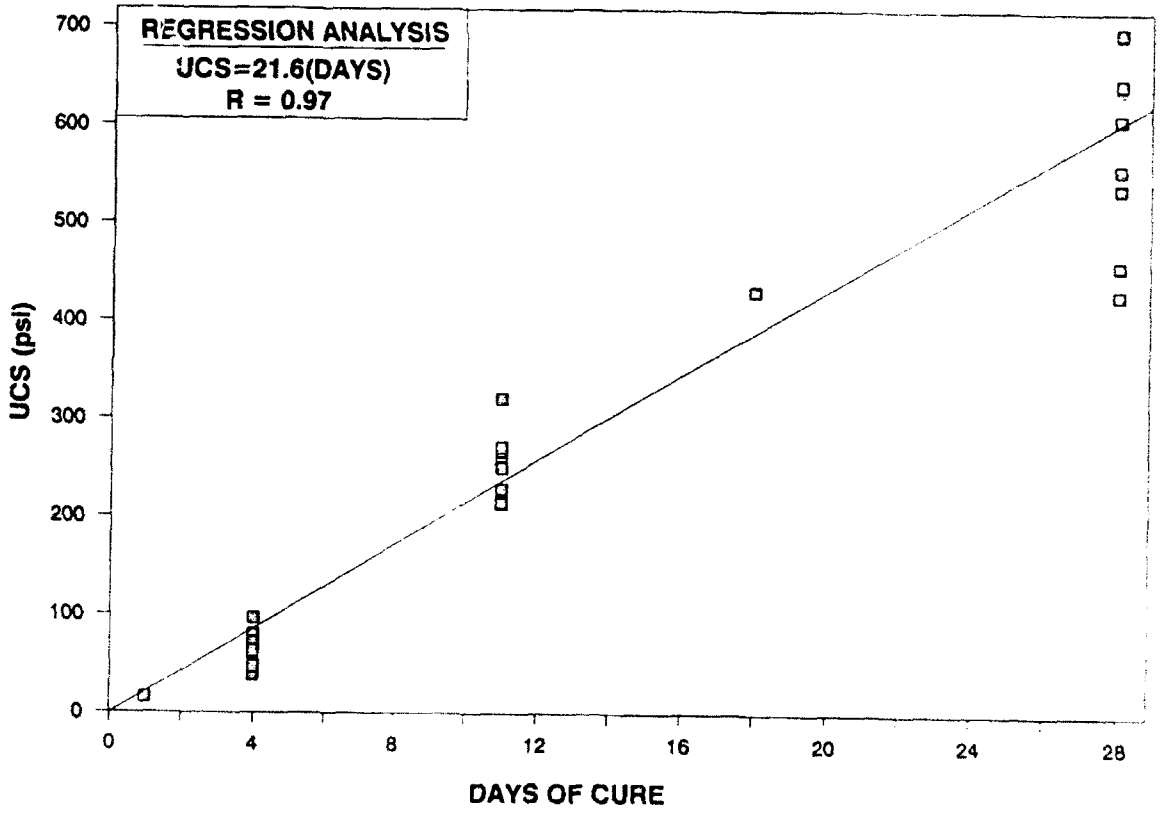

Figure 16. UCS for the LFA solidified/stabilized control samples.

variability in UCS between batches. The CFA mixes varied from a low of 409 to a high of 764 psi after 28 days (CoV = 23 percent), and the LFA mixes from a low of 459 to a high of 697 psi (CoV = 16 percent). The rate of set for both fly ash-containing mixtures was lower than that of the cement mixes, with CFA mixes having only 31 percent and LFA mixes 42 percent of their 28-day UCS at 7 to 11 days of cure compared to 75 percent for CEM samples. From the shape of the UCS versus curing time curves, the strengths of the CFA and LFA mixes would be expected to continue to increase beyond 28 days of cure.

Effects of Interferent Addition

The effects of interferent addition on UCS are summarized in Table 15 for the first week of curing, in Table 16 for the second week, and in Table 17 for 28 days of curing. Each value is the mean of four to five measurements for each time of cure and each concentration of interferent. Also, in the right-most three columns, the percent of the UCS for the control samples (those samples in each batch to which no interferent was added) is indicated.

Metal Interferents--

Three metal ions that have been implicated in interference with the setting properties of cementitious materials (copper, lead, and zinc) were included in the study. All three had pronounced effects upon the rate of attainment of strength and the final 28-day UCS of all three binder systems.

Copper produced different effects on all three binder systems depending upon its concentration. Addition of 2- or 5-percent Cu increased the UCS values throughout the curing period for the cement binder (Figure 17), with the strength at 28 days for 2 percent Cu being twice that of the control, and for 5 percent, 3 times the control. However, addition of 8-percent Cu delayed the onset of the setting reactions. At 28 days of curing, the UCS of the 8-percent addition samples had surpassed the control. The 5-percent Cu samples also show evidence of an early delay in set in the 1-day UCS.

Addition of fly ash to the cement binder in the CFA samples lessened the increase in UCS for the 2-percent copper additions and caused a consistent and dramatic loss of UCS for the 5- and 8-percent additions, as seen in Figure 18. An addition of 8-percent $Cu(NO_3)_2$ practically eliminated the development of UCS in those samples. The LFA samples were consistently weakened by the addition of any of the Cu concentrations (Figure 19). Copper interferes with the setting of all the mixes containing fly ash, as evidenced by the reduction of UCS.

Zinc had the greatest effect on all three binder systems, lowering the strength of all samples at all times and concentrations except the 2-percent Zn in cement. At 5 percent, Zn limited the 28-day UCS to less than 30 percent of that of the controls, and at 8-percent zinc, to less than 15 percent of controls. Even at 2 percent, Zn lowered the 28-day UCS to about 50 percent of the control for both CFA and LFA. The 2-percent Zn in cement samples showed a consistent strength increase in UCS, with a 28-day UCS over 4 times the UCS of the control.

Lead addition to both LFA and CFA samples lowered the UCS to levels comparable to those of Zn. The UCS of these samples were inversely proportional

TABLE 15. UNCONFINED COMPRESSIVE STRENGTH FOR INTERFERENT ADDITIONS AND PERCENT OF CONTROL IN THE FIRST WEEK OF CURE

Binder	Interferent	Age (days)	Unconfined compressive strength				Percent of control		
			Control	2%	5%	8%	2	5	8
CEM	Cu	4	31.4	55.4	25.4	14.8	177	81	50
CFA	Cu	-	ND	ND	ND	ND	--	--	--
LFA	Cu	1	156.3	19.8	31.2	37.3	129	204	243
CEM	Pb	4	42.1	31.1	30.0	27.4	74	71	65
CFA	Pb	4	59.4	57.0	45.0	31.6	96	76	53
LFA	Pb	4	38.5	14.4	10.8	8.6	37	28	22
CEM	Zn		ND	ND	ND	ND	--	--	--
CFA	Zn		ND	ND	ND	ND	--	--	--
LFA	Zn	4	70.6	25.0	36.8	31.0	35	52	44
CEM	Sulfate	4	24.3	16.6	18.5	19.8	68	76	81
CFA	Sulfate	4	32.7	32.3	21.8	22.0	99	67	67
LFA	Sulfate	4	46.9	357.7	212.4	21.2	763	453	45
CEM	NaOH	1	14.1	37.5	10.1	3.0	266	72	21
CFA	NaOH	1	15.3	30.3	39.6	29.1	198	259	190
LFA	NaOH	4	56.6	394.6	323.1	198.7	697	571	351
CEM	Grease	1	54.8	61.1	46.6	33.8	111	85	62
CFA	Grease	1	23.7	18.9	16.8	19.0	80	71	80
LFA	Grease	4	79.9	72.6	43.0	25.2	91	54	31
CEM	Oil	1	44.3	36.5	23.0	19. 5	82	52	44
CFA	Oil	1	19.9	13.2	12.6	17.0	66	63	85
LFA	Oil	4	62.0	54.6	47.8	36.7	88	77	59
CEM	TCE	4	44.9	34.5	34.4	38.0	77	72	85
CFA	TCE	4	52.6	42.0	36.0	42.2	80	68	80
LFA	TCE	4	47.8	78.4	66.3	49.9	164	139	104
CEM	HCB	4	46.8	39.7	43.9	52.5	85	94	110
CFA	HCB	4	58.7	59.1	56.5	57.2	101	96	97
LFA	HCB	4	48.2	50.0	41.8	498	104	87	103
CEM	Phenol	4	70.1	60.5	33.4	19.2	86	48	27
CFA	Phenol	5	85.2	50.0	26.4	13.6	59	31	16
LFA	Phenol	4	96.5	28.8	17.4	7.1	30	18	7

TABLE 16. UNCONFINED COMPRESSIVE STRENGTH FOR INTERFERENT ADDITIONS AND PERCENT OF CONTROL IN THE SECOND WEEK OF CURE

Binder	Interferent	Age (days)	Unconfined Compressive Strength Control	2%	5%	8%	Percent of control 2	5	8
CEM	Cu	11	54.9	74.5	96.2	22.3	136	175	41
CFA	Cu	11	249.7	277.7	46.9	6.9	111	19	3
LFA	Cu	11	320.5	182.5	145.9	24.6	57	46	8
CEM	Pb	11	49.5	49.3	47.6	47.9	100	96	97
CFA	Pb	11	243.4	117.0	67.7	52.4	48	28	22
LFA	Pb	11	220.0	92.4	34.4	27.8	42	16	13
CEM	Zn	11	40.4	230.2	11.7	5.9	570	29	15
CFA	Zn	11	308.5	19.5	14.7	20.2	6	5	7
LFA	Zn	11	255.8	77.5	51.1	42.0	30	20	16
CEM	Sulfate	11	45.2	34.8	31.3	31.2	77	69	69
CFA	Sulfate	11	117.5	133.1	117.5	46.0	113	100	39
LFA	Sulfate	11	250.7	772.1	623.1	157.6	308	249	63
CEM	NaOH	8	90.0	129.4	54.0	28.7	144	60	32
CFA	NaOH	7	129.2	265.6	299.4	239.9	206	232	183
LFA	NaOH	11	228.7	665.4	581.0	384.7	291	254	168
CEM	Grease	7	191.8	161.1	145.8	112.0	89	76	58
CFA	Grease	7	130.3	106.6	92.0	94.5	82	71	73
LFA	Grease	18	431.2	410.7	308.9	214.9	95	72	50
CEM	Oil	7	137.9	111.3	79.1	72.7	81	57	53
CFA	Oil	7	82.3	51.8	56.2	76.0	63	68	92
LFA	Oil	11	267.0	217.9	178.6	130.3	82	67	49
CEM	HCB	11	67.7	58.6	63.2	74.9	87	93	111
CFA	HCB	11	219.9	198.4	210.0	202.8	90	95	92
LFA	HCB	11	214.0	222.2	254.4	222.6	104	119	104
CEM	Phenol	11	105.1	90.9	63.3	34.9	86	60	33
CFA	Phenol	11	263.3	129.0	51.4	23.8	49	20	9
LFA	Phenol	11	271.4	92.6	23.6	5.8	34	9	2
CEM	TCE	11	59.5	52.6	46.1	48.7	88	77	82
CFA	TCE	11	199.0	166.9	142.2	140.9	84	71	71
LFA	TCE	11	214.6	296.2	262.6	274.5	138	122	128

TABLE 17. UNCONFINED COMPRESSIVE STRENGTH FOR INTERFERENT ADDITION AND PERCENT OF CONTROL AFTER 28 DAYS OF CURE

Binder	Interferent	Age (days)	Unconfined compressive strength Control	2%	5%	8%	Percent of control 2	5	8
CEM	Cu	31	42.0	85.4	130.7	47.7	204	311	114
CFA	Cu	28	409.5	478.8	65.2	7.5	117	16	2
LFA	Cu	28	608.8	317.9	315.8	10.0	52	52	2
CEM	Pb	28	60.6	68.4	82.2	76.7	98	118	110
CFA	Pb	28	764.0	324.0	168.7	136.2	42	122	18
LFA	Pb	28	538.1	325.5	122.3	55.7	60	23	10
CEM	Zn	28	73.3	309.3	19.8	10.6	422	27	14
CFA	Zn	28	409.4	216.3	33.9	25.4	53	8	6
LFA	Zn	28	697.4	324.8	161.8	81.6	47	23	12
CEM	Sulfate	28	53.6	49.7	46.4	25.1	93	87	47
CFA	Sulfate	28	475.9	612.8	547.9	186.21	129	115	39
LFA	Sulfate	28	642.3	1061.2	883.5	116.4	165	138	18
CEM	NaOH	28	151.5	173.3	102.1	73.3	114	67	48
CFA	NaOH	28	645.4	792.7	676.3	519.2	123	105	80
LFA	NaOH	28	645.5	1104.2	689.6	622.5	171	107	96
CEM	Grease	28	230.3	202.7	173.5	127.3	88	75	55
CFA	Grease	28	816.2	654.9	492.0	423.0	80	60	52
LFA	Grease	28	640.1	594.2	469.0	297.5	93	73	46
CEM	Oil	28	211.7	169.9	131.0	118.3	80	62	56
CFA	Oil	28	646.3	377.3	395.9	597.3	58	61	92
LFA	Oil	28	557.4	517.6	406.8	380.2	93	73	68
CEM	HCB	28	89.6	85.6	84.6	103.0	96	94	115
CFA	HCB	28	429.0	455.6	467.4	424.0	106	109	99
LFA	HCB	28	701.2	631.2	637.3	631.7	90	91	90
CEM	TCE	28	78.1	56.2	49.9	56.8	72	64	73
CFA	TCE	28	629.6	587.8	424.4	444.9	93	67	71
LFA	TCE	28	459.4	693.8	552.6	617.5	151	120	134
CEM	Phenol	28	119.7	93.2	88.7	55.4	78	74	46
CFA	Phenol	28	474.3	385.1	134.9	62.3	52	18	8
LFA	Phenol	28	696.1	242.7	82.9	27.0	35	12	4

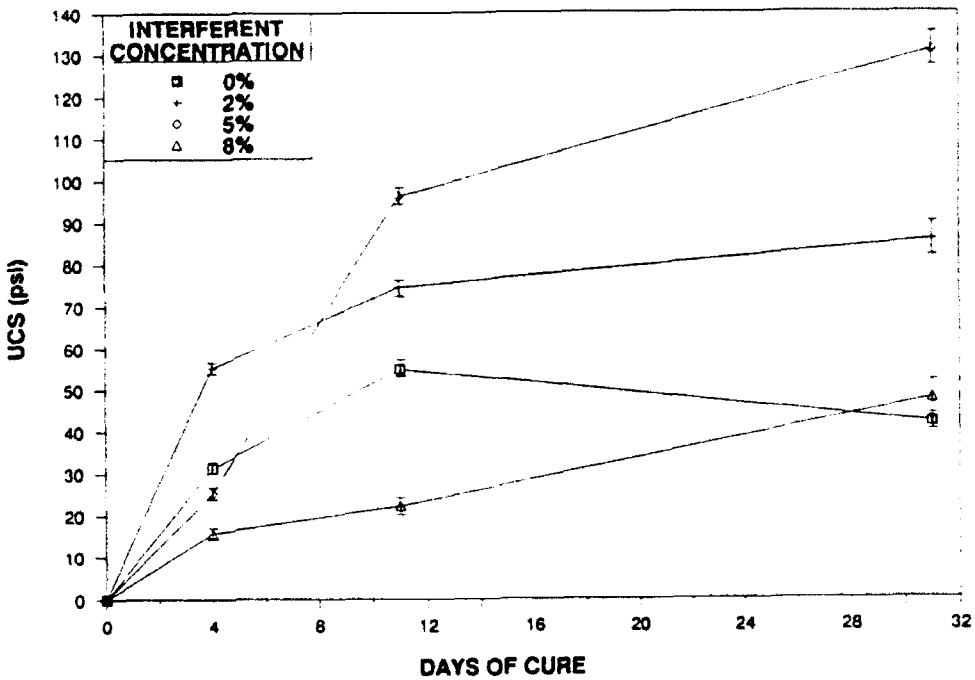

Figure 17. UCS for the CEM solidified/stabilized samples with copper interference.

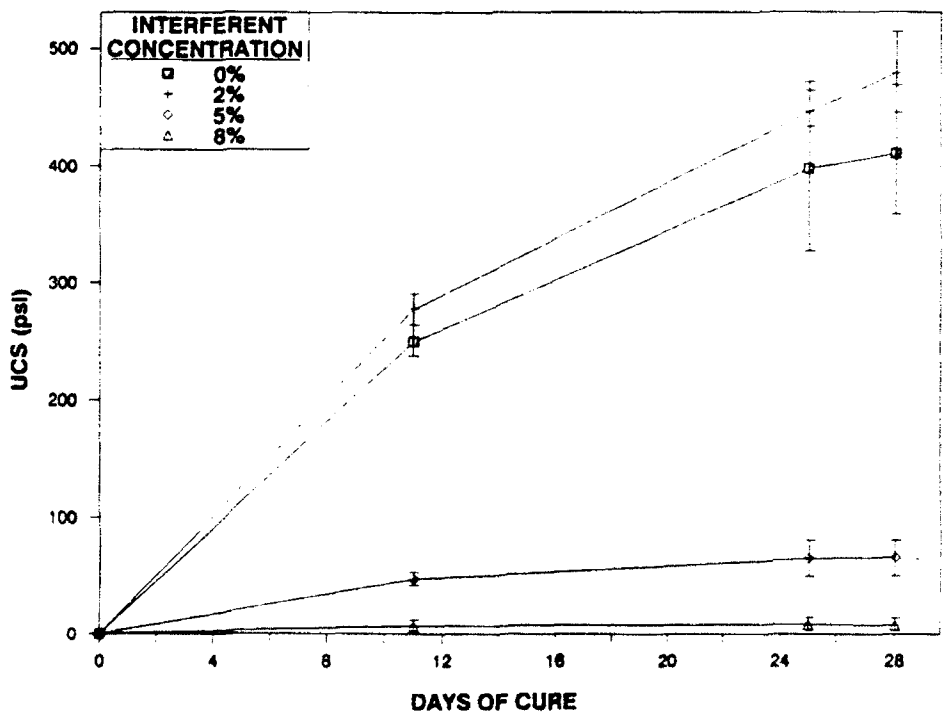

Figure 18. UCS for the CFA solidified/stabilized samples with copper interference.

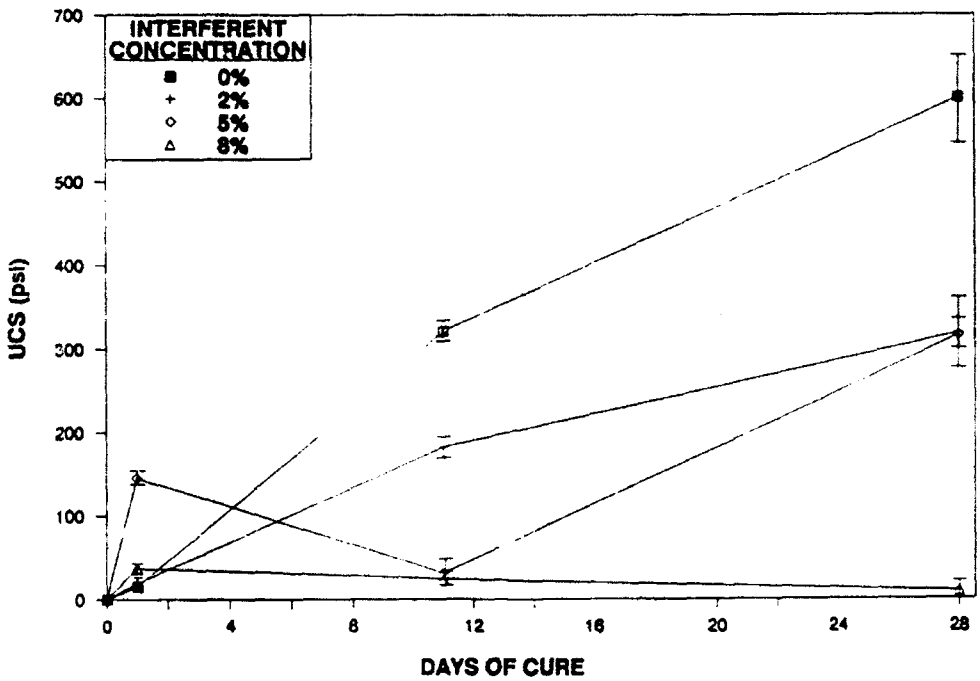

Figure 19. UCS for the LFA solidified/stabilized samples with copper interference.

to the added percentage of Pb. However, at the concentrations added, Pb had only minor effects on the UCS of the cement samples.

The higher levels of Cu and Zn ions depressed or delay the onset of UCS development of the final products. Although strength development is delayed, UCS may possibly develop to satisfactory levels after longer curing periods; however, such tests were not performed in this study.

Sodium Sulfate Interference--

The effects of sulfate addition on strength development varied greatly for the different binder systems. For cement, 5- and 8-percent sulfate levels consistently lowered the strength and rate of strength development throughout the 28-day curing period. LFA showed rapid set and high early strength development with both the 2- and 5-percent sulfate additions (Figure 20). However, after 28 days, 8-percent sulfate addition reduced the UCS of the LFA samples to about 20 percent of the control. Effects on the CFA samples were intermediate between those on the other two binders.

Sulfate in the concrete environment is known to cause long-term scaling and disintegration of type I cement. LFA had a more rapid rate of cure at low sulfate levels and an inhibition of strength formation at the highest sulfate level (Figure 20). In this study, small amounts of scaling were observed for some samples; however, the long-term effects of sulfate addition were not assessed by this short-term testing.

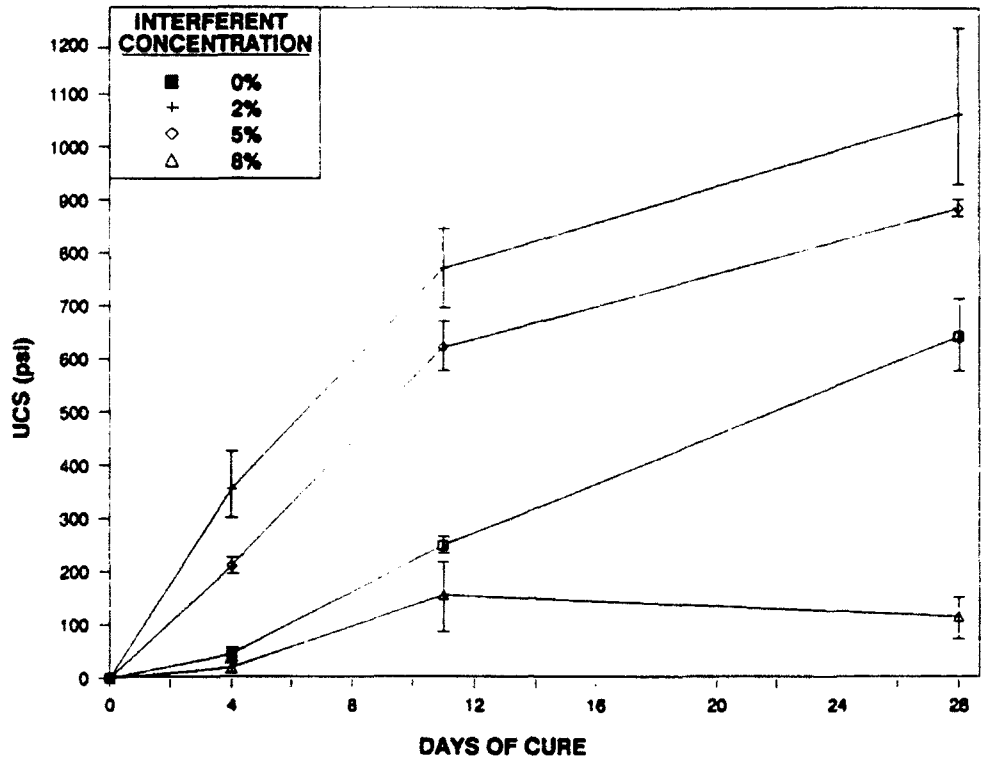

Figure 20. UCS for the LFA solidified/stabilized samples with sulfate interference.

Strong Base Interferents--

Addition of sodium hydroxide to the solidified/stabilized samples generally promoted very rapid early strength development, especially at the lower concentrations. However, results varied depending upon its concentration and the binder. Addition of 2-percent NaOH caused rapid early strength development and a higher UCS for all binders at 28 days. Only CEM samples with 5- and 8-percent NaOH additions had consistently lower UCS throughout the 28 days of curing.

The LFA and CFA samples were affected to a greater extent with all levels of NaOH, showing a rapid set and very high early UCS values (Figures 21 and 22). LFA was especially affected. However, after 28 days of curing, the samples with 5- and 8-percent additions had about the same or lower UCS values than the controls. If these trends continued, the controls would be expected to continue to have higher UCS after periods longer than 28 days.

Organic Compound Interferents--

The nonpolar organic molecules used as interferents (grease, oil, and TCE) differed in size and molecular characteristics. However, they all brought about consistent and significant decreases in UCS at all concentrations in all three binder systems. An example is seen for the effects of grease on CEM and CFA UCS in Figures 23 and 24.

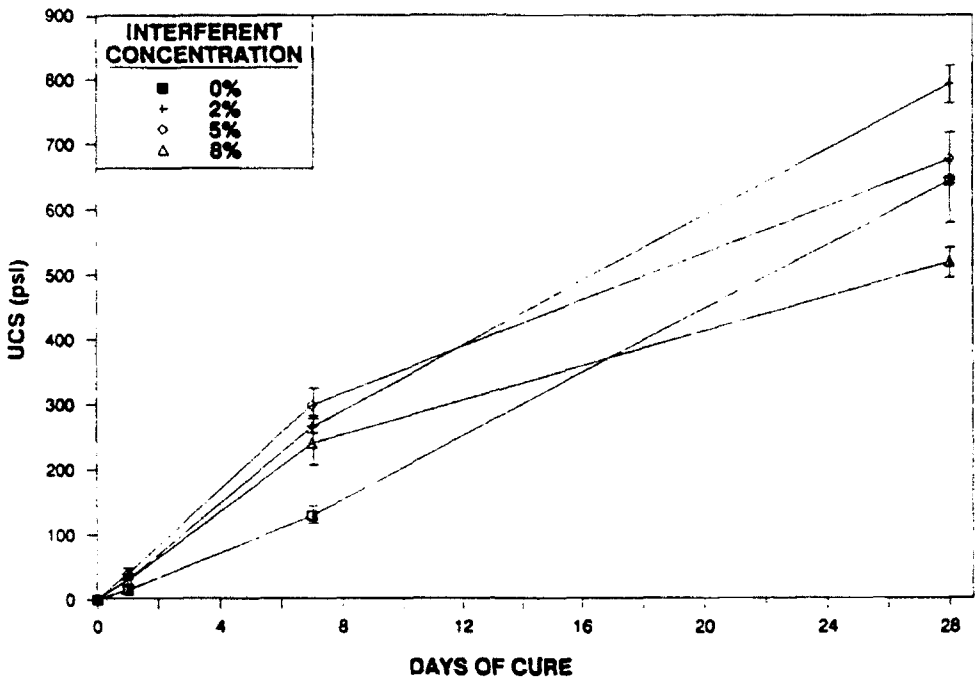

Figure 21. UCS for the CFA solidified/stabilized samples with NaOH interference.

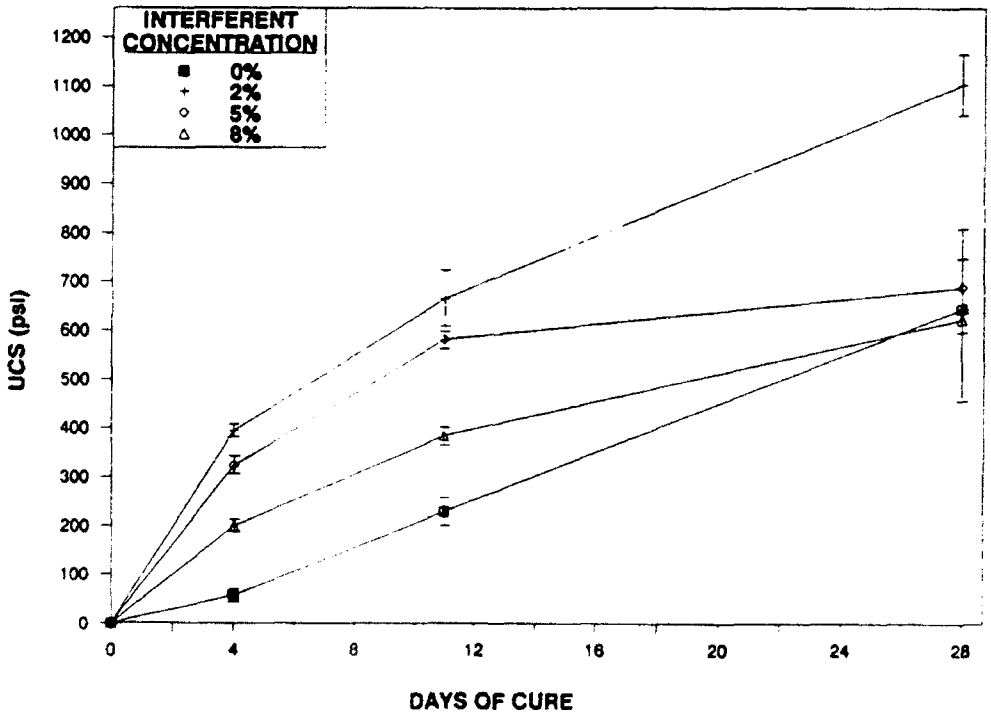

Figure 22. UCS for the LFA solidified/stabilized samples with NaOH interference.

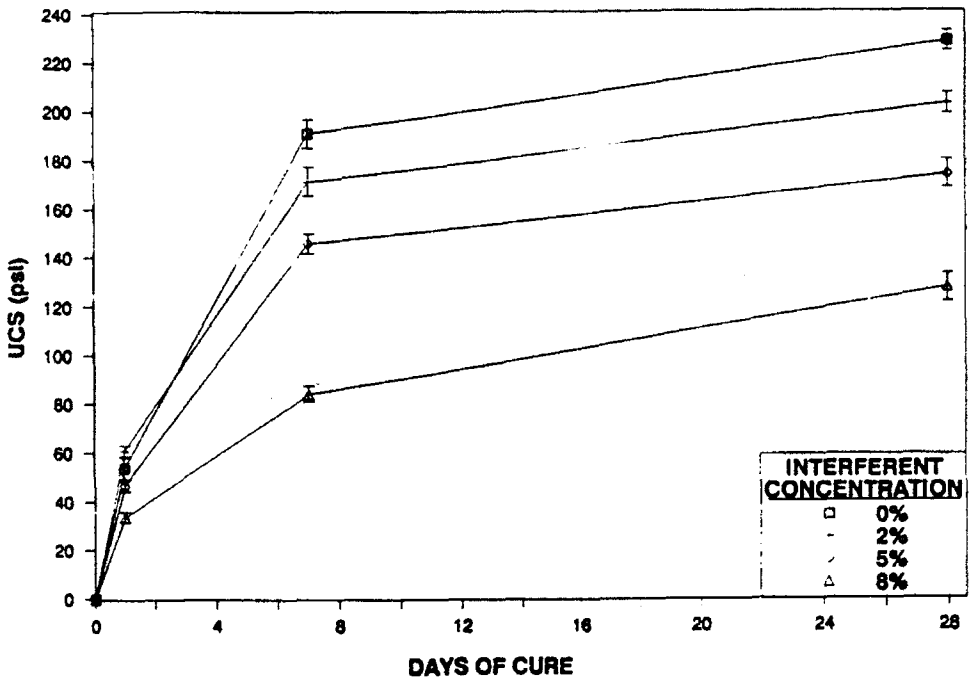

Figure 23. UCS for the CEM solidified/stabilized samples with grease interference.

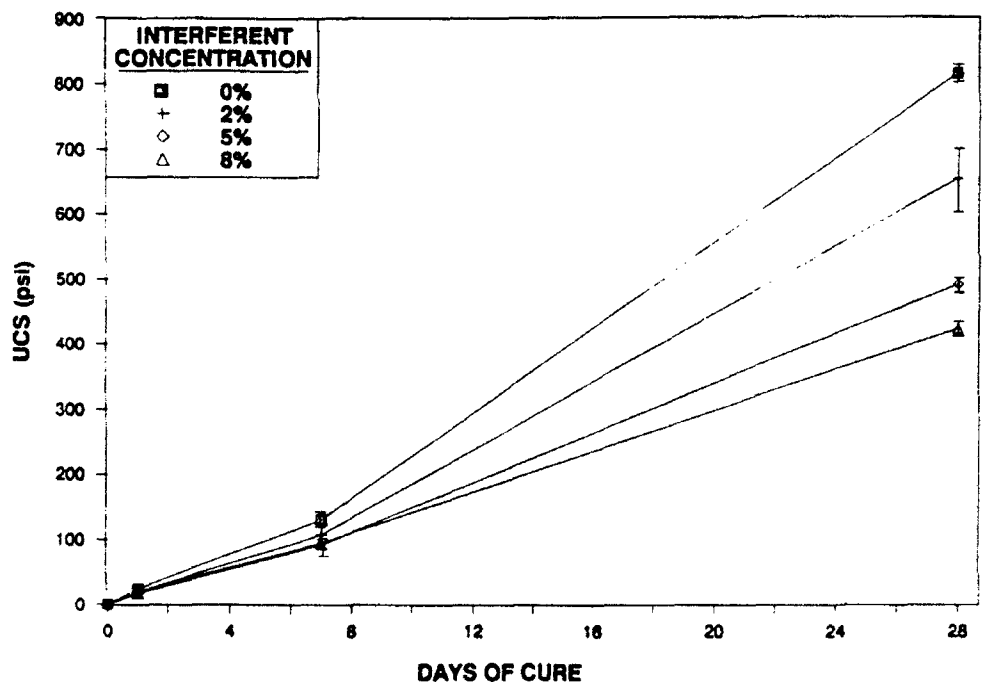

Figure 24. UCS for the CFA solidified/stabilized samples with grease interference.

The only exception to the general reduction of strength by the nonpolar organics is seen in the effects of TCE on the LFA binder system. All levels of TCE consistently increased the UCS at all cure times (see Figure 25).

Hexachlorobenzene (HCB) had little effect upon any of the binder systems at any of the concentrations investigated. The 2-percent addition slightly delayed strength development in the CEM binder system, while the 8-percent addition increased the UCS by only 10 to 15 percent of the control samples. By 28 days of cure, only the 8-percent addition was significantly different from the control.

Phenol, the most polar and water-soluble of the organic interferents in this study, produced the greatest effect on UCS. All samples with phenol addition showed pronounced reduction of UCS in direct proportion to its concentration. The LFA samples were more substantially affected than the other binders. The LFA samples were consistently lower in UCS; those with added phenol retained about 30 percent of controls for the 2-percent addition, 10 percent for the 5-percent phenol addition, and around 5 percent for the 8-percent addition rate. CFA showed similar, consistent, but less dramatic decreases in UCS (Figure 26). The CEM samples showed the least strength loss; those with added phenol retained 80 percent of their UCS for 2-percent phenol addition, 70 percent for 5-percent, and 40 percent for 8-percent. Phenol, with its active hydroxyl group, would be expected to interfere with the hydration reaction and cause a delayed set in a manner similar to commonly used organic cement set retarders (Mehta 1987).

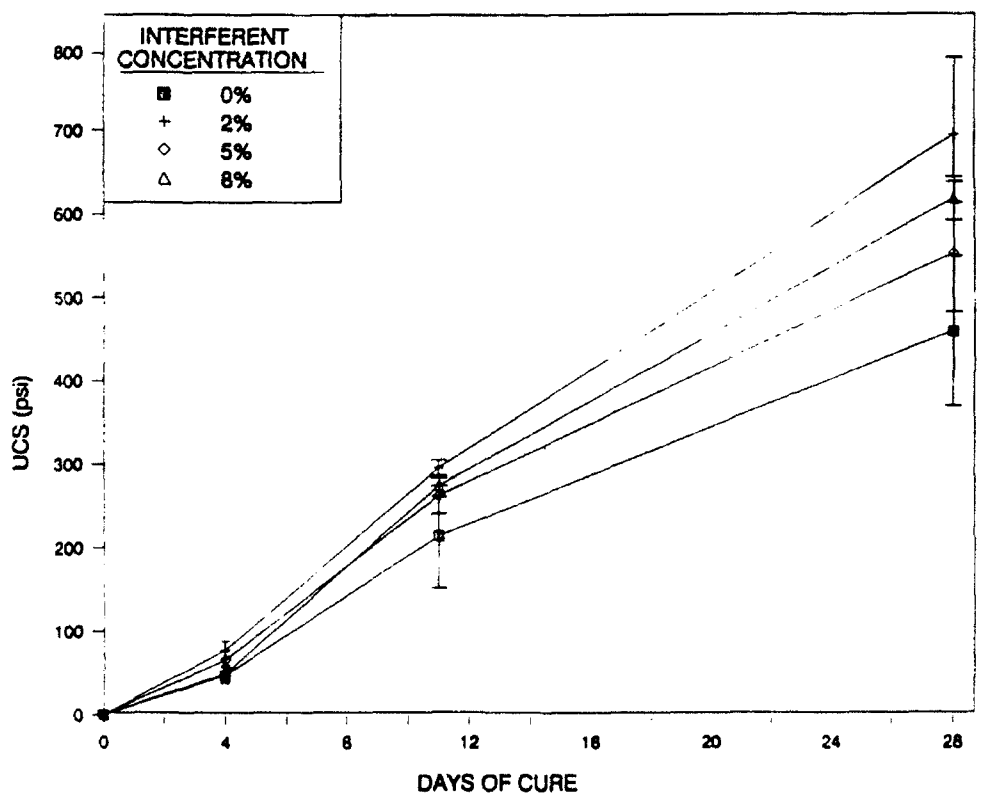

Figure 25. UCS for the LFA solidified/stabilized samples with TCE interference.

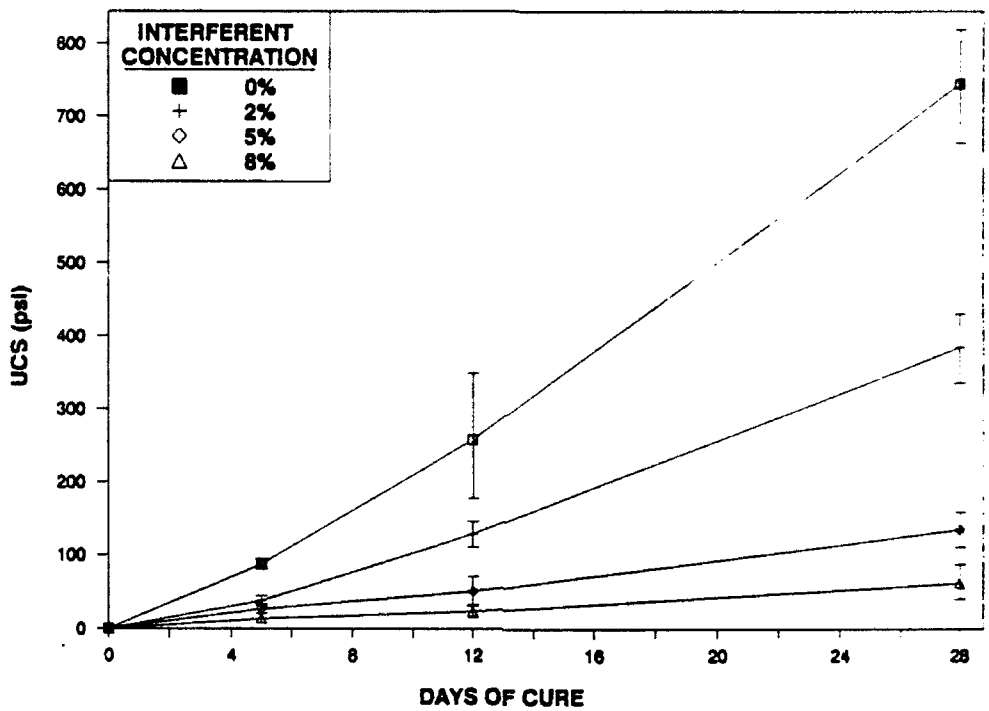

Figure 26. UCS for the CFA solidified/stabilized samples with phenol interference.

CONE INDEX

Cone index measurements were made during the first day of cure and continued until the sample CI exceeded the measuring range of the apparatus (750 psi). Four replicate measurements were made in all cases.

Samples Without Interferent

The mean values for the number of days necessary to attain 750 psi CI are shown in Table 18. The CEM samples that were designed to produce a low-strength product took the longest to reach the limit CI value, at about 16 days. The CFA and LFA samples both reached this value at 11 to 12 days of cure.

Figure 27 illustrates the variability in the CI for the CEM binder by plotting the CI versus time of cure for all CEM control samples. While substantial variability was observed for the complete set of CEM control samples, much less within-batch variation was observed. This indicates that it is difficult to produce identical samples of low-strength product from different batches. The high between-batch variability makes comparisons between batches using absolute values more difficult. The regression line drawn in the figure

TABLE 18. MEAN NUMBER OF DAYS FOR CI VALUES OF CONTROL MIXES TO REACH 750 psi CI

Binder	Mean days to reach CI of 750 Avg	CoV*
Cement	15.7	24%
Cement/fly ash	0.7	27%
Lime/fly ash	12.0	14%

* Coefficient of variation (standard deviation/treatment mean) times 100.

