Technical Report Documentation Page

1. Report No. FHWA/TX-95+1315-2	2. Government Accession No.	3. Recipient's Catalog No.

4. Title and Subtitle

SOLIDIFICATION/STABILIZATION OF USED ABRASIVE MEDIA FOR NON-STRUCTURAL CONCRETE USING PORTLAND CEMENT

5. Report Date

November 1994

6. Performing Organization Code

7. Author(s)

Matthew T. Webster, R. L. Carrasquillo, Raymond C. Loehr, and David W. Fowler

8. Performing Organization Report No.

Research Report 1315-2

9. Performing Organization Name and Address

Center for Transportation Research
The University of Texas at Austin
3208 Red River, Suite 200
Austin, Texas 78705-2650

10. Work Unit No. (TRAIS)

11. Contract or Grant No.

Research Study 0-1315

12. Sponsoring Agency Name and Address

Texas Department of Transportation
Research and Technology Transfer Office
P. O. Box 5051
Austin, Texas 78763-5051

13. Type of Report and Period Covered

Interim

14. Sponsoring Agency Code

15. Supplementary Notes

Study conducted in cooperation with the U.S. Department of Transportation, Federal Highway Administration.
Research study title: "Solidification/Stabilization of Hazardous Blast Sand"

16. Abstract

Highway bridges in the United States are painted to resist corrosion and to help maintain the structural integrity of the bridge. Periodically, it is necessary to remove the existing paint so that the surface can be repainted. Most often the removal process consists of blasting the surface with an abrasive such as sand or slag. The blast media then contains elements present in the paint, such as cadmium, chromium and lead. The spent media may be a hazardous waste as defined by EPA's Toxicity Characteristic (TC) criterion. This criterion uses the Toxicity Characteristic Leaching Procedure (TCLP) to determine whether a waste is classified as a hazardous waste. This procedure subjects the waste to a highly acidic environment in which chemicals can leach out of the waste. The leachate environment is then analyzed to determine the concentration of chemical leached, which must fall within the TC criterion. Some spent blasting material has been shown to have TCLP metal concentrations exceeding the TC criterion.

The Texas Department of Transportation (TxDOT) has begun to recycle spent abrasive media in portland cement-based concrete using solidification/stabilization (S/S) techniques. This technology is designed to immobilize the metals, while recycling the spent abrasive media as a component in non-structural concrete. This study has revealed the effectiveness of portland cement-based S/S systems in recycling contaminated spent abrasive media in portland cement-based concrete. The long-term leaching behavior of metals from these concrete products was examined using sequential extraction leaching tests.

17. Key Words

Highway bridges, corrosion, paint removal process, abrasives, sand, slag, blast media, Toxicity Characteristic (TC) criterion, Toxicity Characteristic Leaching Procedure (TCLP), hazardous waste, leaching

18. Distribution Statement

No restrictions. This document is available to the public through the National Technical Information Service, Springfield, Virginia 22161.

19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 212	22. Price

Form DOT F 1700.7 (8-72) Reproduction of completed page authorized

SOLIDIFICATION/STABILIZATION OF USED ABRASIVE MEDIA FOR NON-STRUCTURAL CONCRETE USING PORTLAND CEMENT

by

Matthew T. Webster
Ramon L. Carrasquillo
Raymond C. Loehr
David W. Fowler

Research Report 1315-2

Research Project 0-1315
Solidification/Stabilization of Hazardous Blast Sand

conducted for the

TEXAS DEPARTMENT OF TRANSPORTATION

in cooperation with the

U.S. Department of Transportation
Federal Highway Administration

by the

CENTER FOR TRANSPORTATION RESEARCH
Construction Materials Research Group
Bureau of Engineering Research
The University of Texas at Austin

November 1994

IMPLEMENTATION STATEMENT

This report summarizes the findings of a two-year study on the Solidification/Stabilization (S/S) of contaminated spent abrasive media. The results show that spent abrasive media can be successfully recycled as a component in portland cement-based concrete.

The results of this study have led to the development of guidelines for the recycling of spent abrasive media in portland cement-based concrete. The approach stresses the following: establish the job requirements and environmental limits to be met by the concrete products in terms of compressive strength, setting time and TCLP leachability; determine the mix design through trial batches and adjustment of mix proportions to achieve the desired physical and chemical properties; ensure that the same materials and job conditions are used both in the trial batches and in the field applications; and ensure that the proper mixing sequence is used and that adequate mixing is achieved.

The guidelines developed in this study have been successfully implemented in the recycling of spent abrasive media at the Rainbow Bridge in Port Arthur, Texas. Mix designs have also been provided for use at many other sites throughout Texas.

Study conducted in cooperation with the Texas Department of Transportation and
the U.S. Department of Transportation, Federal Highway Administration

DISCLAIMERS

This report reflects the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the Texas Department of Transportation. This report does not constitute a standard, specification, or regulation.

NOT INTENDED FOR CONSTRUCTION,
BIDDING, OR PERMIT PURPOSES

Ramon L. Carrasquillo, P.E. (Texas No. 63881)
David W. Fowler, P.E. (Texas No. 27859)

Research Supervisors

TABLE OF CONTENTS

SUMMARY

Highway bridges in the United States are painted to resist corrosion and to help maintain the structural integrity of the bridge. Periodically, it is necessary to remove the existing paint so that the surface can be repainted. Most often the removal process consists of blasting the surface with an abrasive such as sand or slag. The blast media then contains elements present in the paint, such as cadmium, chromium and lead. The spent media may be a hazardous waste as defined by EPA's Toxicity Characteristic (TC) criterion. This criterion uses the Toxicity Characteristic Leaching Procedure (TCLP) to determine whether a waste is classified as a hazardous waste. This procedure subjects the waste to a highly acidic environment in which chemicals can leach out of the waste. The leachate environment is then analyzed to determine the concentration of chemical leached, which must fall within the TC criterion. Some spent blasting material has been shown to have TCLP metal concentrations exceeding the TC criterion.

The Texas Department of Transportation (TxDOT) has begun to recycle spent abrasive media in portland cement-based concrete using solidification/stabilization (S/S) techniques. This technology is designed to immobilize the metals, while recycling the spent abrasive media as a component in non-structural concrete. This study has revealed the effectiveness of portland cement-based S/S systems in recycling contaminated spent abrasive media in portland cement-based concrete. The long-term leaching behavior of metals from these concrete products was examined using sequential extraction leaching tests.

CHAPTER 1: INTRODUCTION

1.1 Background

Highway bridges in the United States are painted to resist corrosion, and this helps to maintain the structural integrity of the bridge. Periodically, it is necessary to remove the existing paint so that the surface can be repainted. Most often, the removal process consists of blasting the surface with an abrasive such as sand or slag. The blast media then contains elements present in the paint, such as cadmium, chromium and lead. The spent media may be a hazardous waste as defined by the EPA's Toxicity Characteristic (TC) criterion. This criterion uses the Toxicity Characteristic Leaching Procedure (TCLP) to determine whether a waste is classified as a hazardous waste. This procedure subjects the waste to a highly acidic environment in which chemicals can leach out of the waste. The leachate is then analyzed to determine the concentration of chemical leached, which must fall below the TC criterion. Some spent abrasive media have been shown to have TCLP metal concentrations exceeding the TC criterion.

The Texas Department of Transportation has begun to recycle spent abrasive media in portland cement based concrete using solidification/stabilization (S/S) techniques. This technology is designed to immobilize the metals, while recycling the spent abrasive media as a component in non-structural concrete.

Among the possible mechanisms for metal immobilization are adsorption, absorption, ion exchange, coprecipitation, chemical bonding and physical entrapment. While the success of S/S as a treatment process for metal bearing wastes is well documented, the exact mechanisms controlling the fixation and leaching of metals have not been completely determined. It is clear that pH control is necessary to prevent significant metal leaching. In S/S systems, the pH is controlled by the alkalinity of the cement, which is present primarily as calcium hydroxide (Shively et al 1986).

Many metals are converted to insoluble precipitates in S/S processes, and are subsequently trapped within the pores of the cement matrix. Metals may also be adsorbed on pore surfaces. In each of these cases, sequential extractions could show that the metals were being released along with the alkalinity. Because pH control is essential to the fixation of metals in S/S processes, the acidic environment of the TCLP ensures a worst case scenario. When subjected to extraction fluids such as seawater and freshwater, S/S products should release considerably fewer metals, as sufficient alkalinity will be present to maintain the pH at high levels.

1.2 Objectives

The objectives of this research were:

1) To immobilize the metals in spent abrasive media using portland cement.
2) To provide sufficient compressive strength for these concrete products to be used as non-structural concrete.
3) To examine the leaching behavior of metals in concrete products and elucidate the possible mechanisms that control the leaching of the metals.
4) To compare the leaching behavior of concrete product made from spent abrasive media with varying characteristics.
5) To compare the leaching behavior of these concrete products under the influence of different leachants.

1.3 General Approach

Spent abrasive media were obtained from the Texas Department of Transportation. Various mixtures of Portland cement based S/S products were analyzed. The mixtures included control mixes containing no spent abrasive media, mixes with increasing amounts of spent abrasive media, and mixes with varying compositions of strength enhancing additives. The mixes were subjected to both leachability and strength analyses in which the TCLP concentrations of chromium, cadmium and lead, along with the compressive strengths, were determined. The success of the mixes was measured with respect to the TC criteria for leaching and the Texas Department of Transportation requirements for compressive strength.

Performing sequential extractions aids in the understanding of long-term leaching behavior of S/S products and can provide insight into the mechanisms involved. Use of leaching media other than that used in the TCLP provides more information about the fixation and leaching of metals from S/S products. By replacing the TCLP extraction fluid with seawater or freshwater, leaching behavior under conditions encountered in the environment can be observed.

CHAPTER 2: LITERATURE REVIEW

2.1 The Bridge Repainting Problem

Steel highway bridges in the United States are painted to resist corrosion, and helps to maintain the structural integrity of the bridge. Periodically, it is necessary to repaint the bridges to ensure adequate protection. Before a new coat of paint can be applied, the existing coat must be removed. This is often accomplished by blasting the surface with an abrasive media. Two main problems exist with paint removal operations. First, containment of blasting abrasives, paint residue, and dust presents a challenging problem. Secondly, once captured, spent abrasives present a disposal problem.