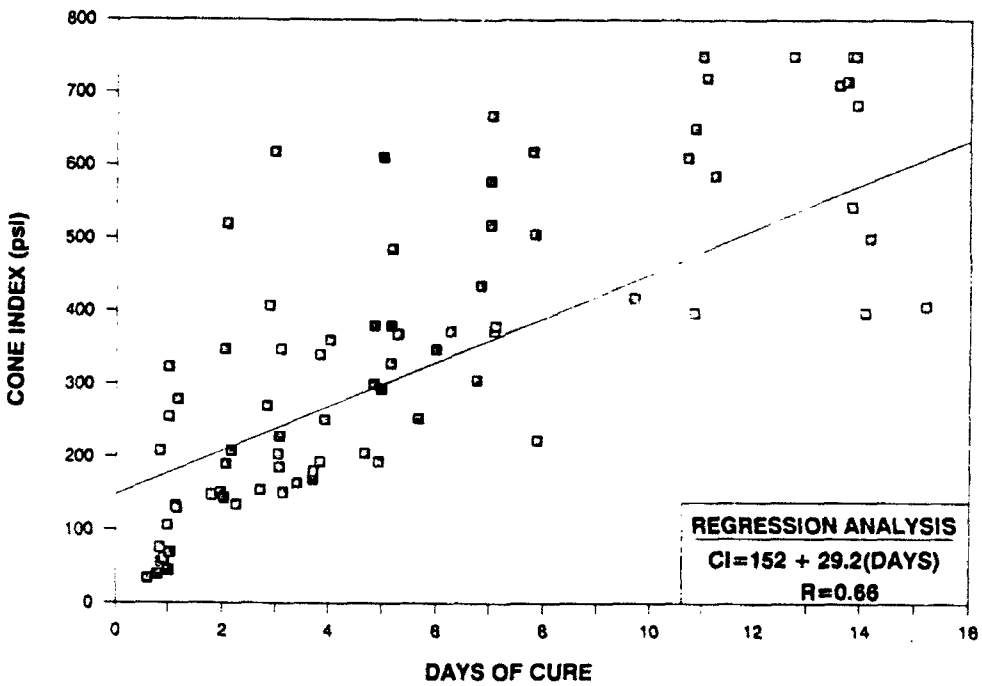

Figure 27. Cone index for the CEM solidified/stabilized control samples.

has a correlation coefficient (r) of 0.66 and a large positive Y-intercept. Plotting these same data for CEM by the square root of time of cure in Figure 28 produces a higher correlation with a near-zero intercept and an r of 0.79. As would be expected, cement shows a hyperbolic setting pattern over this time frame.

The CFA (Figure 29) and LFA (Figure 30) CI setting curves for control samples have much less variability, with Y-intercepts near zero and correlation coefficients over 0.9. These samples have a near-linear setting pattern

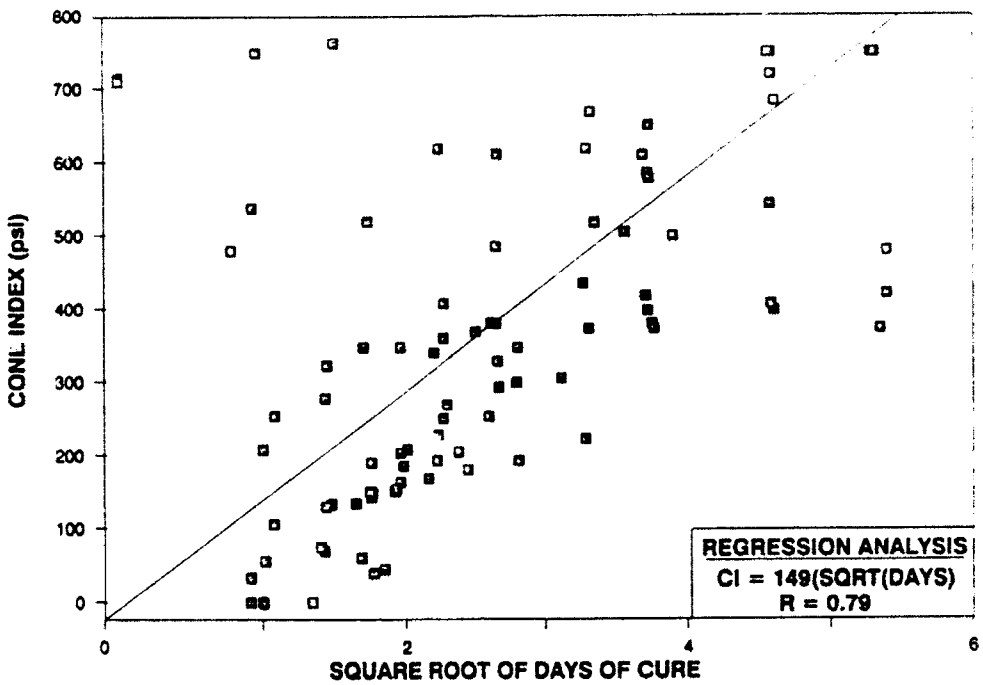

Figure 28. Cone index for the CEM solidified/stabilized control samples, where the square root of the day of cure is plotted versus the cone index value.

over the time frame of these measurements, showing that they have a constant rate of set during this time.

Effects of Added Interferents on Sample CI

The CI measurements were taken at various times during the initial cure period for the solidified/stabilized samples. To simplify the comparison of CI values between treatments, values for 5 and 10 days of cure were predicted using linear regression procedures and are shown in Tables 19 and 20. The percent of control (no added interferent) values are also presented in the right-most column. Patterns of the effects of interferent on the CI of S/S products generally are similar to their effects on UCS and will be discussed in this frame of reference.

The metal nitrates generally lowered sample CI, especially in the fly ash-containing samples as shown in Figures 31-33. Zinc was especially deleterious to all three binder system. In contrast, the 2-percent concentration of all metals uniformly increased the CI of the CEM samples, but at higher concentrations the CI was reduced.

Except for the 8-percent sulfate addition to CFA, sulfate uniformly increased the CI for all fly ash-containing samples. The greatest effect was

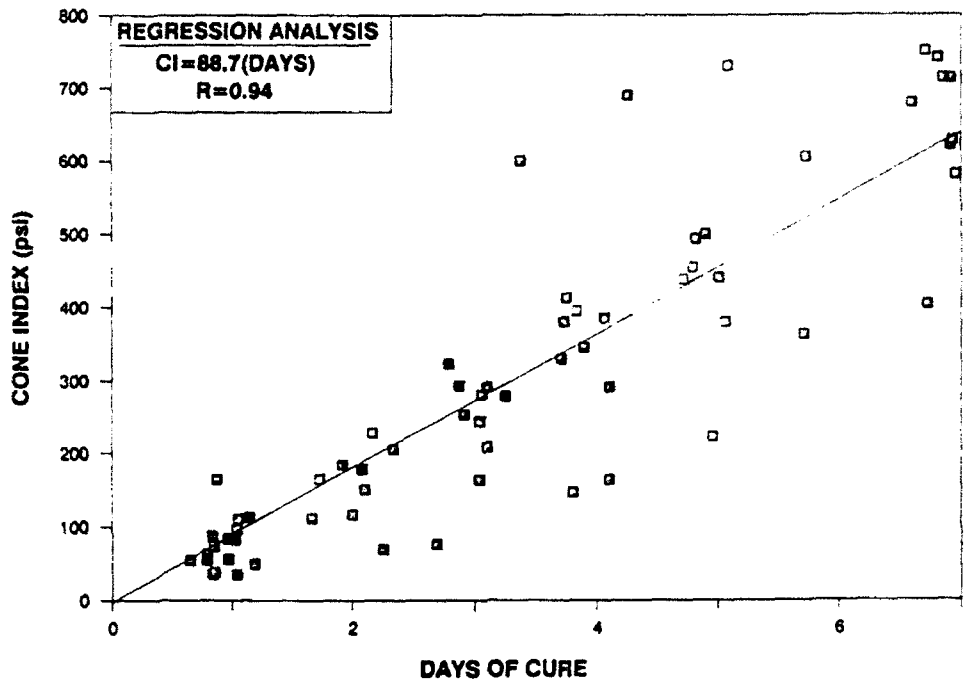

Figure 29. Cone index for the CFA solidified/stabilized control samples.

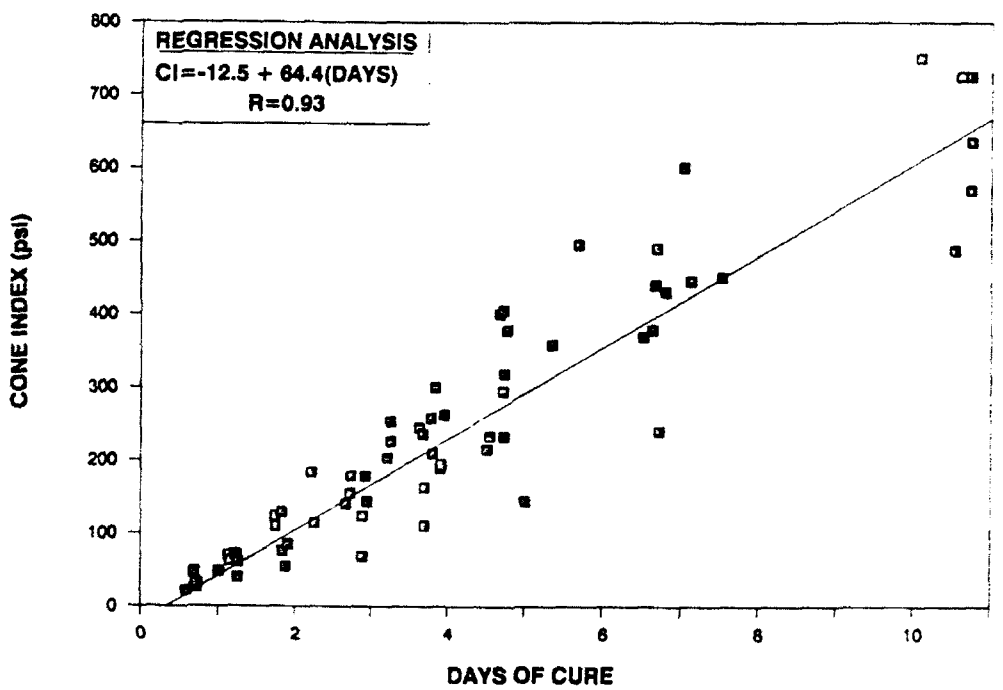

Figure 30. Cone index for the LFA solidified/stabilized control samples.

TABLE 19. CONE INDEX AT 5 DAYS PREDICTED FROM REGRESSION ANALYSIS

		Cone index at 5 days (%)				Percent of control		
Binder	Interferent	0	2	5	8	2	5	8
CEM	Copper	300	457	136	72	152	45	24
CFA	Copper	214	200	ND	ND	94	--	--
LFA	Copper	370	238	354	182	64	96	49
CEM	Lead	187	199	158	139	107	85	74
CFA	Lead	453	391	218	147	86	48	32
LFA	Lead	223	69	32	32	31	14	14
CEM	Zinc	192	231	15	12	121	8	6
CFA	Zinc	437	31	18	38	7	4	9
LFA	Zinc	345	123	155	136	36	45	40
CEM	Sulfate	236	186	196	205	79	83	87
CFA	Sulfate	273	356	300	232	130	110	85
LFA	Sulfate	240	609	906	324	254	378	135
CEM	NaOH	335	648	121	50	193	36	15
CFA	NaOH	465	156	1936	1160	34	416	249
LFA	NaOH	379	2536	4212	1168	670	1112	308
CEM	Grease	500	374	239	156	75	48	31
CFA	Grease	413	329	228	161	80	55	39
LFA	Grease	291	229	216	134	79	74	46
CEM	Oil	385	290	183	136	75	47	35
CFA	Oil	486	291	254	240	60	52	49
LFA	Oil	273	247	205	162	90	75	59
CEM	TCE	332	230	199	190	69	60	57
CFA	TCE	511	429	352	350	84	69	69
LFA	TCE	250	261	238	322	105	95	129
CEM	HCB	307	305	320	364	99	104	119
CFA	HCB	466	490	504	484	105	108	104
LFA	HCB	270	295	370	375	109	137	139
CEM	Phenol	359	230	122	68	64	34	19
CFA	Phenol	517	237	117	45	46	23	9
LFA	Phenol	372	124	54	28	33	14	8

TABLE 20. CONE INDEX AT 10 DAYS PREDICTED FROM REGRESSION ANALYSIS

		Cone index at 5 days (%)				Percent of control		
Binder	Interferent	0	2	5	8	2	5	8
CEM	Copper	609	903	299	199	148	49	33
CFA	Copper	430	400	ND	ND	93	--	--
LFA	Copper	741	423	630	294	57	85	40
CEM	Lead	273	311	263	225	114	96	82
CFA	Lead	911	765	408	286	84	45	31
LFA	Lead	487	158	66	60	32	14	12
CEM	Zinc	375	462	53	40	123	14	11
CFA	Zinc	891	119	38	56	13	4	6
LFA	Zinc	690	221	259	230	32	37	33
CEM	Sulfate	481	389	399	411	81	83	85
CFA	Sulfate	571	741	616	482	130	108	84
LFA	Sulfate	409	1228	1832	689	300	448	169
CEM	NaOH	659	1236	199	90	187	30	14
CFA	NaOH	925	138	3834	2287	15	414	247
LFA	NaOH	780	5072	8425	2302	650	1080	295
CEM	Grease	834	550	329	217	66	39	26
CFA	Grease	814	646	445	306	79	55	38
LFA	Grease	594	468	462	287	79	78	48
CEM	Oil	628	464	267	202	74	3	32
CFA	Oil	981	565	498	477	58	1	49
LFA	Oil	547	503	433	331	92	79	61
CEM	TCE	616	349	294	286	57	48	46
CFA	TCE	1027	866	701	700	84	68	68
LFA	TCE	487	530	484	676	109	99	139
CEM	HCB	579	574	591	658	99	102	114
CFA	HCB	949	999	1031	977	105	109	103
LFA	HCB	557	617	788	777	111	142	140
CEM	Phenol	682	455	256	166	67	38	24
CFA	Phenol	1059	485	159	61	46	15	6
LFA	Phenol	737	252	108	58	34	15	8

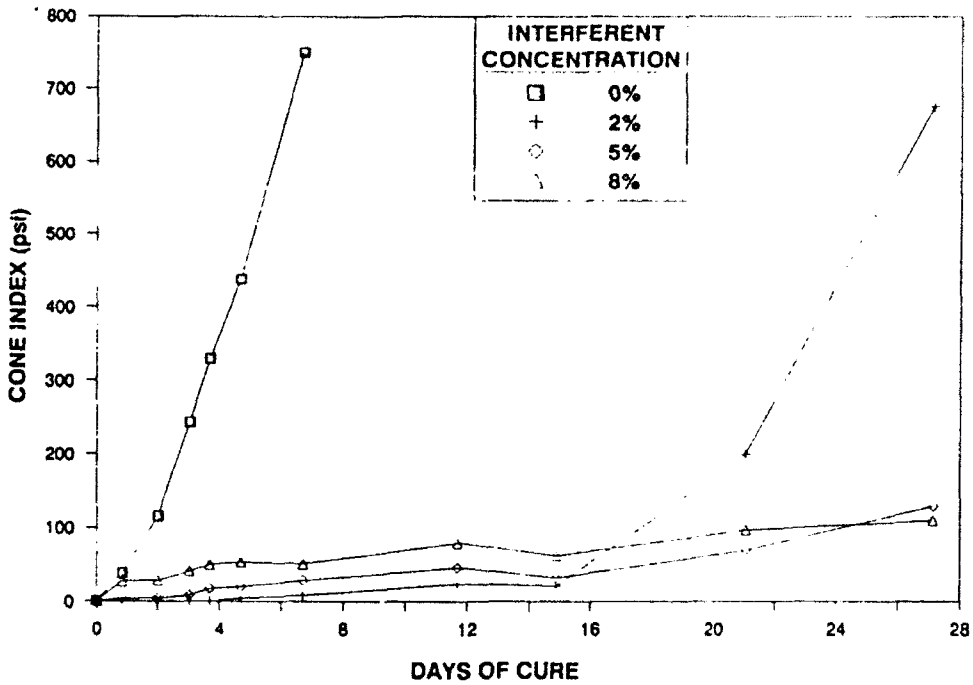

Figure 31. Cone index for the CFA solidified/stabilized samples with zinc interference.

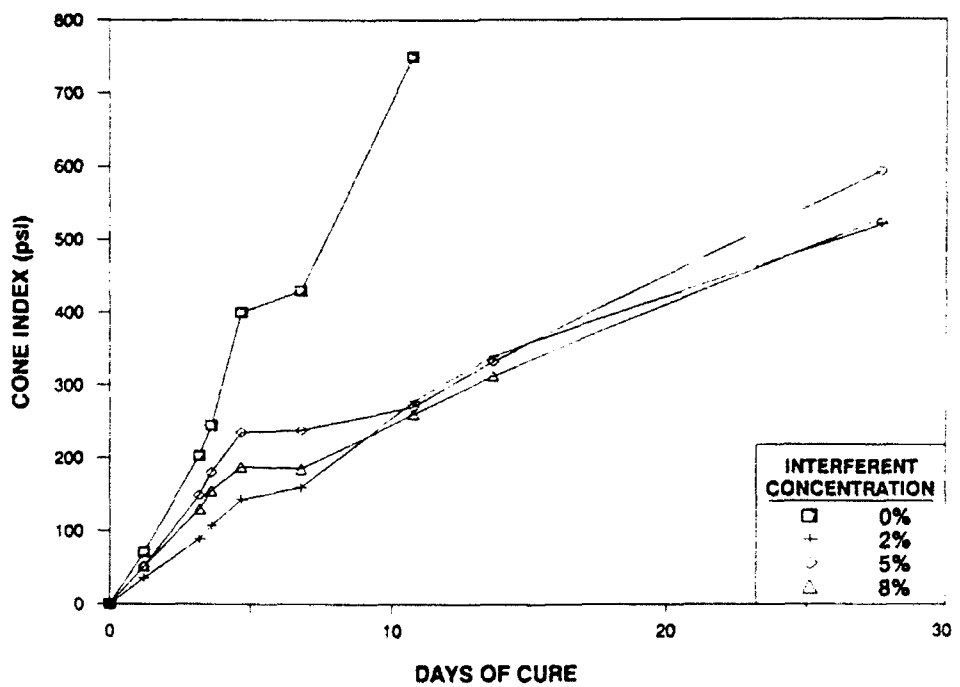

Figure 32. Cone index for the LFA solidified/stabilized samples with zinc interference.

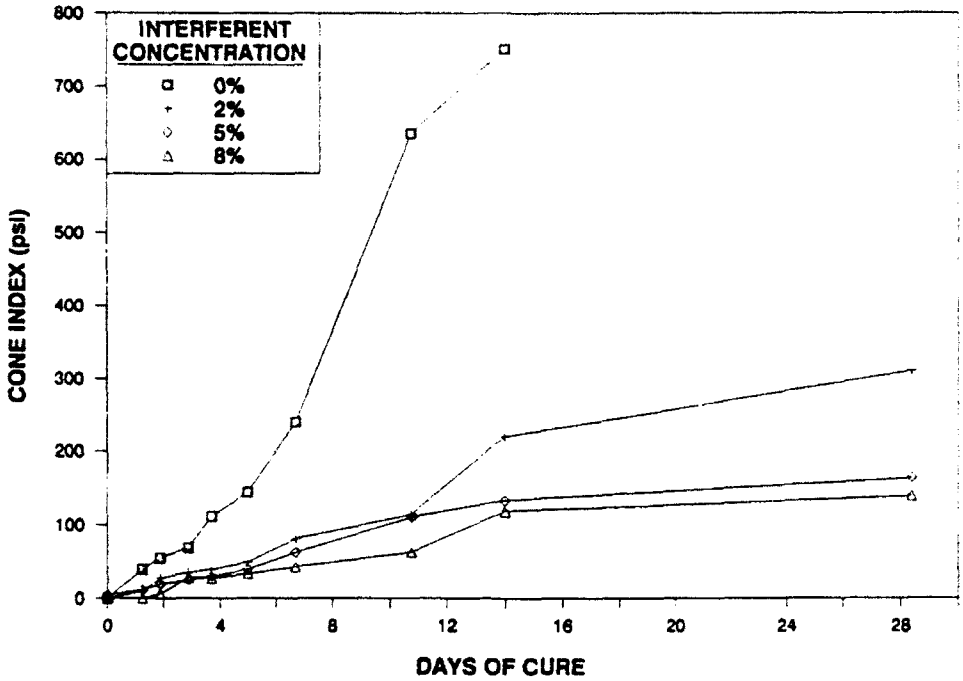

Figure 33. Cone index for the LFA solidified/stabilized samples with lead interference.

on the LFA at 5-percent sulfate addition, which gave UCS values over 10 times the control of both 5 and 10 days of cure. Cement samples, on the other hand, had consistently lower CI values upon sulfate addition.

Sodium hydroxide increased the rate of CI development in both fly ash-containing mixtures. The LFA samples were substantially affected, showing CI increases of 6 and 10 times at 2- and 5-percent addition rates, respectively (Figure 34). The CFA samples also exhibited increased CI at the higher NaOH additions. These samples exceeded the CI maximum of 750 psi in less than 3 days, a very rapid setting reaction. The CEM samples (Figure 35) again showed a nearly opposite response, showing a more rapid setting rate with 2-percent NaOH additions, but a much delayed set at higher levels of sodium hydroxide.

The most nonpolar organics (grease, oil, and TCE) produced a general lowering of the rate of CI development in proportion to their addition rates. Grease (Figure 36) had the largest effect, and TCE the least effect. An exception was the effect of TCE on LFA, which showed a slight increase in the rate of CI development.

Hexachlorobenzene had little net effect on CEM or CFA samples, and showed only a slight increase in CI in the LFA (Figure 37). Phenol again reduced the rate of CI development dramatically, lowering the CI to less than 10 percent of the control for both CFA and LFA at 8-percent addition, and less than 20 percent for 5-percent addition. The CEM samples were also substantially affected, but to a lesser extent than the fly ash-containing samples (Figure 38).

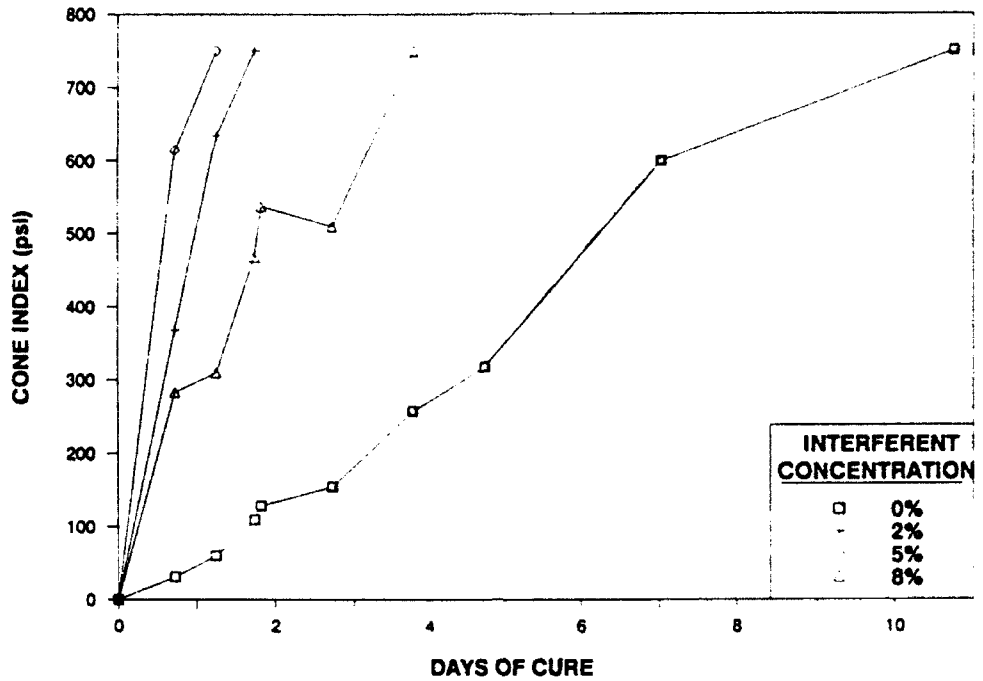

Figure 34. Cone index for the LFA solidified/stabilized samples with NaOH interference.

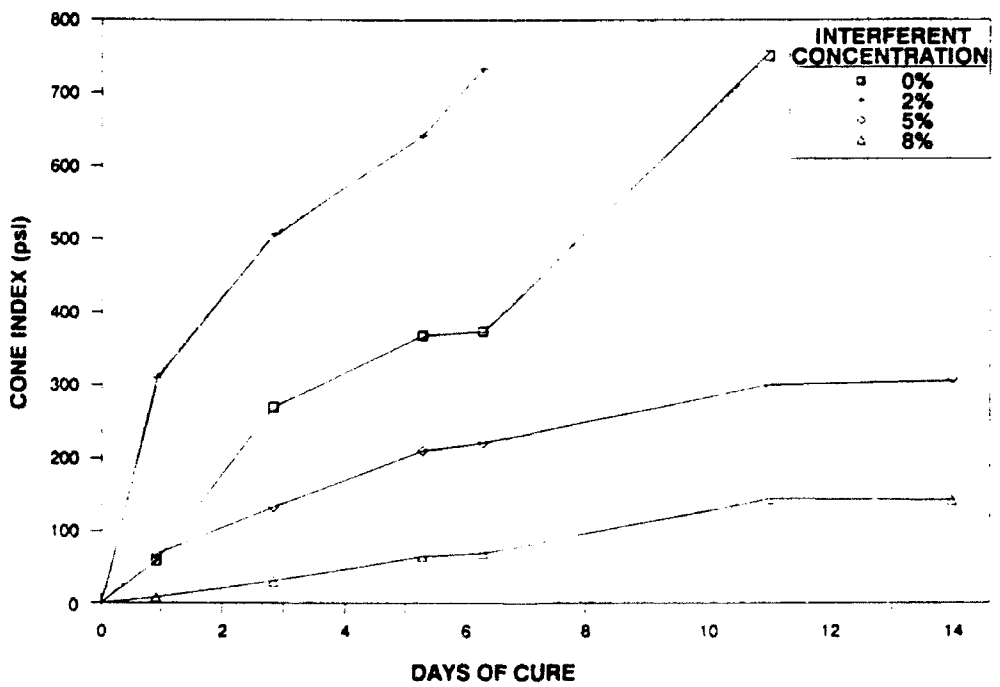

Figure 35. Cone index for the CEM solidified/stabilized samples with NaOH interference.

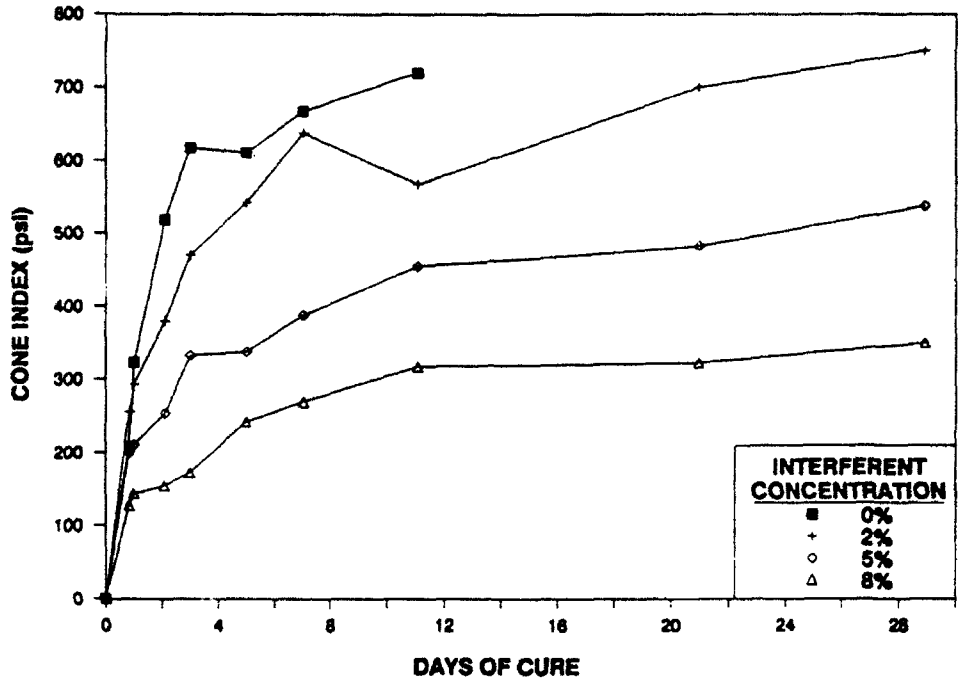

Figure 36. Cone index for the CEM solidified/stabilized samples with grease interference.

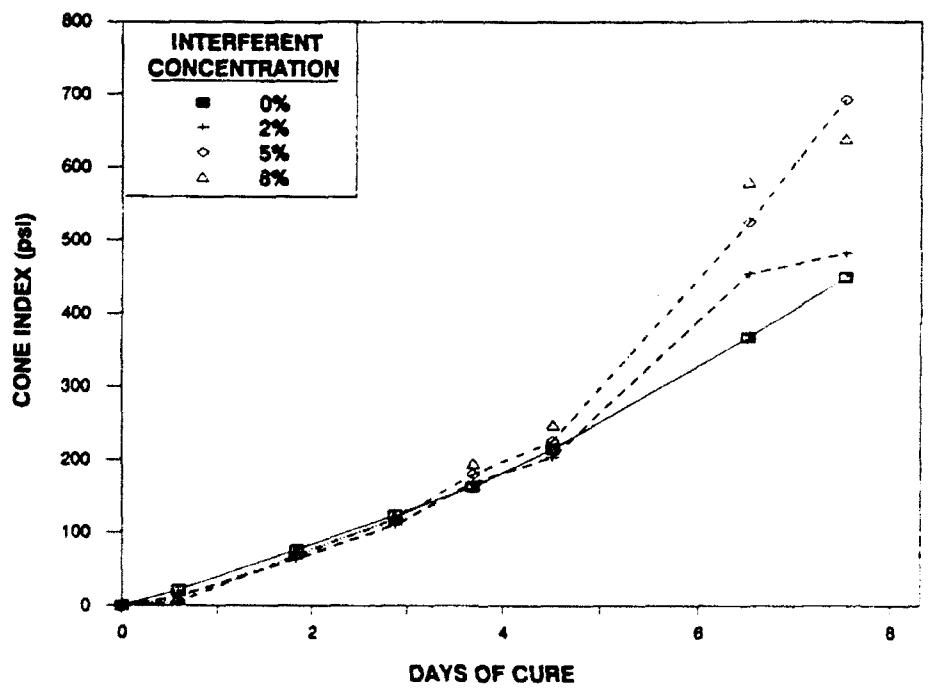

Figure 37. Cone index for the LFA solidified/stabilized samples with HCB interference.

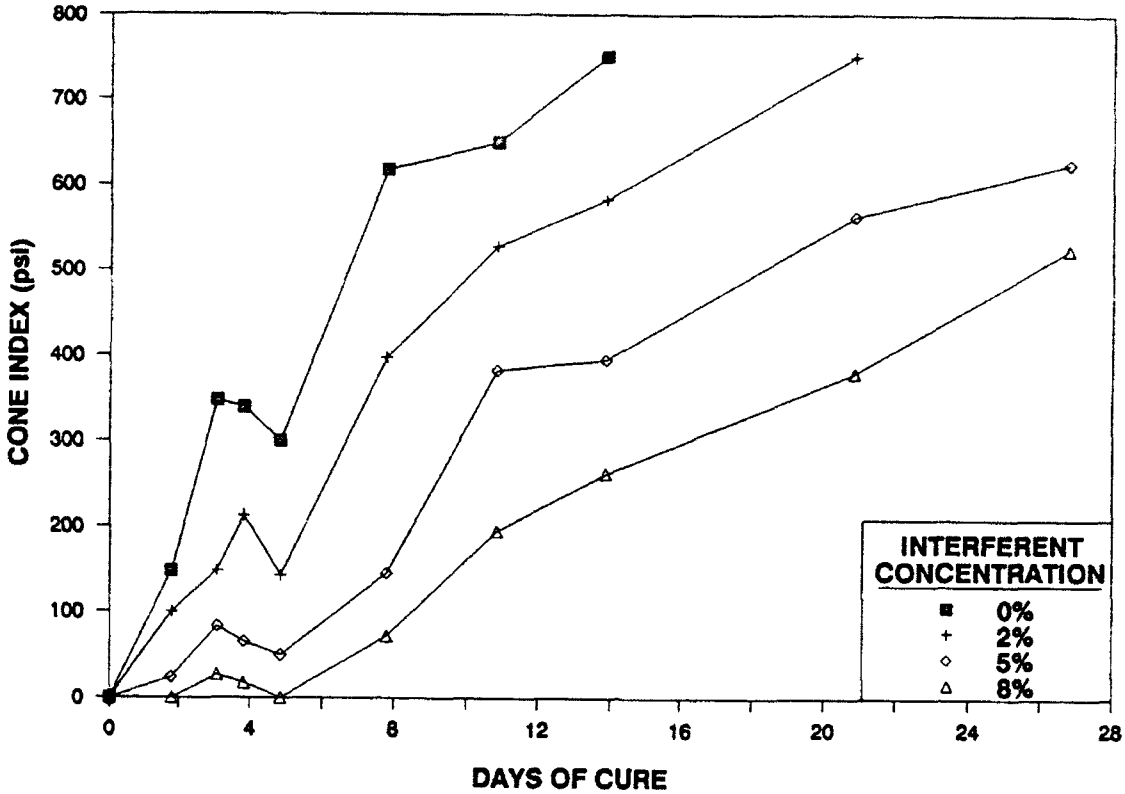

Figure 38. Cone index for the CEM solidified/stabilized samples with phenol interference.

REGRESSION ANALYSIS OF UCS AND CI

A regression analysis was performed to compare the results of the UCS and CI. Because CI only measures sample properties during the initial early set, UCS and CI can only be compared during this early-set period. The model for the analysis includes UCS or CI as the dependent variable and the number of days of curing as independent variables, and determines a best-fit regression line. Due to the strong statistical interactions present in the data for the different binders and interferents, each interferent concentration and binder combination was analyzed individually and independently.

This analysis can be visualized using an example of a hypothetical data set, as shown in Figure 39 Three data sets are shown along with their calculated "best-fit" regression lines and correlation coefficients. The top line represents a sample with a decreasing rate of set over the sampling time, which is similar to that seen for the curing of cement paste. In this case, the Y-intercept of the regression line is positive, and its slope underestimates the initial slope of the actual curve. As we have seen, plotting the data against the square root of the curing time tends to give a linear plot and a zero Y-intercept.

The lower line represents a sample with an increasing rate of set (or a delayed set). Note that the best-fit regression line has a negative Y-intercept and overestimates the initial slope of the actual curve. Plotting the data against the square of the time of curing would tend to give a linear plot of the data. A negative Y-intercept is indicative of a delay in set.

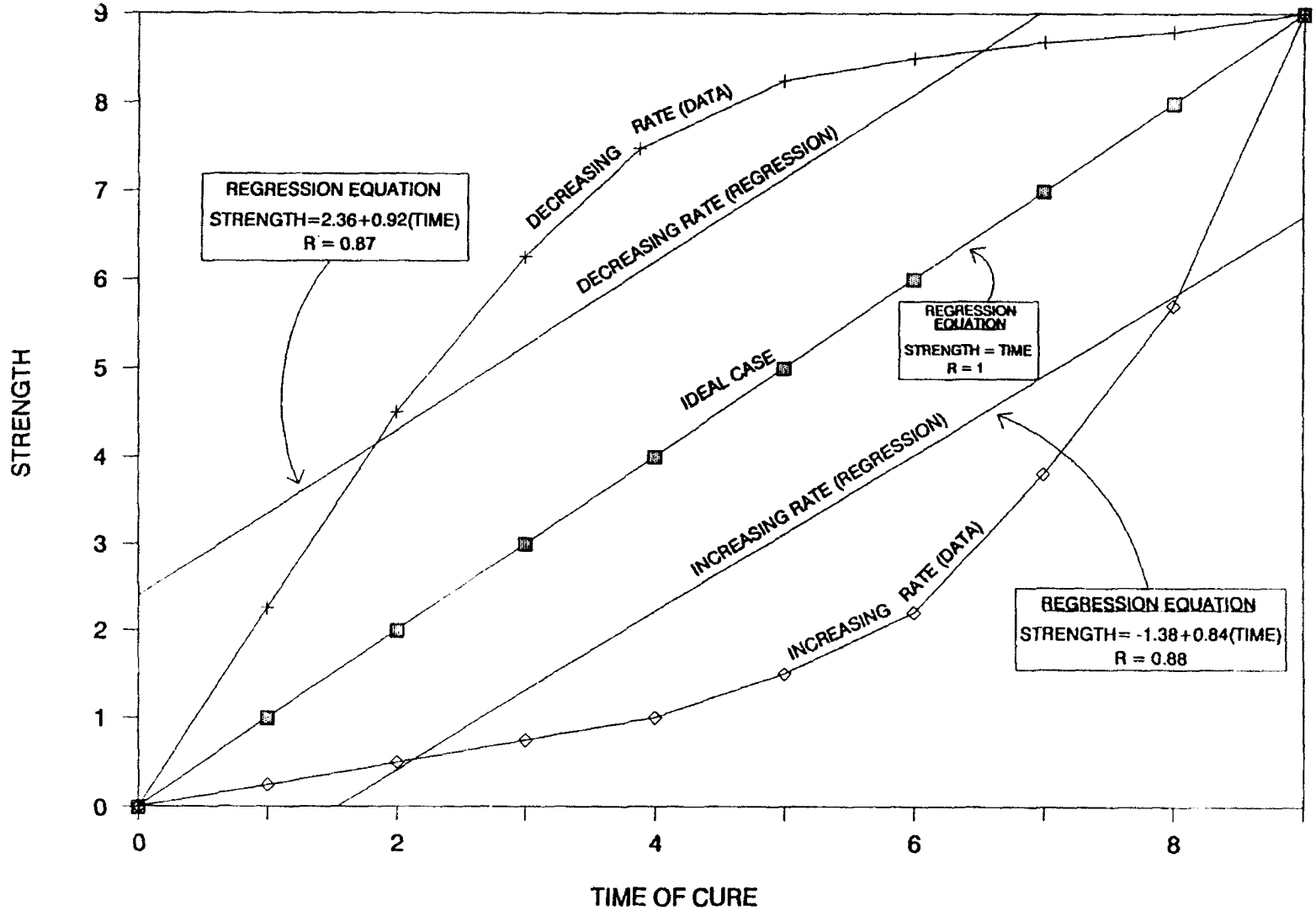

Figure 39. Hypothetical strength versus cure time curves.

The center line represents the ideal case of a sample having a linear setting pattern that is similar to that seen for CFA and LFA over the time periods used in this study. The regression line falls exactly on the line through the data. Note that the correlation coefficients for the first two examples fall around 0.88, indicating a reasonable fit to the regression line. The linear plot, of course, has a correlation coefficient of 1.0.

Similar regression analysis data for both CI and UCS are summarized in Tables 21-23. All of the data listed in these tables are significantly different from zero at a 99-percent level of confidence. The data include the root mean square error (RMSE), which is an estimate of the standard deviation after accounting for the differences between groups, and the coefficients for the best-fit line. The zero intercept (b_o) represents the point where the regression line crosses the UCS or CI axis. Values of b_o above zero indicate that the rate of gain of strength of the samples is hyperbolic in that the rate of set decreases with time; this is the typical situation for the setting of cement. Values below zero indicate an increasing rate of set with time or an initial delay in setting, which is more typical of CFA and LFA. An intercept not significantly different from zero indicates a near-linear rate of set for the period tested. The correlation coefficients are also presented in the tables.

The parameter b_1 represents the coefficient for the rate of increase in UCS or CI over the time of cure. It is the slope of the "best-fit" regression line. A positive value of either b_1 indicates a direct relationship between the variable and the UCS or CI; a negative value indicates an indirect relationship.

Samples Without Added Interferents

The analysis is illustrated by looking only at the UCS and CI of control samples. Control samples from all three binder systems have a significant, positive coefficient (b_1) for the time of curing of the samples; i.e., all samples were going through the setting reactions over the time of the testing. The rate at which they were setting, however, varied considerably.

The control sample values that were pooled and analyzed for the different binder systems independently are shown in Table 24. The analysis of CI, which is representative of early setting rates (from about day 1 to day 7 or 10), indicates that the CFA samples have the greatest rate of initial set, followed by LFA and CEM. Both CFA and LFA have CI and UCS intercepts that are very close to (not significantly different from) zero, indicating a nearly constant rate of set over the CI testing period. CEM, on the other hand, has a large, positive intercept, showing that its rate of set decreases with time. A large, positive intercept also results in an underestimate of the slope of the cure term, so that this value is not readily comparable with the other parameters. Running a regression line of CI against the log of days of curing for cement results in an intercept not significantly different from zero. The RMSE values are relatively large, indicating a large variability in the data within each binder system.

The UCS data for the control samples have a similar pattern. The CFA and LFA samples have very similar rates of CI and UCS development and intercepts that are not significantly different from zero. This is indicative of a near-linear rate of set over the 28-day testing period. The variation in the

TABLE 21. COMPARISON OF UCS AND CI REGRESSION ANALYSIS - CEM SAMPLES

Binder	Inter-ferent	Conc. (%)	Cone index RMSE	Cone index Intercept (b_o)	Cone index Cure (b_1)	UCS RMSE	UCS Interc. (b_o)	UCS Cure (b_1)
CEM	Cu	0	19.9	NS*	61.7	16.8	20.8	0.97
CEM	Cu	2	19.3	NS	89.1	22.3	29.3	2.13
CEM	Cu	5	39.4	-26.9	32.6	21.9	NS	4.05
CEM	Cu	8	97.5	NS	25.2	3.9	5.2	1.40
CEM	Pb	0	97.5	99.7	17.3	13.3	18.5	2.02
CEM	Pb	2	134.4	87.3	22.3	11.4	NS	2.14
CEM	Pb	5	131.5	NS	21.0	8.5	11.2	2.67
CEM	Pb	8	99.6	52.4	17.2	8.7	11.0	2.50
CEM	Zn	0	31.4	NS	36.7	4.7	NS	2.54
CEM	Zn	2	26.8	NS	46.1	8.7	NS	10.85
CEM	Zn	5	56.1	NS	7.5	1.9	NS	0.70
CEM	Zn	8	41.2	NS	5.5	0.8	NS	0.36
CEM	Sulfate	0	24.0	NS	49.0	10.4	12.6	1.68
CEM	Sulfate	2	30.5	NS	40.6	6.6	NS	1.63
CEM	Sulfate	5	18.2	NS	40.6	6.4	8.0	1.49
CEM	Sulfate	8	24.9	NS	41.2	9.8	11.8	NS
CEM	NaOH	0	35.6	NS	64.8	19.3	NS	5.15
CEM	NaOH	2	89.3	NS	117.6	33.8	34.3	5.48
CEM	NaOH	5	54.8	43.7	15.5	10.6	NS	3.47
CEM	NaOH	8	21.9	NS	8.1	3.5	NS	2.59
CEM	Grease	0	161.6	166.3	66.8	53.7	56.5	6.96
CEM	Grease	2	159.0	196.7	35.3	50.4	55.4	5.92
CEM	Grease	5	108.6	148.2	18.1	43.0	45.0	5.16
CEM	Grease	8	71.1	94.9	12.2	31.7	NS	3.82
CEM	Oil	0	118.5	142.2	48.5	33.8	38.6	6.65
CEM	Oil	2	109.9	117.0	34.6	28.8	31.5	5.32
CEM	Oil	5	78.5	97.7	16.9	19.4	20.4	4.20
CEM	Oil	8	53.1	70.5	13.1	18.1	18.2	3.81
CEM	TCE	0	61.9	49.2	56.6	15.0	20.7	2.30
CEM	TCE	2	115.5	110.9	23.8	14.7	18.2	1.63
CEM	TCE	5	102.8	103.7	19.0	13.2	16.7	1.42
CEM	TCE	8	89.1	94.7	19.1	14.2	18.6	1.60
CEM	HCB	0	47.5	35.2	54.4	16.0	21.8	2.71
CEM	HCB	2	49.5	36.6	53.7	13.3	17.3	2.66
CEM	HCB	5	59.4	49.1	54.2	15.7	20.3	2.56
CEM	HCB	8	84.4	70.5	58.7	18.0	23.3	3.15
CEM	Phenol	9	64.8	NS	64.5	27.2	36.1	3.51
CEM	Phenol	2	36.7	NS	45.0	26.1	32.4	2.66
CEM	Phenol	5	47.5	NS	26.7	13.0	15.4	2.87
CEM	Phenol	8	32.9	-30.5	19.7	6.3	NS	1.82

* Not significant at 99-percent level of confidence.

TABLE 22. COMPARISON OF UCS AND CI REGRESSION ANALYSIS-CFA SAMPLES

			Cone index			UCS		
Binder	Inter-ferent	Conc. (%)	RMSE	Intercept (b_o)	Cure (b_1)	RMSE	Interc. (b_o)	Cure (b_1)
CFA	Cu	0	7.8	NS	43.2	36.74	NS	14.3
CFA	Cu	2	11.4	NS	40.1	38.89	NS	16.5
CFA	Cu	5	ND	ND	ND	9.20	NS	2.2
CFA	Cu	8	ND	ND	ND	1.78	NS	0.2
CFA	Pb	0	9.7	NS	91.5	64.1	NS	24.2
CFA	Pb	2	55.3	NS	74.7	7.7	NS	11.4
CFA	Pb	5	62.4	NS	38.0	8.7	NS	5.7
CFA	Pb	8	41.7	NS	27.8	5.4	NS	4.6
CFA	Zn	0	25.3	NS	90.7	62.9	NS	13.3
CFA	Zn	2	107.7	-57.3	17.5	28.1	NS	8.1
CFA	Zn	5	10.5	NS	4.0	1.5	NS	1.2
CFA	Zn	8	13.9	18.9	3.7	4.6	NS	0.7
CFA	Sulfate	0	39.2	NS	59.6	34.8	NS	18.7
CFA	Sulfate	2	53.2	NS	76.9	55.2	NS	24.3
CFA	Sulfate	5	31.0	NS	63.2	48.5	NS	21.9
CFA	Sulfate	8	26.8	-18.7	50.0	12.0	NS	6.7
CFA	NaOH	0	24.8	NS	92.0	13.0	NS	23.2
CFA	NaOH	2	223.9	174.1	NS	30.7	NS	28.0
CFA	NaOH	5	82.7	NS	379.6	57.6	NS	23.4
CFA	NaOH	8	59.1	NS	225.3	49.0	NS	17.9
CFA	Grease	0	21.6	NS	80.1	31.44	NS	29.5
CFA	Grease	2	15.2	11.8	63.3	25.52	NS	23.6
CFA	Grease	5	14.2	10.3	43.4	14.18	NS	17.7
CFA	Grease	8	13.4	16.0	28.9	6.01	NS	15.0
CFA	Oil	0	23.7	NS	99.0	37.2	NS	22.5
CFA	Oil	2	44.7	NS	54.6	19.5	NS	13.6
CFA	Oil	5	25.1	NS	48.7	19.4	NS	14.3
CFA	Oil	8	10.1	NS	47.4	33.0	NS	21.6
CFA	TCE	0	4.6	-4.5	103.1	21.3	NS	23.0
CFA	TCE	2	10.6	-7.6	87.3	28.7	-32.7	21.5
CFA	TCE	5	17.7	NS	69.8	12.5	NS	15.5
CFA	TCE	8	28.8	NS	69.8	15.1	NS	16.1
CFA	HCB	0	64.1	NS	96.6	25.31	NS	25.6
CFA	HCB	2	59.5	NS	101.8	22.12	NS	22.9
CFA	HCB	5	66.6	NS	105.4	19.3	-23.9	23.2
CFA	HCB	8	52.7	NS	98.4	20.8	NS	23.0
CFA	Phenol	0	29.8	-24.2	108.2	26.1	NS	27.2
CFA	Phenol	2	12.2	-10.2	49.5	16.4	NS	13.9
CFA	Phenol	5	174.1	NS	8.4	3.4	NS	4.7
CFA	Phenol	8	65.3	NS	3.1	2.0	NS	2.1

TABLE 23. COMPARISON OF UCS AND CI REGRESSION ANALYSIS-LFA SAMPLES

			Cone index			UCS		
Binder	Inter-ferent	Conc. (%)	RMSE	Intercept (b_o)	Cure (b_1)	RMSE	Interc. (b_o)	Cure (b_1)
LFA	Cu	0	15.2	NS	74.2	39.9	NS	22.0
LFA	Cu	2	68.7	52.4	37.0	25.6	NS	11.3
LFA	Cu	5	101.7	77.1	55.2	10.5	NS	10.9
LFA	Cu	8	77.3	69.2	22.4	14.8	20.5	NS
LFA	Pb	0	58.2	-40.2	52.7	17.4	NS	19.7
LFA	Pb	2	32.1	-20.1	17.8	17.3	-21.7	12.0
LFA	Pb	5	14.1	NS	6.9	5.9	NS	4.4
LFA	Pb	8	15.1	NS	5.5	2.6	NS	1.9
LFA	Zn	0	32.6	NS	69.0	11.7	NS	35.3
LFA	Zn	2	29.3	24.6	19.5	21.7	NS	11.8
LFA	Zn	5	46.8	51.2	20.7	9.6	NS	5.5
LFA	Zn	8	35.8	42.3	18.8	7.9	10.3	[illegible]
LFA	Sulfate	0	155.3	NS	33.8	18.1	NS	23.5
LFA	Sulfate	2	24.3	NS	123.8	156.9	170.8	35.0
LFA	Sulfate	5	48.0	NS	185.2	119.7	102.5	30.4
LFA	Sulfate	8	92.0	NS	73.0	51.1	29.1	4.1
LFA	NaOH	0	29.6	-22.8	80.3	14.8	NS	23.5
LFA	NaOH	2	1.1	NS	507.2	119.1	145.7	36.0
LFA	NaOH	5	0.0	NS	842.4	146.0	159.5	21.7
LFA	NaOH	8	69.7	NS	226.7	65.4	NS	20.6
LFA	Grease	0	25.2	NS	60.5	10.4	NS	23.2
LFA	Grease	2	16.8	NS	47.6	14.8	NS	21.7
LFA	Grease	5	35.5	-30.7	49.3	11.2	NS	17.2
LFA	Grease	8	20.5	-18.90	30.6	14.0	NS	11.1
LFA	Oil	0	21.1	NS	54.6	24.6	NS	20.1
LFA	Oil	2	13.9	-9.8	51.3	12.4	NS	18.7
LFA	Oil	5	36.8	-22.3	45.5	11.0	NS	14.6
LFA	Oil	8	25.1	NS	33.8	9.4	NS	13.8
LFA	TCE	0	23.9	NS	47.3	18.8	NS	16.6
LFA	TCE	2	22.6	NS	53.6	16.3	NS	25.0
LFA	TCE	5	23.2	NS	49.2	23.5	NS	19.9
LFA	TCE	8	53.1	NS	70.8	25.7	NS	22.6
LFA	HCB	0	22.3	-15.7	57.2	22.4	NS	15.5
LFA	HCB	2	39.7	-27.4	64.4	23.0	NS	16.5
LFA	HCB	5	68.3	-48.6	83.6	35.5	NS	17.0
LFA	HCB	8	56.2	NS	80.3	27.9	NS	15.3
LFA	Phenol	0	37.6	NS	73.0	1.2	NS	24.9
LFA	Phenol	2	55.1	NS	25.6	2.4	NS	8.7
LFA	Phenol	5	12.2	NS	10.7	5.4	NS	2.8
LFA	Phenol	8	6.7	NS	5.8	3.0	NS	0.9

TABLE 24. REGRESSION ANALYSIS BY BINDER SYSTEM FOR CI AND UCS OF SAMPLES WITHOUT INTERFERENT ADDITION

	Cone index			Unconfined Compressive strength		
Binder	RMSE	Intercept (b_o)	Cure (b_1)	RMSE	Intercept (b_o)	Cure (b_1)
CEM	151.0	151.8	29.2	46.2	21.9	3.2
CEM*	123.7	NS	149			
CFA	71.8	NS	88.7	118.9	NS	21.9
LFA	55.8	NS	64.4	56.1	NS	21.6

Note: All values are significantly different from zero at the 99-percent level of confidence.
* Regression using logarithm of days of cure.

CFA control samples is about twice as great as that of the LFA. CFA again shows a much lower setting rate and a positive intercept, indicating a faster initial set followed by a slowing rate of set.

Effects of Interferent Additions

Addition of the interferents generally decreased the rate of set, as indicated by the b_1 values in Tables 21-23. Interferents having the greatest effect on CI and UCS were Cu, Pb, Zn, NaOH, grease, oil, and phenol.

Addition of any of the metals had profound effect upon the rate of set of samples of all three binders. Addition of the metals had the least effect on CFA samples. At low levels (2 percent), Cu, Pb, and Zn produced faster set as indicated by larger rate of CI and UCS development (b_1). Higher levels of Cu and Pb had less effect, while the higher levels of Zn lowered the rate of both CI and UCS development for CEM samples.

In CFA and LFA samples, all three metals uniformly decreased both the CI and UCS to very low values, especially at the higher concentrations. This indicates an almost complete lack of setting reactions in these samples, at least over the maximum 28-day curing period used in this study.

Both sodium hydroxide and sodium sulfate increase the rate of CI and UCS development, indicating a more rapid set, for all three binder systems. Increases were 2 to 5 times the rate of control samples at 2- and 5-percent additions for CFA and LFA samples. However, for the binders containing fly ash, an 8-percent addition increased CI development to a lesser extent and produced lower UCS development rates. For the CEM samples, sulfate had little effect over the time of cure studied; however, NaOH produced higher UCS and CI at 2-percent addition but caused significant decreases in both parameters at 5- and 8-percent additions.

Of the organics, HCB had the smallest effect on the development of CI and UCS, producing only a slightly faster set (higher rate of CI development) in the LFA samples. Grease, oil, and TCE consistently lowered the development of CI and UCS of all three binder systems, the only exception being the

increasing of both CI and UCS development in LFA samples. Phenol produced dramatic decreases in both CI and UCS slopes, with the 8-percent addition having only 3 to 6 percent of the control rate for both CFA and LFA. The CEM CI and UCS development was similarly lowered to about 25 percent of the control values.

CI As a Predictor of UCS:
Correlation of UCS and CI Measurements

The development of short-term tests that would accurately predict the ultimate strength of solidified/stabilized waste would be of great use to the waste treatment industry. The quick, reproducible cone index test method has been suggested as a rapid, inexpensive method for predicting the long-term properties of solidified/stabilized waste (Myers 1986). The data collected in this study were analyzed to determine the ability of the CI measurements to predict 28-day UCS. To compare CI data with those of the 28-day UCS, a best-fit regression line was determined for each interferent concentration and each binder. The values of CI for 5 and 10 days of curing were calculated from these regression lines and compared to the 28-day UCS value. The three values are listed for each of the treatment levels and binders in Tables 25-27. Note that there is good apparent agreement between the two measurements.

The actual correlation between CI and UCS for the different binder systems is shown in Figures 40-42. The 28-day UCS is plotted against 5-day CI for each binder system; also included are the best-fit regression lines and 95-percent confidence intervals. A fair amount of scatter in the data produces moderately wide confidence intervals and low correlation coefficients.

The equation for the regression line is included in Figures 40-42. The correlation coefficients are listed in Table 28; they may be relatively low (only near 0.5) because of the high variability between the different S/S batches. However, because of the large number of measurements, the coefficients are highly significant (significantly different from zero at the 99-percent level of confidence). The significant correlation coefficients for the 5- and 10-day CI values support a link between the early CI and 28-day UCS. The relationship between UCS and CI is illustrated in Figures 43-46.

As an added note, attempts were made to correlate the results of all the test methods performed in this study with each other. The only conclusive correlation was that between the UCS and the CI values.

WET/DRY TESTING

Samples Without Interferent Additions

The results of the wet/dry test are expressed as the percent of the specimen remaining in the monolithic form after being subjected to a number of cycles of wetting and drying. The percent of the control specimens remaining, averaged over each of the binder systems, is listed in Table 29 and shown in Figure 47. Data for control samples receiving only the oven treatment are summarized in Table 30 and plotted in Figure 48. As expected, the cement binder system that was formulated to give the lowest UCS also had the least durability to wet/dry cycling and to oven treatment. Although the wet/dry-cycled LFA samples have similar losses over the 11 cycles, the LFA samples maintained near 90 percent of their mass over the first five wet/dry cycles.

TABLE 25. COMPARISON FOR CEM OF CALCULATED CI AT 5 AND 10 DAYS WITH 28-DAY UCS

Binder	Interferent	Concentration (%)	UCS (28-Day)	CI (5-Day)	CI (10-Day)
CEM	Cu	0	51.1	299.4	616.1
CEM	Cu	2	95.4	458.1	893.2
CEM	Cu	5	142.2	128.5	304.3
CEM	Cu	8	48.9	54.4	192.3
CEM	Pb	0	75.2	211.7	282.5
CEM	Pb	2	74.2	223.6	315.2
CEM	Pb	5	86.1	134.4	199.2
CEM	Pb	8	81.2	134.2	203.0
CEM	Zn	0	76.0	193.9	374.7
CEM	Zn	2	311.1	8.0	462.2
CEM	Zn	5	17.9	7.2	48.8
CEM	Zn	8	9.6	231.6	36.7
CEM	Sulfate	0	59.8	233.2	481.7
CEM	Sulfate	2	53.6	182.7	396.5
CEM	Sulfate	5	49.8	195.1	402.7
CEM	Sulfate	8	30.5	204.5	411.0
CEM	NaOH	0	160.5	335.3	647.5
CEM	NaOH	2	187.8	637.6	1150.9
CEM	NaOH	5	106.8	154.0	224.3
CEM	NaOH	8	74.9	53.6	90.0
CEM	Grease	0	251.6	526.3	797.6
CEM	Grease	2	221.3	413.7	537.4
CEM	Grease	5	189.5	275.5	343.3
CEM	Grease	8	137.5	179.9	226.4
CEM	Oil	0	224.8	413.9	616.6
CEM	Oil	2	180.5	334.9	482.1
CEM	Oil	5	138.2	208.3	282.7
CEM	Oil	8	125.1	154.5	210.7
CEM	TCE	0	85.4	343.6	307.9
CEM	TCE	2	64.0	264.9	602.6
CEM	TCE	5	56.7	231.0	356.4
CEM	TCE	8	63.5	219.7	300.6
CEM	HCB	0	97.9	313.6	576.9
CEM	HCB	2	92.0	312.0	571.5
CEM	HCB	5	92.2	329.1	587.9
CEM	HCB	8	111.8	377.0	653.0
CEM	Phenol	0	134.5	367.8	669.8
CEM	Phenol	2	107.0	230.8	451.0
CEM	Phenol	5	96.0	119.4	249.9
CEM	Phenol	8	58.8	56.3	161.2

TABLE 26. COMPARISON FOR CFA OF CALCULATED CI AT 5 AND 10 DAYS WITH 28-DAY UCS

Binder	Interferant	Concentration (%)	UCS (28-Day)	CI (5-Day)	CI (10-Day)
CFA	Cu	0	435.5	214.2	434.2
CFA	Cu	2	499.2	200.0	401.8
CFA	Cu	5	70.8	ND	ND
CFA	Cu	8	8.7	ND	ND
CFA	Pb	0	787.5	453.0	915.1
CFA	Pb	2	321.3	389.9	749.8
CFA	Pb	5	169.0	223.9	406.2
CFA	Pb	8	135.9	148.5	286.7
CFA	Zn	0	445.7	444.7	923.6
CFA	Zn	2	209.6	14.9	109.3
CFA	Zn	5	35.8	17.8	38.2
CFA	Zn	8	27.3	42.5	59.1
CFA	Sulfate	0	452.4	274.1	595.8
CFA	Sulfate	2	577.5	357.1	769.0
CFA	Sulfate	5	523.2	300.6	632.1
CFA	Sulfate	8	179.2	228.6	488.3
CFA	NaOH	0	640.2	465.0	921.4
CFA	NaOH	2	804.5	156.5	18.2
CFA	NaOH	5	698.4	1802.5	3513.8
CFA	NaOH	8	538.0	251.0	156.9
CFA	Grease	0	803.5	412.9	804.1
CFA	Grease	2	645.1	328.2	636.6
CFA	Grease	5	486.6	229.9	444.5
CFA	Grease	8	420.8	164.1	304.5
CFA	Oil	0	614.0	486.2	988.9
CFA	Oil	2	369.8	294.8	563.2
CFA	Oil	5	388.4	255.6	496.7
CFA	Oil	8	584.6	240.2	476.7
CFA	TCE	0	617.2	511.5	1030.5
CFA	TCE	2	571.3	429.8	872.2
CFA	TCE	5	418.1	352.4	700.6
CFA	TCE	8	436.2	350.6	699.5
CFA	HCB	0	686.9	469.1	984.3
CFA	HCB	2	618.5	493.7	1037.4
CFA	HCB	5	627.0	508.3	1078.5
CFA	HCB	8	620.0	487.9	998.4
CFA	Phenol	0	730.7	519.2	1083.5
CFA	Phenol	2	374.8	238.4	495.7
CFA	Phenol	5	133.1	138.7	171.3
CFA	Phenol	8	61.5	52.9	65.1

TABLE 27. COMPARISON FOR LFA OF CALCULATED CI AT 5 AND 10 DAYS WITH 28-DAY UCS

Binder	Interferent	Concentration (%)	UCS (28-Day)	CI (5-Day)	CI (10-Day)
LFA	Cu	0	634.0	370.2	742.5
LFA	Cu	2	333.8	248.6	405.7
LFA	Cu	5	320.7	369.9	605.1
LFA	Cu	8	13.3	196.2	271.5
LFA	Pb	0	540.5	217.2	500.3
LFA	Pb	2	316.4	63.8	157.6
LFA	Pb	5	118.8	31.1	65.0
LFA	Pb	8	57.2	33.3	60.7
LFA	Zn	0	692.8	345.2	690.2
LFA	Zn	2	311.9	130.1	224.7
LFA	Zn	5	158.5	170.5	267.4
LFA	Zn	8	84.1	149.2	237.5
LFA	Sulfate	0	642.1	255.9	403.5
LFA	Sulfate	2	1152.6	633.8	1292.7
LFA	Sulfate	5	954.8	955.2	1960.0
LFA	Sulfate	8	145.4	303.5	659.9
LFA	NaOH	0	639.2	381.0	798.1
LFA	NaOH	2	1155.4	74.4	-62.5
LFA	NaOH	5	769.2	42.7	-55.9
LFA	NaOH	8	658.4	1059.0	2035.3
LFA	Grease	0	648.6	295.2	613.3
LFA	Grease	2	606.5	232.2	482.6
LFA	Grease	5	472.5	209.3	468.0
LFA	Grease	8	307.7	129.3	287.8
LFA	Oil	0	569.9	273.0	546.6
LFA	Oil	2	521.4	245.5	505.8
LFA	Oil	5	411.7	201.2	434.6
LFA	Oil	8	375.3	160.4	332.1
LFA	TCE	0	470.2	253.2	485.7
LFA	TCE	2	700.0	259.3	530.2
LFA	TCE	5	564.5	235.3	484.2
LFA	TCE	8	625.9	315.7	687.3
LFA	HCB	0	440.8	260.9	567.0
LFA	HCB	2	466.9	293.8	635.0
LFA	HCB	5	481.8	367.9	819.6
LFA	HCB	8	438.6	372.8	820.4
LFA	Phenol	0	695.5	372.9	734.9
LFA	Phenol	2	242.0	125.4	257.3
LFA	Phenol	5	80.6	53.9	107.5
LFA	Phenol	8	25.7	28.4	58.0

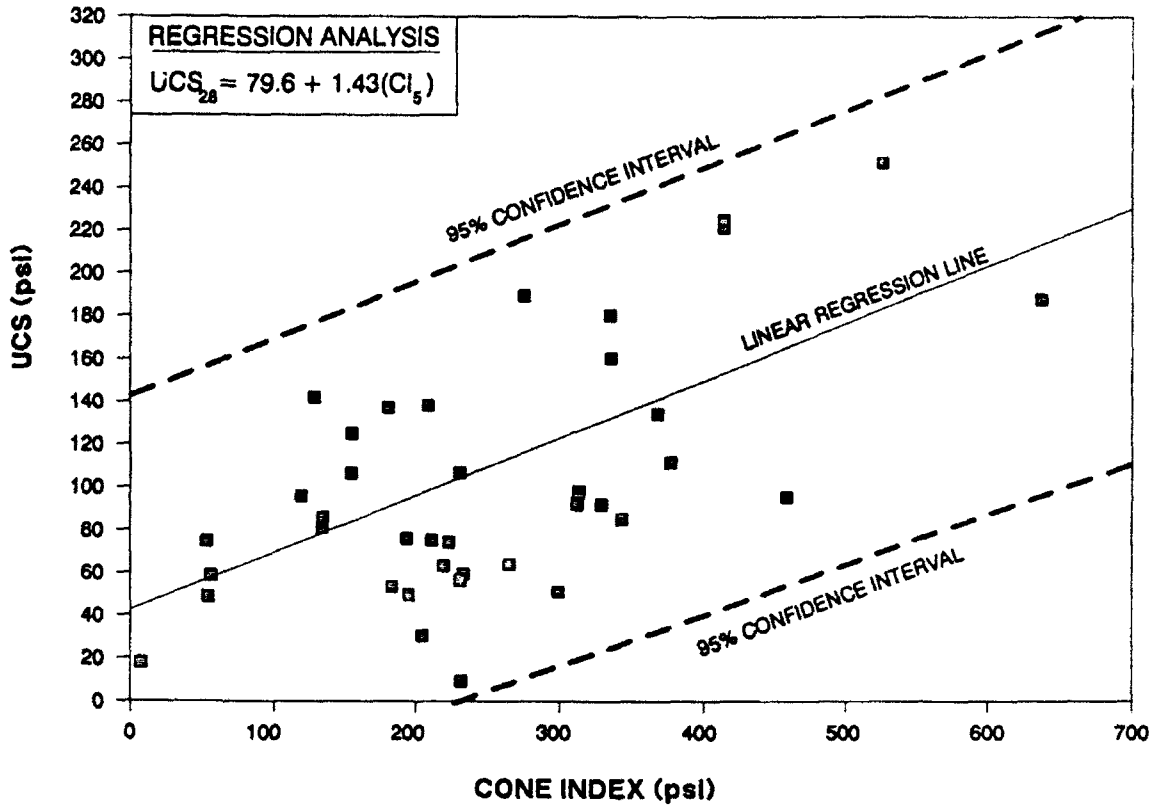

Figure 40. Predicted 5-day CI versus the 28-day UCS for the CEM solidified/stabilized samples, with the regression line and the 95-percent confidence interval.

The LFA samples, which undergo a pozzolanic setting process, set at a much slower rate than either the CEM or CFA samples, their lower durability may be a function of their slower setting rate. The CFA control samples showed the greatest durability to wet/dry cycling, maintaining around 95 percent of their integrity over all 11 wet/dry cycles; the bulk of this loss probably represents losses during the first drying cycle.

The specimens without added interferent that were only left in the oven (not wetted) lost weight over the first few cycles, asymptotically approaching a relatively constant percentage of their original weight. This is thought to be caused by a continued loss of water over the first few days in the 60° C oven. Following the first several days of drying, the weight of CFA and LFA specimens remained constant at around 85 to 86 percent of their original weight. Similarly, the weight of CFA specimens leveled out at about 80 percent of their original weight. As evident in Table 30, the lower CFA value is caused largely by two samples that fragmented and lost an appreciable amount of weight (the lead sample in the first cycle, and the zinc in the third and ninth cycles). Otherwise, the percentages of mass remaining for all specimens for all three binder systems after oven treatment are nearly identical.

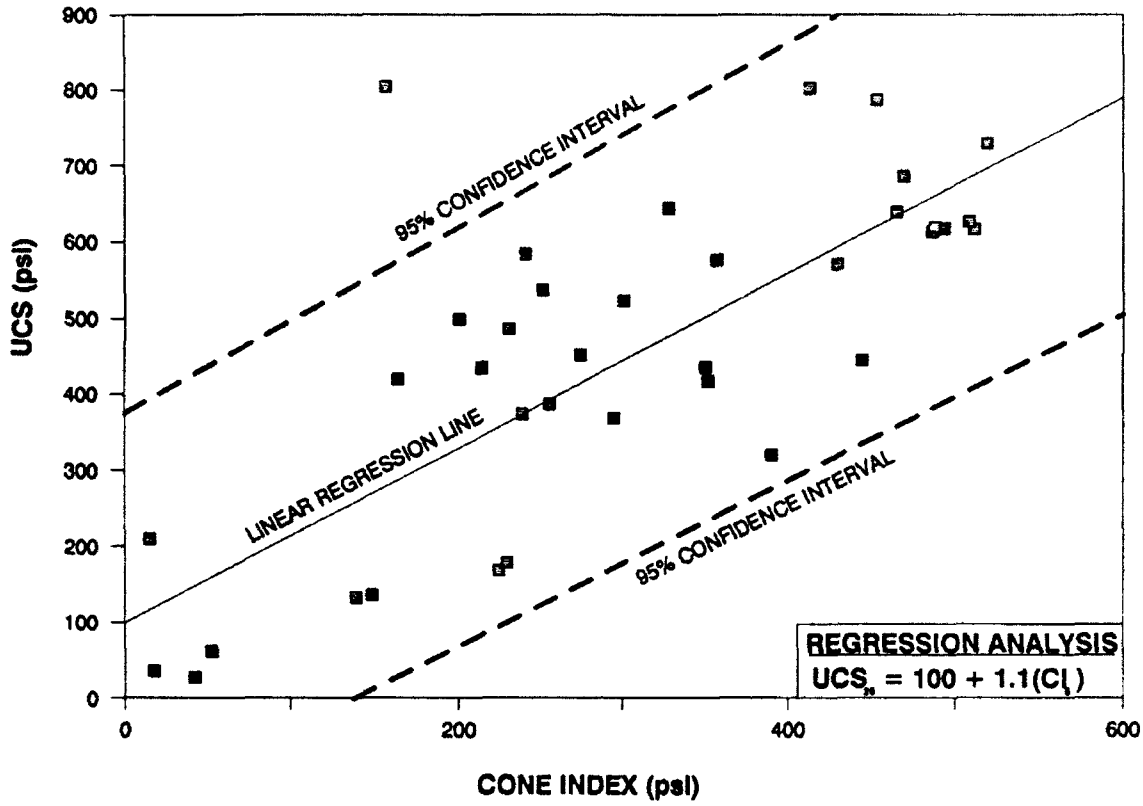

Figure 41. Predicted 5-day CI versus the 28-day UCS for the CFA solidified/stabilized samples, with the regression line and the 95-percent confidence interval.

Effects of Interferent Addition

The mean weights remaining after each wet/dry cycle (before failure) are listed in Tables 31-33. Failure of the specimen represents a loss of over 70 percent of its original mass. Failure in these tables refers to the failure of both specimens; failure of one sample is merely averaged as zero for that sample. The mean number of cycles before failure (with a maximum of 11 cycles) is summarized by interferent type in Tables 34-36. The mean number of cycles before failure in these tables is the average number of cycles each of the duplicate samples survived, and thus will not agree with the values in Tables 31-33, which represent failure of both samples.

Metal Interferents --

All three metal nitrate salts had significant effects on the wet/dry durability of all three binder systems. At 8-percent metal addition, specimens from all three binder systems averaged only 4 to 5 cycles before failure, compared 10 to 11 cycles before failure of the controls. An example of failure is illustrated in Figure 49, which shows specimens with 8 percent added lead after only two wet/dry cycles. Note that the sample left only in the oven has maintained its integrity. The LFA specimens appear to be somewhat more susceptible to metal interference at the lower levels of interferent addition, the 2-percent addition having little effect on the number of CEM and CFA cycles to failure, as illustrated for copper in the CEM binder (Figure 50).

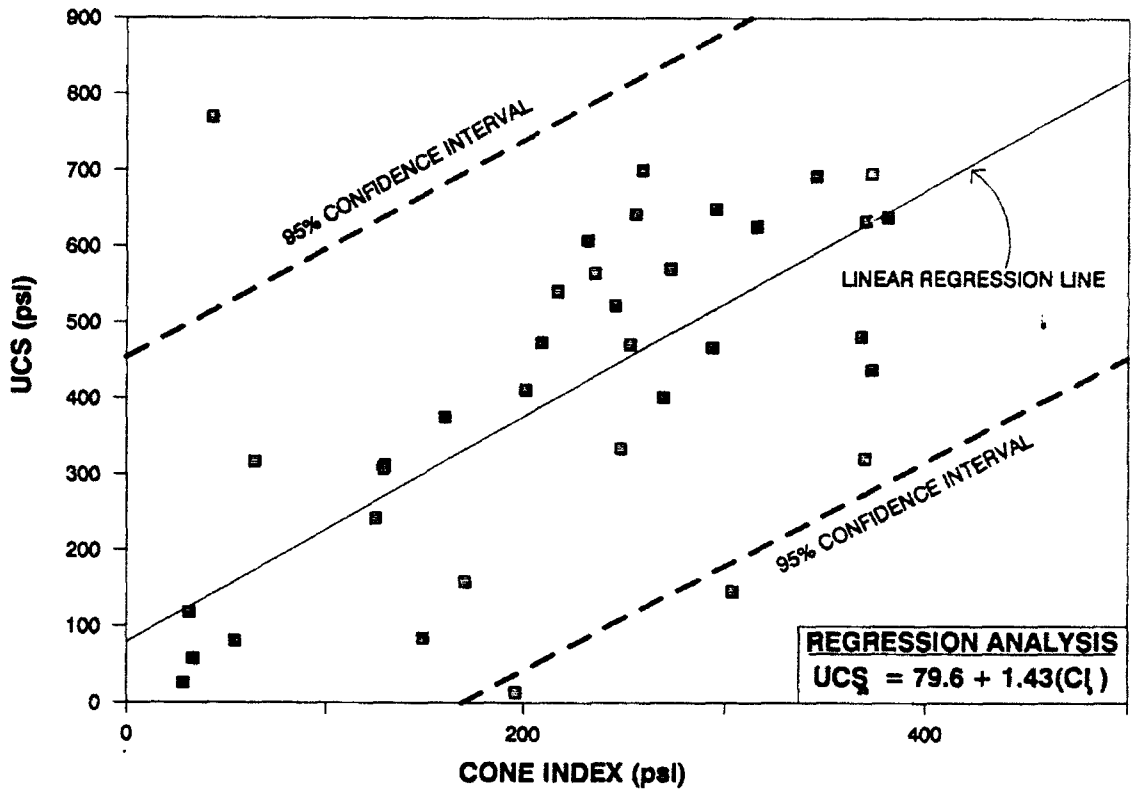

Figure 42. Predicted 5-day CI versus the 28-day UCS for the LFA solidified/stabilized samples, with the regression line and the 95-percent confidence interval.

TABLE 28. CORRELATION BETWEEN 5- AND 10-DAY CI AND UCS FOR EACH BINDER SYSTEM

Binder system	Correlation coefficient between 28-day UCS and 5-day CI	10-day CI
CEM	NS*	0.45
CFA	0.53	0.51
LFA	0.51	0.49

NOTE: Pearson correlation coefficients calculated on treatment means. All correlation coefficients listed are significantly different from zero at the 99-percent level of confidence.

* Not significant at 99-percent level of confidence.

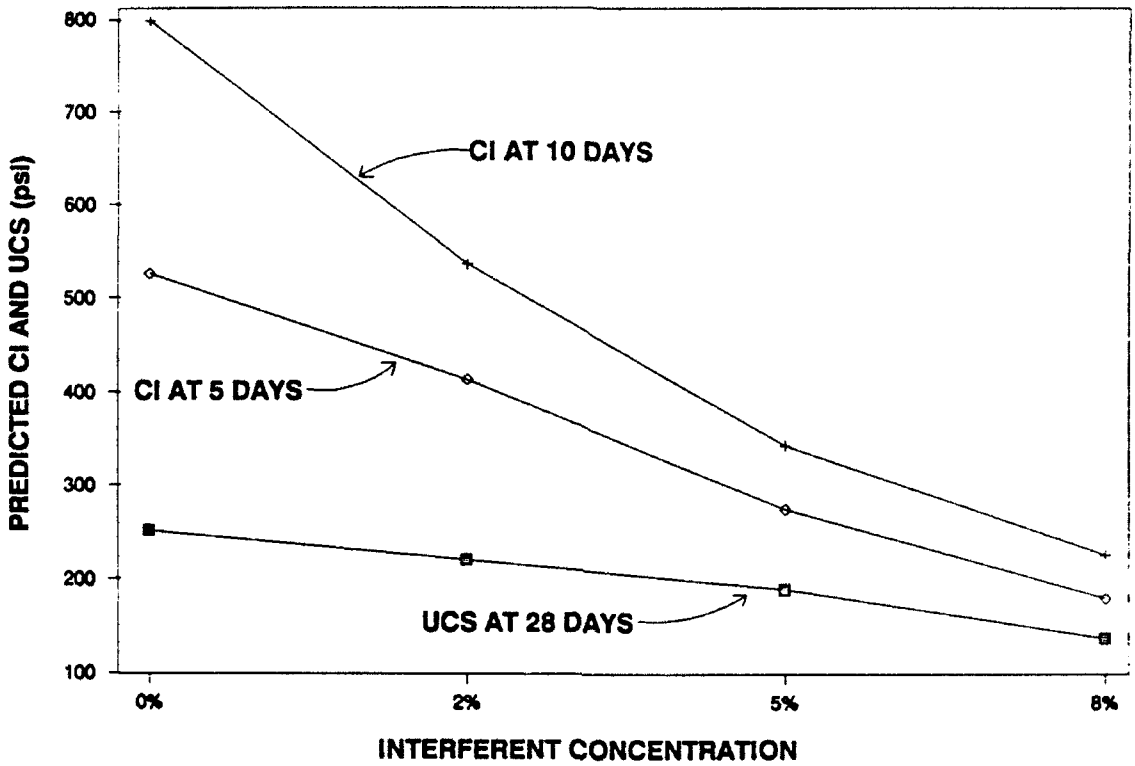

Figure 43. Interference concentration versus the 28-day UCS and the predicted 5- and 10-day CI for the CEM solidified/ stabilized samples with grease interference.

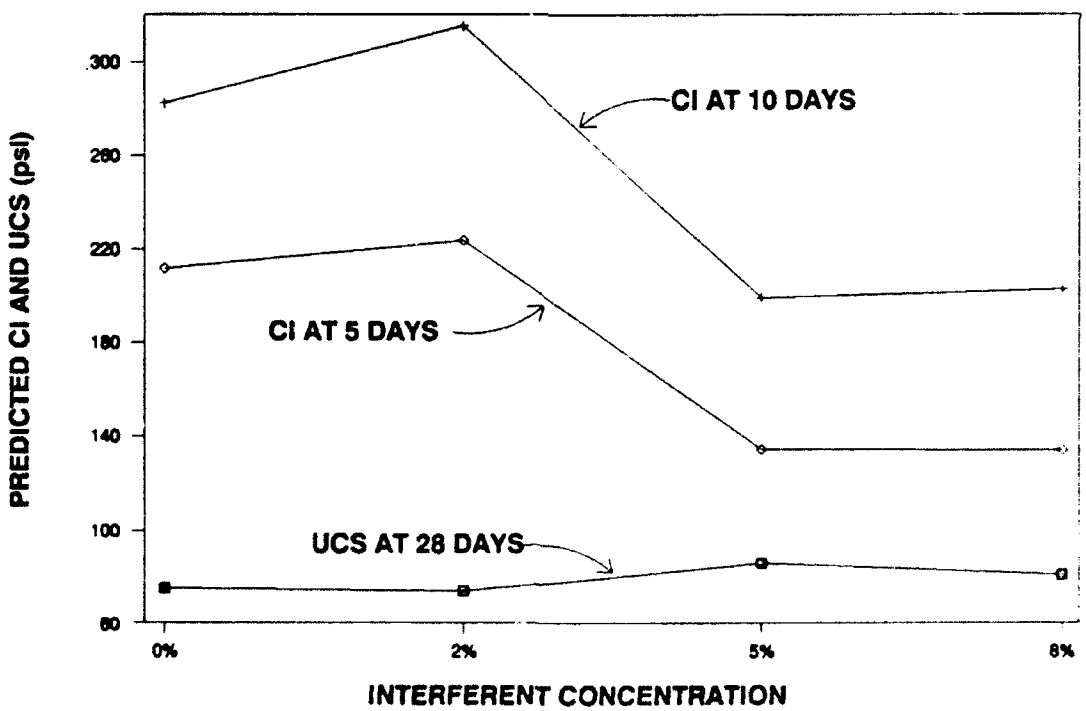

Figure 44. Interference concentration versus the 28-day UCS and the predicted 5- and 10-day CI for the CEM solidified/ stabilized samples with lead interference.

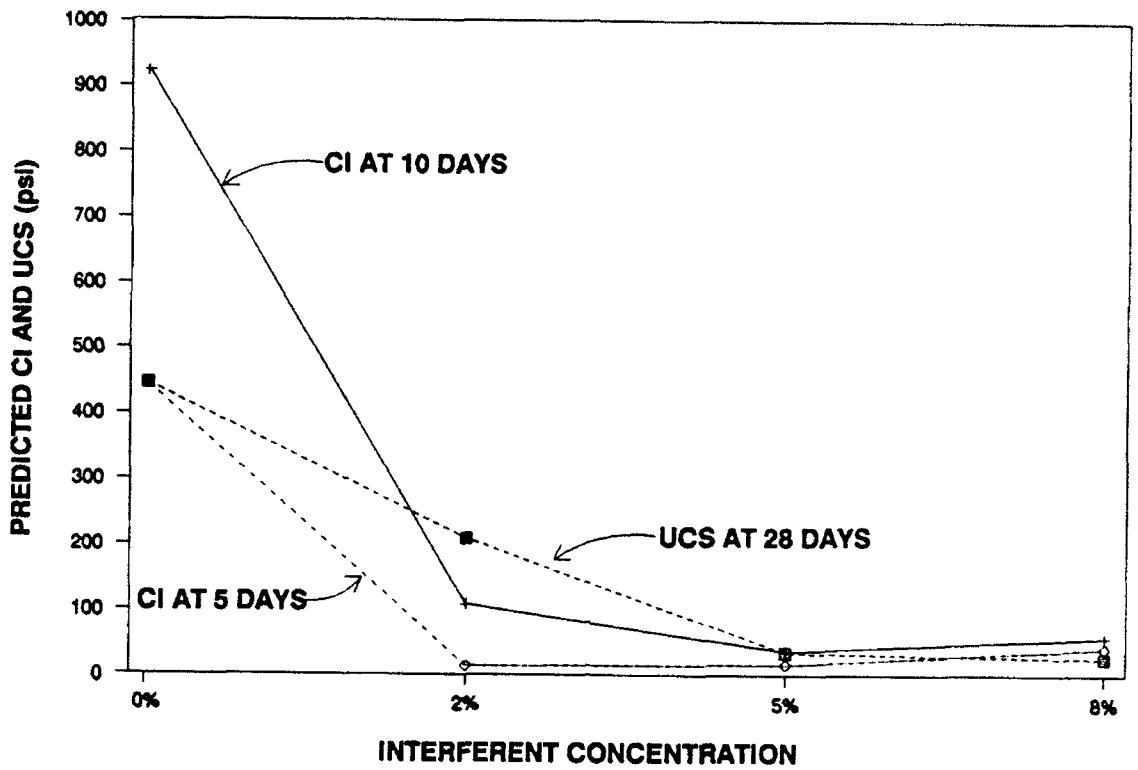

Figure 45. Interference concentration versus the 28-day UCS and the predicted 5- and 10-day CI for the CFA solidified/ stabilized samples with zinc interference.

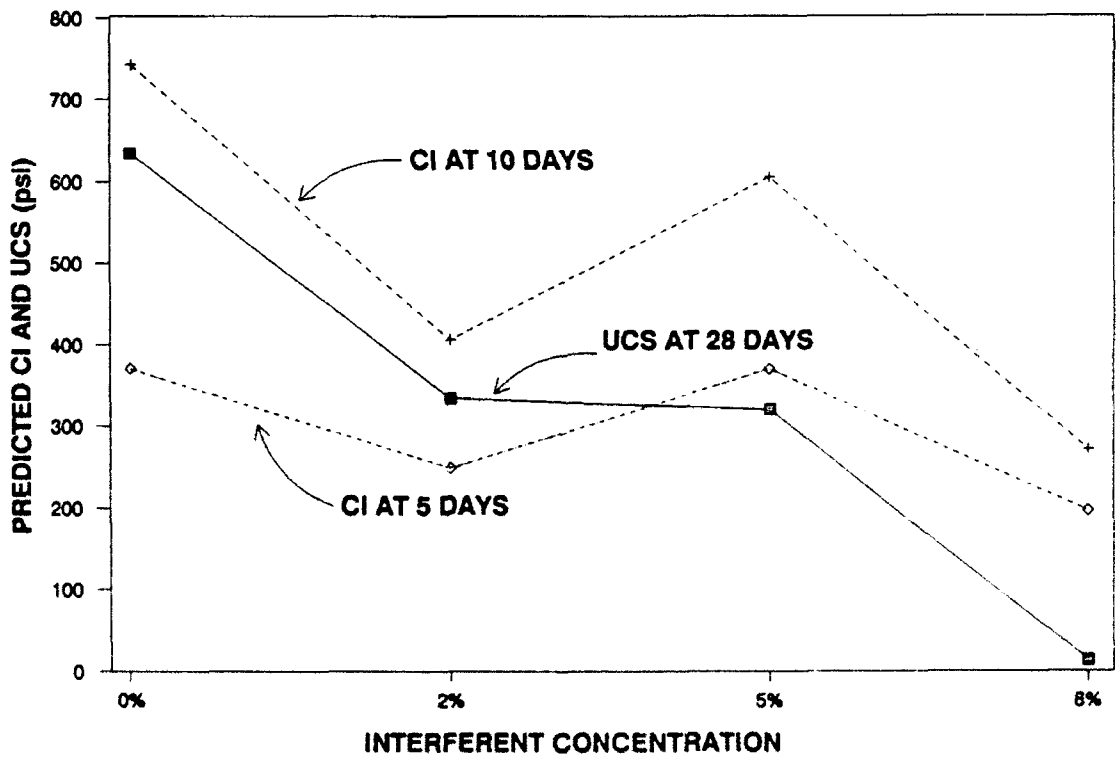

Figure 46. Interference concentration versus the 28-day UCS and the predicted 5- and 10-day CI for the LFA solidified/ stabilized samples with copper interference.