The driving force behind the containment and disposal problems is the existence of metals in the paints used to coat steel bridge surfaces. A survey of state highway agencies revealed that approximately 80 percent of all state-maintained steel highway bridges have been coated with lead-based paints (TRB 1992). If blasting operations are carried out with no containment measures taken, debris is free to migrate off-site. This debris becomes a source of air pollution, and eventually of water and land pollution due to deposition. Because of the metals present, a health hazard may be associated with the migration of paint removal debris.

The metals associated with spent blasting abrasives pose various hazards to human health and the environment. Chromium exists in one of two valence states, +3 or +6. Hexavalent chromium (+6) is more toxic than trivalent chromium (+3), due in part to its higher rate of adsorption through intestinal tracts. The use of hexavalent chromium compounds in algaecides demonstrates their extreme toxicity to plant life. Chromium compounds also have been found to be carcinogenic to animals. Acute health effects of hexavalent chromium are exerted primarily on the skin and mucous membranes (Conner 1990).

The toxicity of cadmium and its compounds is high (Conner 1990). The most common method of exposure is through inhalation of dust and fumes. Examples of water-borne cadmium intoxication also exist. The outbreak of Itai-itai disease in Japan was attributed to the discharge of metal mine wastes into local rivers. The cadmium present led to skeletal deformations and cadmium-induced kidney damage. Cadmium accumulates in organs and has a long half-life, leading to a high risk of intoxication with prolonged exposure. Many fish and aquatic organisms concentrate cadmium and pose a risk to human health if these organisms are consumed, not to mention the toxicity to the organisms themselves (Conner 1990).

Lead exists in the +2 and +4 valence states and can be very toxic. The history of lead poisoning is well documented, dating back to Roman times. The use of lead-based storage vessels is believed to have caused gout, mental retardation, and even personality changes. The major source of lead in humans is inhalation of airborne particles into the respiratory tract (Conner 1990). Lead poisoning also can result from ingestion. The introduction of the "Lead Exposure Reduction Act of 1990" resulted in large part from the fact that lead poisoning is the most prevalent disease of environmental origin among American children (JPCL 1990, p 45).

Clearly, the health risks associated with the metals present in spent abrasive media point to the need to control the release of these chemicals into the environment. Controls must be implemented that will minimize the release of airborne contamination and prevent the migration of these compounds from the waste during treatment and disposal.

Techniques are available that can contain 85 to 90 percent of the abrasives, paint particles and dust resulting from resurfacing operations (TRB 1992). Unfortunately, as the degree of containment increases, working conditions deteriorate. Health hazards for workers become more significant as exposure is increased. This leads to the need for limited exposure time, which decreases the efficiency and increases the cost of paint removal operations. Proper engineering controls are required to decrease worker health risk, such as adequate ventilation and personal respirators. While containment of debris at bridge repainting operations poses a serious problem and a stiff technical challenge, the research described in this report focuses on the management of spent abrasive media that has been contained and collected.

Once the abrasive media has been captured, the agency or contractor in charge of the operation must then deal with the material. Previously, most spent abrasive material was disposed of in landfills. This was a convenient, low-cost method of dealing with the spent abrasive media. Due to regulatory developments such as the Resource Conservation and Recovery Act (RCRA) and the Hazardous and Solid Waste Amendments (HSWA), management and disposal of the potentially hazardous spent abrasive media were subject to new regulatory constraints. This changing regulatory environment provided the impetus for the development of a low-cost treatment technique for spent abrasive media.

2.2 Regulations

Whether a state agency or a private contractor is responsible for the treatment and disposal of spent abrasive media, an understanding of the applicable regulations is required. Initially, it is necessary to define a hazardous waste and to understand how hazardous waste needs to be managed.

Until the mid-1970's, the generation of hazardous waste was continuing to increase. However, there were no substantial steps taken to ensure the proper management of such waste. This resulted in disposal practices that were not protective of human health and the environment. The United States Environmental Protection Agency (EPA) has documented hundreds of cases of hazardous waste mismanagement which resulted in damage to human health and the environment (EPA 1986). To address the problem of solid and hazardous waste management on a nationwide scale, Congress passed the Resource Conservation and Recovery Act (RCRA) in 1976 (40CFR Part 261-267).

The goals of RCRA are:

- To protect human health and the environment.
- To reduce waste and conserve energy and natural resources.
- To reduce or eliminate the generation of hazardous waste as expeditiously as possible.

Three distinct programs were implemented under RCRA. Subtitle D regulates primarily non-hazardous solid waste. Subtitle C established a system for controlling hazardous waste from generation to disposal, or "cradle to grave." Subtitle I was developed later to address issues related to underground storage tanks.

Subtitle C of RCRA was developed to identify hazardous waste and to regulate the generation, handling, treatment and disposal of hazardous waste. The EPA developed four conditions to identify hazardous waste. A solid waste is a hazardous waste if it:

1) Has been named as a hazardous waste and listed.
2) Exhibits, on analysis, any of the four characteristics of a hazardous waste.
3) Is a mixture containing a listed hazardous waste and a non-hazardous solid waste, or is derived from the treatment, storage or disposal of a listed waste.
4) Is not excluded from regulation as a hazardous waste.

The EPA developed three lists of hazardous wastes: non-specific source wastes, specific source wastes, and commercial chemical properties. The lists were developed to include those wastes that meet the statutory definition of a hazardous waste, are acutely toxic or acutely hazardous, or are otherwise toxic.

If a waste is not listed, it may still be a characteristic hazardous waste. EPA identified four characteristics of a hazardous waste:

- Ignitability,
- Corrosivity,

- Reactivity , and
- TCLP toxicity.

In developing these four characteristics, EPA considered two criteria. First, the characteristics must be capable of being defined in terms of physical, chemical, or other properties that cause the waste to meet the definition of hazardous waste presented in RCRA. The second criterion required that the properties defining the characteristics be measurable by standardized and available testing protocols (EPA 1986).

If a solid waste is a listed waste, a characteristic waste, or a mixture of a hazardous waste and a non-hazardous solid waste, and is not specifically excluded from regulation under RCRA, it must be managed according to the regulations contained in Subtitle C of the Act. More specifically, the waste must be tracked by manifest from generation to disposal, or "cradle to grave." Specific requirements for generators and transporters of hazardous waste, as well as treatment, storage and disposal facilities were detailed in Subtitle C of RCRA (40 CFR Part 261-267).

Spent abrasive media is not a listed waste. It also does not exhibit the characteristics of ignitability, corrosivity and reactivity. The concern over spent abrasive media generated in bridge resurfacing operations is a result of the potential for the material to be considered hazardous by the TCLP toxicity criteria. Specifically, when subjected to the Toxicity Characteristic Leaching Procedure (TCLP), the metals present in spent abrasive media may leach in concentrations exceeding the TCLP limits. This would mean that the media is considered a hazardous waste and must be managed as such.

In 1984, Congress passed the Hazardous and Solid Waste Amendments (HSWA) to RCRA. The Amendments were developed to further protect groundwater by restricting hazardous waste land disposal practices that were not protective of human health and the environment. The set of regulations that resulted are referred to as the Land Disposal Restrictions (LDR's).

The Amendments, through the LDR's, largely prohibit the land disposal of untreated, listed hazardous wastes. Once a waste is prohibited, it must either meet the treatment standards established for the waste prior to land disposal, or be disposed of in a land disposal unit that has been found to satisfy the statutory no migration test. Figure 2.1 is a schematic of the land disposal options for listed hazardous wastes.

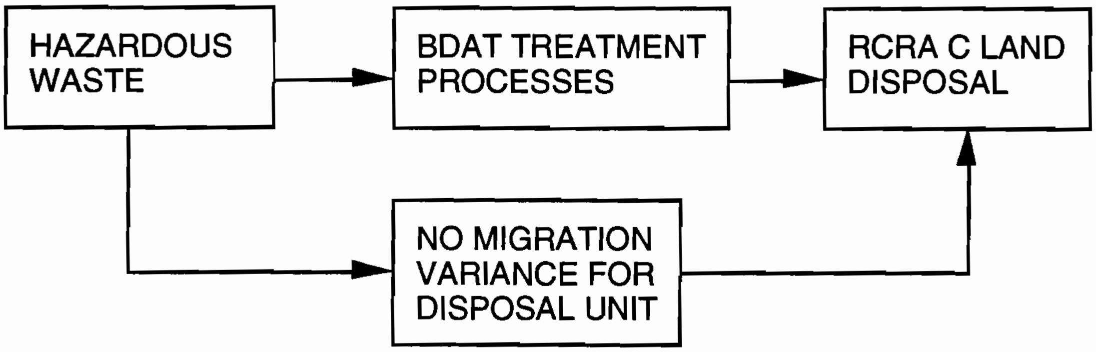


Figure 2.1: Land Disposal Options for Hazardous Wastes

Under HSWA, EPA was required to set "levels or methods of treatment, if any, which substantially diminish the toxicity of the waste or substantially reduce the likelihood of migration of hazardous constituents from the waste so that long and short term threats to human health and the environment are minimized." These treatment standards are technology based standards, developed by evaluating the Best Demonstrated Available Technologies (BDAT) for listed hazardous wastes. While these technologies were used to set the levels to which a waste must be treated prior to land disposal, any technology that achieves the standards is acceptable. Hazardous wastes that meet the treatment standards are not subject to land disposal prohibitions. These wastes are then eligible for disposal in a permitted RCRA C land disposal facility.

HSWA contained statutory deadlines for EPA to develop prohibitions and treatment standards for currently listed hazardous wastes, which it did. The Amendments also contained a deadline for promulgating prohibitions and treatment standards for newly listed wastes, those listed after the passing of the Amendments. EPA failed to meet this deadline and was subsequently sued by the Environmental Defense Fund.

EPA answered this suit with a consent decree which has developed into a proposed rule on "Land Disposal Restrictions for Newly Identified and Listed Hazardous Wastes and Hazardous Soil." This proposed rule was published in the Federal Register on September 14, 1993 (Federal Register, September 14, 1993).

Included in this proposed rule are treatment standards for Toxicity Characteristic (TC) wastes. Previously, characteristic hazardous wastes were not subject to BDAT standards. These wastes were usually treated to a level at which they no longer exhibited any of the four characteristics.

The proposed rule prohibits the land disposal of TC wastes that do not meet the newly established treatment standards. If a TC waste has constituents that exceed these levels at the point of generation of the waste, the constituents must be treated all the way to the proposed levels, even if these levels are below the characteristic levels. In other words, the proposed treatment standards are not constrained by characteristic levels.