TABLE 29. AVERAGE PERCENT OF MASS REMAINING AFTER WET/DRY CYCLING--CONTROL SAMPLES ONLY

Binder	Interferent	Original weight (grams)	Cycle 1	2	3	4	5	6	7	8	9	10	11
CEM	Copper	657.0	96.9	95.0	92.0	87.0	84.5	80.0	75.4	76.0	74.6	71.6	74.5
CEM	Grease	781.0	84.8	85.8	82.0	80.1	82.5	82.3	81.9	77.1	36.2	36.0	36.2
CEM	HCB	732.2	88.7	90.9	83.6	80.7	79.1	78.9	77.3	77.6	77.7	76.5	77.5
CEM	Lead	1,183.8	59.5	46.6	45.8	42.7	40.9	34.7	32.5	Fail			
CEM	NaOH	699.1	83.8	85.9	83.4	78.5	80.5	80.8	78.1	78.3	78.2	76.0	76.8
CEM	Oil	800.0	85.7	88.7	88.6	83.5	86.0	85.9	80.2	82.2	80.3	76.2	73.6
CEM	Phenol	734.6	86.6	89.6	89.8	83.4	86.9	87.0	84.7	88.7	88.7	81.8	84.8
CEM	Sulfate	689.7	91.6	78.0	73.4	65.6	Fail						
CEM	TCE	702.0	91.1	91.0	88.4	75.5	79.6	67.5	64.7	64.6	63.5	62.3	63.1
CEM	Zinc	718.0	93.8	85.1	76.1	63.6	59.3	53.0	52.8	48.6	Fail		
Cement average		769.7	86.2	83.7	80.3	74.1	67.9	65.0	62.8	59.3	49.9	48.1	48.7
CFA	Copper	942.4	98.1	101.4	99.0	96.7	98.1	97.2	93.1	95.5	95.5	92.6	95.8
CFA	Grease	1,006.7	93.0	92.9	96.6	93.3	96.4	96.0	92.4	99.2	100.5	90.0	94.3
CFA	HCB	1,004.7	93.9	94.9	97.3	96.3	93.8	96.3	96.0	93.3	95.4	87.8	88.3
CFA	Lead	906.5	93.2	97.7	95.9	93.9	98.5	96.2	90.4	93.6	94.8	93.1	93.6
CFA	NaOH	1,002.1	95.2	99.4	96.8	91.5	96.3	94.5	91.7	94.3	94.0	93.0	93.2
CFA	Oil	1,006.6	95.1	97.3	97.0	85.4	97.1	96.[illegible]	[illegible].3	96.2	95.6	92.8	97.3
CFA	Phenol	979.1	94.4	99.2	98.0	93.9	96.5	96.6	92.0	94.0	93.9	92.0	92.4
CFA	Sulfate	902.1	95.2	101.8	96.4	92.4	95.3	95.0	90.3	92.8	92.9	88.9	92.3
CFA	TCE	1,025.1	95.9	97.8	96.2	94.6	95.1	93.0	90.7	95.6	94.0	90.0	93.4
CFA	Zinc	945.4	99.0	101.9	101.0	96.9	99.7	100.0	96.5	98.8	97.1	94.6	96.5
CFA average		972.1	95.3	98.4	97.4	93.5	96.7	96.2	92.5	95.3	95.4	91.5	93.7
LFA	Copper	1,111.8	93.4	96.6	94.3	88.0	92.3	89.4	84.8	82.3	53.1	Fail	
LFA	Grease	1,046.0	96.3	93.1	94.9	89.8	91.6	89.1	87.5	89.6	86.9	81.2	80.9
LFA	HCB	968.6	97.4	100.8	100.2	98.9	98.5	100.4	93.1	99.9	Fail		
LFA	Lead	998.2	93.4	96.5	94.1	79.3	83.9	72.5	65.0	66.9	67.8	63.2	65.9
LFA	NaOH	1,124.7	93.2	96.0	95.0	91.2	96.6	91.1	90.3	91.7	90.8	86.9	89.3
LFA	Oil	1,082.2	98.5	102.0	101.7	95.8	95.0	97.0	88.0	92.1	90.7	88.3	89.5
LFA	Phenol	1,066.6	93.9	98.3	97.5	93.3	97.5	89.7	79.6	81.3	73.9	70.7	72.7
LFA	Sulfate	1,050.1	94.8	94.9	87.2	76.4	77.4	Fail					
LFA	TCE	1,058.5	95.1	98.9	99.0	95.5	93.4	93.5	83.1	81.1	82.1	73.7	Fail
LFA	Zinc	1,050.0	90.4	86.0	86.5	79.2	79.2	70.8	Fail				
LFA average		1,055.7	94.6	96.3	95.0	88.7	90.5	79.3	67.1	68.5	54.5	46.4	39.8

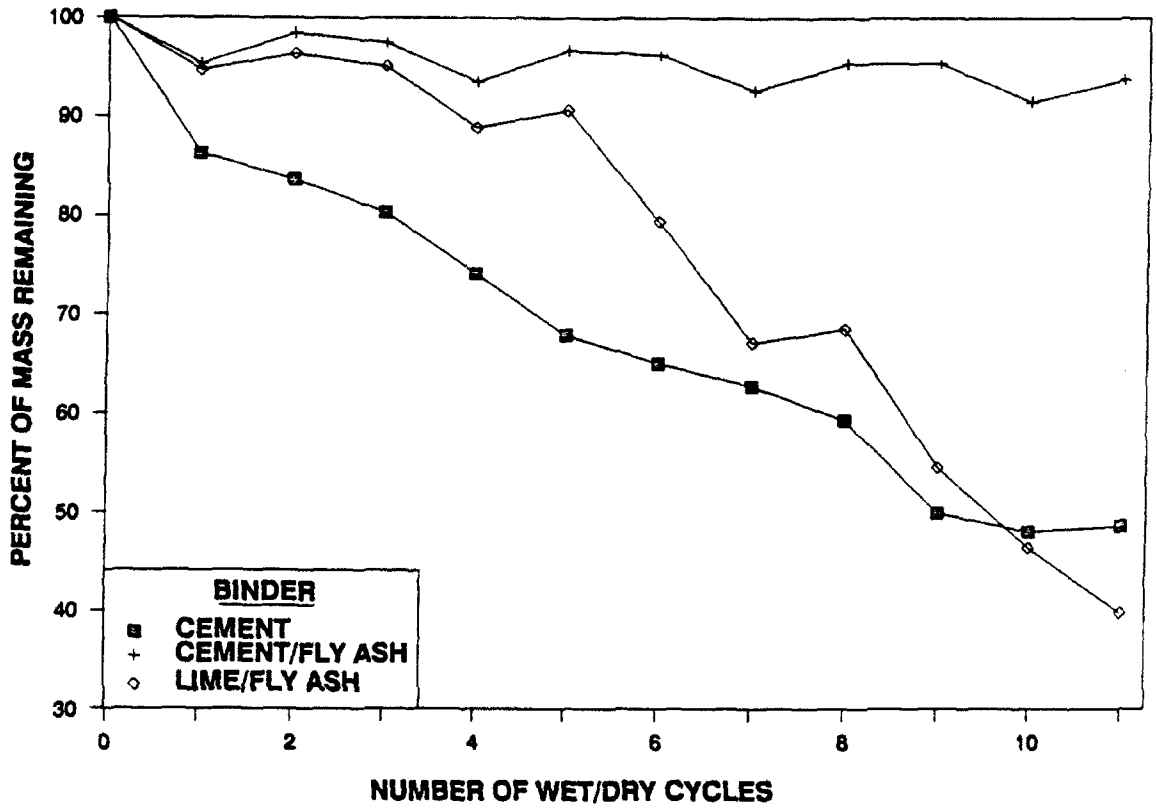

Figure 47. Average wet dry data for the CEM, CFA, and LFA

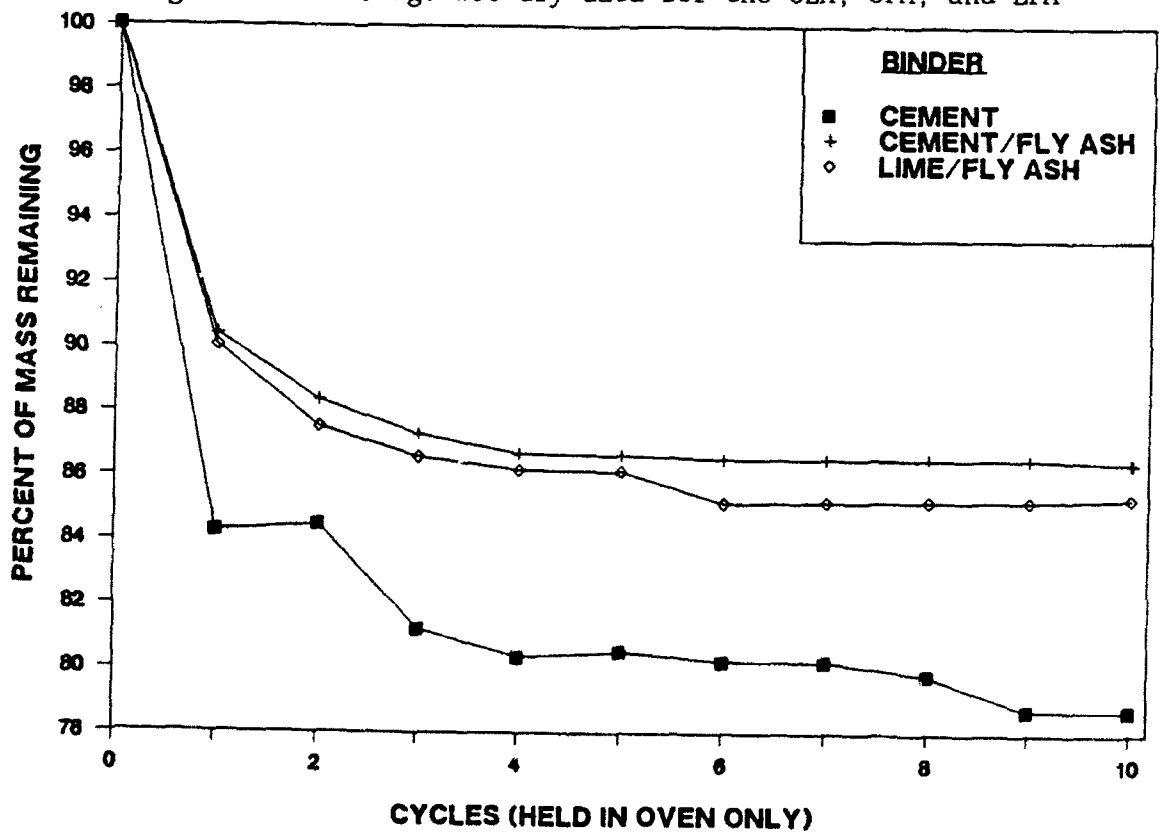

Figure 48. Average wet/dry data for the CEM, CFA, and LFA solidified/ stabilized control samples exposed to only oven drying.

TABLE 30. AVERAGE PERCENT OF MASS REMAINING FOR CONTROL SAMPLES LEFT ONLY IN OVEN

Binder	Interferent	Original weight (grams)	Cycle 1	2	3	4	5	6	7	8	9	10	11
CEM	Copper	669	87.3	94.9	87.5	87.2	87.4	87.6	87.7	87.7	85.0	85.0	85.4
CEM	Grease	758	86.6	86.2	86.1	85.4	86.0	86.8	86.6	86.6	86.8	86.7	87.0
CEM	HCB	739	84.9	88.1	82.8	83.0	83.2	83.2	82.9	82.9	83.2	82.9	82.7
CEM	Lead	1,174	62.4	54.9	54.0	54.0	52.6	49.3	48.1	47.8	47.7	47.8	47.8
CEM	NaOH	720	84.6	84.4	84.4	84.4	84.6	84.7	84.7	84.7	84.7	85.0	85.0
CEM	Oil	753	87.2	87.2	87.2	86.0	85.9	85.8	86.0	86.0	86.0	85.3	85.3
CEM	Phenol	721	87.5	87.7	86.2	84.4	84.6	84.8	86.6	85.9	85.5	85.9	86.0
CEM	Sulfate	688	88.0	88.1	88.7	88.6	88.8	89.1	89.0	89.5	89.6	89.4	89.3
CEM	TCE	720	87.8	87.5	87.9	87.7	89.5	88.3	88.0	85.5	85.8	86.0	86.0
CEM	Zinc	773	86.2	85.8	67.3	62.6	62.6	62.5	62.5	61.0	52.9	53.0	52.4
Cement average		772	84.3	84.5	81.2	80.3	80.5	80.2	80.2	79.8	78.7	78.7	78.7
CFA	Copper	932	93.1	91.7	91.1	91.2	91.1	90.9	91.0	90.8	90.8	91.0	90.9
CFA	Grease	1,022	90.0	88.9	88.2	88.2	88.5	88.0	88.0	88.0	87.9	87.7	87.6
CFA	HCB	1,026	88.3	84.0	82.4	81.1	80.9	81.1	80.7	80.6	81.1	80.6	80.6
CFA	Lead	936	88.8	87.1	86.0	86.0	86.1	86.2	86.1	86.1	86.6	86.6	86.8
CFA	NaOH	995	90.4	88.6	87.7	87.3	87.1	86.9	87.0	87.2	87.0	86.8	86.6
CFA	Oil	1,019	90.2	88.4	87.9	87.2	86.9	87.4	87.3	87.4	87.4	87.4	88.0
CFA	Phenol	982	89.4	86.5	84.6	83.0	82.8	82.6	82.5	82.3	82.2	82.1	82.2
CFA	Sulfate	923	90.8	90.1	88.8	88.5	88.6	88.6	88.6	88.5	88.5	88.5	88.5
CFA	TCE	1,026	89.3	86.2	84.3	82.8	82.7	82.3	82.6	82.9	82.7	82.3	82.2
CFA	Zinc	942	93.6	92.3	91.8	91.7	91.6	91.4	91.7	91.6	91.3	91.5	91.3
CFA average		980	90.4	88.4	87.3	86.7	86.6	86.5	86.5	86.5	86.5	86.4	86.5
LFA	Copper	1,131	87.5	84.8	83.2	82.4	82.2	82.1	82.1	82.1	82.0	82.1	82.1
LFA	Grease	1,048	92.8	90.5	89.5	89.2	89.2	89.2	89.2	89.1	89.2	89.1	89.3
LFA	HCB	996	89.0	86.8	85.8	85.8	85.7	85.5	85.6	85.7	85.5	85.9	85.8
LFA	Lead	998	88.4	85.4	85.3	85.3	85.1	85.2	85.2	85.4	85.3	85.5	85.5
LFA	NaOH	1,116	90.5	85.9	84.1	83.0	83.1	83.0	83.1	83.2	83.2	83.4	83.2
LFA	Oil	1,061	93.6	90.9	89.5	88.8	88.8	88.7	88.7	88.6	88.5	88.6	88.7
LFA	Phenol	1,070	89.0	87.6	86.8	86.6	86.7	76.8	76.9	76.8	76.8	77.1	77.0
LFA	Sulfate	1,048	89.2	88.4	87.8	87.5	87.6	87.7	87.8	87.8	87.9	88.0	87.9
LFA	TCE	1,064	89.6	87.0	85.9	85.5	85.1	85.4	85.5	85.5	85.7	85.5	85.7
LFA	Zinc	1,066	90.7	88.0	87.8	87.9	87.9	87.9	87.9	88.0	88.1	88.1	88.2
LFA average		1,060	90.0	87.5	86.6	86.2	86.1	85.2	85.2	85.2	85.2	85.3	85.3

TABLE 31. AVERAGE PERCENT OF MASS REMAINING AFTER WET/DRY CYCLING FOR CEMENT BINDER SAMPLES

Inter-ferent	Conc. (%)	Original weight	Cycle 1	2	3	4	5	6	7	8	9	10	11
Copper	0	657.0	96.9	95.0	92.0	87.0	84.5	80.0	75.4	76.0	74.6	71.6	74.5
Copper	2	744.5	91.3	93.0	90.9	84.4	83.3	79.8	70.8	71.1	69.2	64.6	59.3
Copper	5	899.3	83.8	73.7	28.9	Fail							
Copper	8	904.8	59.1	Fail									
Lead	0	1,183.8	59.5	46.6	45.8	42.7	40.9	34.7	32.5	Fail			
Lead	2	1,151.6	59.5	53.5	52.1	52.3	49.9	49.9	47.8	44.9	45.2	45.1	44.8
Lead	5	1,158.0	61.1	47.1	46.3	42.0	38.4	36.4	34.5	16.7	16.6	16.6	16.2
Lead	8	1,270.7	63.2	42.6	18.7	15.3	14.7	Fail					
Zinc	0	718.0	93.8	85.1	76.1	63.5	59.3	53.0	52.8	48.6	Fail		
Zinc	2	671.1	81.9	77.7	74.1	30.6	Fail						
Zinc	5	719.4	44.4	40.7	45.0	Fail							
Zinc	8	866.2	Fail										
NaOH	0	699.1	83.8	85.9	83.4	78.5	80.5	80.8	78.1	78.3	78.2	76.0	78.8
NaOH	2	764.7	78.8	74.5	71.6	42.6	21.3	21.5	Fail				
NaOH	5	876.8	75.5	35.7	34.7	23.5	18.4	Fail					
NaOH	8	903.5	76.0	71.2	70.4	35.7	35.7	35.8	35.8	35.6	35.0	34.1	25.8
Sulfate	0	689.7	91.6	78.0	73.4	65.5	Fail						
Sulfate	2	786.4	87.2	87.3	89.2	80.7	45.6	46.3	43.3	44.2	42.4	40.9	41.5
Sulfate	5	923.9	86.5	84.3	85.3	80.5	82.5	82.1	76.8	77.5	75.1	38.1	38.0
Sulfate	8	1,066.6	Fail										
Grease	0	781.0	84.8	85.8	82.0	80.1	82.5	82.3	81.9	77.1	36.2	36.0	36.2
Grease	2	768.0	88.4	89.5	87.1	83.7	85.1	44.4	43.7	44.9	43.9	42.5	44.0
Grease	5	771.4	87.5	85.1	81.8	78.3	76.3	76.5	74.8	74.0	73.3	72.4	71.5
Grease	8	751.0	88.6	88.8	42.2	41.3	Fail						
Oil	0	800.0	85.7	88.7	88.6	83.5	86.0	85.9	80.2	82.2	80.3	76.2	73.6
Oil	2	739.5	88.4	89.9	90.0	86.1	85.0	83.7	82.0	38.9	36.2	36.0	Fail
Oil	5	733.2	84.0	84.1	80.8	80.0	80.1	79.8	Fail				
Oil	8	708.0	89.0	46.2	Fail								
TCE	0	702.0	91.1	91.0	88.4	75.5	79.6	67.5	64.7	64.6	63.5	62.3	63.1
TCE	2	697.4	86.9	87.3	84.1	40.1	43.6	40.8	38.9	39.2	39.1	38.0	38.5
TCE	5	677.6	90.5	88.6	87.8	83.3	87.3	81.1	79.4	42.4	41.0	40.5	41.2
TCE	8	672.8	91.4	91.6	92.2	86.8	93.2	84.7	80.7	41.7	38.6	38.4	38.6
HCB	0	732.2	88.7	90.9	83.6	80.7	79.1	78.9	77.3	77.6	77.7	76.5	77.5
HCB	2	717.7	90.9	91.8	89.4	83.7	42.0	40.2	37.7	24.4	23.8	21.7	21.6
HCB	5	723.4	91.6	90.4	86.2	81.6	80.4	80.3	76.4	76.1	74.9	73.9	72.9
HCB	8	772.7	91.1	88.4	88.6	84.6	86.5	86.3	84.3	84.5	83.2	81.2	81.6
Phenol	0	734.6	86.6	89.6	89.8	83.4	86.9	87.0	84.7	88.7	88.7	81.8	84.8
Phenol	2	753.4	79.9	73.2	70.1	Fail							
Phenol	5	831.1	66.4	19.3	Fail								
Phenol	8	826.5	54.7	Fail									

TABLE 32. AVERAGE PERCENT OF MASS REMAINING AFTER WET/DRY CYCLING FOR CEMENT/FLY ASH BINDER SAMPLES

Inter-ferent	Conc. (%)	Original weight	Cycle 1	2	3	4	5	6	7	8	9	10	11
Copper	0	942.4	98.1	101.4	99.0	96.7	98.1	97.2	93.1	95.5	95.5	92.6	95.8
Copper	2	966.2	97.4	99.5	97.5	95.1	96.1	93.7	89.2	90.5	90.5	43.4	44.6
Copper	5	994.1	Fail										
Copper	8	1,077.1	Fail										
Lead	0	906.5	93.2	97.7	95.9	93.9	98.5	96.2	90.4	93.6	94.8	93.1	93.6
Lead	2	933.9	92.4	96.0	93.7	91.8	95.9	93.8	ND	92.7	91.9	89.8	92.0
Lead	5	1,087.7	83.3	75.0	60.1	37.9	38.9	Fail					
Lead	8	1,154.2	99.7	Fail									
Zinc	0	945.4	99.0	101.9	101.0	96.9	99.7	100.0	96.5	98.8	97.1	94.6	96.5
Zinc	2	887.8	81.5	79.3	77.9	75.0	75.2	74.2	71.7	72.5	70.5	37.3	37.1
Zinc	5	1,016.7	Fail										
Zinc	8	1,017.3	Fail										
Sulfate	0	902.1	95.2	101.8	96.4	92.4	95.3	95.0	90.3	92.8	92.9	88.9	92.3
Sulfate	2	1,022.1	93.7	99.6	94.6	90.5	93.2	92.9	88.4	90.6	90.6	86.6	89.4
Sulfate	5	1,162.4	94.7	98.3	95.2	90.3	91.8	91.2	87.2	88.4	88.4	85.2	87.2
Sulfate	8	1,323.8	90.5	95.0	90.8	86.3	87.6	86.3	81.6	83.1	82.3	78.3	80.4
NaOH	0	1,002.1	95.2	99.4	96.8	91.5	96.3	94.5	91.7	94.3	94.0	93.0	93.2
NaOH	2	1,130.8	94.2	96.6	94.9	90.7	93.8	92.6	90.5	92.0	91.2	90.4	90.0
NaOH	5	1,202.9	94.9	98.0	95.8	91.1	94.2	93.0	90.5	91.9	91.6	90.9	90.7
NaOH	8	1,195.7	94.1	95.9	93.9	89.3	91.6	90.8	88.1	89.0	88.7	87.5	86.9
Grease	0	1,006.7	93.0	92.9	96.6	93.3	96.4	96.0	92.4	99.2	100.5	90.0	94.3
Grease	2	1,004.6	92.8	96.2	95.7	92.7	97.0	95.3	91.6	98.5	102.2	89.8	92.6
Grease	5	987.4	93.7	98.4	97.1	94.0	98.1	96.1	92.5	99.1	102.4	91.1	94.9
Grease	8	986.7	93.1	96.8	95.7	93.2	95.7	94.9	92.0	97.8	98.4	90.7	93.6
Oil	0	1,006.6	95.1	97.3	97.0	85.4	97.1	96.9	92.3	96.2	95.6	92.8	97.3
Oil	2	995.3	92.7	94.5	94.3	90.6	93.5	93.4	89.3	92.4	92.3	89.6	93.2
Oil	5	976.3	91.5	91.8	91.6	89.1	90.1	Fail					
Oil	8	878.9	94.0	90.7	88.4	45.9	45.7	Fail					
TCE	0	1,025.1	95.9	97.8	96.2	94.6	95.1	93.0	90.7	95.6	94.0	90.0	93.4
TCE	2	1,033.1	95.9	97.6	95.4	93.4	94.0	93.1	89.8	95.5	93.1	89.1	92.9
TCE	5	982.6	95.4	97.8	95.9	90.1	95.0	94.1	90.5	95.6	93.8	89.8	93.5
TCE	8	970.9	94.5	97.8	95.5	95.3	95.2	94.4	90.9	93.9	94.5	90.3	94.1
HCB	0	1,004.7	93.9	94.9	97.3	96.3	93.8	96.3	96.0	93.3	95.4	87.8	88.3
HCB	2	1,011.5	94.7	97.3	97.3	93.2	95.3	98.6	92.4	96.1	100.6	91.7	95.1
HCB	8	1,019.2	92.6	96.1	95.2	91.6	94.8	96.6	90.9	92.9	95.0	88.8	92.4
HCB	5	1,015.1	96.8	98.7	99.5	95.1	97.2	100.3	94.6	97.0	99.7	92.9	96.8
Phenol	0	979.1	94.4	99.2	98.0	93.9	96.5	96.8	92.0	94.0	93.9	92.0	92.4
Phenol	2	933.9	94.3	98.0	95.9	90.9	94.8	92.9	87.3	90.3	90.0	84.8	87.7
Phenol	5	896.8	89.6	93.8	91.3	85.9	87.7	88.2	82.7	84.8	84.2	81.5	83.0
Phenol	8	897.4	81.2	79.4	73.5	68.5	69.8	65.3	61.2	61.1	59.2	53.2	52.8

TABLE 33. AVERAGE PERCENT OF MASS REMAINING AFTER WET/DRY CYCLING FOR LIME/FLY ASH BINDER SAMPLES

Interferent	Conc. (%)	Original weight	Cycle 1	2	3	4	5	6	7	8	9	10	11
Copper	0	1,111.8	93.4	96.6	94.3	88.0	92.3	89.4	84.8	82.3	53.1	Fail	
Copper	2	1,172.5	Fail										
Copper	5	1,153.3	87.1	85.4	81.0	37.1	36.0	34.3	32.2	Fail			
Copper	8	1,128.1	35.3	31.2	Fail								
Lead	0	998.2	93.4	96.5	94.1	79.3	83.9	72.5	65.0	66.9	67.8	63.2	65.9
Lead	2	907.5	85.2	85.0	Fail								
Lead	5	981.6	Fail										
Lead	8	1,134.8	Fail										
Zinc	0	1,050.0	90.4	86.0	86.5	79.2	79.2	70.8	Fail	Fail			
Zinc	2	1,122.1	35.0	Fail									
Zinc	5	1,178.2	79.7	Fail									
Zinc	8	1,333.8	72.5	53.2	Fail								
Sulfate	0	1,050.1	94.8	94.9	87.2	76.4	77.4	Fail		90.6			
Sulfate	2	1,235.9	92.2	96.2	92.6	89.0	90.5	90.8	87.3	78.8	91.0	86.8	89.6
Sulfate	5	1,275.2	91.2	93.7	89.4	83.5	84.6	82.7	78.3	39.3	77.2	ND*	72.1
Sulfate	8	1,383.0	88.7	90.9	86.2	81.0	81.6	81.0	73.7		40.3	38.1	39.7
Grease	0	1,046.0	96.3	93.1	94.9	89.8	91.6	89.1	87.5	89.6	86.9	81.2	80.9
Grease	2	1,064.3	94.2	97.6	97.9	92.9	87.7	85.6	81.7	85.4	82.4	77.2	76.8
Grease	5	1,066.0	92.0	93.7	94.6	90.8	88.1	86.2	82.3	83.1	82.7	78.0	78.8
Grease	8	1,021.8	92.3	93.8	93.4	90.0	79.4	34.8	Fail				
NaOH	0	1,124.7	93.2	96.0	95.0	91.2	96.6	91.1	90.3	91.7	90.8	86.9	89.3
NaOH	2	1,218.9	91.2	89.6	89.5	85.3	89.4	85.4	83.3	85.1	84.6	81.3	83.5
NaOH	5	1,201.5	82.1	77.4	70.1	66.1	69.2	67.6	63.5	66.1	64.8	62.6	64.3
NaOH	8	1,144.0	74.5	33.6	Fail								
Oil	0	1,082.2	98.5	102.0	101.7	95.8	95.0	97.0	88.0	92.1	90.7	88.3	89.5
Oil	2	1,018.8	96.8	97.0	94.2	46.1	46.5	47.0	39.2	Fail			
Oil	5	1,003.5	96.2	95.9	94.7	91.0	36.3	Fail					
Oil	8	990.6	94.9	94.9	93.9	92.6	95.0	28.2	Fail				
TCE	0	1,058.5	95.1	98.9	99.0	95.5	93.4	93.5	83.1	81.1	82.1	73.7	Fail
TCE	2	1,054.9	94.9	99.7	97.9	91.0	89.4	90.1	84.3	77.6	76.6	67.8	64.7
TCE	5	1,034.3	96.4	101.3	101.5	98.0	98.2	100.6	95.6	99.6	98.0	94.2	97.3
TCE	8	1,074.7	93.5	97.5	97.7	94.6	95.8	98.3	94.9	98.2	97.5	94.2	98.3
HCB	0	968.6	97.4	100.8	100.2	98.9	98.5	100.4	93.1	99.9	Fail		
HCB	2	999.9	47.8	99.1	98.6	94.4	96.5	94.2	90.4	93.7	89.9	85.6	89.9
HCB	5	1,024.1	95.6	98.1	100.4	96.1	100.1	100.0	97.5	101.1	97.6	95.1	98.9
HCB	8	1,050.8	97.8	98.7	98.7	95.7	100.4	101.2	97.3	101.2	99.2	96.9	100.7
Phenol	0	1,066.6	93.9	98.3	97.5	93.3	97.5	89.7	79.6	81.3	73.9	70.7	72.7
Phenol	2	1,094.3	78.8	Fail									
Phenol	5	1,284.9	78.3	Fail									
Phenol	8	1,333.7	Fail										

* No data.

TABLE 34. AVERAGE NUMBER OF WET/DRY CYCLES WITHOUT FAILURE FOR SAMPLES WITH METAL INTERFERENTS

Interferent	Concentration (%)	Binder CEM	CFA	LFA	Avg
Copper	0			9.0	10.3
Copper	2	11.0	10.0	0.0	7.0
Copper	5	2.5	0.0	5.0	2.5
Copper	8	1.0	0.0	1.0	0.7
Lead	0	7.0	11.0	11.0	9.7
Lead	2	11.0	11.0	2.0	8.0
Lead	5	9.0	4.0	0.0	4.3
Lead	8	3.5	1.0	0.0	1.5
Zinc	0	8.0		6.0	8.3
Zinc	2	3.5	10.0	0.5	4.7
Zinc	5	1.5	0.0	1.0	0.8
Zinc	8	0.0	0.0	1.0	0.3
Average metals	0	8.7	11.0	8.7	9.4
	2	8.5	10.3	0.8	6.6
	5	4.3	1.3	2.0	2.6
	8	1.5	0.3	0.7	0.8

TABLE 35. AVERAGE NUMBER OF WET/DRY CYCLES WITHOUT FAILURE FOR SAMPLES WITH SODIUM HYDROXIDE OR SULFATE INTERFERENTS

Interferent	Concentration (%)	Binder CEM	CFA	LFA	Avg
NaOH	0	11.0	11.0	11.0	11.0
NaOH	2	5.0	11.0	11.0	9.0
NaOH	5	3.0	11.0	11.0	8.3
NaOH	8	7.0	11.0	1.5	6.5
Sulfate	0	4.0	11.0	5.0	6.7
Sulfate	2	7.5	11.0	11.0	9.8
Sulfate	5	10.0	11.0	11.0	10.7
Sulfate	8	0.0	11.0	9.0	6.7

TABLE 36. AVERAGE NUMBER OF WET/DRY CYCLES WITHOUT FAILURE FOR SAMPLES WITH ORGANIC INTERFERENTS

Interferent	Concentration (%)	Binder CEM	CFA	LFA	Avg
Grease	0	11.0	11.0	11.0	11.0
Grease	2	8.0	11.0	11.0	10.0
Grease	5	11.0	11.0	11.0	11.0
Grease	8	3.0	11.0	5.5	6.5
Oil	0	11.0	11.0	11.0	11.0
Oil	2	9.0	11.0	5.0	8.3
Oil	5	6.0	5.0	5.0	5.3
Oil	8	1.5	4.0	5.5	3.7
TCE	0	11.0	11.0	10.0	10.7
TCE	2	7.0	11.0	11.0	9.7
TCE	5	9.0	11.0	11.0	10.3
TCE	8	9.0	11.0	11.0	10.3
HCB	0	11.0	11.0	7.0	9.7
HCB	2	7.5	11.0	11.0	9.8
HCB	5	11.0	11.0	11.0	11.0
HCB	8	11.0	11.0	11.0	11.0
Phenol	0	11.0	11.0	11.0	11.0
Phenol	2	3.0	11.0	1.0	5.0
Phenol	5	2.0	11.0	1.0	4.7
Phenol	8	1.0	11.0	0.0	4.0
Average (excluding phenol)	0	11.0	11.0	9.8	10.6
	2	7.9	11.0	9.5	9.5
	5	9.3	9.5	9.5	9.4
	8	6.1	9.3	8.3	7.9

Figure 49. Illustration of failure in the wet/dry cycling test. The 4-in. cylinders of CFA solidified/stabilized sludge containing 8-percent lead are shown in their sample holders. The sample on the left is the "A" sample, which was kept in the oven; the two "B" samples on the right underwent two wet/dry cycles.

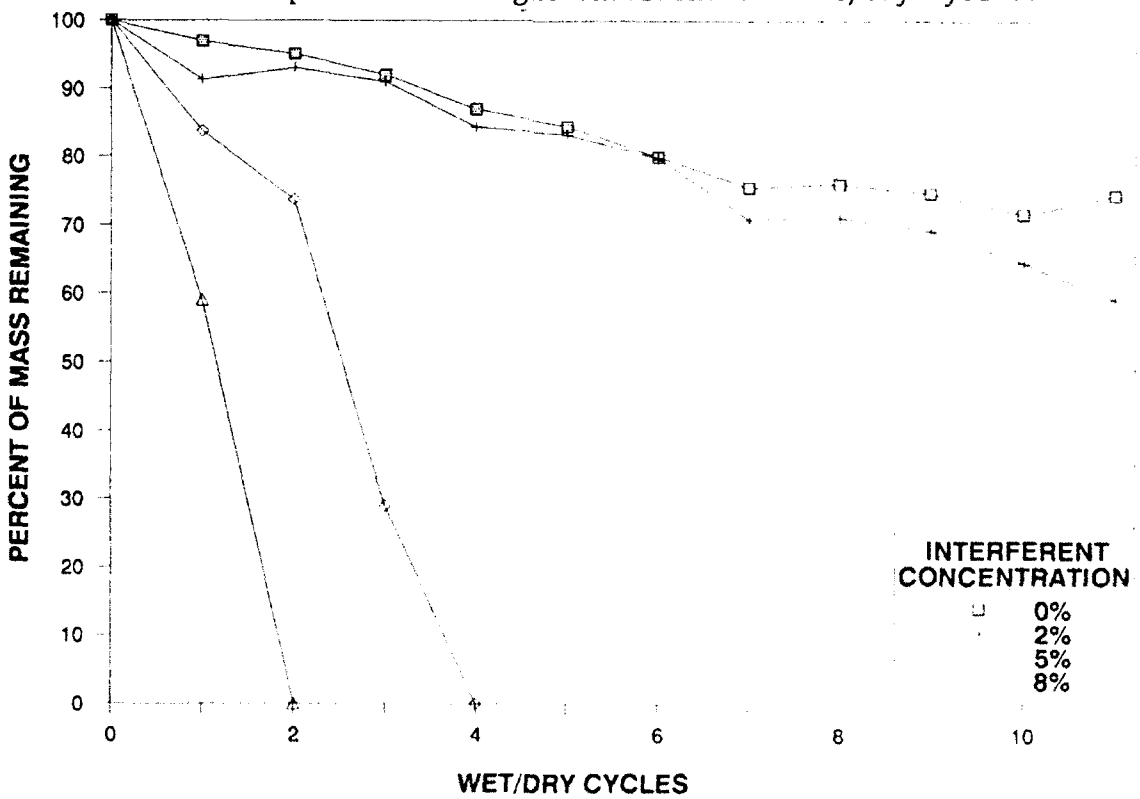

Figure 50. Wet/dry cycling for the CEM solidified/stabilized samples with copper interference.

Strong Base and Sulfate Interferents--

Sodium hydroxide addition had its greatest effect on the durability of the CEM samples, having a pronounced effect at the 2-percent addition. However, only the 8-percent addition rate caused a nearly complete loss of durability in the LFA samples. None of the NaOH addition rates affected the CFA durability appreciably.

Sulfate addition appeared to affect the CEM samples to the greatest extent. However, the variability in the results, including the lower durability of the control in this batch, makes an evaluation difficult. Sulfate had little effect on the CFA or LFA sample durability at any addition rate.

Organic Interferents--

The nonpolar organic interferents had much less effect on the wet/dry durability of the solidified/stabilized sludge. TCE and HCB had no effect on any of the binder systems at any concentration (See Figure 51), except for CEM with added TCE, which lasted only an average of nine cycles at all concentrations. This is shown in Figure 52 for CFA samples with 8-percent HCB in the 11th wet/dry cycle. Note that there is very little degradation of any of the specimens. Grease had a noticeable effect on the CEM and LFA specimens, but only at an 8-percent addition rate (Figure 53). Oil lowered the durability of all three binder systems, the effects becoming apparent at the 2-percent addition in the CEM (Figure 54) and LFA samples, and at 5 percent in the CFA samples. Figure 55 shows there was only slight degradation of the CFA specimens with 2-percent oil after 11 cycles.

Phenol, the most polar and water-soluble of the organics, produced the greatest effect on the CEM and LFA samples at all concentrations and had lesser but measurable effects on the CFA sample. This is shown in Figure 56 for CEM specimens with 8-percent phenol after a single wet/dry cycle. Note the general degradation of the samples that underwent the wet/dry treatment. Even the specimen subjected only to oven storage indicated some breakdown after 48 hr.

On the average, the organic interferents produced the greatest effect on the CEM and LFA specimens, but had only minor effects on the CFA specimens. Since the CEM was designed to have only marginal strength (50 to 100 psi UCS, averaging 112 psi), the effects of interferents were expected to be more pronounced than upon the CFA, which had much higher UCS (controls averaging 624 psi). The higher susceptibility of LFA (which also had higher UCS than the CEM, averaging 591 psi) may be due either to its slower rate of strength development or to its greater susceptibility to the addition of the organic interferents.

Effects of 60° C Oven Treatment

Some of the interferent materials affected the specimens that were not subjected to the immersion cycle but were left in the 60° C oven and weighed after each cycle. These data are presented in Tables 37-39.

The most pronounced effect was caused by the NaOH interferent, which caused failure of two of the three specimens for all three binder systems. No explanation is known for this effect of NaOH. The metal interferants also

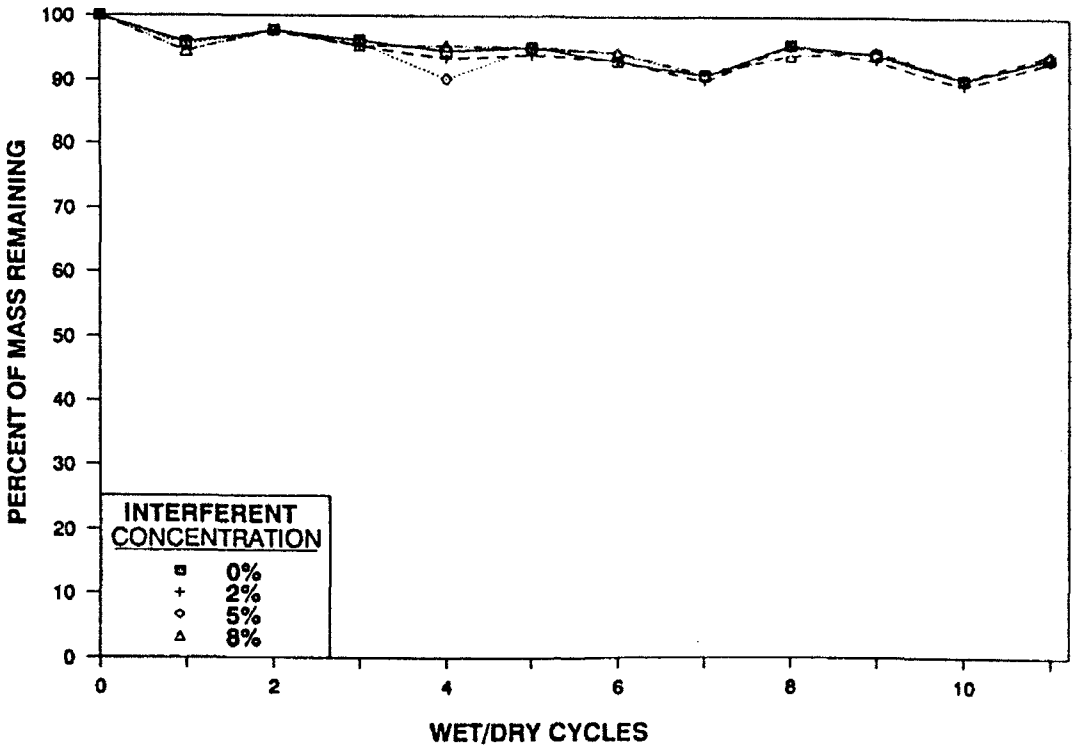

Figure 51. Wet/dry cycling for the CFA solidified/stabilized samples with TCE interference.