The proposed levels for chromium, cadmium and lead in TC wastes, however, were set at 5.0 mg/L, 1.0 mg/L and 5.0 mg/L, respectively, based on the TCLP (Federal Register, September 14, 1993). These standards are the same as the TCLP limits for these metals.

At the time the LDR's were being promulgated, many state highway agencies foresaw the prohibition of TC wastes, such as spent abrasive media, from bridge repainting operations. While such prohibitions have only recently been proposed, the anticipation of such regulations caused many states to drastically rethink the way they handled this material.

As RCRA defines a TC hazardous waste in terms of TCLP concentrations, and as the land disposal of TC hazardous waste bearing metals is contingent upon TCLP concentrations, it is necessary to understand the Toxicity Characteristic Leaching Procedure.

2.3 Toxicity Characteristic Leaching Procedure (TCLP)

The changing regulatory environment resulting from RCRA created the need for a test to determine whether a waste poses an unacceptable risk to groundwater. If so, the waste should be managed as a hazardous waste. Initially, the Extraction Procedure Toxicity (EPT) test was used for this purpose.

EPT was designed to simulate the conditions found in municipal solid waste landfills. In this environment, anaerobic conditions lead to the production of organic acids, which can aid in the mobilization of contaminants through leaching processes. The test subjected the waste to an acetic acid solution to simulate the situation found in landfills. The assumption was that the hazardous waste was being codisposed of in a solid waste landfill. The waste could be subjected to particle size reduction, or it could be tested as a monolith. To be tested as a monolith, it must first pass a structural integrity impact test. If a waste survived the impact, it was assumed to remain in monolithic form in the disposal environment. The assumption is not supported by data. This allowable variation in sample preparation makes good replication of results difficult. Even with particle size reduction, only a maximum allowable size is specified, not a minimum particle size. Thus sample preparation is dependent on the equipment used and the person doing the testing (Conner 1990).

The Toxicity Characteristic Leaching Procedure (TCLP) was designed to identify additional characteristics of wastes that may pose a threat to the environment. Specifically, the number of organic constituents tested for was increased. The TCLP also attempted to address some of the shortcomings of the EPT (Bricka et al 1992).

The TCLP calls for particle size reduction of all samples, so that the waste passes through a 9.5 mm sieve. No minimum particle size is specified, however. The TCLP provided for one of two extraction fluids to be used, depending on the alkalinity of the waste. It eliminated the pH monitoring and adjustment that existed in the EPT. The TCLP specified different filtration criteria as well. Perhaps the biggest changes came in the area of volatiles extraction. A procedure was developed to address the problems of extraction and sample preparation of volatile compounds. Among other considerations, a Zero Headspace Extraction (ZHE) Vessel is required when the mobility of volatile compounds is being analyzed. In the case of nonvolatile constituents, such as metals, ZHE's are not required (Bricka et al 1992).

The TCLP consists of four major steps. First, the waste is subjected to particle size reduction if necessary. Then the appropriate extraction fluid to be used is determined based on the alkalinity of the waste. This extraction fluid is then added to the sample and the rotation is carried out at 30 rpm for 18 hours. The final step includes sample preparation and subsequent concentration analysis.

Concentration limits have been established for 25 organic compounds, 8 metals and 6 pesticides. Table 2.1 shows the concentration limits for the 8 metals. This report focuses on cadmium, chromium and lead, as they are contaminants commonly encountered in spent blasting media.

Table 2.1: EPA TCLP Concentration Limits for Metals (40 CFR Part 261.24)

CHROMIUM	**5.0 mg/L**
CADMIUM	**1.0 mg/L**
LEAD	**5.0 mg/L**
ARSENIC	5.0 mg/L
BARIUM	100.0 mg/L
MERCURY	0.2 mg/L
SELENIUM	1.0 mg/L
SILVER	5.0 mg/L

The TCLP, as the EPT before it, has come under attack as not being an appropriate testing procedure. It assumes codisposal of hazardous wastes in a municipal landfill. There is a lack of evidence supporting the similarity of the test to actual conditions encountered in the environment (Conner 1990). As applied to S/S materials, a single extraction allows for the leaching of high levels of alkalinity, maintaining a high pH at which metals are highly insoluble and immobile (Bishop 1988). This situation may be exacerbated by particle size reduction, which can lead to more surface area for the leaching of alkalinity. Particle size reduction may also enhance sorption of metals to the waste matrix (Cheng and Bishop 1992).

Despite the shortcomings of the TCLP and the dispute over its validity, it is still the procedure used by the EPA to determine the mobility of contaminants from hazardous waste matrices, and to classify certain wastes as hazardous under the TC criterion.

2.4 Management Of Used Abrasive Media

2.4.1 Used Abrasive Media Management Alternatives

As previously stated, the two main problems associated with bridge repainting operations are containment of debris and management of recovered materials. The current regulatory environment has state agencies searching for solutions to the problems they face. In some states, bridge repainting operations have come to a virtual standstill as options are explored. While strides have been made in the area of abrasive containment, the question of cost-effective disposal remains a problem.

Traditionally, spent abrasive media has been disposed of in municipal landfills. Some of this material has been deposited on roadways or in rivers and some has been used along with other construction aggregate waste (TRB 1992). The potential for the waste to be classified as a hazardous waste has made disposal by such methods more costly or even unavailable.

Current management alternatives for spent abrasive media include disposal as a waste, recycling and reuse. While these are the most commonly used alternatives, many others are being explored. Pretreatment with iron filings, repainting without abrasive blasting, and superstructure rehabilitation have all been considered by various state agencies.

The Maine Department of Transportation (Maine DOT) has considered adding iron filings to the abrasive media prior to surface preparation. Recent developments have shown that treating spent abrasive media with iron filings has been effective in reducing the TCLP lead concentration to below 5 mg/L. Adding iron filings to the material decreases the solubility of the lead contained in the spent abrasive material (JPCL 1992). The filings could be added after blast operations, but this could be construed as hazardous waste treatment, which would therefore subject the operation to additional regulations under RCRA.

Previously, the spent abrasive media had been shipped to a treatment facility in Michigan. Maine DOT has identified a cement company in Maine that would take the spent abrasive media for use in its products, but only if the material was not classified as a hazardous waste. By treating the material with iron filings so that it no longer exhibited the toxicity characteristic, Maine DOT could save substantially on transportation and disposal costs for its spent abrasive media (JPCL 1992).

Some states have investigated the repainting of bridges without surface preparation using abrasive media. In some cases, bridges can be repainted without first removing the existing coat. The New Jersey DOT has planned an experimental project that would use a strippable coating to remove lead-based paint from highway bridges (Ovenden 1990). Superstructure rehabilitation is a potential alternative to bridge repainting. The state of Connecticut has found superstructure rehabilitation to be more cost-effective than repainting because of the availability of federal funds. Federal funding is available for bridge replacement or rehabilitation but not for repainting (Ovenden 1990).

Because of the cost of repainting operations, many states have considered partial or total bridge replacement as an alternative to repainting. In some cases, beam replacement may be a viable option, while in others, total replacement of the bridge may prove more cost effective. The comparative analysis of the cost of repainting versus the cost of replacement over the life span of the bridge is a useful tool in this decision making process (Ovenden 1990).

While these management practices are being investigated by state agencies, the most common ways of dealing with spent abrasive media are conventional treatment and disposal methods or reuse of the material. Recycling or reuse of spent abrasive media is an attractive option, as it can greatly reduce the amount of material requiring disposal.

The Pennsylvania Department of Transportation (PennDot) has begun repainting operations on the Tarentum Bridge, a 2,788 ft bridge over the Allegheny River. The bridge was last painted in 1971 using a lead-based alkyd coating. The operation is using recyclable steel grit as an abrasive media (Civil Engineering 1993). By using a recyclable abrasive, the amount of waste requiring disposal is greatly reduced. The projected amount of steel grit that will require disposal is 150 to 170 tons. In comparison, 5,500 tons of nonrecyclable grit required disposal as a result of repainting operations at a bridge of comparable size, the Mckees Rock Bridge. The use of recyclable steel grit as opposed to nonrecyclable steel grit results in substantial cost savings for PennDot, considering that disposal costs can run up to $500 per ton of material (Civil Engineering 1993).

In many cases, some used abrasive media will require disposal. As such a material is often a characteristic hazardous waste, disposal is usually preceded by a treatment step to immobilize the metals present. Solidification/Stabilization (S/S) technologies provide an opportunity to immobilize the metals in spent abrasive media and to reuse the material as a component in non-structural concrete. The Texas Department of Transportation has begun to use S/S as a treatment technology for spent abrasive media (Brabrand 1993).

2.4.2 Solidification/Stabilization Of Used Abrasive Media

Solidification/Stabilization processes provide for the treatment of waste material as well as for the beneficial reuse of waste material. The recycling of wastewater solids ash in non-structural concrete has been demonstrated in Virginia. The Virginia Center for Innovative Technology (CIT) has developed a process to use the wastewater solids ash as an ingredient in concrete revetment blocks used to control shoreline erosion. The technology allows for the beneficial reuse of the waste material, and this has resulted in a substantial cost savings through the avoidance of the high cost of landfilling (WET 1993).

Solidification/Stabilization has been shown to be an effective means of dealing with spent abrasive media from bridge repainting operations. The Michigan Department of Transportation uses a solidification/stabilization process as a treatment for spent abrasive media prior to land disposal. By mixing the media with a wetted silicate and cement kiln dust, leachable lead levels can be reduced to an extent such that the material will not be classified as a toxicity characteristic hazardous waste. The hardened treatment residues are then disposed of at a non-hazardous waste land disposal facility (Ovenden 1990).

The North Carolina Department of Transportation (NC DOT) examined the recycling of spent abrasive media in portland cement based concrete and in asphalt cement based concrete for bituminous pavement (Khosla 1988). In this case, solidification/stabilization processes allow for the recycling of the abrasive media, as well as provide for the immobilization of the metals present.

While portland cement was effective in immobilizing the lead contained in the spent abrasive media, the existence of aluminum in the media caused problems. Aluminum will corrode rapidly in the moist, highly alkaline environment found in portland cement mixes. This reaction produces large amounts of hydrogen gas which results in swelling and cracking of the concrete. NC DOT determined that this concrete was not suitable for structural use (Khosla 1988).

Potential solutions to this problem are to design mixes which set either very rapidly or very slowly. A rapidly setting mix will harden before substantial hydrogen gas can be produced, thereby eliminating expansion during hardening. A slowly setting mix will allow the production of gas to occur before setting, again eliminating expansion of the matrix during hardening. The extra treatment needed to use portland cement as a binding material for spent abrasive media containing aluminum puts this process at an economic disadvantage (Khosla 1988). North Carolina DOT has decided to use the material in bituminous pavement (Ovenden 1990).

By incorporating the spent abrasive media in an asphalt concrete mix, the leaching of metals can be minimized while using the abrasive media in the pavement structure. North Carolina DOT has found that the resulting asphalt concrete meets the EPA criteria for leachable lead (Ovenden 1990). There is also no decrease in pavement performance resulting from the use of lead containing abrasive sand in bituminous pavements (Khosla 1988).

The Texas Department of Transportation also has begun to use solidification/stabilization (S/S) as a treatment technology for spent blasting abrasives (Brabrand 1993). The research described in this report is a continuation of work initiated by Brabrand at The University of Texas at Austin. Brabrand (1992) investigated the leaching behavior of cadmium, chromium and lead from S/S products made with varying amounts of spent abrasive materials. Abrasives were obtained from the Texas Department of Transportation (TxDOT) and originated from actual bridge repainting operations.

Abrasives were vacuum separated on-site, producing separated spent sand and separated spent dust. Mixes were prepared using incremental amounts of these two forms of media. Two different cement contents were also used to study the effect of cement content on the structural properties and leaching behavior of S/S products.

Results showed that up to 100% replacement of clean river sand with contaminated separated abrasive sand could be achieved with TCLP concentrations remaining below the TC criterion. While the compressive strength of the concrete decreased with increasing replacement of sand, all mixes had compressive strengths greater than 6,895 kN/m^2 (1,000 psi).

Incremental additions of spent separated dust were made to mixes prepared with clean sand. Up to 25% addition of spent dust to the portland cement resulted in adequate compressive strengths and TCLP concentrations below the TC criterion.

Throughout the experiments, lead TCLP concentrations were below or near detection limits. Cadmium TCLP concentrations were well below TC criterion limits and remained relatively constant with changes in abrasive media content. TCLP concentrations were greatest for chromium. While chromium leaching increased with increased dust addition, little effect was found with increased replacement of clean sand with contaminated sand. For all mixes, the TCLP chromium concentrations were well below the TC criterion.

The results of Brabrand (Brabrand 1993) indicated that spent abrasive media can be effectively recycled as a component in non-structural concrete. Recommendations for mix designs were made to TxDOT, and mixing was carried out at a site in Port Arthur, Texas. Concrete blocks were obtained from this site and have been analyzed for structural properties and leachability. This allowed for verification of the effectiveness of S/S in recycling spent abrasive media. The results of these analyses appear later in this report.

To properly implement a solidification/stabilization strategy for the management of spent abrasive media, it is necessary to understand the fundamentals of solidification/stabilization technologies.

2.4.3 Solidification/Stabilization Background

Solidification/Stabilization (S/S) is a technology that is frequently used as a final treatment step prior to land disposal of hazardous wastes. It accomplishes immobilization through the binding of hazardous constituents into a solid that is resistant to leaching. S/S processes can effectively immobilize harmful constituents in a waste and transform the waste into a form that may have beneficial reuse such as construction material or filler.

Stabilization refers to processes by which wastes are converted to a more stable form. These processes use chemical reactions to transform toxic or hazardous constituents into a form that is less toxic or hazardous, and/or less mobile (Nehring 1992). Stabilization does not imply a change in the physical characteristics of the waste (Conner 1990).

Solidification refers to processes in which materials are added to a waste to produce a solid of improved structural integrity (Nehring 1992). No chemical reaction between waste constituents and solidification media is implied, although such reactions can occur. The final product may be of monolithic or granular form.

The goals of S/S processes are to:

1) improve the handling characteristics of the waste,
2) decrease the surface area available for transport of waste constituents, and
3) limit the solubility of harmful constituents in the waste (Jackman 1991).

Two of the most common S/S processes are lime/pozzolan-based processes and portland cement-based processes.

Lime/pozzolan processes rely on the reaction of lime with fine siliceous material and water to produce a concrete-like material. A matrix of calcium silicate and alumino hydrates forms and entraps the waste. Materials commonly used in such processes include fly ash, blast furnace slag, ground brick and cement-kiln dust (Jackman 1991).

Cement-based processes commonly use portland cement, water, and, frequently, pozzolanic additives to form a concrete product. Immobilization is achieved through physical entrapment and the formation of hydration products from silicate compounds and water. In the cementation process, a calcium silicate hydrate gel is formed which swells and forms a concrete matrix of interlocking silicate fibrils (Jackman 1991).

The addition of pozzolans can improve portland cement based concrete products through the formation of additional cementitious compounds and the adsorption of excess water, metals and organics. Silica fume and fly ash are frequently added to portland cement based concrete for this purpose (The Hazardous Waste Consultant 1992).

Soluble silicates can also be added to cement based S/S products. These silicates gel rapidly with the calcium hydroxide produced during cement hydration. Addition of soluble silicates may reduce the leachability of metal constituents but may also weaken the concrete product and increase its porosity (The Hazardous Waste Consultant 1992).

The success of portland cement based S/S processes in treating metal-bearing wastes results from the highly alkaline environment present in cement systems. This allows for the maintenance of pH at high levels through the neutralization of excess acidity. At these high pH values, metals are relatively insoluble. The leaching of metals from S/S products is therefore highly dependent on pH control (Conner 1990). To further understand the immobilization of metals (such as those found in spent abrasive media) from S/S products, the process of leaching must be investigated.

2.5 Leaching

The dissolution of constituents from a waste form is known as leaching. Leaching occurs when a waste comes into contact with water (the leachant). All materials have a finite solubility in water and will therefore leach to some extent. Water that has passed through the waste and now contains waste constituents is termed the leachate. Leachability is the capacity of a material to leach constituents. It is expressed either as a rate or as a concentration of a constituent in the leachate (Conner 1990).

Leachability is affected by material properties and characteristics of the test method used. Test method factors include the surface area of the waste, the leachate used, the leachate to waste ratio, the type of vessel and agitation equipment used, contact time, temperature, pH and the concentration analysis method used. These factors have been previously discussed as they pertain to the TCLP (Section 2.3).

Waste material factors affecting leachability include pH, redox potential, and chemical reactions such as precipitation, adsorption and encapsulation. These factors control which leaching mechanisms are important for a waste. Leaching mechanisms include solubilization, convective and diffusive transport through the solid, bulk diffusion and chemical reaction in the leachant, and biological attack.

In S/S systems designed for the immobilization of metals, pH control is the most important factor in leaching (Conner 1990). The solubility of metal species is highly pH-dependent. If the pH of the leachate can be held at a level where metal solubility is low, the level of leaching will be minimized. The solubilities of chromium, cadmium and lead hydroxides as a function of pH are shown in Figure 2.2. These metal hydroxides are amphoteric in nature. This means that there is a narrow pH range at which the solubility is minimum. The solubility increases as the pH diverges from this range. To minimize leaching, the pH could be held at the pH of minimum solubility for the metal of concern. Unfortunately, the pH of minimum solubility is different for each metal, making the choice of an optimum pH a compromise when more than one metal is present.

The effect of pH on metal leaching from S/S products has been well documented. Shively et al (1986) performed sequential extractions on portland cement based S/S products containing metal sludges. The pH of the leachate was gradually decreased with each subsequent extraction. It was found that at high pH levels (around 10), little metal leaching occurred. As the pH dropped, more metals were leached, with a peak occurring as the pH fell below 6.0. pH control was determined to be an integral part of metal stabilization (Shively 1986).

Metals may also precipitate as silicates, sulfides, sulfates and carbonates. Each of these systems will exhibit different solubilities for the metals of interest. Determining which systems control leaching of metals from a S/S matrix is very difficult. Evidence has shown that hydroxide solubilities cannot adequately explain the leaching of metals from solidified/stabilized waste forms.

Bishop (1988) also performed sequential extractions on similar waste forms. It was shown that alkalinity was leached out of the matrix with each successive extraction. As a result, the pH was gradually reduced. Again, limited metal leaching was observed until the pH dropped below 6.0. Cadmium was found to leach at higher levels, and to leach sooner (at higher pH) than chromium and lead. The chromium and lead concentrations in the leachate were much lower than their hydroxide solubilities.

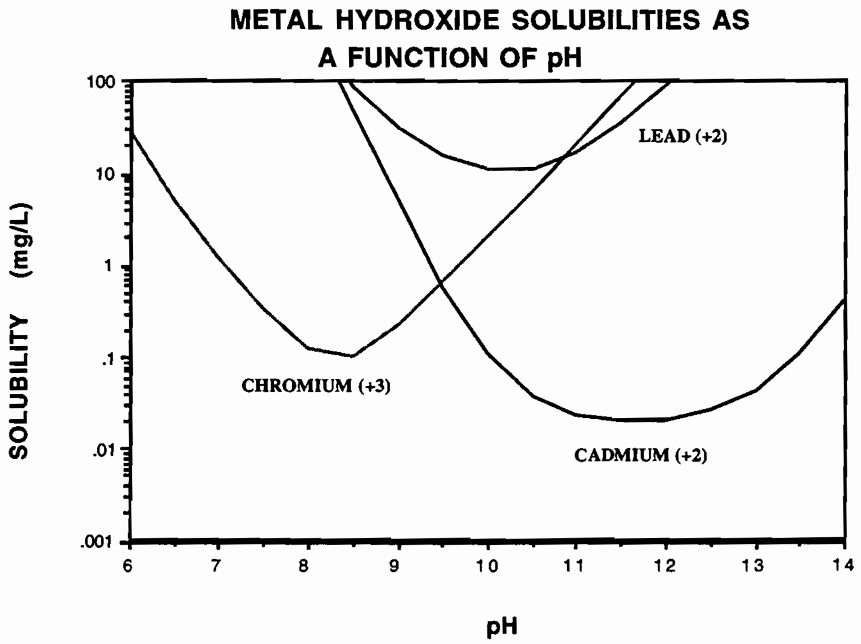


Figure 2.2: Hydroxide Solubility Curves for Chromium, Cadmium, and Lead (Stumm and Morgan 1981, Pankow 1991).

Bishop observed that the leaching of cadmium closely followed the alkalinity leaching, while chromium and lead leaching closely resembled that of silicon. It was concluded that cadmium was bound in the pores of the calcium matrix and would therefore be released with alkalinity. Chromium and lead, on the other hand, were assumed to be tightly bound in the silica matrix, and would not be released unless this matrix was broken down (Bishop 1988). The results indicate that different mechanisms are at work for different metals.