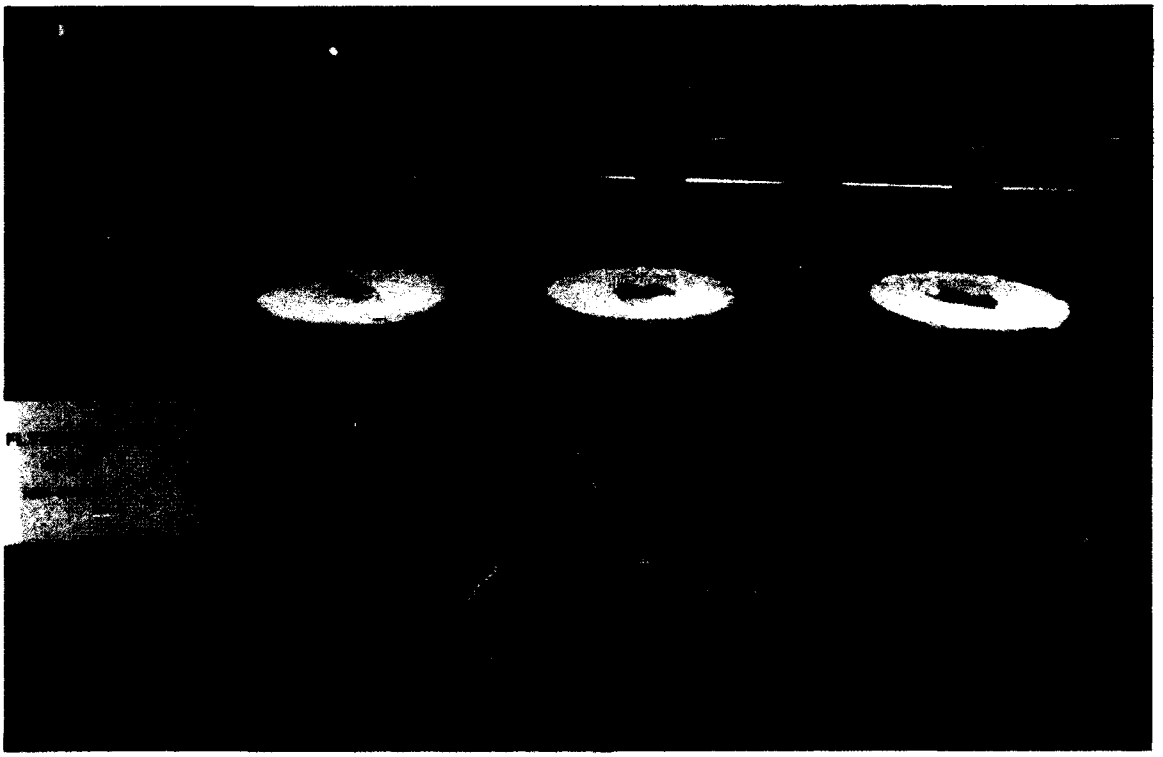

Figure 52. Successful completion of the wet/dry test. These cylinders of CFA solidified/stabilized sludge containing 8 percent HCB have undergone 11 wet/dry cycles. These show no indication of degradation compared with the sample on left, which was exposed to only oven drying.

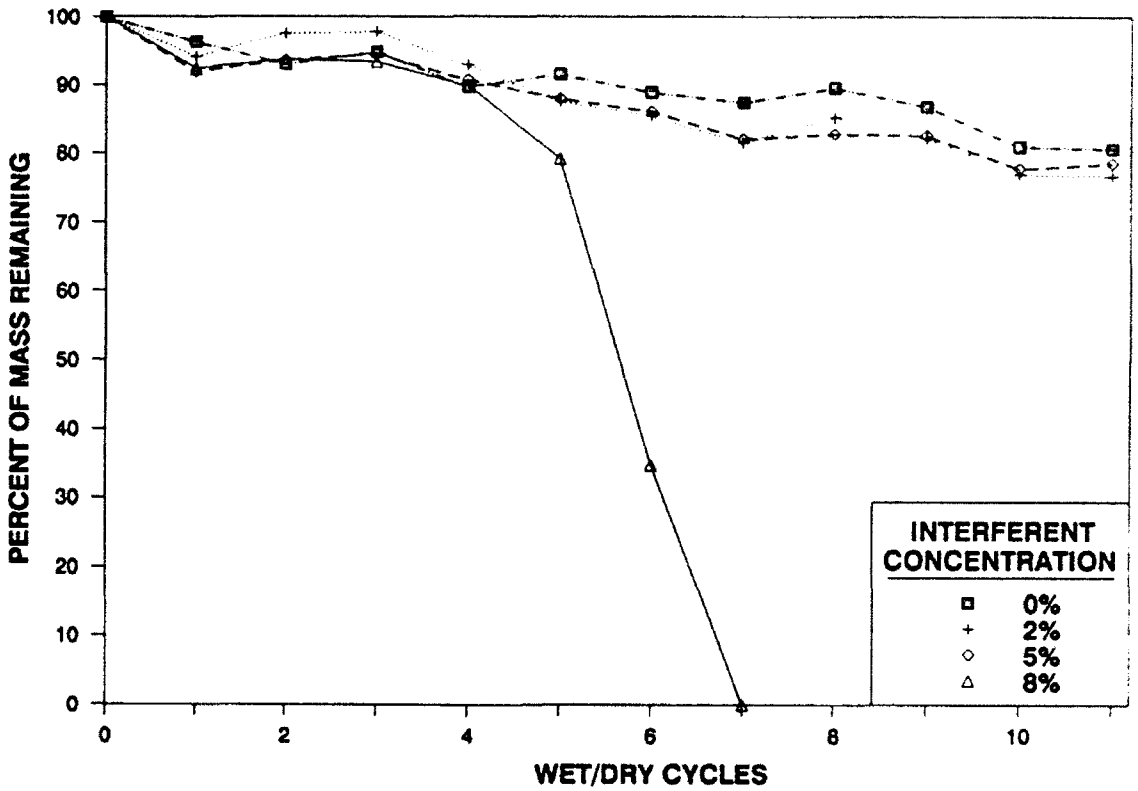

Figure 53. Wet/dry cycling for the LFA solidified/stabilized samples with grease interference.

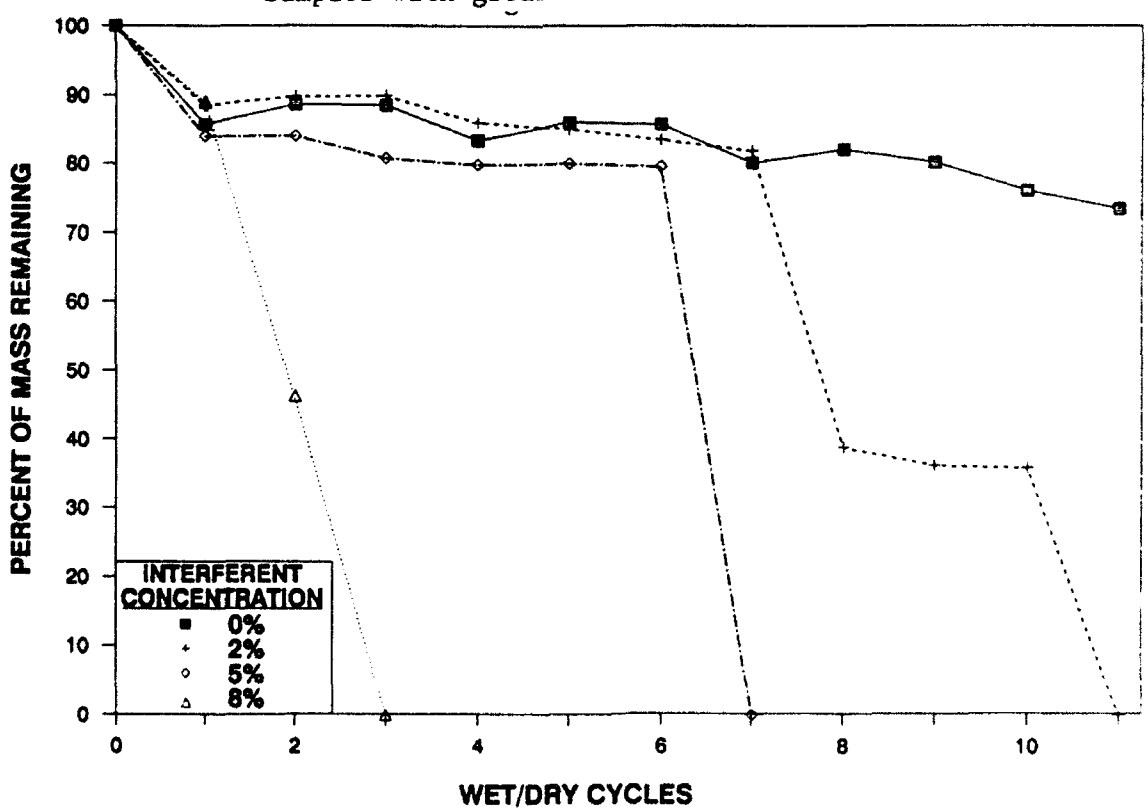

Figure 54. Wet/dry cycling for the CEM solidified/stabilized samples with oil interference.

Figure 55. CFA solidified/stabilized sludge with 2 percent added oil after 11 wet/dry cycles. Note the beginning of degradation for the immersed specimens (center and right).

Figure 56. CEM solidified/stabilized sludge with 8 percent added phenol after the first wet/dry cycle. The samples that have been immersed (center and right) show advanced degradation; samples exposed to oven drying only show some damage.

TABLE 37. PERCENT OF MASS REMAINING FOR CEMENT SAMPLES KEPT ONLY IN OVEN

Inter-ferent	Conc. (%)	Cycle 1	2	3	4	5	6	7	8	9	10	11
Copper	0	87.3	94.9	87.5	87.2	87.4	87.6	87.7	87.7	85.0	85.0	85.4
Copper	2	90.6	88.9	84.1	83.5	83.6	68.2	67.2	59.0	Fail		
Copper	5	Fail										
Copper	8	84.3	81.3	79.9	80.0	79.6	80.9	79.6	79.6	80.5	79.5	80.6
Lead	0	62.4	54.9	54.0	54.0	52.6	49.3	48.1	47.8	47.7	47.8	47.8
Lead	2	58.7	53.1	52.3	52.4	51.6	51.6	48.6	48.3	48.2	48.3	48.0
Lead	5	58.5	52.3	49.0	49.0	46.0	44.0	43.5	42.4	42.4	42.4	42.3
Lead	8	62.8	52.0	47.0	45.1	44.8	42.9	41.8	40.8	Fail		
Zinc	0	86.2	85.8	67.3	62.6	62.6	62.5	62.5	61.0	52.9	53.0	52.4
Zinc	2	89.2	87.8	87.6	87.4	87.5	88.0	86.7	80.9	87.9	88.2	88.2
Zinc	5	85.9	85.4	85.3	85.3	84.8	85.5	84.9	84.9	85.0	85.4	85.2
Zinc	8	82.5	81.9	81.8	81.6	81.6	79.4	79.0	79.0	79.0	79.5	79.2
Sulfate	0	88.0	88.1	88.7	88.6	88.8	89.1	89.0	89.5	89.6	89.4	89.3
Sulfate	2	87.0	86.4	86.6	86.5	86.5	86.6	86.4	86.7	86.8	86.6	86.5
Sulfate	5	106.3	98.3	96.6	95.6	94.8	94.7	94.0	93.0	92.8	92.5	92.0
Sulfate	8	78.6	72.3	71.7	70.7	70.3	70.0	69.4	69.3	69.3	69.1	68.7
NaOH	0	84.6	84.4	84.4	84.4	84.6	84.7	84.7	84.7	84.7	85.0	85.0
NaOH	2	79.8	71.6	68.0	Fail							
NaOH	5	79.9	69.1	64.1	Fail							
NaOH	8	83.2	82.0	81.8	82.1	82.4	82.1	82.2	82.0	82.0	82.1	82.0
Grease	0	86.6	86.2	86.1	85.4	86.0	86.8	86.6	86.6	86.8	86.7	87
Grease	2	87.2	86.9	86.8	86.4	86.9	87.3	87.2	81.7	81.7	81.7	82
Grease	5	88.5	88.3	87.8	87.5	88.0	88.3	88.3	80.2	80.3	74.3	74.5
Grease	8	88.3	88.2	88.2	87.9	88.5	88.8	88.7	88.8	88.8	88.9	89
Oil	0	87.2	87.2	87.2	86.0	85.9	85.8	86.0	86.0	86.0	85.3	85.3
Oil	2	87.0	86.9	86.9	84.0	84.1	83.8	84.0	79.0	72.2	Fail	
Oil	5	88.1	88.2	88.3	88.2	88.2	88.1	88.3	88.2	81.1	75.8	76.4
Oil	8	90.8	91.0	91.1	90.7	89.3	89.5	89.7	89.4	89.3	88.6	89.3
TCE	0	87.8	87.5	87.9	87.7	89.5	88.3	88.0	85.5	85.8	86.0	86.0
TCE	2	86.3	86.0	86.3	86.1	87.8	86.6	86.4	86.5	86.8	86.9	86.9
TCE	5	91.7	91.5	91.9	91.6	93.5	88.5	88.2	88.3	87.4	87.5	87.6
TCE	8	91.1	90.9	90.9	90.7	92.5	91.4	91.1	91.2	91.5	91.6	91.7
HCB	0	84.9	88.1	82.8	83.0	83.2	83.2	82.9	82.9	83.2	82.9	82.7
HCB	2	86.8	86.2	86.3	86.7	86.9	86.9	86.6	86.6	85.9	79.1	78.9
HCB	5	87.0	86.1	86.1	86.5	86.6	86.6	86.2	86.2	85.7	86.2	85.9
HCB	8	89.6	88.8	88.8	89.2	89.3	89.3	88.9	88.9	87.9	88.8	88.6
Phenol	0	87.5	87.7	86.2	84.4	84.6	84.8	86.6	85.9	85.5	85.9	86.0
Phenol	2	85.8	85.7	85.3	85.1	85.3	85.5	87.2	86.6	86.1	86.5	86.5
Phenol	5	75.4	73.9	73.3	72.8	72.6	72.5	73.9	73.2	74.5	74.7	73.0
Phenol	8	78.4	76.9	76.3	68.0	73.8	72.7	74.0	73.3	71.1	71.4	73.0

TABLE 38. PERCENT OF MASS REMAINING FOR CEMENT/FLY ASH SAMPLES KEPT ONLY IN OVEN

Inter-ferent	Conc. (%)	Cycle 1	2	3	4	5	6	7	8	9	10	11
Copper	0	93.1	91.7	91.1	91.2	91.1	90.9	91.0	90.8	90.8	91.0	90.9
Copper	2	94.3	93.5	92.9	93.0	92.9	92.6	92.7	92.5	92.5	92.7	92.6
Copper	5	90.1	88.1	87.3	87.5	87.3	87.1	87.1	86.9	86.9	87.2	87.0
Copper	8	90.4	87.1	84.3	83.8	83.6	83.3	83.3	83.1	83.0	83.2	83.2
Lead	0	88.8	87.1	86.0	86.0	86.1	86.2	86.1	86.1	86.6	86.6	86.8
Lead	2	90.8	90.8	90.3	90.4	90.5	90.6	90.6	90.6	91.1	91.2	91.4
Lead	5	85.5	85.3	75.3	85.1	85.1	85.3	85.3	85.3	85.8	85.8	86.0
Lead	8	84.7	83.6	83.2	83.2	82.8	82.9	82.9	82.9	83.3	83.3	83.6
Zinc	0	93.6	92.3	91.8	91.7	91.6	91.4	91.7	91.6	91.3	91.5	91.3
Zinc	2	95.6	95.5	95.4	95.4	95.3	95.1	95.5	95.3	95.1	95.2	95.1
Zinc	5	91.1	90.7	90.6	90.5	90.6	90.2	90.8	90.5	90.2	90.4	90.3
Zinc	8	90.0	89.3	89.0	89.0	89.0	88.6	89.2	88.9	88.5	88.7	88.5
Sulfate	0	90.8	90.1	88.8	88.5	88.6	88.6	88.6	88.5	88.5	88.5	88.5
Sulfate	2	90.2	88.7	86.4	85.0	84.8	84.7	84.6	84.4	84.2	84.2	84.2
Sulfate	5	90.7	88.4	84.9	81.6	80.3	79.4	78.2	77.7	77.3	76.9	76.8
Sulfate	8	89.7	86.5	81.9	77.6	75.8	74.6	73.2	72.5	72.1	71.8	71.7
NaOH	0	90.4	88.6	87.7	87.3	87.1	86.9	87.0	87.2	87.0	86.8	86.6
NaOH	2	88.4	84.9	82.1	78.9	77.7	76.8	76.5	76.5	Fail		
NaOH	5	88.2	84.6	81.8	78.5	77.1	75.9	75.3	75.1	74.9	74.7	74.5
NaOH	8	88.7	92.2	82.1	78.6	77.7	77.0	77.0	77.1	Fail		
Grease	0	90.0	88.9	88.2	88.2	88.5	88.0	88.0	88.0	87.9	87.7	87.6
Grease	2	89.8	88.5	87.7	87.7	87.8	87.3	87.3	87.2	87.1	86.9	86.8
Grease	5	90.8	89.6	88.6	88.9	89.0	88.5	88.6	88.5	88.4	88.2	88.1
Grease	8	90.9	89.7	89.1	89.4	89.5	89.2	89.2	89.2	89.1	88.9	88.8
Oil	0	90.2	88.4	87.9	87.2	86.9	87.4	87.3	87.4	87.4	87.4	88.0
Oil	2	39.9	88.1	87.8	87.5	87.7	87.8	87.9	88.0	88.0	88.3	88.6
Oil	5	89.6	89.5	89.5	89.3	89.6	89.8	89.7	89.9	89.8	90.2	90.5
Oil	8	93.2	93.1	93.1	93.0	93.2	93.3	93.3	93.5	93.4	93.8	94.1
TCE	0	89.3	86.2	84.3	82.8	82.7	82.3	82.6	82.9	82.7	82.3	82.2
TCE	2	89.9	86.4	84.2	82.6	82.3	81.9	82.2	82.5	82.3	81.8	81.7
TCE	5	89.4	86.2	84.3	83.0	82.9	82.5	82.7	83.1	82.9	82.3	82.3
TCE	8	89.9	86.9	96.9	84.1	84.1	83.8	84.1	84.4	84.2	83.7	83.7
HCB	0	88.3	84.0	82.4	81.1	80.9	81.1	80.7	80.6	81.1	80.6	80.6
HCB	2	89.0	85.4	84.0	82.9	82.9	83.1	82.6	82.6	83.1	82.5	82.5
HCB	5	88.7	84.8	83.6	82.6	82.6	82.7	82.4	82.3	82.8	82.2	82.2
HCB	8	90.0	86.8	85.7	84.7	84.7	84.9	84.3	84.2	84.8	84.3	84.2
Phenol	0	89.4	86.5	84.6	83.0	82.8	82.6	82.5	82.3	82.2	82.1	82.2
Phenol	2	89.2	87.0	85.7	85.0	84.9	84.7	84.8	84.6	84.5	84.5	84.6
Phenol	5	86.9	85.4	84.7	84.2	84.2	84.1	84.1	83.9	83.9	83.8	83.9
Phenol	8	86.7	85.9	85.4	84.8	84.9	84.8	84.8	84.4	84.4	83.8	84.3

TABLE 39. PERCENT OF MASS REMAINING FOR LIME/FLY ASH SAMPLES KEPT ONLY IN OVEN

Inter-ferent	Conc. (%)	Cycle 1	2	3	4	5	6	7	8	9	10	11
Copper	0	87.5	84.8	83.2	82.4	82.2	82.1	82.1	82.1	82.0	82.1	82.1
Copper	2	89.0	86.1	84.6	83.4	83.0	82.8	82.6	82.5	82.4	82.4	82.3
Copper	5	90.3	89.6	89.5	89.6	89.5	89.5	89.6	89.5	89.4	89.6	89.4
Copper	8	92.4	92.1	92.1	92.1	92.0	91.9	92.0	91.9	91.8	91.9	91.9
Lead	0	88.4	85.4	85.3	85.3	85.1	85.2	85.2	85.4	85.3	85.5	85.5
Lead	2	94.8	94.5	94.5	94.5	94.3	94.4	94.3	94.4	94.4	94.6	94.6
Lead	5	90.7	88.9	88.6	88.5	88.4	88.4	88.3	88.4	88.1	88.2	88.2
lead	8	89.6	85.8	84.0	82.8	Fail						
Zinc	0	90.7	88.0	87.8	87.9	87.9	87.9	87.9	88.0	88.1	88.1	88.2
Zinc	2	86.3	84.0	83.1	82.9	82.9	82.9	82.9	82.9	83.0	83.0	83.0
Zinc	5	68.2	65.3	63.9	63.2	Fail						
Zinc	8	83.8	76.1	66.8	65.2	64.6	63.9	63.6	63.5	63.5	63.4	63.4
Sulfate	0	89.2	88.4	87.8	87.5	87.6	87.7	87.8	87.8	87.9	88.0	87.9
Sulfate	2	89.7	87.3	84.4	81.4	79.9	79.0	78.0	77.6	77.5	77.4	77.3
Sulfate	5	90.2	87.9	84.8	81.0	79.6	78.7	77.8	77.4	75.6	Fail	
Sulfate	8	88.9	86.4	82.3	78.5	78.1	77.7	77.8	77.8	77.9	77.9	77.9
NaOH	0	90.5	85.9	84.1	83.0	83.1	83.0	83.1	83.2	83.2	83.4	83.2
NaOH	2	90.0	86.2	83.6	80.0	78.8	77.8	77.4	77.0	77.0	77.0	76.7
NaOH	5	75.5	70.8	66.2	66.1	64.8	Fail					
NaOH	8	81.0	79.4	Fail								
Grease	0	92.8	90.5	89.5	89.2	89.2	89.2	89.2	89.1	89.2	89.1	89.3
Grease	2	90.2	88.0	86.4	85.9	85.9	85.9	85.9	85.8	85.9	85.8	86.0
Grease	5	91.3	89.2	87.7	86.7	86.6	86.7	86.6	86.6	86.7	86.6	86.7
Grease	8	90.9	88.8	87.2	86.4	86.4	86.4	86.4	86.4	86.5	86.4	86.5
Oil	0	93.6	90.9	89.5	88.8	88.8	88.7	88.7	88.6	88.5	88.6	88.7
Oil	2	90.8	88.6	88.0	87.0	81.9	81.9	81.9	81.8	81.8	81.9	82.0
Oil	5	90.7	89.1	88.0	79.0	79.0	79.1	79.1	76.7	76.7	76.9	76.9
Oil	8	90.5	89.1	88.7	87.1	56.8	56.8	56.8	56.7	53.1	53.2	53.2
TCE	0	89.6	87.0	85.9	85.5	85.1	85.4	85.5	85.5	85.7	85.5	85.7
TCE	2	89.8	87.7	86.6	86.2	85.8	86.1	86.2	86.2	86 4	86.2	86.4
TCE	5	91.8	89.7	88.6	88.1	87.8	88.0	88.1	88.1	88.3	88.1	88.2
TCE	8	89.8	86.8	85.7	76.3	85.3	85.5	85.6	85.6	85.9	85.6	85.8
HCB	0	89.0	86.8	85.8	85.8	85.7	85.5	85.6	85.7	85.5	85.9	85.8
HCB	2	89.8	87.5	86.5	86.3	86.1	86.2	86.2	86.3	91.9	86.3	86.3
HCB	5	91.1	89.0	87.8	87.5	87.3	87.3	87.4	87.4	87.2	87.5	87.5
HCB	8	91.7	89.7	88.7	88.6	88.4	88.4	88.5	88.5	88.3	88.6	88.6
Phenol	0	89.0	87.6	86.8	86.6	86.7	76.8	76.9	76.8	76.8	77.1	77.0
Phenol	2	86.0	84.6	84.1	Fail							
Phenol	5	87.6	81.1	76.1	71.9	71.4	65.9	65.8	Fail			
Phenol	8	91.2	86.1	81.7	74.1	70.4	69.0	67.9	Fail			

caused some failures in the CEM and LFA samples (copper and lead in the CEM specimens, and zinc and lead in the LFA specimens). All specimens of LFA containing phenol also failed. Otherwise, just heating the samples to 60° C did not have an appreciable effect.

Combined Effects of Interferent
Addition on the Three Binder Systems

The mean values for the mass remaining for each of the binder systems, averaged over all interferents and broken down by interferent addition concentration, are shown in Table 40 and graphed in Figures 57-59. All three binder systems are affected by interferent additions. In general, the interferents had the largest overall effects on the CEM specimens, which were the least durable to begin with. The LFA specimens were affected about equally by the wet/dry cycling but showed less effect from the interferents. The CFA samples also lost increasing mass with increasing interferent concentration but at a lower rate. The 8-percent samples had an average of about 54 percent of the mass of the control after 11 cycles.

PERMEABILITY

Triplicate permeability tests were run on a single specimen from each of the interferent treatments for each binder system. Good reproducibility was found for the results of permeability testing within the triplicate measurements on each specimen; however, the variability between permeability values for different specimens, even from the same mix or batch, was quite large.

The minimum, maximum, and average permeability values for the control samples for each binder are given in Table 41. All of the control samples for all three binder systems were near 10^{-6} cm/sec, but showed considerable variation between batches, as seen in the high coefficients of variation. The CEM controls had the highest average permeability, the greatest range in permeabilities, and the largest variability. The CFA and LFA products, which had higher more atypical UCS values, also had lower average permeabilities with smaller ranges and variation.

The time of cure (age) of the samples when the permeability was determined varied from about 35 days to over 300 days, with most being over 200 days of age. However, the age of the samples for each interferent group was the same when tested. No correlation was found between sample permeability and the different ages of either the control samples, shown in Figure 60, or the complete data set including the interferent samples (Figure 61).

The permeabilities of all of the specimens, along with the percent of the control value, are presented by binder in Table 42 and by interferent in Table 43. The permeabilities are compared for each interferent and binder by plotting the log of the permeability in Figures 62-64. Because of the large variability, no significant differences were found even between different specimens from the same batch.

Some consistency can be seen when comparing the effects of the interferents on the permeabilities of the three binder systems. Of the metals, copper appeared to lower sample permeability while lead, and to some extent zinc, increased it. Lime/fly ash, however, had lower permeabilities at the

TABLE 40. AVERAGE PERCENT OF MASS REMAINING AFTER WET/DRY CYCLING AVERAGED OVER ALL SAMPLES FOR EACH BINDER

Binder	Conc.	Original mass	Cycle 1	2	3	4	5	6	7	8	9	10	11
CEM	0	769	86.2	83.7	80.3	74.1	67.9	65.0	62.8	59.3	49.9	48.1	48.7
CEM	2	779	83.3	81.8	79.9	58.4	45.6	40.6	36.4	30.8	30.0	28.9	25.0
CEM	5	831	77.1	64.9	57.7	46.9	46.3	43.6	34.2	28.7	28.1	24.1	24.0
CEM	8	874	61.3	42.9	31.2	26.4	23.0	20.7	20.1	16.2	15.7	15.4	14.6
CFA	0	972	95.3	98.4	97.4	93.5	96.7	96.2	92.5	95.3	95.4	91.5	93.7
CFA	2	991	93.0	95.5	93.7	90.4	92.9	92.1	79.0	91.1	91.3	79.2	81.5
CFA	5	1,032	73.6	74.9	72.2	67.0	69.1	55.9	53.4	55.3	55.5	52.7	54.2
CFA	8	1,051	74.4	65.4	63.7	57.4	58.3	53.2	50.8	52.2	52.3	49.3	50.5
LFA	0	1,055	94.6	96.3	95.0	88.7	90.5	79.3	67.1	68.5	54.5	46.4	39.8
LFA	2	1,088	71.6	66.4	57.1	49.9	50.0	49.3	46.6	43.2	42.4	39.9	40.5
LFA	5	1,120	79.9	64.6	63.2	56.3	51.2	47.1	44.9	42.9	42.0	33.0	41.1
LFA	8	1,159	65.0	59.4	47.0	45.4	45.2	34.4	26.6	23.9	23.7	22.9	23.9

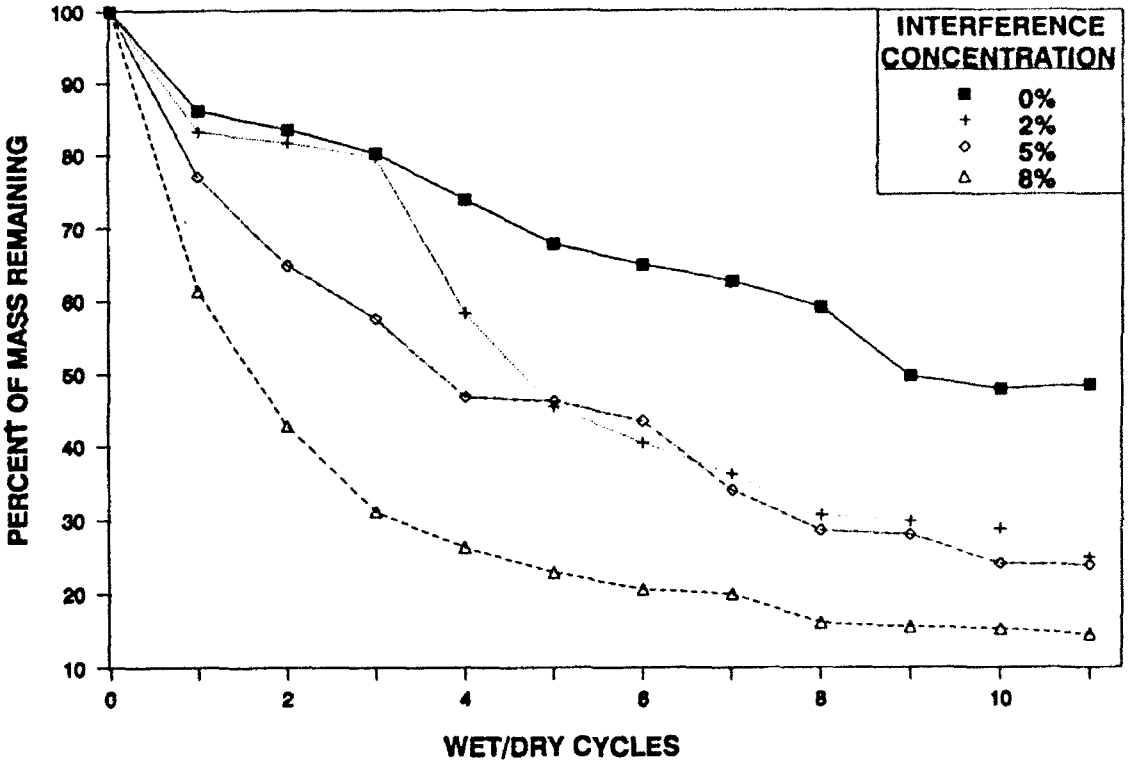

Figure 57. Average wet/dry data for the CEM solidified/ stabilized samples.

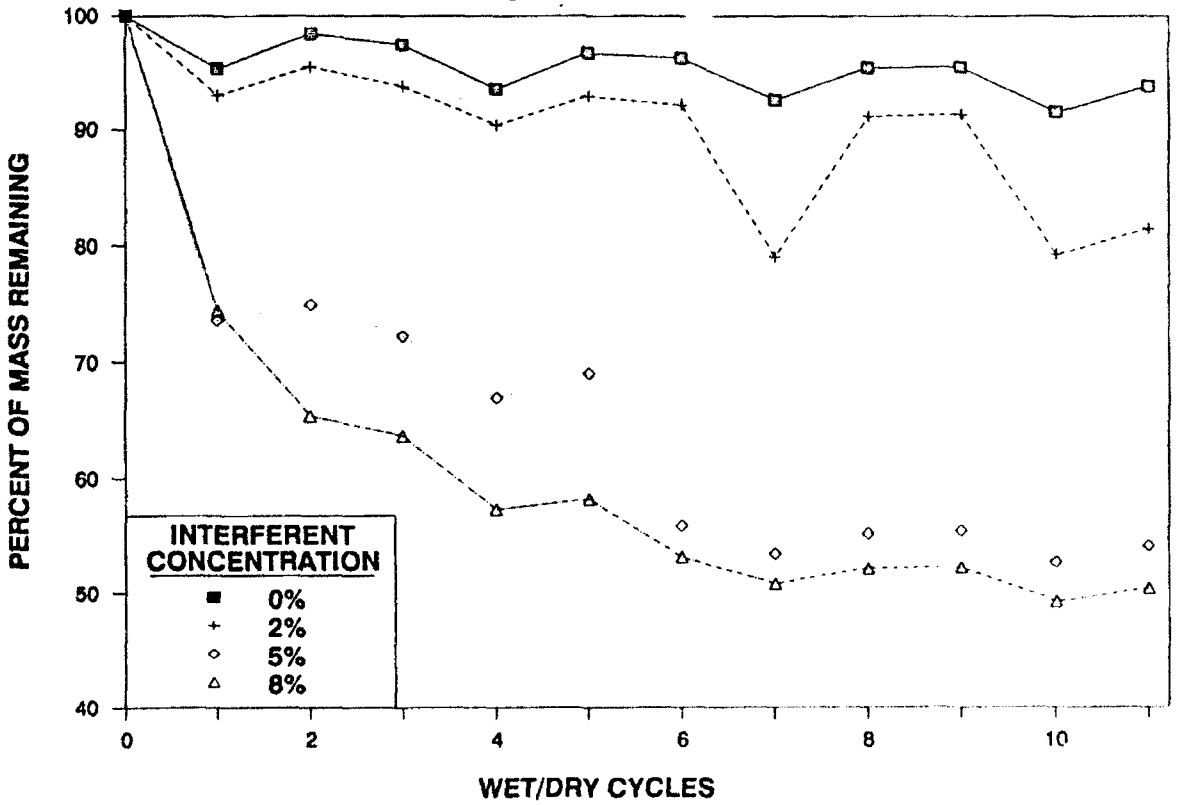

Figure 58. Average wet/dry data for the CFA solidified/ stabilized samples.

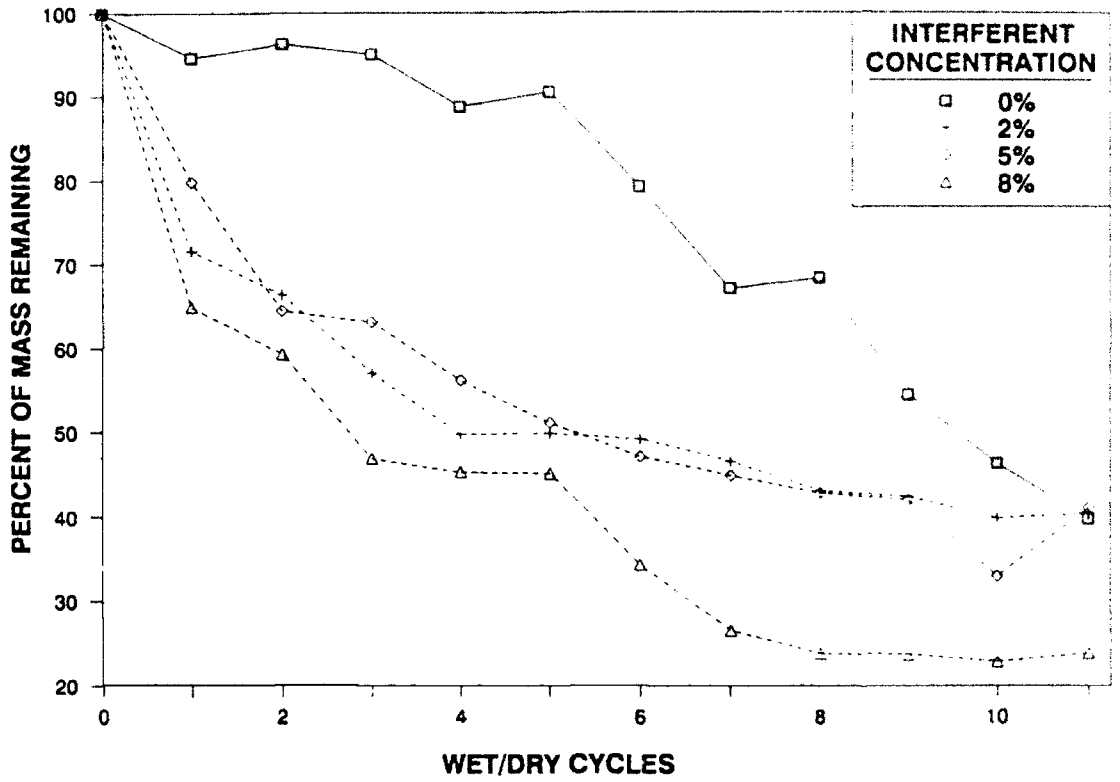

Figure 59. Average wet/dry data for the LFA solidified/ stabilized samples.

TABLE 41. AVERAGE, RANGE, AND VARIABILITY OF PERMEABILITIES OF CONTROL S/S SAMPLES FOR EACH BINDER

Binder	Permeability ($\times 10^{-6}$ cm/sec) Average	Lowest	Highest	Coefficient of variation, %
Cement	5.15	0.13	24.2	147
Cement/ fly ash	3.53	0.72	7.27	73
Lime/ fly ash	0.95	0.20	3.37	99

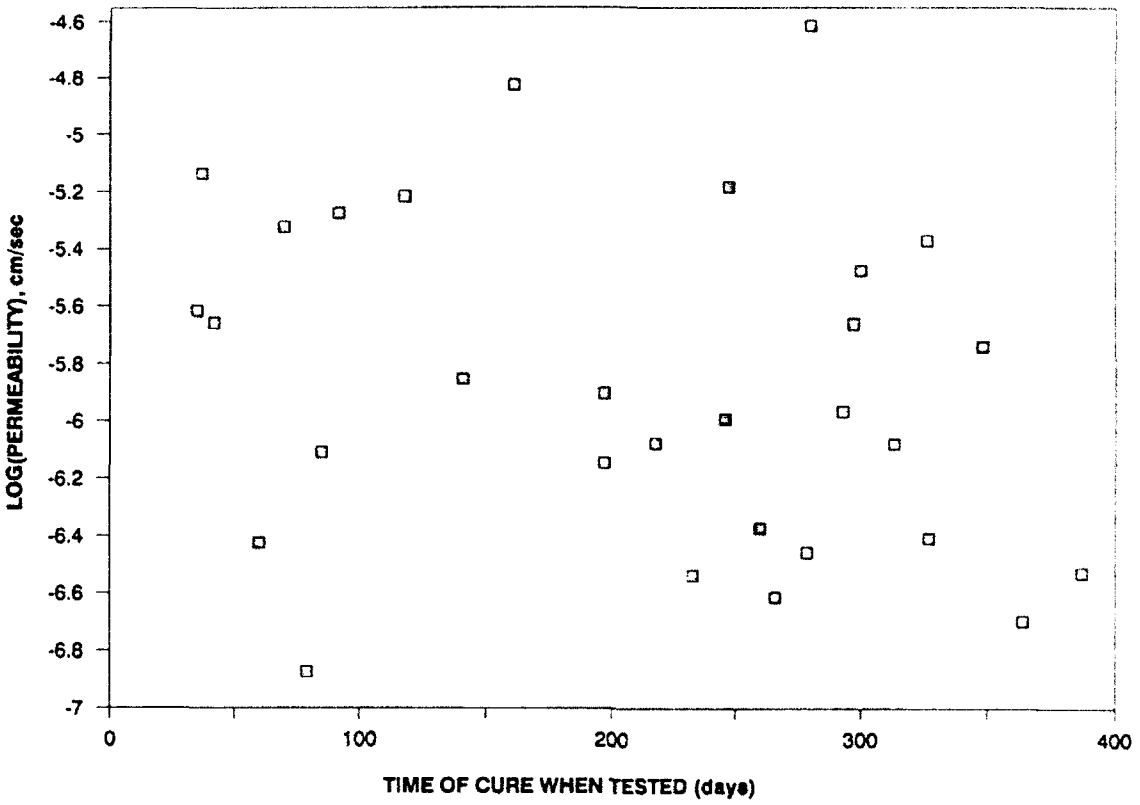

Figure 60. Sample age versus permeability for all control samples.

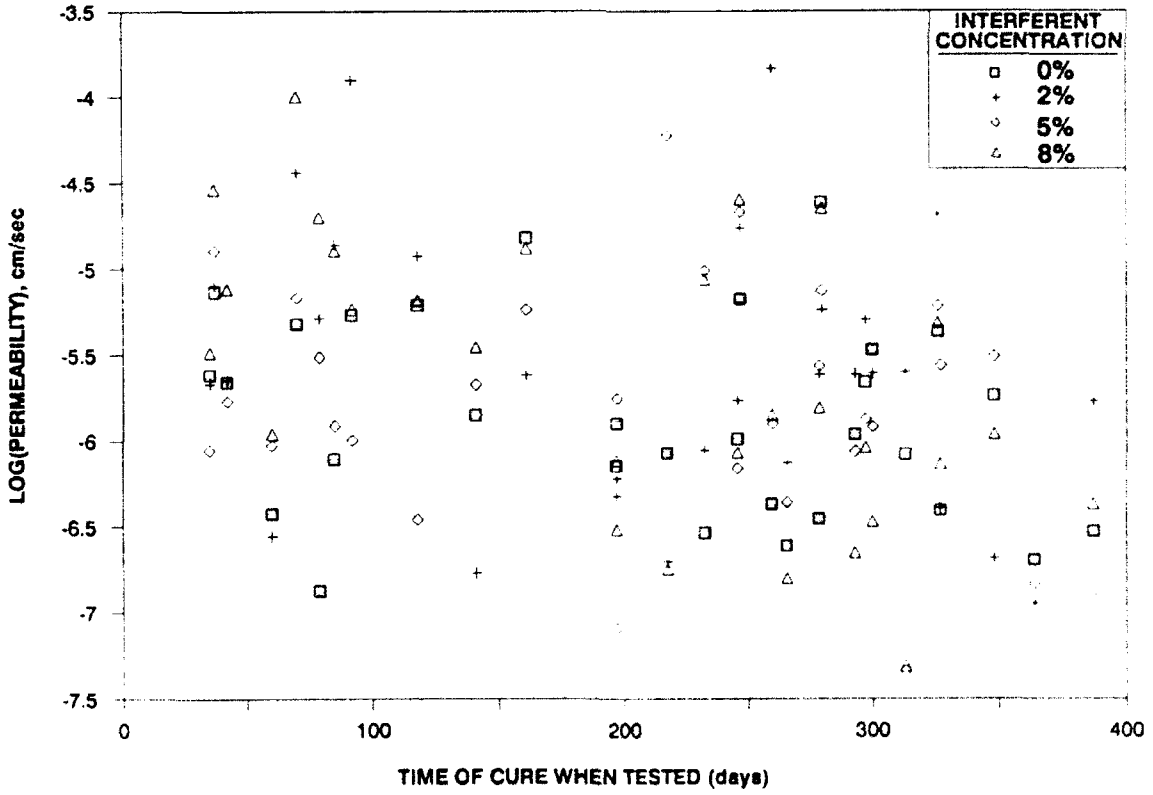

Figure 61. Sample age versus permeability for all samples tested.