Cote (1986) also found that leachate concentrations for cadmium, chromium and lead did not coincide with their hydroxide solubilities.

Actual metal concentrations were much less dependent on pH than the theoretical hydroxide solubilities. Test data showed that concentrations could be maintained at low levels over a wide pH range. However, the minimum theoretical solubility was as much as an order of magnitude less than the minimum observed concentration (Conner 1990). It is apparent that in complex S/S systems, simple hydroxide solubilities cannot adequately explain the leaching of metals from the matrix.

Cheng and Bishop (1992) found that sorption was an important factor in the solidification/stabilization of metal bearing wastes. At high pH, the surface of the S/S matrix is negatively charged, allowing for the adsorption of dissolved metal cations onto the solid surfaces. They used the modified GANC test to study the concentration of leached metals as a function of pH.

As acidic solutions attack the concrete matrix, the calcium structure is leached away, leaving a silica-rich structure. The leaching behavior was compared to that in studies involving sorption on amorphous silica. It was observed that all metal cations were strongly adsorbed at high pH, and that almost no adsorption occurred at low pH. Test results revealed a sorption edge, or pH range, where sorption begins to occur. Results obtained very closely matched those predicted from previous studies of adsorption on amorphous silica. Table 2.2 gives estimates for the sorption edge of chromium, cadmium and lead. Cadmium desorbs at a higher pH than both chromium and lead, with chromium desorbing at a slightly lower pH than lead.

So while the importance of adsorption in S/S systems increases the difficulty in pinpointing exact mechanisms of metal stabilization, this fact reaffirms the importance of pH control. Adsorption is a chemical phenomenon, while precipitation can be important from both a chemical (solubility) and a physical (entrapment) standpoint. Encapsulation is another factor that affects the leachability of metals from S/S products.

Roy et al (1992) studied the microstructure and microchemistry of portland cement based S/S products containing synthetic electroplating sludge. With the use of scanning and transmission electron microscopy,

TABLE 2.2: pH of Sorption Edge for Chromium, Cadmium and Lead on Amorphous Silica (Cheng and Bishop 1992)

Metal	pH of Sorption Edge
Chromium	4.0 - 5.5
Cadmium	6.0 - 8.5
Lead	5.0 - 6.5

optical microscopy, energy-dispersive X-ray microanalysis and X-ray diffractometry, evidence of physical entrapment as a stabilization mechanisms was found. The authors view the binding of metals in the S/S matrix as occurring by either physical or chemical entrapment (Roy et al 1992).

Chemical entrapment occurs when the waste constituents react with the binder material to form a new, stable species. Physical entrapment involves no chemical change, and the waste is protected from the leachant by a coating on the binding matrix. While both mechanisms are expected to play a role in real-world applications, the results of this study indicate that physical entrapment on a microscopic scale was the dominant mechanism of stabilization (Roy et al 1992).

pH control, chemical reaction (such as precipitation), adsorption and encapsulation are all factors that affect the leaching of metals from S/S products. The mechanisms of leaching have been detailed by Cheng (1991). Conclusions drawn indicate that the leaching rate is limited by diffusion. Mechanisms include transport through the solid and solubilization, both of which are controlled by molecular diffusion. This dependence of leaching on diffusion may explain the deviation of observed results from behavior expected based solely on equilibrium solubility information.

CHAPTER 3: MATERIALS AND METHODS

3.1 Introduction

This research examined the structural and environmental properties of solidification/stabilization (S/S) products resulting from the incorporation of spent abrasive media in portland cement mortars. Over 180 mix designs were prepared and tested to determine compressive strength and metals leachability. Spent abrasives used in this study included spent slag, separated spent sand, separated spent dust, and unseparated spent sand/dust. These materials were obtained from actual TxDOT repainting operations. Additives used consisted of silica fume, fly ash, superplasticizer, calcium nitrite and sodium silicate. These materials are commercially available and commonly used to increase the strength and durability of cement.

This research was a collaborative effort between the Environmental and Water Resources Engineering and Structural Engineering programs in the Department of Civil Engineering at The University of Texas at Austin. Mixing of the cement mortars and subsequent physical testing (compressive strength and permeability) were carried out by students in the Structural Engineering program. Bryan Salt has written a report detailing the structural aspects of this research (Salt 1993). Leaching tests were carried out by students in the Environmental and Water Resources program. This report focuses on the environmental aspects of solidification/stabilization of spent abrasive media. Specifically, leaching of chromium, cadmium and lead from the concrete products was investigated.

3.2 Materials

Materials used in the solidification/stabilization mixes included the spent abrasive media, portland cement, siliceous river sand, silica fume, fly ash, superplasticizer, calcium nitrite and sodium silicate.

3.2.1 Portland Cement

The portland cement used in this research was Type I-II cement conforming to ASTM C 150, "Standard Specification for Portland Cement." It was obtained from the LaFarge Corporation in New Braunfels, Texas. The physical properties and chemical composition of the cement are given in Table 3.1 and 3.2. Data were provided by the LaFarge Corporation. Table 3.3 shows the total metal content for chromium, cadmium and lead in the cement.

Data presented in this report are the average of multiple samples. Replicates were run to establish the statistical variability of the samples. As the number of replicates varied for each analysis, the number of replicate samples run for each analysis (n) has been indicated in the respective tables. The coefficient of variation (CV) expresses the standard deviation of the samples as a percentage of the mean (Section 3.14.1), and is also given in the tables.

3.2.2 River Sand (Fine Aggregate)

The concrete river sand used in this research was obtained from the Colorado River in Austin, Texas. The sand had a fineness modulus of 2.79. Table 3.4 gives sieve analysis data for the concrete river sand used, which meet the ASTM C33 limits for fine aggregate.

3.2.3 Water

The water used in all mixes conformed with ASTM C 94, "Standard Specification for Ready-Mixed Concrete."

Table 3.1: Physical Properties* of Type I-II Portland Cement Used in This Study

Specific Surface (cm^2/g)	Procedure		
	Blaine	3,540	
	Wagner	1,900	
Compressive Strength (psi)	Curing Time		
	1 day	2,020	
	2 days	3,530	
	7 days	4,670	
	28 days	6,290	
Time of Setting (min.)		Procedure	
		Vicat	Gilmore
Initial		110	130
Final		200	220

1,000 psi = 6.895 MPa

*Based on Methods in ASTM C150; Data was provided by the LaFarge Corporation

3.2.4 Silica Fume

Condensed silica fume was used in many of the mixes prepared in this research. It was obtained from Master Builders, Inc., in Cleveland, Ohio. Condensed silica fume is a pozzolanic material resulting from the production of silicon metal and ferrosilicon alloys. Silica fume is effective in increasing the compressive strengths of concrete mixes. It also is most effective in lowering the permeability of concrete products.

Table 3.2: Chemical Composition* of Type I-II Portland Cement Used in This Study

Chemical Composition	Notation	% by Weight
Silicon Dioxide	SiO_2	21.5
Aluminum Dioxide	Al_2O_3	4.4
Ferric Oxide	Fe_2O_3	3.9
Calcium Oxide	CaO	64.1
Magnesium Oxide	MgO	1.5
Sulfur Trioxide	SO_3	2.6
Loss on Ignition	LOI	1.3
Insoluble Residue		0.1
Free lime		0.7
Tricalcium Silicate	$C_3S^{\dagger}$	55.0
Tricalcium Aluminate	$C_3A^{\dagger}$	5.1
Total Alkali	Na_2O Equ.	0.6

*Based on Methods in ASTM C150; data were provided by the LaFarge Corporation

†Standard Mineral Nomenclature for Cements (Conner 1990): C= CaO, A= Al_2O_3, S= SiO_2

The pozzolanic nature of silica fume leads to a calcium silicate hydrate gel with a lower C/S ratio than that resulting from cement hydration alone. A more refined pore structure results, leading to lower permeability.

Silica fume can be incorporated in cement mixes as either a percent replacement of or an addition to the portland cement. In this research, silica fume was added as a 12% by weight addition to the portland cement. The total metal content for chromium, cadmium, and lead in the silica fume used in this research is shown in Table 3.5.

Table 3.3: Total Metal Content* for Type I-II Portland Cement Used in This Study

Metal	Total Metal Content (Based on wet weight) (n=5)
	Mean (CV)†
Chromium	92 mg/kg (1.12 %)
Cadmium	47 mg/kg (6.13 %)
Lead	33 mg/kg (17.8 %)

*As determined by procedure explained in Section 3.10.2

†CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.4: Sieve Analysis of Siliceous River Sand Used in This Study

Sieve Size	Cum. Percent Retained (n=1)	ASTM C33 Limits
3/8 in.	0.00	
#4	0.15	0-5
#8	9.54	0-20
#16	30.72	15-50
#30	57.23	40-75
#50	85.94	70-90
#100	95.88	90-98
Pan	---	---
Total	279.46	

Table 3.5: Total Metal Content* for Silica Fume Used in This Study

Metal	Total Metal Content (Based on wet weight) (n=5)
	Mean (CV)†
Chromium	22 mg/kg (9.64 %)
Cadmium	18 mg/kg (11.7 %)
Lead	68 mg/kg (9.35 %)

*As determined by procedure explained in Section 3.10.2
†CV = (standard deviation/mean)*100% (Section 3.14.1)

3.2.5 Fly Ash

Parish Class C fly ash was used in many of the mixes prepared in this research. It was obtained from JTM Industries in Stafford, Texas. Fly ash is a fine residual byproduct of the combustion of coal commonly used in power generation plants. The use of fly ash in the production of concrete leads to improved workability, increased strength and durability, and decreased permeability (Kosmatka, 1990).

Fly ash is a pozzolanic material. The makeup of fly ash depends on the source coal but usually consists primarily of calcium, silica, aluminum, iron, and sulfur oxides. The addition of the hydrated lime, released during hydration of cement, to the silica, alumina and iron oxides from fly ash, results in a pozzolanic reaction. The product of this reaction is a calcium silicate hydrate gel. This process reduces the amount of calcium hydroxide in the cement pores, thereby reducing the permeability and increasing the compressive strength.

Fly ash can be incorporated in cement mixes as either a percent replacement of or an addition to the portland cement. In this research, fly ash was added as a 30% by weight replacement of the portland cement. The total metal content for chromium, cadmium, and lead in the fly ash used in this research is shown in Table 3.6.

3.2.6 Superplasticizer

Superplasticizer was used in many of the mixes to improve workability. Rheobuild 1000, marketed by Master Builders, Inc., in Cleveland, Ohio, was the superplasticizer used in this research. Workability of cement can be increased by adding water, but the compressive strength will then be reduced. By adding superplasticizer, adequate workability can be obtained, while increasing the compressive strength, by reducing the need for additional water. The superplasticizer was added as needed by the mixer, to ensure adequate workability. Therefore, the amount added varied for each mix. The total metal contents for chromium, cadmium and lead in the superplasticizer used in this research are shown in Table 3.7. Superplasticizers are also known as high-range water reducers (HRWR) and will be referred to as such in the mix tables that appear in this report.

3.2.7 Calcium Nitrite

DCI corrosion inhibitor, a calcium nitrite based liquid, was used in some of the mixes. It was obtained from Grace Construction Products. Calcium nitrite blocks the corrosive action of chloride in deicing salts and marine environments, when used in steel reinforced concrete. While the concrete being produced in this research was not steel reinforced, the calcium nitrite also served as an accelerator. It is effective in decreasing the set time of portland cement mixes. This was the primary reason calcium

Table 3.6: Total Metal Content* for Fly Ash Used in This Study

Metal	Total Metal Content (Based on wet weight) (n=5)
	Mean (CV)†
Chromium	129 mg/kg (2.88 %)
Cadmium	63 mg/kg (3.16 %)
Lead	72 mg/kg (5.05 %)

*As determined by procedure explained in Section 3.10.2
†CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.7: Total Metal Content* for Superplasticizer Used in This Study (Brabrand 1992)

Metal	Total Metal Content (Based on wet weight)
	Mean (CV)†
Chromium	16 mg/kg
Cadmium	8 mg/kg
Lead	4 mg/kg

*As determined by procedure explained in Section 3.10.1
†CV = (standard deviation/mean)*100% (Section 3.14.1)

nitrite was used in some of the mixes. Calcium nitrite has also been shown to increase the strength of concrete (Chin 1987).

Typical dosages of calcium nitrite in cement mixes range from 2.0 gallons (7.6 L) to 6.0 gallons (22.7 L) per cubic yard (0.77 m^3) of concrete.

Two dosages were used in this research, 2.0 gallons (7.6L) and 4.0 gallons (15.2 L) per cubic yard (0.77 m^3) of concrete.

3.2.8 Sodium Silicate

Sodium Silicate is used in portland cement based S/S systems for many reasons. It acts as an accelerating agent and can increase the concrete compressive strength. Sodium silicate is commonly used as a stabilization agent for metal-bearing wastes due to the formation of low solubility metal silicates. Metal ions can also be encapsulated in the silicate matrix of such systems. Permeability can be reduced as precipitates block pores in the matrix.

The sodium silicate used in this research was obtained from Spectrum Chemical Mfg. Corp.. Doses were based on the sodium silicate to cement ratio. Values of 0.02 and 0.04 based on weight were used. Sodium silicate was added to the mix water prior to addition of the water to the mortar.

3.2.9 Spent Abrasive Slag

Blast furnace slag is commonly used as abrasive media in repainting operations. Spent abrasive slag used in this research was produced from repainting operations on the Montopolis Bridge over the Colorado River on US 183 in Austin, Texas. It was obtained in June of 1993. The slag had a fineness modulus of 1.93. The sieve analysis data are shown in Table 3.8. Total metal content for chromium, cadmium and lead in the slag is detailed in Table 3.9. TCLP concentrations for these metals are given in Table 3.10.

Table 3.8: Sieve Analysis of Spent Abrasive Slag Used in This Study

Sieve Size	Cum. Percent Retained (n=1)	ASTM C33 Limits
3/8 in.	0.0	
#4	0.2	0-5
#8	0.4	0-20
#16	0.9	15-50
#30	23.4	40-75
#50	76.7	70-90
#100	91.3	90-98
Pan	---	---
Total	192.9	

Table 3.9: Total Metal Content* for Spent Abrasive Slag Used in This Study

Metal	Total Metal Content (Based on wet weight) (n=5)
	Mean (CV)†
Chromium	564 mg/kg (12.8 %)
Cadmium	61 mg/kg (10.1 %)
Lead	1941 mg/kg (14.9 %)

*As determined by procedure explained in Section 3.10.2

†CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.10: TCLP Concentrations* for Spent Abrasive Slag Used in This Study

Metal	TCLP Concentration (n=5)
	Mean (CV)†
Chromium	2.34 mg/L (4.85 %)
Cadmium	0.47 mg/L (4.88 %)
Lead	3.58 mg/L (6.53 %)

*As determined by procedure explained in Section 3.9
†CV = (standard deviation/mean)*100% (Section 3.14.1)

3.2.10 Separated Spent Abrasive Sand

The majority of the S/S mixes were prepared with spent abrasive sand, or separated spent abrasive sand and dust. Separated spent abrasive sand and separated spent abrasive dust resulted from particle separation of spent abrasive sand. The separator separates larger sand particles for reuse in further paint removal operations. The smaller particles and paint chips are separated out and are referred to as dust. These two forms of spent media are stored separately and can be incorporated into mixes in various proportions. Spent abrasive sand that has not been subjected to particle separation is referred to as unseparated spent abrasive sand/dust.

The separated spent abrasive sand was obtained from TxDOT and originated at their Rainbow Bridge Project near Port Arthur, Texas. The material was obtained in August of 1992. The sand had a fineness modulus of 2.25 and the sieve analysis is shown in Table 3.11. The total metal content for chromium, cadmium and lead in the separated sand is shown in Table 3.12. TCLP concentrations for these metals are given in Table 3.13.

Table 3.11: Sieve Analysis of Separated Spent Abrasive Sand Used in This Study

Sieve Size	Cum. Percent Retained (n=1)	ASTM C33 Limits
3/8 in.	0.00	
#4	0.00	0-5
#8	0.02	0-20
#16	1.31	15-50
#30	44.15	40-75
#50	83.44	70-90
#100	95.96	90-98
Pan	---	---
Total	224.88	

Table 3.12: Total Metal Content* for Separated Spent Abrasive Sand Used in This Study

Metal	Total Metal Content (Based on wet weight) (n=5)
	Mean (CV)†
Chromium	462 mg/kg (58.4 %)
Cadmium	27 mg/kg (14.7 %)
Lead	1580 mg/kg (26.4 %)

*As determined by procedure explained in Section 3.10.2

†CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.13: TCLP Concentrations* for Separated Spent Abrasive Sand Used in This Study

Metal	TCLP Concentration (n=3)
	Mean (CV)†
Chromium	1.04 mg/L (2.88 %)
Cadmium	0.80 mg/L (3.19 %)
Lead	7.22 mg/L (13.8 %)

*As determined by procedure explained in Section 3.9
†CV = (standard deviation/mean)*100% (Section 3.14.1)

3.2.11 Separated Spent Abrasive Dust

The separated spent abrasive dust was also obtained from TxDOT's Rainbow Bridge Project near Port Arthur, Texas, in August of 1992. The dust was a fine powdery material that passed through a #200 sieve (0.0074-cm opening). The total metal content for chromium, cadmium and lead is shown in Table 3.14, and TCLP concentrations are shown in Table 3.15.

3.2.12 Unseparated Spent Abrasive Sand/Dust

The unseparated spent abrasive sand/dust used in this study was obtained from a TxDOT project on I-20 near Odessa, Texas. Two barrels were obtained in December of 1992 and handled separately. Barrel #1 had a fineness modulus of 2.02 and Barrel #2 had a fineness modulus of 2.39. Sieve analyses for the two barrels are shown in Table 3.16. Total metal contents are shown in Table 3.17, and TCLP concentrations are shown in Table 3.18.

Table 3.14: Total Metal Content[*] for Separated Spent Abrasive Dust Used in This Study

Metal	Total Metal Content (Based on wet weight) (n=5)
	Mean (CV)[†]
Chromium	968 mg/kg (4.52 %)
Cadmium	85 mg/kg (6.03 %)
Lead	6610 mg/kg (6.50 %)

[*] As determined by procedure explained in Section 3.10.2

[†] CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.15: TCLP Concentrations for Separated Spent Abrasive Dust Used in This Study

Metal	TCLP Concentration (n=3)
	Mean (CV)[†]
Chromium	3.76 mg/L (6.54 %)
Cadmium	1.01 mg/L (2.00 %)
Lead	4.91 mg/L (2.55 %)

[*] As determined by procedure explained in Section 3.9

[†] CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.16: Sieve Analysis of Unseparated Spent Abrasive Sand/Dust Used in This Study

Sieve Size	Cum. Percent Retained		ASTM C33 Limits
	Barrel #1 (n=1)	Barrel #2 (n=1)	
3/8 in.	0.00	0.0	
#4	0.00	0.0	0-5
#8	0.02	0.9	0-20
#16	4.11	8.1	15-50
#30	34.84	51.7	40-75
#50	71.15	83.2	70-90
#100	91.54	94.7	90-98
Pan	---	---	---
Total	201.66	238.6	

Table 3.17: Total Metal Content* for Unseparated Spent Abrasive Sand/Dust Used in This Study

Metal	Total Metal Content	(Based on wet weight)
	Barrel #1 (n=2)	Barrel #2 (n=2)
	Mean (CV)†	Mean (CV)†
Chromium	53 mg/kg (2.88 %)	80 mg/kg (13.8 %)
Cadmium	11 mg/kg (16.4 %)	7 mg/kg (16.0 %)
Lead	246 mg/kg (3.66 %)	184 mg/kg (3.66 %)

*As determined by procedure explained in Section 3.10.1

†CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.18: TCLP Concentrations* for Unseparated Spent Abrasive Sand/Dust Used in This Study

Metal	TCLP Concentration	
	Barrel #1 (n=2)	Barrel #2 (n=2)
	Mean (CV)†	Mean (CV)†
Chromium	0.56 mg/L (16.5 %)	0.45 mg/L (17.4 %)
Cadmium	0.29 mg/L (24.4 %)	0.10 mg/L (10.0 %)
Lead	1.33 mg/L (9.05 %)	2.78 mg/L (10.9 %)

*As determined by procedure explained in Section 3.9
†CV = (standard deviation/mean)*100% (Section 3.14.1)

3.2.13 Seawater

Seawater was used in sequential extraction leaching tests to better simulate the actual conditions S/S products made from spent abrasive media may encounter in tidal areas of Texas and the Gulf Coast. The seawater used in this research was obtained from the Gulf of Mexico near Corpus Christi, Texas on November 4, 1993. The water was collected in 50-L plastic containers and stored in a 4°C temperature-controlled room. Relevant properties of the seawater are presented in Table 3.19.