TABLE 42. PERMEABILITIES OF SOLIDIFIED/STABILIZED PRODUCTS ARRANGED BY BINDER

Binder	Interferent	Age (Days)	Permeability ($\times 10^{-6}$) Control	2%	5%	8%	Percent of control 2	5	8
CEM	Copper	297	2.19	5.05	1.34	0.92	230	61	42
CEM	Grease	35	2.41	2.15	0.88	3.27	89	37	136
CEM	HCB	161	14.98	2.40	5.72	13.05	16	38	87
CEM	Lead	79	0.13	5.13	3.05	19.87	3,826	2,276	14,826
CEM	NaOH	60	0.38	0.28	0.93	1.10	74	248	292
CEM	Oil	42	2.19	2.25	1.70	7.64	103	78	349
CEM	Phenol	233	0.29	0.88	9.67	8.65	302	3,324	2,971
CEM	Sulfate	280	24.20	5.71	7.40	22.40	24	31	93
CEM	TCE	260	0.43	145.67	1.26	1.42	34,194	296	333
CEM	Zinc	326	4.29	20.43	6.01	4.81	476	140	112
CFA	Copper	197	1.26	0.60	1.75	0.77	48	139	61
CFA	Grease	85	0.78	13.67	1.22	12.70	1,754	157	1,630
CFA	HCB	197	0.72	0.48	0.08	0.30	66	11	42
CFA	Lead	70	4.75	36.23	6.73	99.50	762	142	2,093
CFA	NaOH	118	6.10	11.77	0.35	6.49	193	6	106
CFA	Oil	92	5.33	124.33	1.01	5.81	2,331	19	109
CFA	Phenol	141	1.41	0.17	2.11	3.47	12	150	246
CFA	Sulfate	37	7.27	7.98	12.63	29.00	110	174	399
CFA	TCE	246	1.03	1.72	0.69	0.85	168	67	83
CFA	Zinc	247	6.62	17.07	21.20	25.23	258	320	381
LFA	Copper	364	0.20	0.11	0.15	ND*	56	73	ND
LFA	Grease	327	0.39	0.40	2.75	0.74	103	702	188
LFA	HCB	300	3.37	2.47	1.20	0.34	73	36	10
LFA	Lead	293	1.09	2.45	0.87	0.22	225	80	20
LFA	NaOH	313	0.84	2.52	0.05	0.05	301	5	6
LFA	Oil	218	0.85	0.20	59.37	0.18	23	7,026	21
LFA	Phenol	279	0.35	2.43	2.72	1.55	694	778	444
LFA	Sulfate	387	0.30	1.69	0.13	0.43	572	45	146
LFA	TCE	266	0.24	0.75	0.44	0.16	305	180	65
LFA	Zinc	348	1.83	0.21	3.09	1.10	11	169	60

* No data.

TABLE 43. PERMEABILITIES OF SOLIDIFIED/STABILIZED PRODUCTS ARRANGED BY INTERFERENT

Binder	Interferent	Age (Days)	Permeability ($\times 10^{-6}$) Control	2%	5%	8%	Percent of control 2	5	8
CEM	Copper	297	2.19	5.05	1.34	0.92	230	61	42
CFA	Copper	197	1.26	0.60	1.75	0.77	48	139	61
LFA	Copper	364	0.20	0.11	0.15	ND*	56	73	ND
CEM	Lead	79	0.13	5.13	3.05	19.87	3,826	2,276	14,826
LFA	Lead	293	1.09	2.45	0.87	0.22	225	80	20
CFA	Lead	70	4.75	36.23	6.73	99.50	762	142	2,093
CEM	Zinc	326	4.29	20.43	6.01	4.81	476	140	112
CFA	Zinc	247	6.62	17.07	21.20	25.23	258	320	381
LFA	Zinc	348	1.83	0.21	3.09	1.10	11	169	60
CEM	Sulfate	280	24.20	5.71	7.40	22.40	24	31	93
CFA	Sulfate	37	7.27	7.98	12.63	29.00	110	174	399
LFA	Sulfate	387	0.30	1.69	0.13	0.43	572	45	146
CEM	NaOH	60	0.38	0.28	0.94	1.10	74	248	292
CFA	NaOH	118	6.10	11.77	0.35	6.49	193	6	106
LFA	NaOH	313	0.84	2.52	0.05	0.05	301	5	6
CEM	Grease	35	2.41	2.15	0.88	3.27	89	37	136
CFA	Grease	85	0.78	13.67	1.22	12.70	1,754	157	1,630
LFA	Grease	327	0.39	0.41	2.75	0.75	103	702	188
CEM	Oil	42	2.19	2.25	1.70	7.64	103	78	349
CFA	Oil	92	5.33	124.33	1.01	5.81	2,331	19	109
LFA	Oil	218	0.85	0.20	59.37	0.18	23	7,026	21
CEM	TCE	260	0.43	145.67	1.26	1.42	34,194	296	333
CFA	TCE	246	1.03	1.72	0.69	0.85	168	67	83
LFA	TCE	266	0.24	0.75	0.44	0.16	305	180	65
CEM	HCB	161	14.98	2.40	5.72	13.05	16	38	87
CFA	HCB	197	0.72	0.48	0.08	0.30	66	11	42
LFA	HCB	300	3.37	2.47	1.20	0.34	73	36	10
CEM	Phenol	233	0.29	0.88	9.67	8.65	302	3,324	2,971
CFA	Phenol	141	1.41	0.17	2.11	3.47	12	150	246
LFA	Phenol	279	0.35	2.43	2.72	1.55	694	778	444

* No data.

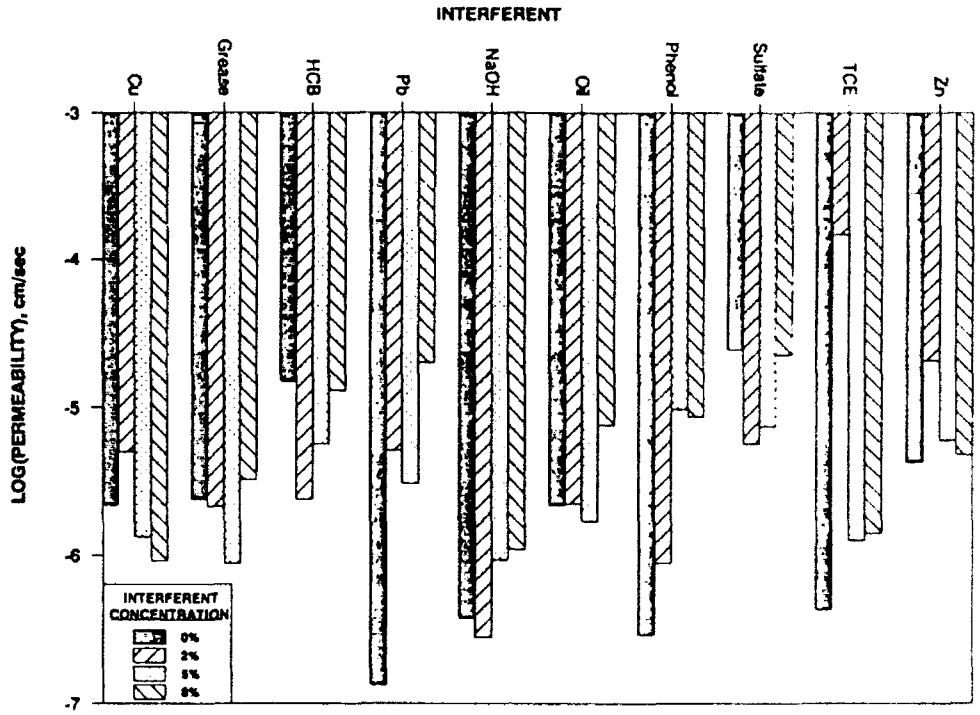

Figure 62. Permeability for the CEM solidified/stabilized samples.

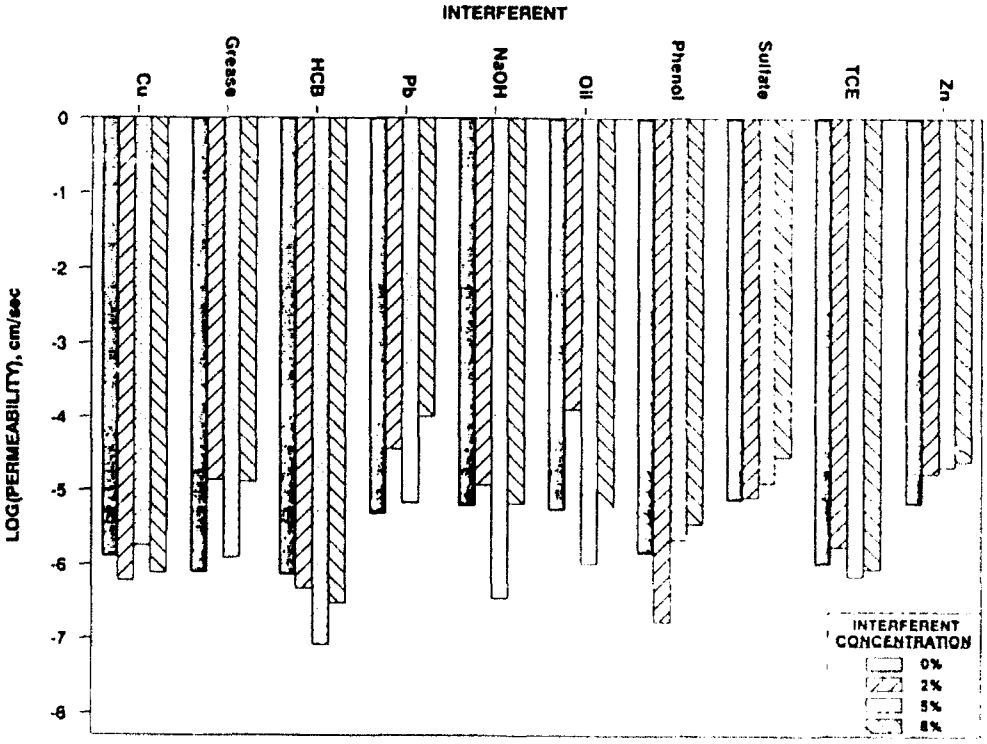

Figure 63. Permeability for the CFA solidified/stabilized samples.

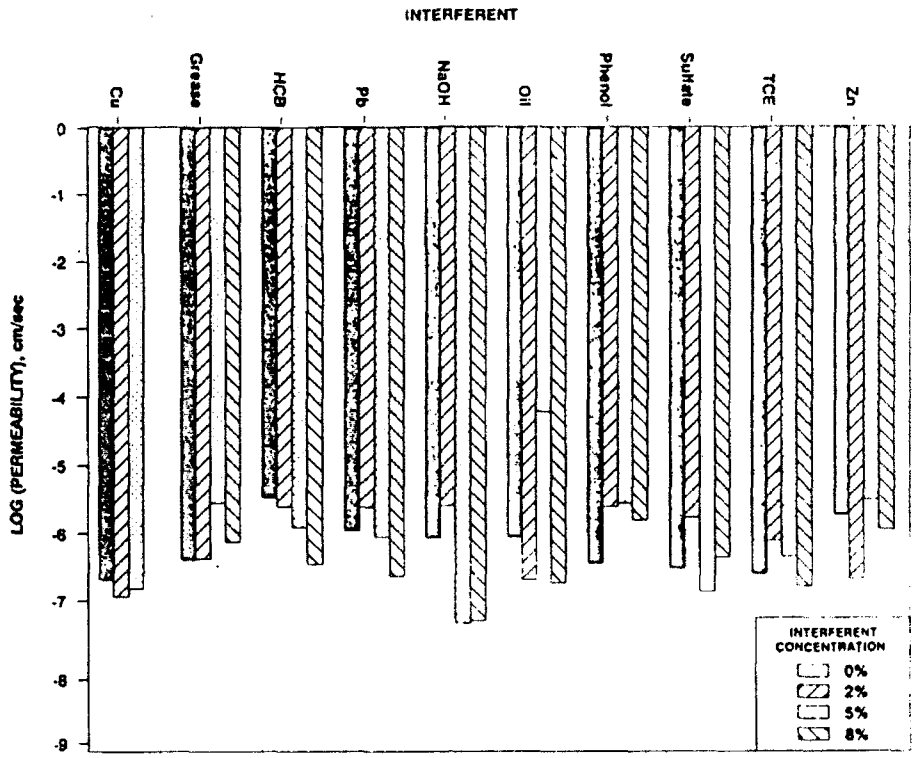

Figure 64. Permeability for the LFA stabilized/solidified samples.

higher concentrations of all three metals. Lead appears to produce the greatest increase in permeability in both cement ar cement/fly ash samples.

The organics, except for HCB, generally increased the permeability in all binder systems. Cement appears to be the most affected while LFA is least affected, although no general pattern is evident. For instance, TCE increased the permeability of cement samples while lowering it for CFA samples and having less effect upon LFA samples; however, the results for grease interference are almost exactly the opposite. HCB appears to consistently decrease permeability for all three binders while phenol increases it. Phenol increased the permeability of cement to a larger extent than that of LFA and CFA.

The high variability encountered even within batches and the lack of replicates makes interpretation of the permeability data nearly impossible. A large number of replicates would be necessary to establish any confidence in permeability values. The standard triaxial permeability test is a time-consuming and expensive procedure. For these reasons, the usefulness of measurements of permeability as an interpretive tool for low-permeability solids such as stabilized/solidified waste products is extremely limited if not impractical.

EXTRACTION PROCEDURE TOXICITY TEST

Duplicate samples from each of the treated and untreated stabilized/solidified products were leached according to the USEPA Extraction Procedure (EP). The leachates were analyzed for the four metals in the synthetic sludge and for the interferent material used in that treatment.

Samples Without Interferent Additions

Parameters for EP leaching of duplicate control specimens for each of the 30 sludge batches are shown in Table 45, arranged by binder. The control leachates show reasonable consistency for all parameters. Mercury concentrations had the highest values and variability between leaching tests. The control samples for the HCB interferent batch using CFA as the binder had the highest concentrations for all of the sludge metals.

The final EP leachate pH for CEM and LFA were both quite basic (median between 11.6 and 11.9), indicating an excess of acid neutralization capacity in the specimens, which overpowered the EP acid addition. The CFA leachates apparently had lower acid neutralization capacity, as their median final pH was 9.13. The difference in the final pH of the EP leachate strongly affects the amount of metals released from the solidified/stabilized sludge, especially Cd and Cr.

For both Cd and Cr, the CEM and LFA have median concentrations less than the average method blank (Appendix B), probably due to the lower metal solubility at the more basic pH of their leachates. CFA leachate concentrations of these metals from the control specimens were about 10 times greater than from the other binder systems for these metals, again due to the lower pH of the leachates. Median nickel leachate concentrations were near the method blanks for LFA samples, but leachates from both the CEM and CFA contained about 10 times higher Ni concentrations. Mercury leachate concentrations exceeded the method blanks by 2 orders of magnitude for all three binder systems, with the LFA control specimens having the highest median concentration.

Interferent materials found at appreciable levels in the control specimen leachates were sodium, sulfate, and phenol, which were found in leachates from all three binder systems (Table 44). Lead was found above the detection limit in all control specimen leachates but was especially elevated in the CFA control leachates.

Considering the high concentrations of metals contained in the sludge (Table 45), the low concentrations of metals found in the EP leachates are indicative of excellent contaminant immobilization. Metal concentrations in the EP leachates were reduced by 3 to 5 orders of magnitude by all binders even though all of the specimens were ground before the EP was performed. All three binder systems exhibit good containment characteristics for the metals in this sludge. Mercury levels in the EP leachates from the solidified/stabilized sludge, however, are about the same as those from the raw sludge. All three binders were essentially ineffective in immobilizing Hg.

Effects of Interferent Additions

The concentrations of the sludge metals and interferents in the EP leaching solutions from the solidified/stabilized sludge with different levels of interferent addition are summarized by interferent and binder in Tables 46-48. Each value is the average of two independent EP leaching procedures on two different specimens from each treatment.

TABLE 44. EP LEACHATE CONCENTRATIONS FOR THE CONTROL SPECIMENS LISTED BY INTERFERENT

Binder	Interferent	Moisture	Final pH	Concentration in EP leachate (mg/l) Cd	Cr	Hg	Ni	Interferent
CEM	Cu	0.279	11.57	0.0002	0.004	0.614	BDL*	BDL
CEM	Grease	0.366	11.80	BDL	0.020	0.303	0.090	BDL
CEM	HCB	0.437	11.83	0.0120	0.027	0.964	0.126	0.02
CEM	NaOH	0.404	11.26	0.0021	0.011	0.156	0.076	129.
CEM	Oil	0.307	11.66	0.0007	0.015	0.006	0.089	BDL
CEM	Pb	0.322	11.25	0.0034	0.009	0.296	0.102	0.01
CEM	Phenol	0.507	11.70	0.0013	0.008	2.140	0.070	34.0
CEM	Sulfate	0.311	11.78	0.0025	0.007	1.014	BDL	10.6
CEM	TCE	0.214	11.36	0.0022	0.013	0.513	0.093	BDL
CEM	Zn	0.262	11.59	0.0007	0.0008	0.950	0.008	0.03
Median for CEM		0.316	11.63	0.0021	0.010	0.564	0.083	--
CFA	Cu	0.197	9.19	0.0370	0.041	0.126	0.002	BDL
CFA	Grease	0.331	8.51	0.0915	0.131	0.299	0.082	BDL
CFA	HCB	0.378	7.73	1.2255	2.937	2.560	0.220	BDL
CFA	NaOH	0.342	8.34	0.1205	0.245	0.289	0.113	77.7
CFA	Oil	0.328	9.53	0.0025	0.038	0.683	0.060	BDL
CFA	Pb	0.296	932	0.0154	0.115	0.254	0.109	62.4
CFA	Phenol	0.378	9.06	0.0190	0.139	2.540	0.075	65.4
CFA	Sulfate	0.288	9.07	0.0624	0.041	0.207	0.013	34.6
CFA	TCE	0.351	9.88	0.0044	0.023	1.680	0.057	0.01
CFA	Zn	0.200	9.46	0.0087	0.031	0.109	0.005	0.03
Median for CFA		0.33	9.13	0.0280	0.078	0.294	0.068	--
LFA	Cu	0.314	11.77	BDL	0.003	0.762	BDL	BDL
LFA	Grease	0.272	11.95	0.0026	0.011	0.330	0.058	BDL
LFA	HCB	ND	11.85	0.0001	0.006	0.291	0.004	BDL
LFA	NaOH	0.298	11.87	0.0009	0.004	0.330	0.013	192.
LFA	Oil	0.308	11.70	0.0034	0.008	0.168	0.047	BDL
LFA	Pb	0.267	11.52	0.0009	0.003	0.625	0.084	0.20
LFA	Phenol	ND	12.05	0.0050	0.006	1.660	0.008	30.5
LFA	Sulfate	ND	11.86	BDL	0.007	0.950	BDL	76.5
LFA	TCE	0.292	12.04	0.0019	0.007	0.927	0.010	0.01
LFA	Zn	0.295	11.82	0.0004	0.007	0.900	BDL	BDL
Median for LFA		0.294	11.86	0.0009	0.007	0.694	0.009	--

* Below detection limits.

TABLE 45. COMPARISON OF THE SOLIDIFIED/STABILIZED EXTRACTS WITH THE RAW WASTE EXTRACTS

Parameter (units)	Cd	Cr	Hg	Ni
Sludge concentrations (mg/kg dry wt)	18,100	81,300	1,390	81,100
EP of raw sludge (mg/l leachate)	57.9	242	0.84	149
Median EP of CEM controls (mg/l)	0.0021	0.010	0.95	0.083
Median EP of CFA controls (mg/l)	0.028	0.078	0.29	0.068
Median EP of LFA controls (mg/l)	0.0009	0.007	0.69	0.009

Effects of Final EP Leachate pH

An important characteristic of the different interferents with regard to leaching of sludge metals was their effect on the final pH of the EP leachates, especially in the specimens treated with CFA. This was most evident for the metal ion interferents, as seen in Tables 46-48. The metals were added as their nitrate salts, which may account for the pronounced decrease in the final EP leachate pH. Copper nitrate had the greatest effect on the final pH. As illustrated in Figure 65, an 8-percent addition of $CuNO_3$ lowered the pH of the CEM leachates by 2.6 units, the CFA leachates by 3.6 units, and the LFA by 1.8 pH units. The lead and zinc nitrates had similar but less pronounced effects.

This pattern held for all of the interferents. The CFA samples had the lowest acid neutralization capacity (median pH of controls = 9.13), followed by the CEM samples (11.63) and, finally, by the LFA samples, which appear to have the highest acid neutralization capacity (median pH = 11.86). As discussed for the control specimen leaching results, this difference in pH is especially significant for the leaching of the sludge metals. The relationship between final pH and the log of the concentration of Cd in the leaching fluid is illustrated for all samples from all controls and interferents in Figure 66. The correlation coefficient (r), including only samples with final pH below 10.5 (n = 145) using the log of Cd concentrations, is -0.91. This correlation coefficient is significantly different from zero at the 99-percent level of confidence. The amphoteric nature of Cd solubility is evident in the plot as Cd concentrations increase as the final leachate pH increases above about 10.5; minimum Cd solubility is around pH 10.5. The concentration of Cd leached can be predicted very closely from the final pH of the EP leaching solution, especially in leachates with final pH below 10.5.

As illustrated in Figure 67, Ni leachate concentrations showed a similar but less pronounced dependence on final leachate pH. An r value of -0.62 is significantly different from zero for final pH values less than 10.5 (n = 91). Leachates contained fairly consistent Ni concentrations above a pH of about 7.5. The Ni concentrations increased about 2 orders of magnitude at pH values below about 7.5. Deviations from this pattern are probably caused by

TABLE 46. SUMMARY OF THE EP LEACHING DATA FOR THE CEM BINDER

Interferent	Conc. (%)	Moisture	Final pH	Concentration in EP leachate (mg/l) Cd	Cr	Hg	Ni	Interferent
Copper	0	0.279	11.57	0.0002	0.004	0.614	BDL*	BDL
Copper	2	0.293	11.01	0.0002	0.031	0.471	0.003	0.17
Copper	5	0.257	9.29	0.0344	0.026	0.288	BDL	0.01
Copper	8	0.215	8.99	0.1055	0.009	0.482	BDL	0.01
Lead	0	0.322	11.25	0.0034	0.009	0.296	0.102	0.01
Lead	2	0.259	11.22	0.0011	0.005	0.296	0.098	3.69
Lead	5	0.226	11.12	0.0008	0.008	0.301	0.101	8.98
Lead	8	0.394	10.97	0.0005	0.007	0.302	0.103	8.18
Zinc	0	0.262	11.59	0.0007	0.008	0.950	0.008	0.03
Zinc	2	0.286	11.00	0.0005	0.016	1.029	0.016	0.13
Zinc	5	0.327	10.27	0.0012	0.132	0.515	0.036	0.09
Zinc	8	0.288	10.99	0.0027	0.078	0.615	0.036	0.18
Sulfate	0	0.311	11.78	0.0025	0.007	1.014	0.002	10.60
Sulfate	2	0.359	11.83	0.0064	0.009	1.010	BDL	497.
Sulfate	5	0.373	11.86	0.0065	0.095	0.915	BDL	1,140.
Sulfate	8	0.400	11.64	0.0012	0.199	0.657	BDL	1,525.
NaOH	0	0.404	11.26	0.0021	0.011	0.156	0.076	129.2
NaOH	2	0.389	11.36	0.0014	0.012	0.156	0.062	616.
NaOH	5	0.374	11.55	0.0029	0.070	0.157	0.045	1,430.
NaOH	8	0.339	11.70	0.0021	0.365	0.158	0.041	1,835.
Grease	0	0.366	11.80	BDL	0.020	0.303	0.090	BDL
Grease	2	0.375	11.83	BDL	0.013	0.300	0.087	BDL
Grease	5	0.352	11.85	BDL	0.021	0.297	0.085	BDL
Grease	8	0.361	11.87	BDL	0.012	0.309	0.081	BDL
Oil	0	0.307	11.66	0.0007	0.015	0.006	0.089	BDL
Oil	2	0.199	11.52	0.0016	0.032	0.001	0.090	BDL
Oil	5	0.262	11.76	0.0007	0.010	0.002	0.094	BDL
Oil	8	0.211	11.68	0.0019	0.011	0.001	0.087	BDL
TCE	0	0.214	11.36	0.0022	0.013	0.513	0.093	0.00
TCE	2	0.183	11.61	0.0018	0.018	1.965	0.090	0.02
TCE	5	0.112	11.48	0.0013	0.013	1.310	0.091	0.01
TCE	8	0.137	11.42	0.0019	0.013	1.290	0.091	0.01
HCB	0	0.437	11.83	0.0120	0.027	0.964	0.126	0.02
HCB	2	0.354	11.89	0.0012	0.012	1.045	0.103	BDL
HCB	5	0.357	11.76	0.0024	0.015	0.986	0.108	BDL
HCB	8	0.341	11.82	0.0037	0.009	0.759	0.112	BDL
Phenol	0	0.507	11.70	0.0013	0.008	2.140	0.070	34.05
Phenol	2	0.542	11.56	0.0014	0.010	6.580	0.064	746.
Phenol	5	0.509	11.59	0.0059	0.011	6.560	0.075	1,570.
Phenol	8	0.503	11.24	0.0023	0.011	7.175	0.073	2,710.

* Below detection limits.

TABLE 47. SUMMARY OF THE EP LEACHING DATA FOR THE CFA BINDER

Inter-ferent	Conc. (%)	Mois-ture	Final pH	Concentration in EP leachate (mg/l) Cd	Cr	Hg	Ni	Inter-ferent
Copper	0	0.197	9.19	0.0370	0.041	0.126	0.002	BDL*
Copper	2	0.176	8.51	0.2050	0.047	0.994	0.003	BDL
Copper	5	0.165	6.05	49.7000	0.010	0.194	0.994	312.00
Copper	8	0.179	5.57	56.0500	0.669	0.272	1.670	1,155.00
Lead	0	0.296	9.32	0.0154	0.115	0.254	0.109	11.15
Lead	2	0.292	9.65	0.0077	0.123	0.312	0.092	10.16
Lead	5	0.351	8.65	0.0780	0.044	0.313	0.118	11.04
Lead	8	0.310	7.24	8.7500	0.196	0.215	2.285	4.04
Zinc	0	0.200	9.46	0.0087	0.031	0.109	0.005	0.03
Zinc	2	0.179	10.07	0.0008	0.092	0.260	0.001	0.03
Zinc	5	0.277	6.61	48.6000	0.020	0.209	4.110	881.50
Zinc	8	0.293	6.36	60.5500	0.010	0.249	5.720	1,675.00
Sulfate	0	0.288	9.07	0.0624	0.041	0.207	0.013	34.60
Sulfate	2	0.312	8.85	0.0905	0.100	0.158	0.009	310.50
Sulfate	5	0.336	7.78	1.4695	0.215	0.075	0.041	1,090.00
Sulfate	8	0.340	7.82	0.9690	0.193	0.051	0.028	1,785.00
NaOH	0	0.342	8.34	0.1205	0.245	0.289	0.113	77.75
NaOH	2	0.368	8.49	0.0870	0.169	0.158	0.086	702.50
NaOH	5	0.379	9.38	0.0074	0.212	0.257	0.057	1,300.00
NaOH	8	0.390	10.45	0.0003	0.405	0.312	0.031	1,930.00
Grease	0	0.331	8.51	0.0915	0.131	0.299	0.082	BDL
Grease	2	0.321	8.20	0.2500	0.165	0.100	0.089	BDL
Grease	5	0.273	9.15	0.0200	0.087	0.161	0.083	BDL
Grease	8	0.247	9.20	0.0158	0.095	0.138	0.086	BDL
Oil	0	0.328	9.53	0.0025	0.038	0.683	0.060	BDL
Oil	2	0.313	9.55	0.0039	0.044	0.065	0.059	BDL
Oil	5	0.290	9.00	0.0161	0.115	0.002	0.064	BDL
Oil	8	0.247	9.45	0.0085	0.084	0.063	0.074	BDL
TCE	0	0.351	9.88	0.0044	0.023	1.680	0.057	0.01
TCE	2	0.345	9.50	0.0106	0.053	0.588	0.059	0.03
TCE	5	0.349	9.69	0.0132	0.092	0.336	0.058	0.04
TCE	8	0.367	9.89	0.0044	0.022	0.441	0.055	0.06
HCB	0	0.378	7.73	1.2255	2.937	2.560	0.220	BDL
HCB	2	0.356	8.79	0.0696	0.083	2.010	0.077	BDL
HCB	5	0.370	7.70	0.5120	0.196	1.660	0.088	0.01
HCB	8	0.384	7.82	0.3410	0.132	2.110	0.082	0.01
Phenol	0	0.378	9.06	0.0190	0.139	2.540	0.075	65.40
Phenol	2	0.414	9.50	0.0066	0.062	7.230	0.074	573.50
Phenol	5	0.098	9.57	0.0085	0.033	3.755	0.073	1,310.00
Phenol	8	0.414	9.48	0.0123	0.031	3.860	0.077	2,350.00

* Below detection limits.

TABLE 48. SUMMARY OF THE EP LEACHING DATA FOR THE LFA BINDER

Interferent	Conc. (%)	Moisture	Final pH	Concentration in EP leachate (mg/l) Cd	Cr	Hg	Ni	Interferent
Copper	0	0.314	11.77	BDL*	0.003	0.762	BDL	BDL
Copper	2	0.231	11.49	BDL	0.008	1.110	BDL	0.32
Copper	5	0.345	10.71	BDL	0.014	0.506	0.002	0.04
Copper	8	0.138	10.02	0.0033	0.015	0.205	0.004	0.03
Lead	0	0.267	11.52	0.0009	0.003	0.625	0.084	0.20
Lead	2	0.193	11.65	0.0021	0.010	0.761	0.087	70.65
Lead	5	0.104	11.66	0.0194	0.009	0.807	0.104	471.
Lead	8	0.120	11.51	0.0113	0.009	0.709	0.104	425.
Zinc	0	0.295	11.82	0.0004	0.007	0.900	BDL	BDL
Zinc	2	0.143	11.18	BDL	0.011	0.831	BDL	0.74
Zinc	5	0.214	10.22	BDL	0.017	0.494	BDL	0.27
Zinc	8	0.318	8.35	BDL	0.054	0.320	BDL	0.12
Sulfate	0	ND	11.86	BDL	0.007	0.950	BDL	76.5
Sulfate	2	ND	11.65	BDL	0.005	0.576	BDL	617.
Sulfate	5	ND	11.49	BDL	0.006	0.594	BDL	993.
Sulfate	8	ND	10.66	BDL	0.091	BDL	0.004	1,620.
NaOH	0	0.298	11.87	0.0009	0.004	0.330	0.013	192.
NaOH	2	0.323	11.9[illegible]	0.0032	0.015	0.315	0.006	667.
NaOH	5	0.337	11.89	0.0030	0.003	0.316	0.007	1,115.
NaOH	8	0.344	12.01	0.0026	0.050	0.323	0.006	1,520.
Grease	0	0.272	11.95	0.0026	0.011	0.330	0.058	BDL
Grease	2	0.272	12.09	0.0121	0.006	0.307	0.056	BDL
Grease	5	0.315	11.99	0.0138	0.002	0.275	0.054	BDL
Grease	8	0.297	11.98	0.0083	0.016	0.270	0.068	BDL
Oil	0	0.308	11.70	0.0034	0.008	0.168	0.047	BDL
Oil	2	0.301	11.79	0.0039	0.019	0.006	0.052	BDL
Oil	5	0.284	11.80	0.0066	0.010	BDL	0.078	BDL
Oil	8	0.268	11.78	0.0031	0.009	BDL	0.068	BDL
TCE	0	0.292	12.04	0.0019	0.007	0.927	0.010	BDL
TCE	2	0.297	12.11	0.0041	0.004	1.250	0.005	0.03
TCE	5	0.283	12.07	0.0013	0.006	0.821	0.003	0.04
TCE	8	0.296	12.05	0.0005	0.004	0.910	0.001	0.12
HCB	0	ND	11.85	0.0001	0.006	0.291	0.004	BDL
HCB	2	ND	11.79	0.0004	0.061	0.477	0.076	0.02
HCB	5	ND	11.70	0.0003	0.005	0.360	0.003	BDL
HCB	8	ND	11.74	0.0002	0.006	0.281	0.003	BDL
Phenol	0	ND	12.05	0.0050	0.006	1.660	0.008	30.5
Phenol	2	ND	12.22	0.0003	0.006	3.920	0.006	676.
Phenol	5	ND	12.17	0.0003	0.006	2.290	0.005	1,640.
Phenol	8	ND	12.06	0.0001	0.007	2.705	0.010	2,650.

* Below detection limits.

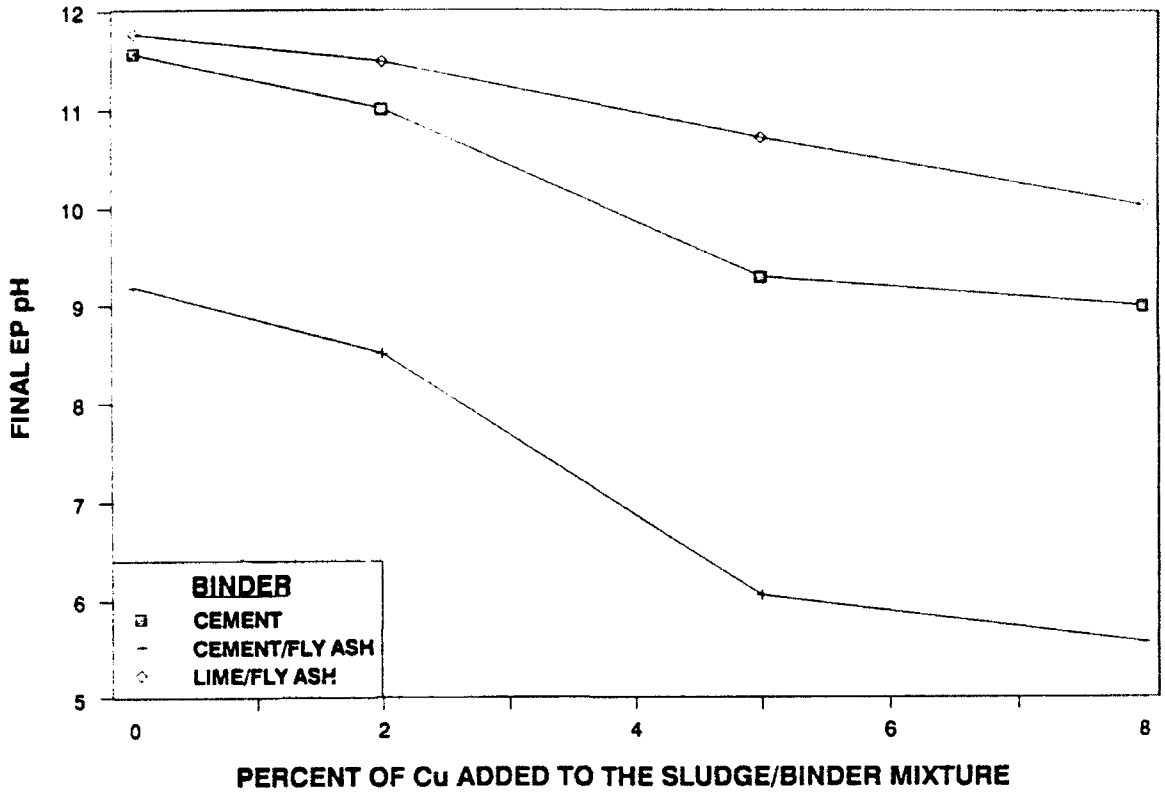

Figure 65. Final pH of EP extracts for the solidified/ stabilized samples with copper addition.

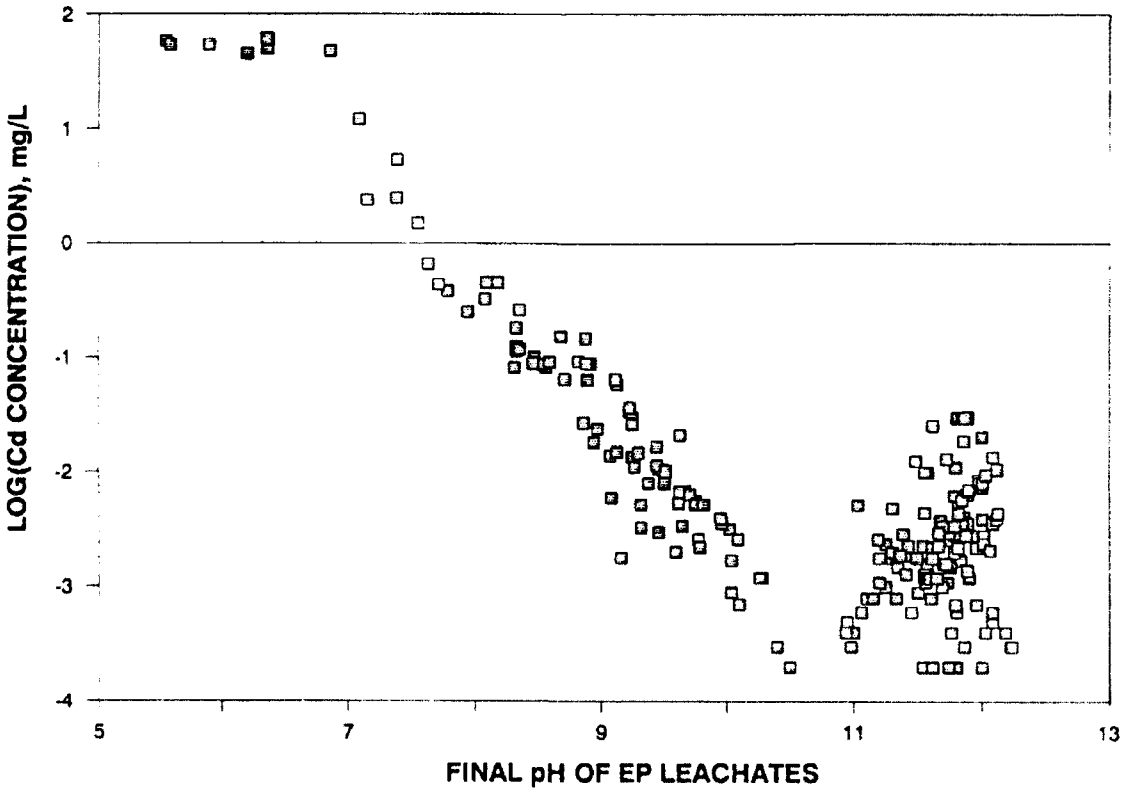

Figure 66. Cadmium concentrations for all EP extracts.

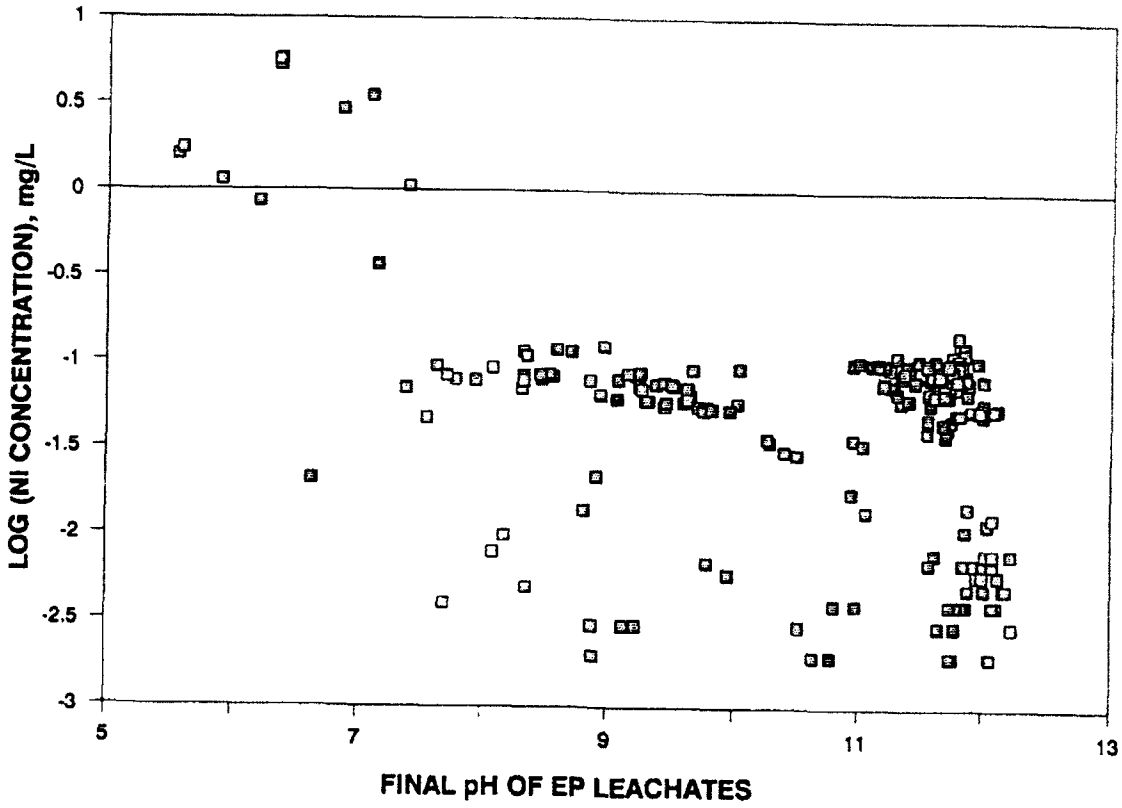

Figure 67. Nickel concentrations for all EP extracts.

differences in the effects of the individual interferents reacting with the Ni in solution. As seen in Figure 68, Cr also tended to increase in concentration as the pH of the leachate decreased but much less dramatically. (For pH values less than 10.5, r = -0.12, where r is not significantly different from zero; however, for values of final pH greater that 7.5, r = -0.51, which is significant.) Mercury concentrations in the leachates showed little or no apparent pH dependence (Figure 69).