3.2.14 Freshwater

Freshwater was used in sequential extraction leaching tests to better simulate the actual conditions S/S products made from spent abrasive media may encounter if there is contact with water in upland streams and lakes. The freshwater used in this research was obtained from the Town Lake portion of the Colorado River in Austin, Texas, on February 23, 1994. The water was collected in 50-L plastic containers and stored in a 4°C temperature-controlled room. Relevant properties of the freshwater are presented in Table 3.20.

Table 3.19: Properties* of Seawater Used in this Study

Property	Mean (CV)† (n=3)
pH	8.3 (0.19 %)
Alkalinity (mg/L $CaCO_3$)	125 (0.71 %)
Total Solids (mg/L)	34,650 (4.2 %)
Na^+ (mg/L)	11,300 (1.7 %)
K^+ (mg/L)	310 (3.1 %)
Cl^- (mg/L)	16,750 (0.95 %)
Conductivity (μMho/cm)	29,700 (3.5 %)

*As determined by SW-846 Methods

†CV = (standard deviation/mean)*100% (Section 3.14.1)

Table 3.20: Properties* of Freshwater Used in this Study

Property	Mean (CV)† (n=3)
pH	8.1 (0.12 %)
Alkalinity (mg/L $CaCO_3$)	150 (1.7 %)
Total Solids (mg/L)	380 (7.4 %)
Na^+ (mg/L)	34 (0.75 %)
K^+ (mg/L)	4.0 (2.8 %)
Cl^- (mg/L)	60 (2.3 %)
Conductivity (μMho/cm)	710 (6.1 %)

*As determined by SW-846 Methods

†CV = (standard deviation/mean)*100% (Section 3.14.1)

3.3 Mix Proportions

Appendix A provides detailed information on mix designations and the corresponding mix proportions for the mixes studied. The mixes were prepared by Bryan Salt (Salt 1993) and were prepared as indicated in Sections 3.4 through 3.7.

3.4 Mixing Procedure

All batches were mixed using the following procedure:

a) all materials were weighed to the nearest one-tenth of a pound;
b) the mixer was charged with the dry materials and then mixed for 10 seconds;
c) water and superplasticizer (if used) were added followed by mixing for three minutes;
d) the batch was then allowed to rest without mixing for two minutes;
e) if needed, additional superplasticizer was added to achieve the required workability; and
f) the batch was mixed for three more minutes.

3.5 Workability of the Mixes

The workability of each mix was measured according to ASTM C109, "Standard Test Method for Compressive Strength and Hydraulic Cement Mortars" (ASTM 1987). The targeted workability was established on the basis of the control mixes, with all subsequent mixes batched to have a similar workability as indicated by the flow table test. The flow table test measures the workability of a cement mix by placing the uncured mix on the prescribed flow table and then measuring the diameter of the mix's spread. Batches were mixed to produce a flow of 110 ± 5 (measured with a standardized flow table test caliper conforming to ASTM C109 specifications).

3.6 Casting the Mixes

Immediately after mixing, specimen molds were filled in two equal layers with each layer vibrated on a vibrating table for 20 seconds according to ASTM C 192, "Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory" (ASTM 1988). The specimens were then trowel finished.

3.7 Curing the Mixes

Curing consisted of placing the specimens under wet burlap and polyethylene for the first 24 hours after casting according to ASTM C192, "Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory" (ASTM 1988). Specimens were then placed in a curing room which was maintained at 23° C and 100% relative humidity, conforming with ASTM C511, "Standard Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes" (ASTM 1985).

3.8 Compressive Strength Testing

Compressive strengths were determined using 3 in. (76 mm) diameter, 152-mm-long (6-in.-long) cylinders tested according to ASTM C39, "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens" (ASTM 1979). Compressive strengths were determined using a Fornay model LD8606 600-kip testing machine, calibrated according to ASTM E4, "Standard Method of Load Verification of Test Machines" (ASTM 1979). The cylinders were capped with unbonded neoprene caps inside steel restraining rings.

3.9 Toxicity Characteristic Leaching Procedure

To determine whether a waste is considered hazardous by the Toxicity Characteristic criterion, the waste is subjected to the Toxicity Characteristic Leaching Procedure. This leaching procedure was carried out on the spent blasting abrasives and the solidified/stabilized products in four stages as specified by the EPA in 40 CFR Part 261 App. II:

- Particle Size Reduction of the Material,
- Extraction Fluid Determination,
- Rotation of the Fluid and Material, and
- Concentration Analysis of Metals.

Each of these four stages is discussed in detail as applied to these experiments. Included are explanations of deviations from the 40 CFR procedure.

3.9.1 Stage 1: Particle Size Reduction

The TCLP states that material under analysis must be reduced to a size that passes through a 9.5-mm (3/8-inch) sieve (40 CFR Part 261 App. II). All of the spent abrasive media used in this study satisfied this size requirement without any particle size reduction being necessary. Size reduction was necessary for the solidified/stabilized product (cured concrete blocks).

A Braun Chipmunk gyratory crusher was used to crush the concrete samples. This crusher was set to a 9.5-mm (3/8") opening, ensuring that after crushing the material was less than 9.5 mm (3/8"). The walls of the crusher were cleaned with a wire brush after each sample was crushed to prevent cross-contamination. Brabrand found that the crusher was able to reproduce the particle size distribution of the crushed blocks with remarkable precision (Brabrand 1992). The particle size distribution of the crushed samples was determined by sieve analysis. Having few variations in particle sizes between samples lessens the role of particle size as a variable when the leaching of the different samples is compared. Figure 3.1 provides the average particle size distribution, with the standard deviations, of 35 crushed samples. The next step in the TCLP is the determination of the extraction fluid to be used for the particular sample.

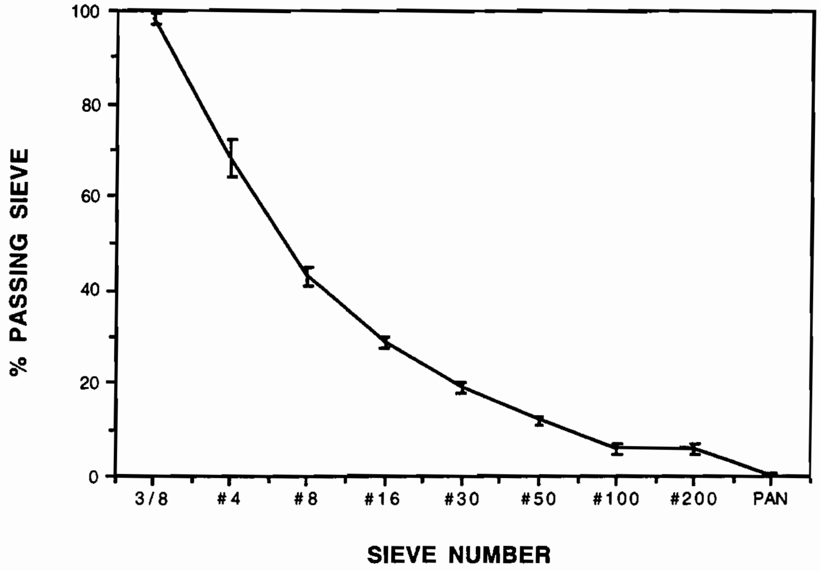


Figure 3.1: Average Particle Size Distribution of 35 S/S samples with Standard Deviation Bars (Brabrand 1992)

3.9.2 Stage 2: Extraction Fluid Determination

Determination of the extraction fluid to be used is based on the alkalinity of the sample to be analyzed. The TCLP allows for one of two different extraction fluids to be used in the procedure. For a sample that does not buffer acid well and is more sensitive to an acidic environment, the procedure specifies that extraction fluid #1 (pH = 4.93) be used. For a sample that is highly alkaline and buffers acid well, the more acidic extraction fluid #2 (pH = 2.88) is used. Fluid #1 is a dilute solution of glacial acetic acid and sodium hydroxide. Fluid #2 is a dilute solution of only glacial acetic acid. Table 3.21 outlines the composition of the two extraction fluids. Extraction fluids have to be made fresh the day of the extraction. Storing extraction fluids is not acceptable.

Table 3.21: Composition of the TCLP Extraction Fluids

Extraction Fluid #1 pH = 4.93	5.7 mL of glacial acetic acid and 64.3 mL of sodium hydroxide diluted to 1 L with distilled deionized water
Extraction Fluid #2 pH = 2.88	5.7 mL of glacial acetic acid diluted to 1 L with distilled deionized water

To objectively determine which fluid to use, the TCLP specifies a simple laboratory procedure. This procedure is outlined in Appendix B.

Briefly, a small portion of the previously crushed sample was diluted with distilled deionized water. While the solution was stirred, the pH was measured using an Orion model 701A digital pH meter. This meter was used throughout the experiments for pH measurement. If the pH was < 5, then extraction fluid #1 was used for the extraction. If the pH was > 5, then a small amount of hydrochloric acid was added to the solution and the sample was heated to 50° C. The pH was measured again. If the pH was < 5, extraction fluid #1 was used. If the pH was > 5, extraction fluid #2 was used. Using this procedure, extraction fluid #1 was found to be the appropriate fluid for all the spent abrasive media. For all of the S/S material, extraction fluid #2 was the prescribed fluid. These results are summarized in Table 3.22.

Next, the appropriate extraction fluid has to be added to the sample in the correct amount. The TCLP specifies that the amount of fluid added should be equal to 20 times the weight of the sample. The TCLP also specifies that the weight of the sample that is subjected to the extraction be at least 100 grams. Since both fluids #1 and #2 are predominantly water, it was assumed that their density is 1 g/cc. Thus, if 100 grams of sample are used for the extraction, then the required amount of fluid added to the sample is 2 liters.

Table 3.22: Correct TCLP Extraction Fluids for the Materials Tested

Spent Media - Slag - Sand - Dust - Sand & Dust	Extraction fluid #1
All Solidified/Stabilized Material	Extraction fluid #2

It was at this point that the TCLP had to be modified for use in the EWRE (Environmental and Water Resources Engineering) laboratories. The rotating device used for the extraction process could hold only 1-liter bottles; therefore, only 50 grams of the crushed sample was used in the extraction process. For every extraction, 1 liter of the proper extraction fluid was added to 50 grams of a sample. The sample and the fluid were placed in 1 liter polyurethane bottles.