S/S Moisture Content

The moisture content of the S/S products varied considerably within and between different batches. The LFA mixtures had consistently lower moisture values (higher solids), the controls averaging 16 and 6 percent lower than the moisture content of the CEM and CFA specimens. Averaging over all of the 480 samples from all treatments at 28 days of cure, LFA averaged 26.7 percent moisture; CFA, 30.6 percent; and CEM, 32.4 percent.

No consistent relationship between moisture content and leachate concentrations of any of the sludge metals was found. Mercury had the highest and only significant correlation coefficient with moisture content (r = 0.38). The overall relationship between sludge metal concentration and moisture content is illustrated for EP leachate final pH in Figure 70 and for Ni in Figure 71.

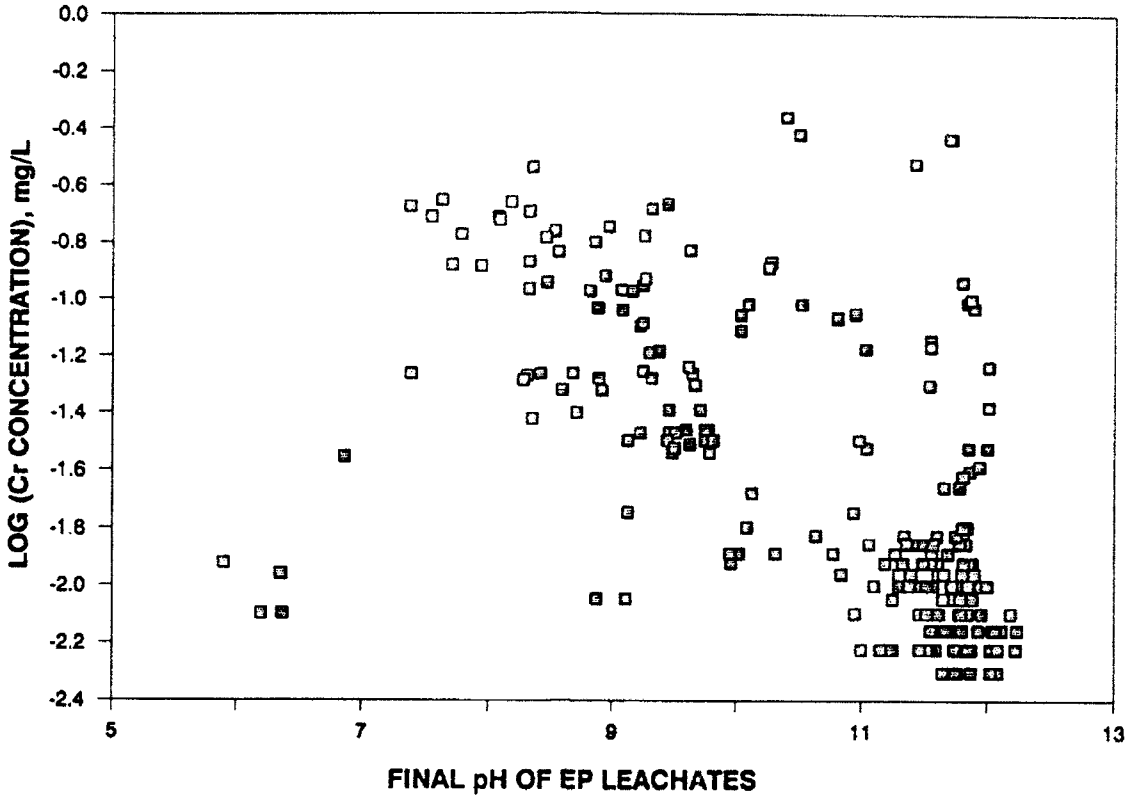

Figure 68. Chromium concentrations for all EP extracts.

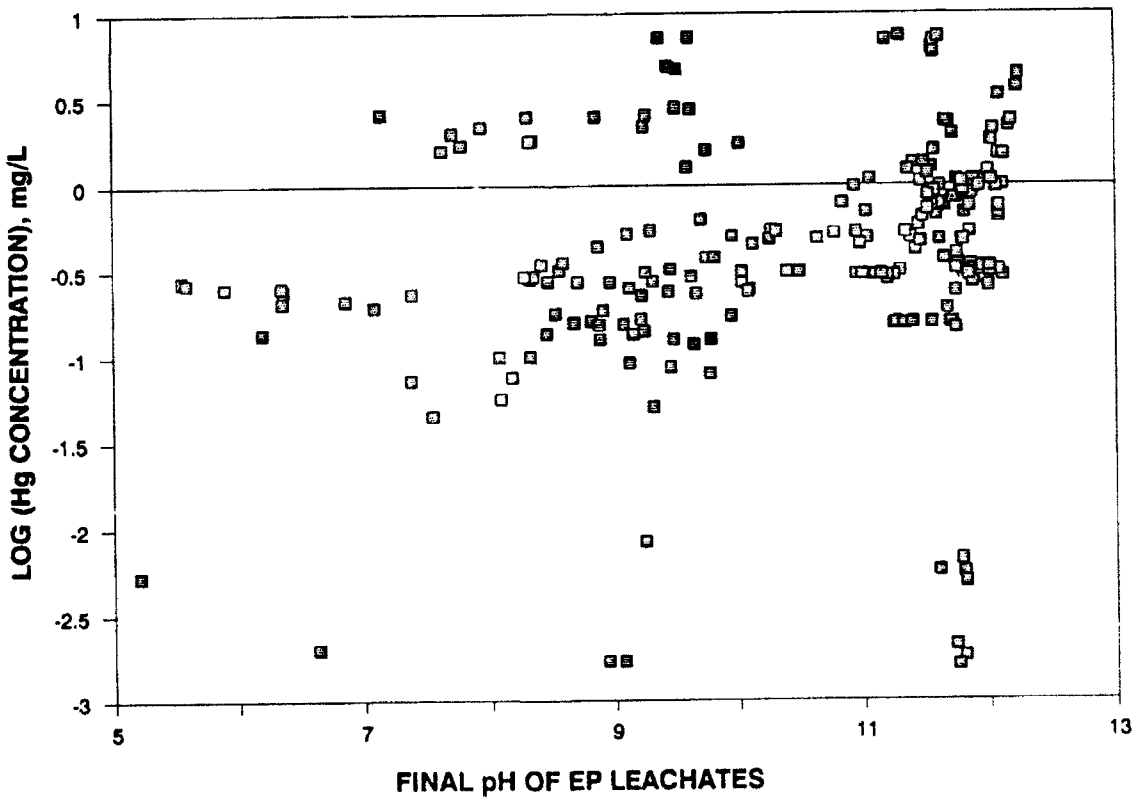

Figure 69. Mercury concentrations for all EP extracts.

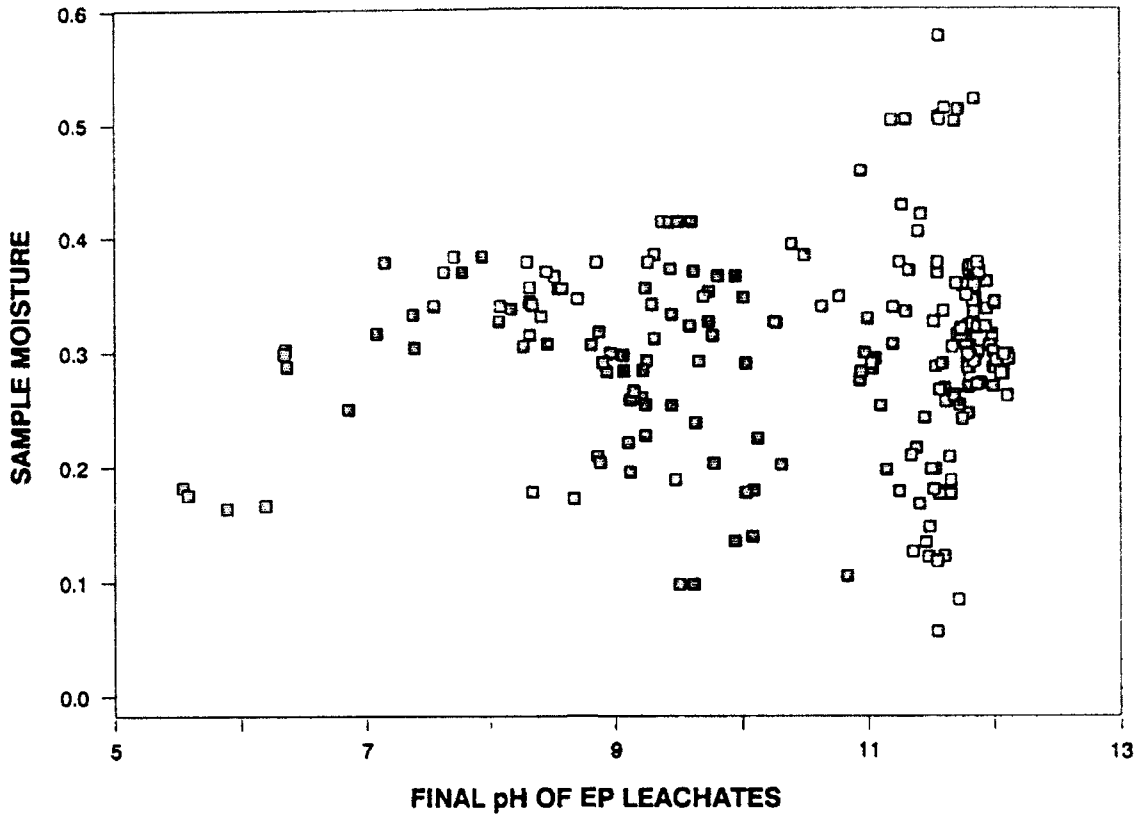

Figure 70. Final pH of the EP extract versus the moisture content for all samples evaluated.

Metal Ion Interferents

The addition of the metal ion interferents Pb, Zn, and Cu (as nitrates) had consistent and measurable effects on the final pH values of the EP leachates. This is especially true for Cu addition, and for all of the CFA mixes that had a lower initial acid neutralization capacity (Table 47). Cadmium concentrations in the leachates were affected most dramatically. The Cd concentrations increased over 3 orders of magnitude at the highest metal ion interferent levels for CFA mixes--from 37 ppb in the control to 56,000 ppb for the 8-percent copper interferent, from 15 ppb to 8,750 ppb for the 8-percent lead interferent, and from 9 ppb to over 60,000 ppb for the 8-percent zinc interferent. Averaging data for all three metal ion interferents (Table 49) gives an average change from 0- to 8-percent metal addition rate of 20 ppb to 42,000 ppb for Cd concentrations in the CFA leachates. Similar but less pronounced decreases in pH and increases in Cd concentrations are seen for the CEM and LFA binder systems. For Cd, the residual pH of the leachate appears to be the major factor that controls its release.

The EP leachate concentrations for Ni and Cr show similar but less pronounced increases at lower pH, especially for pHs at or below 7. When the pH drops below 7 in the CFA leachates, Ni concentrations are higher by a factor of over 100, while Ni concentrations in the CEM and CFA leachates increase less that 30 percent at the highest metal interferent levels. Chromium concentrations in the EP leachates increase about 5 times as the levels of all three metal interferents increase.

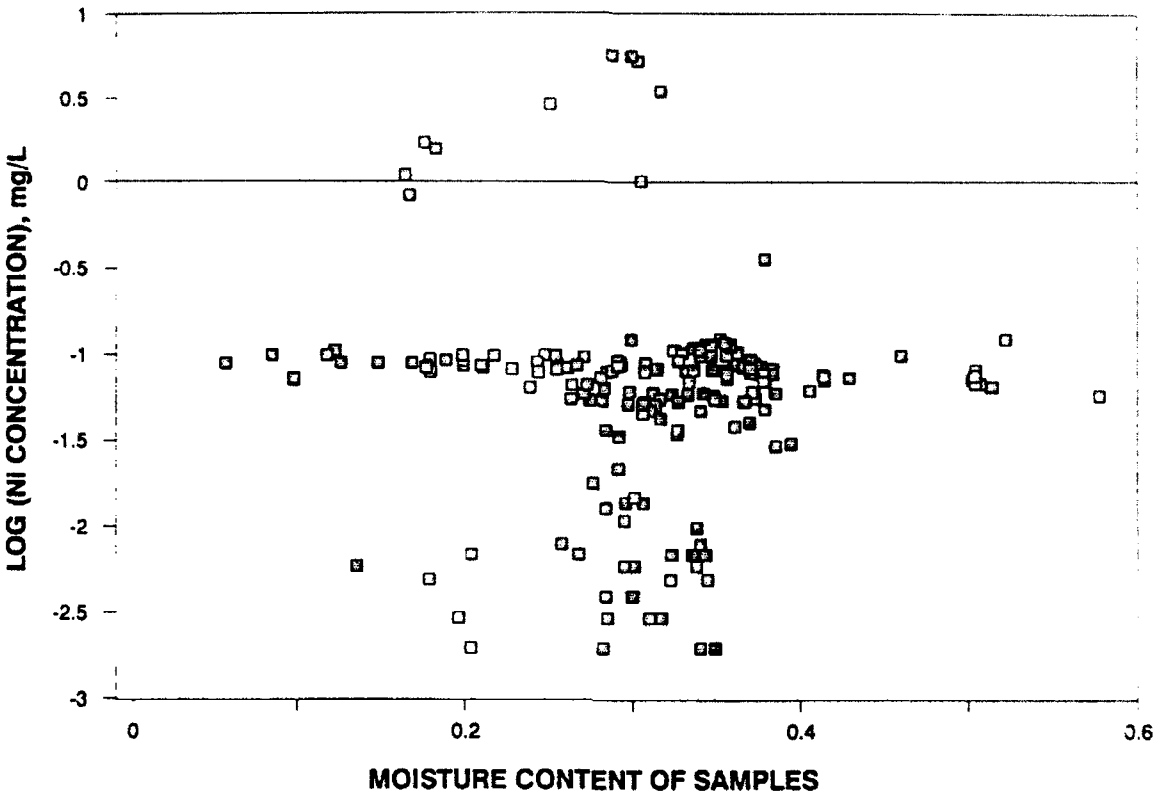

Figure 71. Moisture content versus the log of nickel concentration measured in the EP extracts.

TABLE 49. AVERAGE OF EP LEACHING DATA OVER METAL INTERFERENTS (ONLY CU, PB, AND ZN)

Binder	Conc. (%)	Final pH	Moisture	Concentration in leachate (mg/l) Cd	Cr	Hg	Ni
CEM	0	11.5	0.288	0.0014	0.0065	0.620	0.0367
CEM	2	11.1	0.280	0.0006	0.0173	0.598	0.0388
CEM	5	10.2	0.270	0.0121	0.0553	0.368	0.0458
CEM	8	10.3	0.299	0.0362	0.0313	0.466	0.0463
CFA	0	9.3	0.231	0.0204	0.0622	0.163	0.0383
CFA	2	9.4	0.215	0.0712	0.0872	0.522	0.0318
CFA	5	7.1	0.264	32.8	0.0245	0.239	1.74
CFA	8	6.4	0.261	41.8	0.2912	0.245	3.22
LFA	0	11.7	0.292	0.0005	0.0046	0.762	0.0285
LFA	2	11.4	0.189	0.0008	0.0095	0.901	0.0295
LFA	5	10.9	0.221	0.0065	0.0133	0.602	0.0357
LFA	8	10.0	0.192	0.0049	0.0255	0.412	0.0362

Mercury leaching patterns are quite different from the other sludge constituents. On the average, Hg is leached from the control samples at parts per million concentrations. Reversed from the other sludge constituents, Hg is found at higher concentrations in the CEM and LFA specimen leachates than

in those from the CFA samples; higher levels of metal interferent appear to decrease the levels of Hg in the leachate, although mercury's leaching pattern is not consistent.

As would be expected, the interferent concentrations measured in the EP leachates also increase as their level in the sludge increases. Interferents Cu and Zn are held to low levels in the CEM and LFA sample leachates but are leached at very high levels from the CFA samples, both attaining a concentration of over 0.1 percent in leachates from the 8-percent interferent additions.

The Pb concentration in the CEM and LFA specimen leachates increased with increasing Pb additions, as would be expected, since the leachate pH values are relatively uniform. In contrast, when CFA was used as a binder, the Pb concentration actually decreased with increasing Pb addition. Although the data are somewhat scattered, it is believed that this decrease in solubility can be attributed to the fact that Pb, while amphoteric like Cd and Cr, exhibits a minimum solubility at lower pH values.

Strong Base and Sulfate Interference

Sodium hydroxide generally affected the final leachate in an inverse manner to that of the metal nitrates. As expected, higher levels of NaOH addition increased the final EP leachate pH. The higher final leachate pH is especially prevalent in the CFA samples, which have low buffering capacity. This high pH results in lower concentrations of the metals Cd and Ni in the leachates from the samples with added NaOH. No other effects were seen in the leaching of the sludge metals. The high levels of sodium in the leachates reflected the NaOH additions and the generally high solubility of Na salts.

Sodium sulfate (SO_4) addition did not substantially affect the levels of sludge metals in the leachates. The largest effect observed for SO_4 addition is its own high concentration in the leachates from all three binder systems. The interferents with high water-solubility (phenol, Na, and SO_4) are lost from specimens prepared from all three binders in very similar concentrations and in direct proportion to their addition rates. All three binder systems are equally ineffective in containing very soluble materials.

Organic Interferents

The nonpolar, organic interferents (those with low water-solubilities, i.e., grease, oil, TCE, and HCB) had no significant effect on the final pH of the EP leachates or the concentration of any of the sludge metals leached from the solidified/stabilized sludge. Average concentrations leached from the grease, oil, and TCE specimens follow closely the concentrations leached from the controls (averaged). The CEM and LFA specimen leachates have similar constituent concentrations, which are lower than those found in the CFA sample leachates for Cd and Cr, and higher or the same for Hg and Ni. In addition, only very low levels of these interferent compounds were found in the EP leachates, even from the samples with an 8-percent addition, TCE being the only one of the three found at greater than detection limits.

Hexachlorobenzene shows similar leaching patterns except that the lower leachate pH values found for all of the CFA specimen leachates (including the control) produce leachates with much higher levels of the metals, especially

Cd and Cr. Mercury is highest in the leachates of all of the specimens of CFA, about twice the level in the CEM leachates, and 5 to 10 times the level in the LFA specimen leachates. HCB is found only near or below detection limits in leachates from all binders.

Addition of phenol, which has a much higher water-solubility than the other organics, had little effect on final leachate pH or Cd, Cr, and Ni concentrations. These leachates did have higher Hg levels than the other organics. Phenol was also found at very high levels in the leachates--between 0.2 and 0.3 percent in leachates from all three binders with 8-percent phenol addition. Phenol has a pronounced effect on product strength and durability, but has only a minimal effect on the EP leaching characteristics of sludge constituents.

MICROGRAPHIC STUDY OF S/S SAMPLES

The effects of the interferents on the solidified/stabilized sludge were studied using scanning electron microscopy (SEM), energy dispersive x-ray microanalysis (EDXRA), and x-ray diffraction (XRD). Although the use of these methods for solidified/stabilized waste analysis is in its infancy, such analytic techniques have been used in concrete research to study the microscopic interactions between cement and pozzolans and their contained aggregates. A summary of the results of an extensive examination of samples from this study, accomplished by the Hazardous Waste Research Center at Louisiana State University in Baton Rouge, is presented below. A detailed account of the study will be published elsewhere.

The x-ray diffraction analyses of the raw sludge (sludge control) suggest that the major constituents of the sludge were mixed metal hydroxides and calcite. Calcite results from the carbonation reaction with the excess lime used in the precipitation reaction. The x-ray diffraction analysis also indicated that the solidified/stabilized samples were mechanical mixtures, as evidenced by the fact that most of the same XRD peaks which occurred in the raw sludge also appeared in the solidified/stabilized samples. Hydration products of the 0-percent interference (control specimens) were similar to those expected for the binders alone; however, portlandite was uniformly missing from all CFA control samples.

The EDXRA indicated that Cr and Ni were distributed throughout the samples in highly variable amounts. In some cases, discrete ellipsoidal particles a few hundred microns long that were identical in composition to the sludge were found. In other instances, particles of similar morphology contained significant amounts of silicon and aluminum. The distribution of heavy metals in the samples indicated that some respeciation may have taken place in many of the samples.

The SEM photomicrographs showed pronounced changes in morphology in several of the samples. Examples of these differences can be seen in Figures 72-74, which show SEM photomicrographs of the cement binder alone, cement binder plus sludge, and a CEM sample with 5-percent added copper.

The general effects of the interferents are summarized in Table 50 for organic interferents and in Table 51 for inorganic interferents. Some overall comments are given below.

- The raw sludge was primarily composed of calcite plus minor amounts of complex metal hydroxides of Cr and Ni with trace quantities of Cd and Hg.

- The control samples apparently were mechanical mixtures of the sludge and the respective binders.

- Portlandite was absent from most samples containing CFA.

- Organic interferences were much less reactive with the control mixtures than the inorganics.

- TCE volatilized in the sample preparation and was essentially undetectable (at the 5- to 10-percent detection limit of the method).

- Lead nitrate may have inhibited reaction between the sludge and the binders.

- Samples containing copper nitrate, zinc nitrate, sodium hydroxide, or sodium sulfate usually produced new XRD peaks which may represent new reaction products. These four interferents also seemed to decrease binder reaction products in many cases.

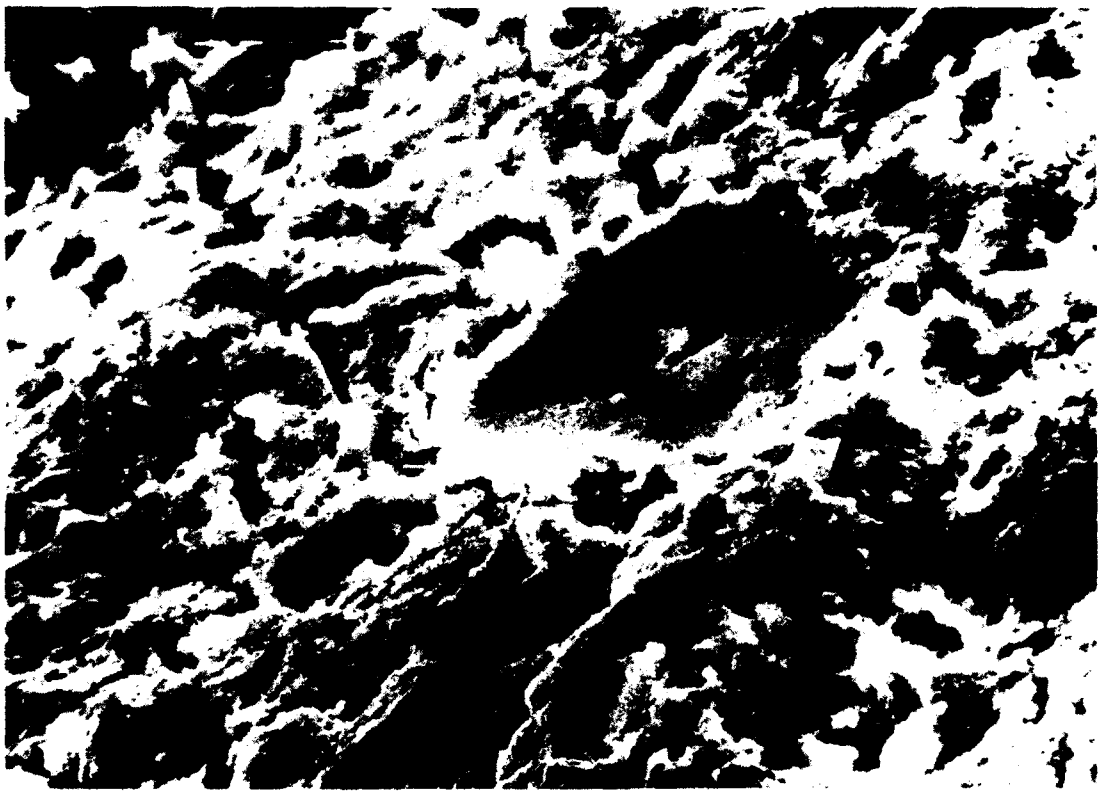

Figure 72. Pure Portland cement with the same water-to-cement ratio as other sludge and interference-containing cement samples. Calcium silicate hydrate (C-S-H) and needles of ettringite are seen. The C-S-H does not have a very well-defined morphology; in some places, however, a crude layering is evident (lower middle). There are also slightly concave, smooth surfaces (center). (1000 × magnification; bar scale at top left is 10 micrometers.)

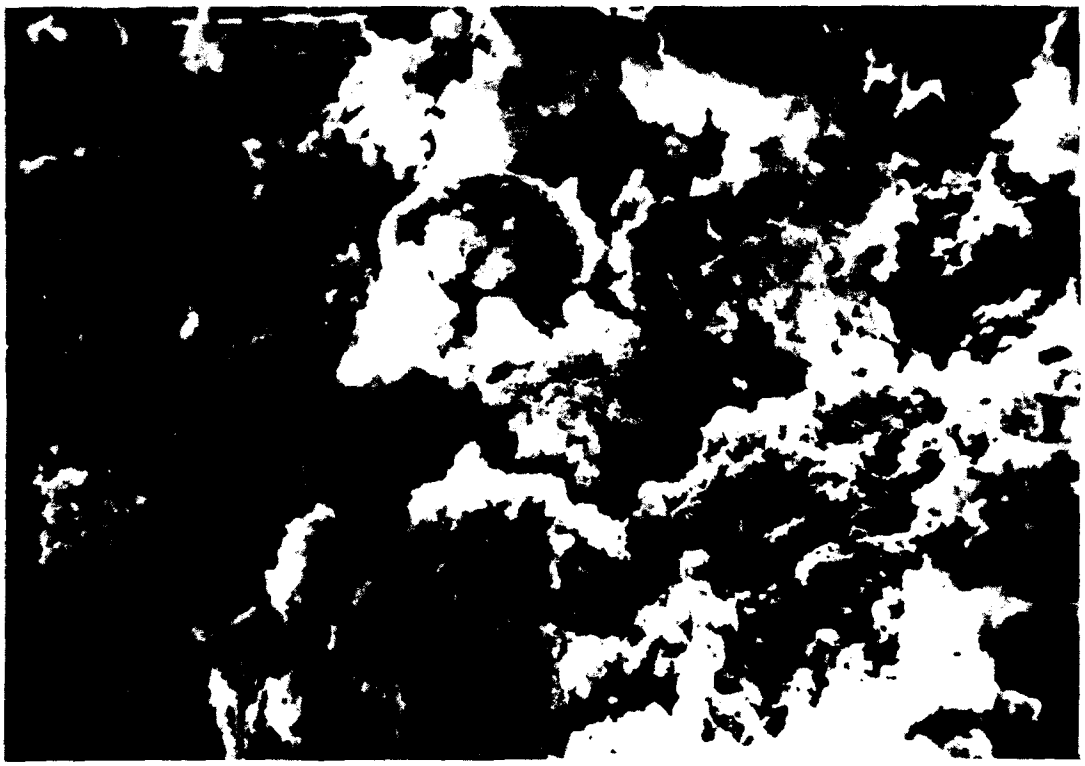

Figure 73. Photomicrograph of a cement sample with the heavy metal sludge. Large spherical to oblate-shaped particles are very common compared to the pure sample. Most of these are unhydrated tri- and di-calcium silicates, but some are sludge particles. The morphology of the C-S-H is also different. The pores and porosity distribution are unlike that of pure cement. (1,000 ×; bar scale at top is 10 micrometers.)

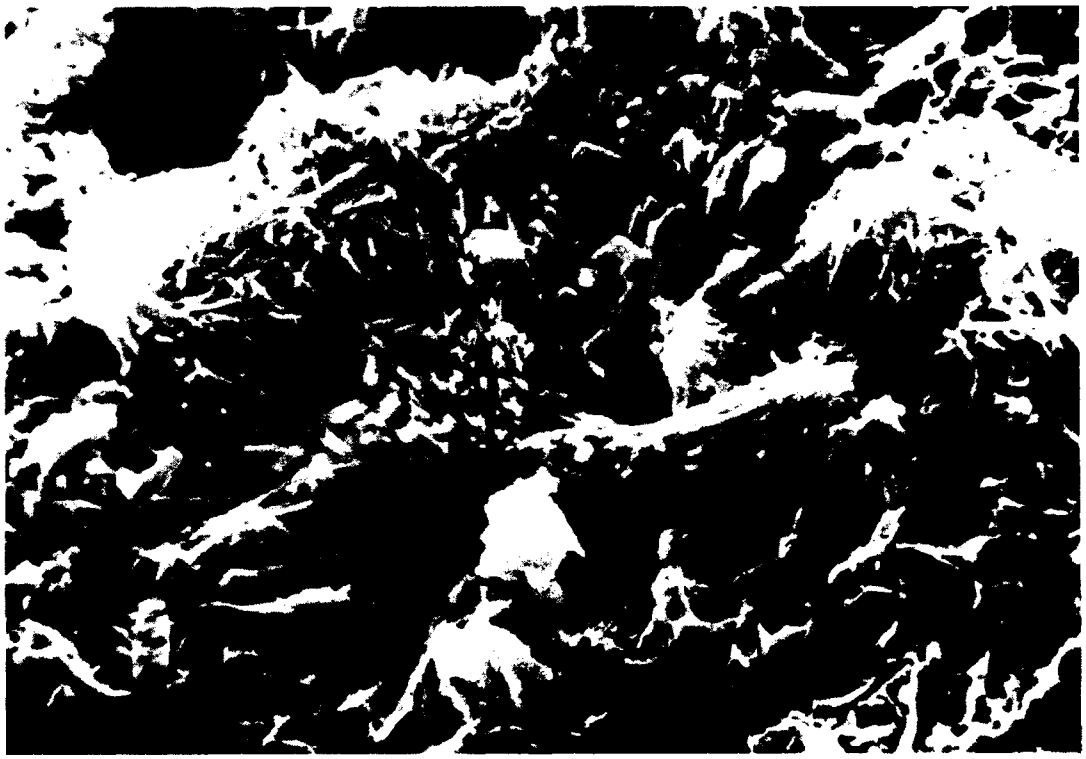

Figure 74. Portland cement sample with the heavy metal sludge and 5-percent copper nitrate as an interferent. The addition of the interference has pronouncedly affected the morphology of the C-S-H. Energy dispersive x-ray microanalysis indicated that the long fibers in the photomicrograph mostly contained calcium and silicon, some minor aluminum, and very little copper. The interference thus appears to act as a catalyst. Other grains with partially developed crystalline faces (lower left), absent in both pure and control cement samples, are also present. (7,000 × magnification; bar scale at top 1 micrometer.)

TABLE 50. GENERAL TRENDS BETWEEN THE ORGANIC INTERFERENCES AND BINDERS

Binder	Grease	Oil	TCE	HCB	PHENOL
CEM	- No effects except calcite decreased as grease increased	- No effects	- No effects - No chlorine found in EDXRA	- Some visible changes as HCB increased - New XRD peaks at high 20 values, but were unidentifiable	- Some visible changes - Porosity increased with phenol - New peak at 6° = 20
CFA	- Ettringite absent in control and 2%, present in 5% and 8%	- Spherical pores increased as oil increased to 5%	- No visible change - Gypsum absent in all	- No effects	- No visible change - Ettringite and calcite only in control
LFA	- No visible change - Ettringite increased in 2%, then decreased in 5% and 8%	- Fly ash reacted less in 5% - No gypsum in 5% and 8%	- Matrix platelets more defined as TCF increased - Porosity decreased as TCE increased - Only on EDXRA showed chlorine - NO XRD trends	- No visible changes - Gypsum only in 8% - HCB XRD peaks absent in 2%, present in 5%, and strong in 8%	- No visible changes - XRD Portlandite peaks inversely proportional to phenol - Ettringite only in control - Gypsum proportional to phenol

TABLE 51. GENERAL TRENDS BETWEEN THE INORGANIC INTERFERENCES AND BINDERS

Binder	Lead Nitrate	Copper Nitrate	Zinc Nitrate	Sodium Hydroxide	Sodium Sulphate
CEM	- Unhydrated calcium silicates proportional to lead nitrate - Portlandite inversely proportional to lead nitrate until absent in 8%	- Minor visible changes - Porosity proportional copper nitrates may be related to plate-like morphology - Portlandite reduced in 2%, absent in 5% and 8% - No calcium aluminate hydrates in copper nitrate samples - More sludge XRD peaks in 8% - New, unidentifiable XRD peaks	- Some visible change - EDXRA suggests an even distribution of zinc nitrate - Portlandite less in 5% and 8% - Ettringite absent in 5% and 8% - No new XRD peaks	- Crystallinity and porosity proportional to NaOh - Visible changes increased with NaOH - Portlandite decreased as NaOH increased	- Visible changes - 8% more porous - EDXRA showed Na, 5, and OPC elements in same spectra - Portlandite up in 2%, then decreased in 5% and 8% - Ettringite absent in 8% - No Na_2 SO_4 XRD peaks - New, unidentifiable peaks
CFA	- Fly ash less reacted in 5% and 8% - New morphology in 8%	- Minor visible changes, - Gypsum inversely proportional to copper nitrate, until absent in 8% - $Cu_2(OH)_3NO_3$ XRD peaks found in 8%	- No visible changes, except porosity higher in the 8% - Portlandite in 5% and (rare for CFA) - Ettringite and gypsum absent in 5% and 89% - New, unidentifiable XRD peaks	- Visible changes in 5% and 8% - Porosity increased in 2% but decreased in 5% and 8% - Crystallinity increased at 2% NaOH - no XRD trends - Strength varied with NaOH content	- Visible changes increased with Na_2 SO_4 - EDXRA; Na and S increased as Na_2 SO_4 increased in fly ash - Gypsum and ettringite increased up to 5% then decreased in 8% - Portlandite in 2% and 5% peaks increased from 5% to 8% - No Na_2 SO_4 XRD peaks
LFA	- Visible changes in 8% - EDXRA: more lead in fly ash than CSH - Gypsum absent in 8% - Portlandite decreased in 2% and absent in 5%	- Minor visible changes - Some evidence, by EDXRA of fly ash, that A1 is proportional to copper nitrate	- Visible changes increased with zinc nitrate - Porosity higher in 8% - EDXRA of fly ash showed same amount of	- Visible changes in 5% and 8% - Gypsum inversely proportional to NaOH, until absent in 8% - Portlandite and ettrin-	- Visible changes increased with Na_2 SO_4 - EDXRA: Na and S increased in fly ash as Na_2 SO_4 increased - Ettringite and gypsum

SECTION 4

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

This study has shown that common components of hazardous wastes interfere with the containment and strength characteristics of solidified/stabilized wastes. All measured parameters were affected by one or more of the interferent materials. Table 52 summarizes the effects of the interferents on the binder systems used in the study. Each interferent was rated as to whether it produced a strong, moderate, or slight positive or negative effect on each of the measured parameters for each binder system. Permeability is not included in the summary table because of the high variability in the results. Bulk density was also omitted from the table because only small differences were observed for this parameter.

Specific conclusions regarding the effects of the interferences were characterized by the test methods used to evaluate the effects, as given below.

UCS, CI, and Wet/Dry

Conclusions based on the UCS, CI, and wet/dry tests are as follows:

(1) The metals, grease and oil, and phenol were generally deleterious to the strength and durability all binder systems. The effects of these interferences generally increase with increasing concentration.

(2) The interferents sulfate, HCB, and TCE had little measurable effect at any concentration.

(3) Sodium hydroxide had mixed effects, increasing early strength (CI) at 2- and 5-percent interference addition, but decreasing early strength at the 8-percent NaOH level.

(4) The strength and durability of the solidified/stabilized products are strongly related, as might be expected.

(5) A highly significant correlation was found between UCS and CI.

Permeability

The results of this study indicate that the permeability measurements do not appear meaningful for the evaluation of solidified/stabilized solid samples due to the difficulty of the test method and the inherent variability of the results. This is unfortunate, as this information is of basic importance to the evaluation of the long-term leaching potential of solidified/stabilized waste forms. New test methods should be developed to give this information.

TABLE 52. SUMMARY OF THE EFFECTS OF INTERFERENTS ON THE SOLIDIFIED/ STABILIZED SAMPLES

Binder	Interferent	UCS	CI	Wet/dry cycles	Final EP pH	EP leachate concentrations Cd	Cr	Hg	Ni
CEM	Copper	+++*	--	---	--	---	---	+	BDL
CFA	Copper	---	ND	---	---	---	---	---	---
LFA	Copper	---	-	---	--	--	--	0	-
CEM	Lead	0	0	-	0	--	-	0	0
CFA	Lead	---	--	---	--	---	0	0	--
LFA	Lead	---	---	---	---	BDL	--	+	BDL
CEM	Zinc	--	---	---	-	-	---	+	--
CFA	Zinc	---	---	---	---	---	+	-	---
LFA	Zinc	---	---	---	---	BDL	--	+	BDL
CEM	Sulfate	-	-	0	0	-	---	0	BDL
CFA	Sulfate	0	0	0	--	---	--	+	0
LFA	Sulfate	+	++	+	--	BDL	---	+	BDL
CEM	NaOH	+	--	---	+	0	---	0	+
CFA	NaOH	-	++	0	++	--	---	+	0
LFA	NaOH	0	+++	-	0	-	--	0	+
CEM	Grease	--	---	---	0	BDL	0	0	0
CFA	Grease	--	--	0	+	++	+	+	+
LFA	Grease	--	-	--	0	--	0	0	0
CEM	Oil	--	--	---	0	-	0	+	0
CFA	Oil	-	-	---	0	-	-	+++	0
LFA	Oil	-	-	---	0	0	-	+++	0
CEM	HCB	0	0	-	0	++	+	0	0
CFA	HCB	0	0	0	0	+	+++	0	+
LFA	HCB	0	+	0	0	-	-	-	-
CEM	TCE	-	-	0	0	0	0	+	0
CFA	TCE	-	-	0	0	-	-	+	0
LFA	TCE	+	0	0	0	-	0	0	+
CEM	Phenol	-	---	---	-	-	0	+	0
CFA	Phenol	---	---	0	+	+	++	0	0
LFA	Phenol	---	---	---	0	+++	0	0	0

Notes: ND = not detected; BDL = below the detection limit. A key to the symbols is given below.

* Symbol	UCS and CI (number times the control)	Wet/Dry (cycles intact vs. control)	Final EP pH	EP Leachate Conc. (number times the control level)
+++	>2 times control	+5 cycles	>+2 units	Less than 0.1 times
++	1.5 to 2.0 times	+2 to 5	+1 to 2	0.25 to 0.1 times
+	1.2 to 1.5 times	+1 to 2	+0.3 to 1	0.5 to 0.25 times
0	0.8 to 1.2 times	-1 to +1	-0.3 to 0.3	2 to 0.5 times
-	0.8 to 0.66 times	-1 to -2	-0.3 to -1	2 to 4 times
--	0.66 to 0.5 times	-2 to -5	-1 to -2	4 to 10 times
---	<0.5 times	-5 cycles	>-2 units	More than 10 times

Bulk Density

Significant volume changes with increasing interference concentrations were observed for the phenol, sulfate, and copper interferents. Although these volume changes were significant, generally these changes were less than 10 percent.

Contaminant Leaching

Conclusions based on the Extraction Procedure are as follows:

(1) The final pH of the EP leachates was lowered appreciably by the metal nitrate and sulfate interferents.

(2) The NaOH addition raised the final pH, as would be expected.

(3) The organic interferents had little effect on the final EP pH.

(4) The changes in final EP leachate pH correlate with the EP leaching losses of Cd and Cr, and to some extent Ni. The propensity of these metals to leach from the solidified/stabilized waste materials was indirectly proportional to the final EP leachate pH.

(5) Generally, the organic interferents did not appreciably affect the leaching of the metals although HCB and phenol appeared to slightly reduce the leaching rates of Cd and Cr and increase the leaching rate of Hg.

(6) Mercury concentrations in the EP leachates were independent not only of the added interferents, but also of the type of binder. Mercury was leached from the solidified/stabilized products at about the same levels as from the raw sludge. Only the addition of oil as an interferent appeared to decrease the concentration of Hg in the EP leachates, and HCB and phenol increased it.

Microchemical/Micromorphological Examinations

No definitive conclusions can be drawn from the results of the microchemical/micromorphological examinations. More work needs to be performed to perfect these methods as a diagnostic tool for hazardous waste evaluation.

RECOMMENDATIONS

Specific objectives were outlined by this study. In addressing these objectives, several items for additional consideration were uncovered. With regard to these issues, the following recommendations are made:

(1) Since the effect of waste constituents on the integrity of the final product cannot be predicted from current knowledge, it is recommended that tests of strength, durability, and leachability be required of each solidified/stabilized waste before disposal.

(2) Cone index measurements are recommended as a rapid and inexpensive method to estimate early strength of solidified/stabilized waste.

(3) The variability associated with the permeability measurements of low-permeability solids needs to be addressed. Alternative measurements that address attributes such as connected pore volume or gas permeation may be of greater value for highly impermeable materials.

(4) Additional studies addressing the long-term durability of solidified/stabilized materials must be conducted. These studies should include at least a small portion of the samples evaluated by this study. This will provide a basis for correlating the short-term testing with long-term treatment success.

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APPENDIX A

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APPENDIX B

QUALITY ASSURANCE AND QUALITY CONTROL DATA

TABLE B-1. EXTERNAL METHOD BLANKS SAMPLES

Binder	Interferent	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference* (mg/l)
CEM	Grease	<0.030	0.009	0.003	<0.03	<5
CEM	Oil	0.0006	0.01	<0.0008	<0.03	<5
CEM	NaOH	0.0009	0.004	0.0019	<0.03	0.4
CEM	Lead	0.0016	0.005	0.0053	<0.03	0.019
CEM	Phenol	0.001	0.005	<0.0008	<0.03	NA**
CEM	HCB	0.01	0.011	<0.002	<0.03	0.00023
CEM	TCE	0.0039	0.012	<0.0004	<0.03	<0.005
CEM	Zinc	0.022	0.028	<0.002	0.028	0.377
CEM	Copper	0.0106	0.039	<0.002	0.042	0.187
CEM	Sulfate	0.0165	0.018	<0.02	0.021	2.19
CFA	Grease	0.0006	<0.030	<0.008	<0.03	<5
CFA	Oil	0.0001	<0.030	0.0077	<0.030	<9
CFA	NaOH	0.0012	<0.030	0.046	<0.03	1.34
CFA	Lead	0.0008	<0.030	<0.0002	<0.03	0.0004
CFA	Phenol	0.0004	<0.03	<0.04	<0.03	0.054
CFA	HCB	0.0018	<0.02	<0.02	<0.001	0.0031
CFA	TCE	0.0012	<0.001	<0.0008	<0.03	<0.005
CFA	Zinc	0.05	0.01	<0.002	0.009	2.54
CFA	Copper	0.0025	0.006	0.009	0.036	<0.0008
CFA	Sulfate	0.05	0.01	<0.002	0.009	2.54
LFA	Grease	0.0082	<0.001	<0.0002	<0.03	<9
LFA	Oil	0.002	0.007	<0.0004	<0.003	<9
LFA	NaOH	0.0013	0.002	<0.0008	0.01	0.863
LFA	Lead	0.002	0.001	<0.0004	<0.03	0.136
LFA	Phenol	0.0017	0.006	<0.002	0.008	<1
LFA	HCB	0.0005	0.004	<0.002	0.021	0.0031
LFA	TCE	0.0007	0.002	<0.0002	0.004	<0.005
LFA	Zinc	<0.0001	0.001	<0.0002	0.006	0.058
LFA	Copper	<0.0001	0.008	<0.0002	0.002	0.008
LFA	Sulfate	0.0045	0.004	<0.0002	0.004	0.017

* Interference chemicals of analysis are listed in Table B-7.
** Not submitted.

TABLE B-2. EXTERNAL STANDARDS

Binder	Interferent	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference* (mg/l)
CEM	Grease**	NA†	NA	NA	NA	NA
		NA	NA	NA	NA	NA
CEM	Oil	NA	NA	NA	NA	NA
		NA	NA	NA	NA	NA
CEM	NaOH	NA	NA	NA	NA	NA
		NA	NA	NA	NA	NA
CEM	Pb	NA	NA	NA	NA	NA
		NA	NA	NA	NA	NA
CEM	Phenol	NA	NA	NA	NA	NA
		NA	NA	NA	NA	NA
CEM	HCB	NA	NA	NA	NA	NA
		NA	NA	NA	NA	NA
CEM	TCE	1.3	6.54	0.246	112.4	NA
		1.28	6.46	0.306	40.76	NA
CEM	Zn	0.0335	0.254	<0.002	0.21	0.397
		0.0364	0.258	0.0085	0.206	0.41
CEM	Cu	<0.0001	0.006	0.011	0.009	<0.002
		0.0087	0.007	0.0173	0.009	0.0069
CEM	Na_2SO_4	0.0024	<0.001	<0.002	<0.001	<0.05
		0.0135	0.0007	0.0007	0.00173	NA
CFA	Grease	0.059	0.305	0.0045	0.163	NA
		0.059	0.304	0.0076	0.165	NA
CFA	Oil	0.048	0.314	0.0069	0.176	NA
		0.059	0.304	0.0076	0.165	NA
CFA	NaOH	0.061	0.309	0.021	0.159	NA
		0.059	0.304	0.0076	0.165	NA
CFA	Pb	0.0035	0.0041	0.099	NA	0.78
		0.00431	0.0042	0.004	NA	0.038
CFA	Phenol	0.0021	0.044	0.032	0.106	NA
		0.0043	0.042	0.04	0.1	NA
CFA	HCB	1.28	6.4	0.257	NA	NA
		1.28	6.46	0.306	NA	NA
CFA	TCE	1.29	6.69	0.294	NA	NA
		1.28	6.46	0.306	NA	NA

(Continued)

* Interference chemicals of analysis are listed in Table B-7.

** For each interferent, the pair of values shown represents the concentration (upper entry) and the true value (lower entry).

† Not submitted.

TABLE B-2 (Concluded)

Binder	Interferent	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CFA	Zn	0.0008	0.006	<0.0008	0.411	0.257
		0.00193	0.0092	0.00103	0.421	0.29
CEM	Sulfate	NA	NA	NA	NA	NA
		NA	NA	NA	NA	NA
CFA	Cu	0.0011	0.008	NA	0.012	NA
		0.0087	0.007	NA	0.017	NA
LFA	Grease	0.608	3.24	0.076	NA	NA
		0.72	1.29	0.034	NA	NA
LFA	Oil	1.3	6.7	NA	NA	NA
		1.28	6.46	NA	NA	NA
LFA	NaOH	1.32	6.7	0.214	NA	NA
		1.28	6.46	0.306	NA	NA
LFA	Pb	NA	NA	NA	0.097	NA
		NA	NA	NA	0.034	NA
LFA	Phenol	0.0105	0.449	<0.002	0.208	NA
		0.0369	0.258	0.0085	0.206	NA
LFA	HCB	0.031	0.255	NA	0.195	NA
		0.0369	0.258	NA	0.206	NA
LFA	TCE	0.0323	0.248	NA	0.198	NA
		0.0369	0.258	NA	0.206	NA
LFA	Zn	0.0212	0.254	0.0067	0.203	0.4
		0.0369	0.258	0.0085	0.205	0.41
LFA	Cu	NA	NA	0.205	0.002	NA
		NA	NA	0.335	0.0085	NA
LFA	Sulfate	0.025	0.259	<0.0002	0.203	0.39
		0.0369	0.258	0.0085	0.205	NA

TABLE B-3. EXTERNAL DUPLICATE SAMPLES

Binder	Interferent		Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference* (mg/l)
CEM	TCE	0%	0.0015	0.0150	0.5330	0.0850	0.00250
		0%	0.0029	0.0100	0.4920	0.1000	0.00530
		2%	0.0013	0.0140	1.5900	0.0860	0.01500
		2%	0.0023	0.0220	2.3400	0.0940	0.01800
		5%	0.0013	0.0120	1.2700	0.0900	0.01300
		5%	0.0013	0.0140	1.3500	0.0910	0.00940
		8%	0.0018	0.0120	1.3500	0.0910	0.00670
		8%	0.0019	0.0140	1.2300	0.0910	0.00450
CEM	Phenol	0%	0.0010	0.0070	2.3100	0.0730	34.8
		0%	0.0016	0.0090	1.9700	0.0660	33.3
		2%	0.0011	0.0130	6.2300	0.0690	749.
		2%	0.0016	0.0070	6.9300	0.0590	744.
		5%	0.0018	0.0110	7.2600	0.0660	1490.
		5%	0.0100	0.0100	5.8600	0.0830	1650.
		8%	0.0020	0.0100	7.3400	0.0690	2820.
		8%	0.0026	0.0120	7.0100	0.0770	2600.
CEM	NaOH	0%	0.0024	0.0090	0.1550	0.0760	96.4
		0%	0.0018	0.0130	0.1570	0.0750	162.
		2%	0.0019	0.0110	0.1570	0.0630	620.
		2%	0.0008	0.0120	0.1550	0.0610	612.
		5%	0.0012	0.0720	0.1570	0.0410	1470.
		5%	0.0045	0.0680	0.1570	0.0490	1390.
		8%	0.0016	0.3660	0.1580	0.0390	1880.
		8%	0.0026	0.3640	0.1570	0.0430	1790.
CEM	HCB	0%	0.0189	0.0300	0.9430	0.1260	0.03170
		0%	0.0050	0.0240	0.9840	0.1260	0.00070
		2%	0.0007	0.0100	1.0700	0.1050	0.00077
		2%	0.0017	0.0140	1.0200	0.1000	0.00150
		5%	0.0020	0.0140	0.9020	0.1010	0.00050
		5%	0.0027	0.0150	1.0700	0.1140	0.00500
		8%	0.0033	0.0060	0.9840	0.1080	0.00630
		8%	0.0041	0.0120	0.5330	0.1160	0.00087
CEM	Grease	0%	0.0300	0.0240	0.3070	0.0910	5.
		0%	0.0300	0.0160	0.2980	0.0890	5.

(Continued)

* Interference chemicals of analysis are listed in Table B-7.

** For each interferent, the pair of values shown represents the concentration (upper entry) and the true value (lower entry).

† Not submitted.

(Sheet 1 of 7)

TABLE B-3 (Continued)

Binder	Interferent		Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CEM	Grease	2%	0.0300	0.0100	0.3010	0.0870	5.
	(Cont.)	2%	0.0300	0.0150	0.2980	0.0870	5.
		5%	0.0300	0.0250	0.2980	0.0880	5.
		5%	0.0300	0.0160	0.2950	0.0820	5.
		8%	0.0300	0.0110	0.3160	0.0830	5.
		8%	0.0300	0.0120	0.3010	0.0790	5.
CEM	Oil	0%	0.0008	0.0150	0.0057	0.0830	5.
		0%	0.0008	0.0150	0.0057	0.0830	5.
		2%	0.0009	0.0140	0.0010	0.0930	5.
		2%	0.0023	0.0500	0.0010	0.0870	5.
		5%	0.0002	0.0110	0.0018	0.0980	5.
		5%	0.0011	0.0090	0.0021	0.0890	5.
		8%	0.0022	0.0120	0.0008	0.0810	5.
		8%	0.0015	0.0100	0.0016	0.0920	5.
CEM	Pb	0%	0.0049	0.0110	0.3140	0.1120	0.01600
		0%	0.0018	0.0060	0.2780	0.0910	0.00900
		2%	0.0011	0.0040	0.2960	0.1000	3.37000
		2%	0.0010	0.0060	0.2960	0.0960	4.02000
		5%	0.0008	0.0060	0.3020	0.1010	11.90000
		5%	0.0008	0.0100	0.3000	0.1000	6.05000
		8%	0.0004	0.0060	0.3020	0.1040	11.30000
		8%	0.0005	0.0080	0.3020	0.1010	5.05000
CEM	Zn	0%	0.0012	0.0070	0.9220	0.0070	0.03000
		0%	0.0002	0.0080	0.9784	0.0080	0.03000
		2%	0.0004	0.0180	0.9780	0.0180	0.10900
		2%	0.0006	0.0140	1.0800	0.0140	0.15500
		5%	0.0012	0.1290	0.4840	0.0370	0.08100
		5%	0.0012	0.1350	0.5450	0.0350	0.10200
		8%	0.0001	0.0890	0.5310	0.0370	0.14600
		8%	0.0052	0.0670	0.6980	0.0340	0.22000
CEM	Cu	0%	0.0002	0.0030	0.7490	0.0010	0.00200
		0%	0.0001	0.0040	0.4790	0.0010	0.00200
		2%	0.0003	0.0320	0.4510	0.0040	0.16700
		2%	0.0001	0.0300	0.4900	0.0010	0.16400
		5%	0.0107	0.0340	0.3240	0.0010	0.00900
		5%	0.0580	0.0180	0.2520	0.0010	0.00900
		8%	0.0650	0.0090	0.5240	0.0010	0.01000
		8%	0.1460	0.0090	0.4390	0.0010	0.00900

(Continued)

(Sheet 2 of 7)

TABLE B-3 (Continued)

Binder	Interferent		Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CEM	Sulfate	0%	0.0020	0.0070	1.0500	0.0030	10.9
		0%	0.0029	0.0070	0.9780	0.0010	10.3
		2%	0.0065	0.0090	1.0700	0.0010	482.
		2%	0.0063	0.0080	0.9500	0.0010	513.
		5%	0.0058	0.0970	0.8660	0.0010	1140.
		5%	0.0071	0.0930	0.9640	0.0010	1140.
		8%	0.0023	0.2980	0.4190	0.0010	1810.
		8%	0.0001	0.1000	0.8940	0.0010	1240.
CFA	TCE	0%	0.0056	0.0320	1.6000	0.0550	0.00650
		0%	0.0032	0.0130	1.7600	0.0590	0.00490
		2%	0.0148	0.0650	0.5460	0.0610	0.04400
		2%	0.0064	0.0410	0.6300	0.0560	0.02500
		5%	0.0052	0.0350	0.3780	0.0540	0.03900
		5%	0.0212	0.1490	0.2940	0.0620	0.03500
		8%	0.0036	0.0120	0.5040	0.0540	0.05700
		8%	0.0052	0.0320	0.3780	0.0550	0.06400
CFA	Phenol	0%	0.0269	0.1590	2.5200	0.0790	62.8
		0%	0.0110	0.1190	2.5600	0.0710	68.
		2%	0.0053	0.0580	7.2600	0.0720	682.
		2%	0.0080	0.0660	7.2000	0.0760	465.
		5%	0.0102	0.0340	4.7400	0.0740	1280.
		5%	0.0067	0.0310	2.7700	0.0720	1340.
		8%	0.0166	0.0320	4.8900	0.0780	2320.
		8%	0.0079	0.0300	2.8300	0.0760	2380.
CFA	NaOH	0%	0.1230	0.2020	0.2850	0.1170	64.
		0%	0.1180	0.2880	0.2930	0.1090	91.5
		2%	0.0860	0.1730	0.1790	0.0860	699.
		2%	0.0880	0.1640	0.1370	0.0850	706.
		5%	0.0116	0.2160	0.2390	0.0570	1270.
		5%	0.0033	0.2080	0.2740	0.0610	1330.
		8%	0.0002	0.3770	0.3120	0.0300	1850.
		8%	0.0003	0.4320	0.3120	0.0310	2010.
CFA	HCB	0%	2.3700	5.8200	2.6100	0.3680	0.00180
		0%	0.0810	0.0540	2.5100	0.0710	0.00150
		2%	0.1130	0.1090	1.8100	0.0790	0.00320
		2%	0.0263	0.0560	2.2100	0.0740	0.00140

(Continued)

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TABLE B-3 (Continued)

Binder	Interferent		Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CFA	HCB	5%	0.3760	0.1690	1.7200	0.0800	0.00490
	(Cont.)	5%	0.6480	0.2220	1.6000	0.0960	0.01380
		8%	0.2480	0.1310	2.2000	0.0790	0.01360
		8%	0.4340	0.1320	2.0200	0.0840	0.00610
CFA	Grease	0%	0.1010	0.1140	0.2770	0.0810	5.
		0%	0.0820	0.1470	0.3210	0.0830	5.
		2%	0.3210	0.1940	0.1000	0.0930	5.
		2%	0.1790	0.1360	0.1000	0.0840	5.
		5%	0.0060	0.0920	0.1550	0.0810	5.
		5%	0.0341	0.0810	0.1660	0.0850	5.
		8%	0.0298	0.0830	0.1410	0.0840	5.
		8%	0.0018	0.1070	0.1350	0.0880	5.
CFA	Oil	0%	0.0020	0.0350	1.2800	0.0600	9.
		0%	0.0030	0.0410	0.0860	0.0600	9.
		2%	0.0052	0.0530	0.0507	0.0610	9.
		2%	0.0026	0.0350	0.0791	0.0570	9.
		5%	0.0139	0.1080	0.0017	0.0620	9.
		5%	0.0184	0.1210	0.0017	0.0650	9.
		8%	0.0034	0.0550	0.1180	0.0650	9.
		8%	0.0136	0.1120	0.0084	0.0830	9.
CFA	Pb	0%	0.0240	0.1800	0.2730	0.1250	114.
		0%	0.0068	0.0500	0.2340	0.0920	10.9
		2%	0.0017	0.0780	0.3120	0.0940	9.33000
		2%	0.0137	0.1680	0.3120	0.0890	11.
		5%	0.0650	0.0400	0.2730	0.1170	10.40000
		5%	0.0910	0.0480	0.3530	0.1190	11.60000
		8%	12.2000	0.3360	0.1950	3.5200	6.11000
		8%	5.3000	0.0550	0.2340	1.0500	1.97000
CFA	Zn	0%	0.0152	0.0320	0.0920	0.0030	0.03000
		0%	0.0022	0.0290	0.1260	0.0070	0.03000
		2%	0.0009	0.0880	0.2700	0.0010	0.03000
		2%	0.0007	0.0960	0.2490	0.0010	0.03000
		5%	49.6000	0.0110	0.2060	5.2800	1480.
		5%	47.6000	0.0280	0.2120	2.9400	283.
		8%	59.7000	0.0080	0.2430	5.7400	1670.
		8%	61.4000	0.0110	0.2550	5.7000	1680.
CFA	Cu	0%	0.0635	0.0530	0.1260	0.0020	0.00300
		0%	0.0105	0.0290	0.1260	0.0010	0.00200

(Continued)

(Sheet 4 of 7)

TABLE B-3 (Continued)

Binder	Interferent		Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CFA	Cu	2%	0.2570	0.0380	1.8300	0.0050	0.00500
	(Cont.)	2%	0.1530	0.0550	0.1580	0.0010	0.00400
		5%	54.0000	0.0120	0.2520	1.1300	515.
		5%	45.4000	0.0080	0.1360	0.8580	109.
		8%	58.1000	0.4680	0.2760	1.6000	1080.
		8%	54.0000	0.8690	0.2670	1.7400	1230.
CFA	Sulfate	0%	0.0367	0.0340	0.2270	0.0030	37.8
		0%	0.0880	0.0480	0.1870	0.0220	31.4
		2%	0.0890	0.0930	0.1540	0.0030	310.
		2%	0.0920	0.1070	0.1610	0.0140	311.
		5%	0.4490	0.2190	0.0760	0.0100	1140.
		5%	2.4900	0.2110	0.0730	0.0710	1040.
		8%	1.4900	0.1950	0.0450	0.0480	1790.
		8%	0.4480	0.1900	0.0570	0.0080	1780.
LFA	TCE	0%	0.0002	0.0100	1.2100	0.0060	0.00500
		0%	0.0036	0.0040	0.6430	0.0130	0.00500
		2%	0.0039	0.0040	1.0000	0.0040	0.02500
		2%	0.0044	0.0040	1.5000	0.0060	0.02700
		5%	0.0005	0.0050	0.6780	0.0040	0.03500
		5%	0.0021	0.0070	0.9640	0.0020	0.05500
		8%	0.0006	0.0040	0.7500	0.0010	0.09900
		8%	0.0004	0.0050	1.0700	0.0010	0.15000
LFA	Phenol	0%	0.0005	0.0060	1.5100	0.0070	29.4
		0%	0.0095	0.0060	1.8100	0.0080	31.7
		2%	0.0003	0.0060	3.6700	0.0080	637.
		2%	0.0003	0.0070	4.4200	0.0030	754.
		5%	0.0004	0.0080	2.3700	0.0050	1720.
		5%	0.0001	0.0030	2.2100	0.0050	1560.
		8%	0.0001	0.0060	3.3000	0.0080	2700.
		8%	0.0001	0.0070	2.1100	0.0120	2600.
LFA	NaOH	0%	0.0003	0.0040	0.3400	0.0110	151.
		0%	0.0014	0.0040	0.3190	0.0150	233.
		2%	0.0036	0.0040	0.3120	0.0050	673.
		2%	0.0028	0.0260	0.3170	0.0070	662.
		5%	0.0022	0.0080	0.3310	0.0060	1160.
		5%	0.0037	0.0080	0.3000	0.0070	1070.
		8%	0.0024	0.0580	0.3340	0.0050	1540.
		8%	0.0028	0.0420	0.3120	0.0070	1500.

(Continued)

(Sheet 5 of 7)

TABLE B-3 (Continued)

Binder	Interferent		Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
LFA	HCB	0%	0.0001	0.0060	0.3110	0.0040	0.00004
		0%	0.0001	0.0060	0.2700	0.0040	0.00030
		2%	0.0001	0.1150	0.4750	0.1470	0.03600
		2%	0.0007	0.0070	0.4780	0.0040	0.00130
		5%	0.0004	0.0050	0.3490	0.0020	0.00050
		5%	0.0001	0.0050	0.3700	0.0030	0.00360
		8%	0.0001	0.0050	0.3230	0.0020	0.00340
		8%	0.0002	0.0060	0.2380	0.0040	0.00140
LFA	Grease	0%	0.0012	0.0110	0.3340	0.0550	9.
		0%	0.0039	0.0100	0.3250	0.0600	9.
		2%	0.0136	0.0050	0.3190	0.0550	9.
		2%	0.0106	0.0070	0.2940	0.0560	9.
		5%	0.0074	0.0020	0.2760	0.0510	9.
		5%	0.0202	0.0020	0.2730	0.0560	9.
		8%	0.0086	0.0020	0.2820	0.0540	9.
		8%	0.0080	0.0300	0.2570	0.0820	9.
LFA	Oil	0%	0.0030	0.0090	0.1450	0.0480	9.
		0%	0.0038	0.0070	0.1900	0.0460	9.
		2%	0.0044	0.0160	0.0049	0.0520	9.
		2%	0.0034	0.0220	0.0066	0.0522	9.
		5%	0.0022	0.0120	0.0008	0.0740	9.
		5%	0.0110	0.0080	0.0008	0.0820	9.
		8%	0.0028	0.0050	0.0008	0.0680	9.
		8%	0.0034	0.0130	0.0008	0.0670	9.
LFA	Pb	0%	0.0006	0.0008	0.5820	0.0800	0.12500
		0%	0.0012	0.0060	0.6680	0.0870	0.27500
		2%	0.0030	0.0110	0.7540	0.0850	90.4
		2%	0.0012	0.0090	0.7670	0.0880	50.9
		5%	0.0256	0.0080	0.7790	0.1070	627.
		5%	0.0132	0.0100	0.8340	0.1010	316.
		8%	0.0126	0.0100	0.6500	0.1070	385.
		8%	0.0100	0.0070	0.7670	0.1010	466.
LFA	Zn	0%	0.0001	0.0060	0.7500	0.0010	0.03000
		0%	0.0007	0.0090	1.0500	0.0010	0.03000
		2%	0.0001	0.0110	0.7870	0.0010	0.52700
		2%	0.0001	0.0100	0.8750	0.0010	0.95700
		5%	0.0001	0.0210	0.4500	0.0010	0.24300
		5%	0.0001	0.0130	0.5370	0.0010	0.30600

(Continued)

(Sheet 6 of 7)

TABLE B-3 (Concluded)

Binder	Interferent		Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
LFA	Zn	8%	0.0001	0.0520	0.2930	0.0010	0.10700
	(Cont.)	8%	0.0001	0.0550	0.3470	0.0010	0.12500
LFA	Cu	0%	0.0001	0.0030	0.8370	0.0010	0.00200
		0%	0.0001	0.0030	0.6870	0.0010	0.00200
		2%	0.0001	0.0080	1.0500	0.0010	0.28200
		2%	0.0001	0.0080	1.1700	0.0010	0.36400
		5%	0.0001	0.0150	0.4870	0.0020	0.04700
		5%	0.0001	0.0130	0.5250	0.0020	0.04200
		8%	0.0026	0.0160	0.2380	0.0010	0.01000
		8%	0.0040	0.0130	0.1720	0.0060	0.05600
LFA	Sulfate	0%	0.0001	0.0070	0.9720	0.0010	83.4
		0%	0.0001	0.0070	0.9280	0.0010	69.6
		2%	0.0001	0.0060	0.7560	0.0010	622.
		2%	0.0001	0.0050	0.3960	0.0010	612.
		5%	0.0001	0.0060	0.4680	0.0010	966.
		5%	0.0001	0.0060	0.7200	0.0010	1020.
		8%	0.0001	0.0960	0.0002	0.0030	1470.
		8%	0.0001	0.0860	0.0002	0.0040	1770.

(Sheet 7 of 7)

TABLE B-4. INTERNAL SPLIT SAMPLES

Binder	Interferent	Sample*	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CEM	Grease	A	<0.03	0.024	0.307	0.091	NA**
		A-DUP	NA	NA	0.2	0.088	NA
		B	<0.03	0.012	0.301	0.079	NA
		B-DUP	NA	NA	0.338	NA	NA
CEM	Oil	A	0.0008	0.015	0.0057	0.083	219
		A-DUP	NA	NA	0.0058	0.086	220
		B	0.0006	0.01	<0.0008	<0.030	0.159
		B-DUP	NA	NA	<0.0020	NA	NA
CEM	NaOH	A	0.0024	0.009	0.155	0.076	NA
		A-DUP	NA	NA	0.169	0.08	NA
		B	0.0009	0.004	0.0019	<0.03	0.4
		B-DUP	NA	NA	0.002	NA	NA
CEM	Pb	A	0.0049	0.011	0.314	0.112	0.016
		A-DUP	NA	NA	0.317	0.11	NA
		B	0.0016	0.005	0.0053	<0.03	0.019
		B-DUP	NA	NA	0.0065	NA	NA
CEM	Phenol	A	0.003	0.007	2.31	0.073	NA
		A-DUP	NA	NA	NA	0.068	NA
		B	0.001	0.005	<0.0008	<0.03	NA
		B-DUP	0.0011	0.004	<0.002	NA	NA
CEM	HCB	A	0.0041	0.012	0.533	0.166	NA
		A-DUP	NA	NA	0.492	0.110	NA
CEM	TCE	A	0.0019	0.014	1.23	0.091	NA
		A-DUP	NA	NA	1.15	0.094	NA
		B	0.0039	0.012	<0.0004	<0.03	NA
		B-DUP	NA	NA	<0.0008	NA	NA
CEM	Zn	A	0.0002	0.008	0.978	0.008	<0.03
		A-DUP	NA	NA	0.95	NA	NA
		B	0.0052	0.067	0.698	0.034	0.22
		B-DUP	NA	0.062	0.698	0.032	0.218
CEM	Cu	A	0.0002	0.003	0.749	<0.001	0.002
		A-DUP	NA	NA	0.755	NA	NA
		B	0.065	0.009	0.524	<0.001	0.01
		B-DUP	NA	NA	0.529	NA	NA
CEM	Na_2SO_4	A	0.002	0.007	1.05	0.003	10.9
		A-DUP	NA	NA	1.06	NA	10.5

(Continued)

* Interference chemicals of analysis are listed in Table B-7.
** Split not analyzed.

(Sheet 1 of 4)

TABLE B-4 (Continued)

Binder	Interferent	Sample*	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CEM	Na_2SO_4 (Cont.)	B	0.0023	0.298	0.419	<0.001	1810
		B-DUP	NA	0.304	0.408	NA	NA
CFA	Grease	A	0.082	0.147	0.321	0.083	NA
		A-DUP	0.078	0.142	0.31	0.086	NA
		B	0.059	0.305	0.0045	0.163	NA
		B-DUP	0.056	0.3	0.005	0.164	NA
CFA	Oil	A	0.002	0.035	0.154	0.06	NA
		A-DUP	NA	0.036	0.144	0.064	NA
		B	0.0001	<0.03	0.0077	<0.03	NA
		B-DUP	NA	NA	0.106	NA	NA
		C	0.048	0.314	0.0069	0.176	NA
		C-DUP	0.052	0.316	NA	0.176	NA
CFA	NaOH	A	0.118	0.288	0.293	0.109	91.5
		A-DUP	0.12	0.282	0.281	0.102	91
		B	0.0012	<0.03	0.046	<0.03	1.34
		B-DUP	NA	NA	0.046	NA	NA
		C	0.061	0.309	0.021	0.159	0.133
		C-DUP	0.062	0.318	NA	0.164	NA
CFA	Pb	A	0.024	0.18	0.125	114	0.273
		A-DUP	0.176	NA	0.122	NA	0.234
		B	0.0008	<0.03	<0.03	0.0004	<0.0002
		B-DUP	NA	NA	NA	NA	<0.0002
		C	0.0035	0.0041	0.099	0.053	0.78
		C-DUP	NA	0.0045	0.1	NA	NA
CFA	Phenol	A	0.011	0.119	2.56	0.071	NA
		A-DUP	NA	0.116	2.44	0.074	NA
CFA	HCB	A	0.081	0.054	2.51	0.0071	NA
		A-DUP	0.088	0.05	2.47	0.076	NA
		B	0.248	0.131	2.2	0.079	NA
		B-DUP	0.252	0.133	NA	0.082	NA
CFA	TCE	A	0.056	0.032	1.6	0.055	NA
		A-DUP	NA	0.031	NA	0.058	NA
		B	0.0052	0.032	0.378	0.055	NA
		B-DUP	0.0054	0.032	0.336	0.058	NA
		C	0.0012	<0.001	<0.0008	<0.03	NA
		C-DUP	0.0012	<0.001	<0.002	NA	NA
CFA	Zn	A	0.0022	0.029	0.126	0.007	<0.03
		A-DUP	NA	NA	0.182	NA	NA

(Continued)

(Sheet 2 of 4)

TABLE B-4 (Continued)

Binder	Interferent	Sample*	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
CFA	Zinc (Cont.)	B	61.4	0.011	0.255	5.7	1680
		B-DUP	62.7	NA	0.23	5.68	1680
CFA	Cu	A	0.0105	0.029	0.001	0.002	0.126
		A-DUP	NA	NA	NA	NA	0.158
		B	54	0.869	1.74	1230	0.267
		B-DUP	54.5	0.874	1.71	1230	NA
CFA	Na_2SO_4	A	0.0367	0.034	0.227	0.003	37.8
		A-DUP	NA	0.036	0.22	NA	36.9
		B	1.49	0.195	0.045	0.04	1790
		B-DUP	1.52	0.189	0.039	0.048	1780
LFA	Grease	A	0.012	0.011	0.334	0.055	NA
		A-DUP	NA	NA	NA	0.054	NA
		B	0.008	0.03	0.257	0.082	NA
		B-DUP	0.0092	0.028	NA	0.084	NA
		C	0.0082	<0.001	<0.0008	<0.030	NA
		C-DUP	0.01	<0.001	<0.0002	NA	NA
		D	0.608	3.24	0.076	272	NA
		D-DUP	NA	3.24	NA	276	NA
LFA	Oil	A	0.0028	0.005	<0.008	0.068	NA
		A-DUP	0.003	0.004	<0.0002	0.064	NA
		B	0.002	0.007	<0.0004	<0.003	NA
		B-DUP	NA	0.007	<0.0008	NA	NA
		C	1.3	6.7	<0.0004	41.8	NA
		C-DUP	1.3	66.6	NA	41.8	NA
LFA	NaOH	A	0.003	0.004	0.34	0.011	151
		A-DUP	NA	NA	NA	NA	155
		B	0.0024	0.058	0.334	0.005	1540
		B-DUP	NA	0.058	0.408	NA	1510
		C	0.0013	0.002	<0.0008	0.01	0.863
		C-DUP	NA	0.002	<0.002	NA	NA
		D	1.32	6.7	0.214	41.2	0.407
		D-DUP	1.31	6.7	NA	41.2	NA
LFA	Pb	A	0.0006	0.008	0.08	0.125	0.582
		A-DUP	NA	NA	0.082	NA	NA
		B	0.0125	0.01	0.107	385	0.65
		B-DUP	0.128	NA	0.11	379	0.65
		C	0.601	3.23	275	4.14	0.104
		C-DUP	0.604	NA	271	NA	NA

(Continued)

(Sheet 3 of 4)

TABLE B-4 (Concluded)

Binder	Interferent	Sample*	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference (mg/l)
LFA	Pb (Cont.)	D	0.002	0.001	<0.0004	<0.03	0.136
		D-DUP	0.0022	NA	<0.0008	NA	NA
LFA	Phenol	A	0.0095	0.006	1.81	0.008	NA
		A-DUP	NA	NA	1.79	NA	NA
		B	0.0105	0.249	<0.0020	0.208	NA
		B-DUP	NA	0.248	<0.004	0.204	NA
LFA	HCB	A	0.0001	0.006	0.27	0.004	NA
		A-DUP	NA	NA	0.264	NA	NA
		B	0.0002	0.006	0.238	0.004	NA
		B-DUP	NA	NA	0.235	NA	NA
LFA	TCE	A	0.0002	0.01	1.21	0.006	NA
		A-DUP	NA	NA	1.28	NA	NA
		B	0.0007	0.002	<0.0002	0.004	NA
		B-DUP	NA	NA	<0.0004	NA	NA
LFA	Zn	A	0.0007	0.009	1.05	<0.001	<0.030
		A-DUP	NA	NA	1.02	NA	NA
		B	<0.0001	0.001	<0.0002	0.006	0.058
		B-DUP	NA	NA	<0.0004	NA	0.05
LFA	Cu	A	<0.0001	0.003	<0.001	0.002	0.837
		A-DUP	NA	NA	NA	NA	0.8
		B	0.004	0.013	0.006	0.056	0.172
		B-DUP	NA	NA	NA	0.06	0.172
LFA	Na_2SO_4	A	<0.0001	0.007	0.928	<0.001	69.6
		A-DUP	NA	NA	0.857	NA	NA
		B	0.0045	0.004	<0.0002	0.004	0.017
		B-DUP	NA	NA	<0.0004	NA	NA

(Sheet 4 of 4)

TABLE B-5. INTERNAL SPIKED SAMPLES

Binder	Interferent	Sample	Metal (% recovery) Cd	Cr	Hg	Ni	Interference* (% recovery)
CEM	Grease	A	NA**	NA	NA	103	NA
CEM	Oil	A	NA	NA	87	109	108
		B	NA	NA	94	NA	NA
CEM	NaOH	A	NA	NA	87	109	108
		B	NA	NA	94	NA	NA
CEM	Pb	A	NA	NA	106	NA	108
		B	NA	NA	106	NA	NA
CEM	Phenol	A	NA	NA	NA	108	NA
		B	105	100	101	NA	NA
CEM	HCB	A	NA	NA	113	105	NA
CEM	TCE	A	NA	NA	113	110	NA
		B	NA	NA	94	NA	NA
CEM	Zn	A	NA	NA	111	NA	NA
		B	NA	106	112	110	100
CEM	Cu	A	NA	NA	84	NA	NA
		B	NA	NA	100	NA	NA
CEM	Sulfate	A	NA	NA	111	NA	110
		B	NA	108	93	NA	NA
CFA	Grease	A	102	95.4	82	105	NA
		B	100	103	93	99.8	NA
CFA	Oil	A	NA	99	110	105	NA
		B	NA	NA	86	NA	NA
		C	108	107	NA	92	NA
CFA	NaOH	A	103	107	95	110	102
		B	NA	NA	75	NA	NA
		C	94.8	99.2	NA	98.8	NA
CFA	Pb	A	101	NA	85	107	NA
		B	NA	NA	101	NA	NA
		C	NA	101	NA	99	NA
CFA	Phenol	A	NA	102	102	110	NA
CFA	HCB	A	103	103	94.5	108	NA
		B	111	104	NA	111	NA
CFA	TCE	A	NA	102	NA	110	NA
		B	125	102	95	108	NA
		C	100	100	92	NA	NA
CFA	Zn	A	110	NA	97	103	104
CFA	Cu	A	110	104	103	99.4	NA

(Continued)

* Interference chemicals of analysis are listed in Table B-7.
** Not analyzed.

TABLE B-5 (Concluded)

Binder	Interferent	Sample	Metal (% recovery) Cd	Cr	Hg	Ni	Interference (% recovery)
CFA	Sulfate	A	NA	107	97	NA	107
		B	105	107	100	109	93
LFA	Grease	A	NA	NA	NA	101	NA
		B	110	108	NA	103	NA
		C	110	90	97	NA	NA
		D	NA	102	NA	103	NA
LFA	Oil	A	115	110	106	108	NA
		B	NA	110	84	NA	NA
		C	98.5	99.4	NA	102	NA
LFA	NaOH	A	NA	NA	NA	NA	107
		B	NA	103	NA	NA	105
		C	NA	85	104	NA	NA
		D	101	101	NA	102	NA
LFA	Pb	A	NA	NA	110	NA	NA
		B	NA	NA	109	97.5	90
		C	105	NA	89.8	NA	NA
		D	95	NA	104	NA	NA
LFA	Phenol	A	NA	NA	103	NA	
		B	NA	96.8	104	100	
LFA	HCB	A	NA	NA	111	NA	
		B	NA	NA	108	NA	
LFA	TCE	A	NA	NA	107	NA	
		B	NA	NA	104	NA	
LFA	Zn	A	NA	NA	112	NA	NA
		B	NA	NA	107	NA	NA
LFA	Cu	A	NA	NA	NA	NA	112
		B	NA	NA	NA	109	107
LFA	Na_2SO_4	A	NA	NA	114	108	NA
		B	NA	NA	97	NA	NA

TABLE B-6. INTERNAL STANDARDS

Binder	Interferent	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference* (mg/l)
CEM	Grease**	0.48	0.168	1.49	0.17	
		0.48	0.166	1.52	0.179	
CEM	Oil	0.048	0.161	1.54	0.182	70
		0.048	0.166	1.52	0.179	80
CEM	NaOH	0.023	0.162	1.52	0.18	240
		0.020	0.166	1.52	0.179	256
CEM	Pb	0.0241	0.019	1.5	0.185	0.105
		0.0240	0.019	1.49	0.179	0.109
CEM	Phenol	0.99	0.217	1.49	0.35	NA†
		0.97	0.227	1.46	0.361	NA
CEM	HCB	0.0203	0.261	1.49	0.282	NA
		0.0200	0.271	1.54	0.275	NA
CEM	TCE	0.048	0.28	1.49	0.29	NA
		0.048	0.271	1.54	0.271	NA
CEM	Zn	0.059	0.186	1.49	0.294	0.22
		0.063	0.2	1.46	0.294	0.228
CEM	Cu	0.06	0.192	0.392	1.49	0.298
		0.063	0.2	0.4	1.52	0.294
CEM	Sulfate	0.046	0.192	1.49	0.298	303
		0.046	0.2	1.46	0.294	311
CFA	Grease	0.043	0.16	1.5	0.174	NA
		0.048	0.16	1.49	0.179	NA
CFA	Oil	0.05	0.164	1.49	0.177	NA
		0.048	0.166	1.49	0.179	NA
CFA	NaOH	0.022	0.018	1.43	0.045	233
		0.02	0.019	1.49	0.049	256
CFA	Pb	0.022	0.018	0.025	1.5	0.048
		0.02	0.019	0.024	1.49	0.049
CFA	Phenol	0.044	0.258	1.49	0.292	NA
		0.046	0.271	1.5	0.275	NA
CFA	HCB	0.041	0.263	1.49	0.295	NA
		0.046	0.271	1.49	0.275	NA
CFA	TCE	0.103	0.219	1.49	0.374	NA
		0.097	0.227	1.46	0.361	NA

(Continued)

* Interference chemicals of analysis are listed in Table B-7.

** For each interferent, the pair of values shown represents the concentration (upper entry) and the true value (lower entry).

† Not analyzed.

TABLE B-6 (Concluded)

Binder	Interferent	Metal concentration (mg/l) Cd	Cr	Hg	Ni	Interference* (mg/l)
CFA	Zn	0.067	0.132	1.49	0.233	0.18
		0.067	0.14	1.49	0.239	0.182
CFA	Sulfate	0.061	0.19	1.49	0.313	295
		0.063	0.2	1.54	0.294	293
LFA	Grease	0.097	0.215	1.49	0.355	NA
		0.097	0.227	1.54	0.361	NA
LFA	Oil	0.105	0.02	1.49	0.364	NA
		0.19	0.19	1.52	0.361	NA
LFA	NaOH	0.94	0.951	1.49	0.046	257
		0.94	1.03	1.53	0.049	280
LFA	Pb	0.022	0.018	1.49	0.051	0.027
		0.02	0.019	1.48	0.049	0.024
LFA	Phenol	0.055	0.215	1.49	0.350	NA
		0.048	0.217	1.48	0.361	NA
LFA	HCB	0.099	0.215	1.49	0.357	NA
		0.103	0.227	1.51	0.354	NA
LFA	TCE		0.21	1.49	0.354	NA
			0.227	1.51	0.361	NA
LFA	Zn	0.062	0.19	1.49	0.294	0.229
		0.063	0.2	1.53	0.294	0.228
LFA	Cu	0.063	0.186	1.49	0.256	0.358
		0.063	0.2	1.53	0.254	0.4
LFA	Sulfate	0.06	0.156	1.49	0.273	310
		0.063	0.2	1.51	0.294	293

TABLE B-7. INTERFERENCE CONTAMINANTS OF ANALYSIS

Interferent	Constituent Analyzed for
Grease	--
Oil	--
NaOH	Na
$Pb(NO_3)$	Pb
Phenol	--
HCB	--
TCE	--
$Zn(NO_3)$	Zn
Cu	Cu
Sulfate	Na

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13. ABSTRACT *(Maximum 200 words)*

Solidification/stabilization of hazardous waste involves mixing the waste with a binder material to enhance the physical properties of the waste and to immobilize contaminants that may be detrimental to the environment. Many hazardous wastes contain materials that are known to inhibit the setting and strength development properties of cement and pozzolan binding agents commonly used in solidification/stabilization processes.

This study presents the results of an evaluation into the effects of ten interfering substances (oil, grease, lead, copper, zinc, sodium hydroxide, sodium sulfate, phenol, trichloroethylene, and hexachlorobenzene) on the physical and contaminant mobility properties of a solidified/stabilized heavy metal sludge. Three binder materials (Portland cement, lime/fly ash, and cement/fly ash) were used to solidify/stabilize a synthetic metal plating waste sludge containing substantial concentrations of four metals (cadmium, chromium, nickel, and mercury). The effects of these interfering materials were evaluated using

(Continued)

14. SUBJECT TERMS
See reverse.

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five physical tests: unconfined compressive strength (UCS), cone index, bulk density, wet/dry cycling, and permeability. Contaminant leaching properties were evaluated using the U.S. Environmental Protection Agency's Extraction Procedure test. Microchemical/micromorphological analyses were also performed on the treated sludges.

Test results indicate that copper, lead, zinc, grease, oil, and phenol have a significant detrimental effect on the physical properties of the solidified/stabilized sludge. In contrast, the effects of hexachlorobenzene, trichloroethylene, and sodium sulfate on the physical properties were much less marked. Sodium hydroxide increased the rate of set and 28-day UCS of the fly ash-containing mixtures, but reduced the 28-day UCS of the cement specimens. The Extraction Procedure leaching tests indicated that contaminant leaching was highly dependent on the extraction solution's final pH, except for the mercury contaminant. The metal nitrate salts had the greatest effect on the extract solution's final pH and, thus, contaminant leaching. No definitive conclusions could be drawn from the results of the microchemical/micromorphological examinations.

The results of this research confirm the need for waste and binder-specific studies prior to the selection of a chemical solidification/stabilization process for the treatment of hazardous waste.

14. (Concluded).

Cement	Metals
Fixation	Pozzolan
Hazardous waste	Solidification
Heavy metals	Stabilization
Interferences	