3.9.3 Stage 3: Rotation of the Fluid and Material

The bottles filled with the sample and the fluid were then placed into a rotary agitation device where they were rotated for 18 hours at 30 rpm as specified by the TCLP. The rotating device was kept in a temperature-controlled room where the temperature was kept at 23° C in accordance with the TCLP. Following the 18 hours of rotation, the fluid in the bottles was separated from the sample material by vacuum filtration. The filters used were Whatman GF/F glass fiber filters with an effective pore size of 0.7 μm as specified by the TCLP. Prior to filtration, filters were acid washed with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 liter per rinse is recommended).

The sample was filtered into a 50-ml glass test tube and the pH was recorded. Figure 3.2 provides a schematic of stages 1, 2, and 3.

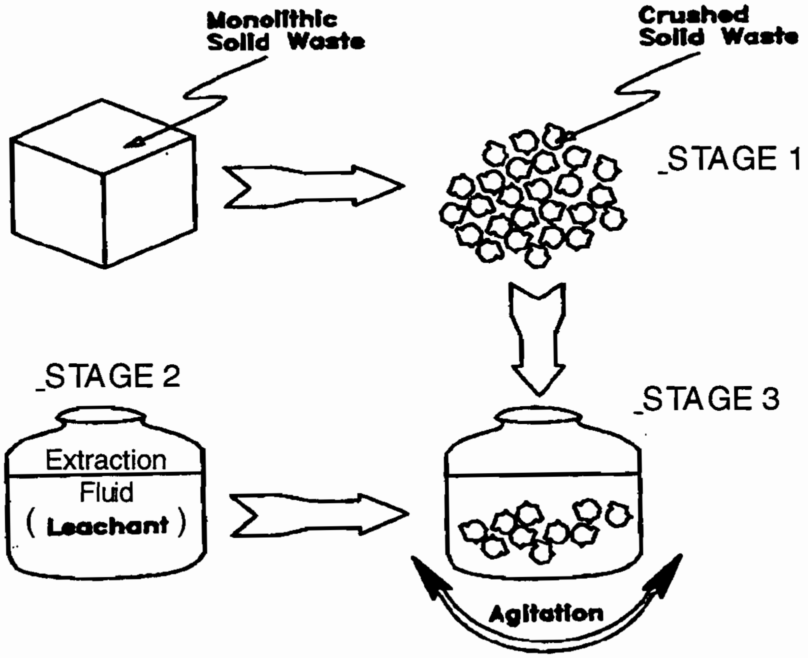


Figure 3.2: An illustration of the first 3 stages of the TCLP (Environ. Canada 1991)

A modification of the TCLP also occurred at this point. After filtration, the TCLP states that TCLP extracts to be analyzed for metals shall be acid digested. This step was omitted from the procedure. Brabrand (1992) compared the metal concentrations that resulted when the acid digestion step was used to the concentrations that resulted without acid digestion.

The extracts of three samples, two S/S samples made with spent slag media and one of the slag by itself, were used for the comparison. Three replicates of each were included. Out of the three samples, both extraction fluids were represented; fluid #1 for the slag by itself and fluid #2 for two S/S samples. After filtration, the TCLP extracts of the three samples and their replicates were acid digested according to the SW-846

Method 3005, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis By FLAA or ICP Spectroscopy (EPA 1987). The TCLP concentrations from the acid digested samples were then compared to those from the undigested samples to determine if statistically different results would occur with this TCLP modification. The statistics used are explained in Section 3.14.2.

In a statistical comparison of the concentrations of the three pairs (undigested, digested) of samples, none of the chromium or lead concentrations were statistically different. The cadmium concentrations did have statistical differences between the two S/S pairs. However, the cadmium concentrations for these pairs were very low (0.14 - 0.23 mg/L) and were within 0.09 mg/L. It was determined these statistical differences would not adversely affect the results of the experiments (Brabrand 1992).

3.9.4 Stage 4: Concentration Analysis for Metals

A Plasma 40 Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES), manufactured by Perkin Elmer, was used for concentration analyses of cadmium, chromium, lead and aluminum. Figure 3.3 is a schematic of the major components of the ICP .

Atomic emission spectrometry subjects a sample to high temperatures which excite the atoms present, promoting an electron to a higher energy level. Atoms are less stable in an excited state and tend to return to their ground state. In the process, energy is released through the emission of a photon. Each element will emit photons of a given wavelength and thus can be identified through its emission wavelength. The intensity of the emission is proportional to the concentration of the element in the sample.

The ICP machine uses an argon plasma flame to provide the energy of excitation. A radio frequency (RF) generator supplies power to a load coil, providing an alternating current within the coil. The resulting electric and magnetic fields accelerate electrons stripped from argon gas as it is passed through a spark in the torch. The continual stripping and acceleration of electrons breaks down the gas into a plasma of argon atoms, ions, and electrons, known as the ICP discharge.

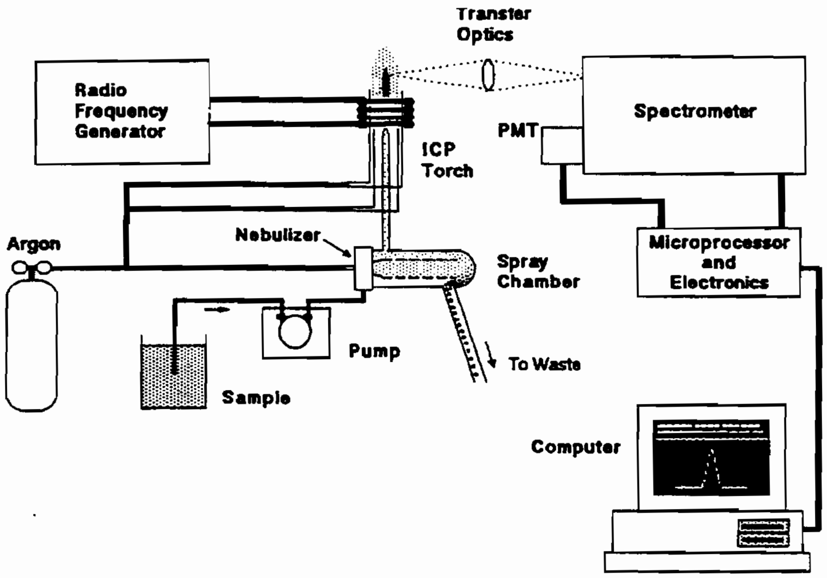


Figure 3.3: Major components of the Inductively Coupled Argon Plasma emission spectrometer (Boss 1989)

Sample introduction involves nebulizing the liquid sample into an aerosol of very fine droplets. Excitation can then take place in the 6,000 °C plasma discharge. Polychromatic light is emitted in the process and must be divided into individual wavelengths for element identification. This is accomplished through the use of a monochromator. A photomultiplier tube is then used to detect the emissions. Photomultiplier tubes contain photosensitive materials that release electrons when struck by light. The electron signal is then amplified and a current is measured at the anode of the photomultiplier tube.

This current is converted into a voltage, which is then sent to the computer. The computer then converts this voltage to a relative intensity and then a concentration. This is done using a calibration curve. A blank solution and two standards of known concentration are introduced into the ICP. The intensity of emissions measured for each metal's wavelength then provides a relationship that can be used to determine a sample's metal concentrations. Figure 3.4 illustrates the concept of a calibration curve. In this research, standards were used that contained 1 mg/L and 5 mg/L of chromium, cadmium, lead and aluminum.

An internal standard was used as a Quality Assurance/Quality Control (QA/QC) measure. Scandium was chosen because it is not reactive with the other metals. Through trial and error, it was found that a 1:6 dilution of 250 mg/L scandium solution gave well-defined emission peaks (Brabrand 1992). The calibration blank, standards, and all samples were prepared with 250 mg/L scandium in a 1:6 ratio.

3.10 Procedure for Determining Total Metals

The TCLP concentration is used to determine if a waste is considered hazardous by the Toxicity Characteristic criteria. The TCLP gives a measure of the leachability of a constituent from a waste matrix. It is also useful to know the total amount of a constituent present in the material. From the ratio of the TCLP concentration to the acid digestion concentration, the percent leached in the TCLP for a constituent can be determined. The total metal content of a material is determined by subjecting the material to an acid digestion procedure.

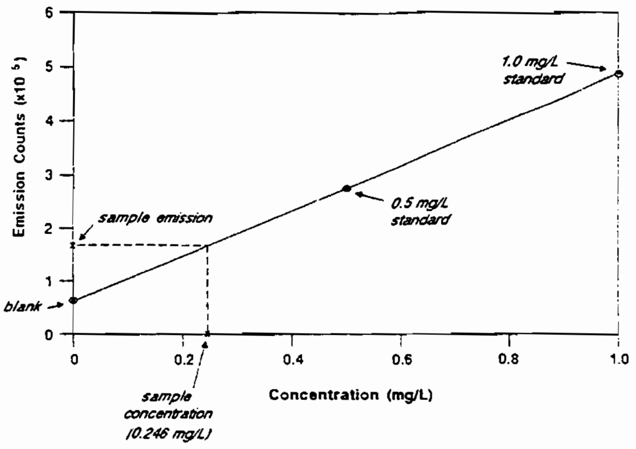


Figure 3.4: An illustration of a calibration curve and how the concentration is determined from the emission (Boss 1989).

Traditionally, SW-846 Method 3050, "Acid Digestion of Sediments, Sludges and Soils" (EPA 1987) has been used as the appropriate procedure. More recently, microwave technology has been used for the digestion of samples. SW-846 Method 3051, "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils" (EPA 1987) details the procedure for use of a microwave to determine the total metal content of a material. Microwave Digestion became an EPA approved procedure in 1992 (Federal Register, Friday, September 11, 1992).

At the beginning of this project, Method 3050 was used. A microwave digestion unit was then obtained, and Method 3051 was used from then on. The spent abrasive media, the additives and some of the S/S mixes were analyzed for total metal content. Mixes were subjected to the procedures in the same crushed form as they were in for the TCLP. After digestion, the samples were prepared for metal analysis and concentrations were determined using the ICP.

However, acid digestion concentrations for a sample could not be directly compared to the TCLP concentrations of the sample. The TCLP concentrations were determined from a 50-g sample, while the acid digestion concentrations were determined from a 2.0-g sample. Therefore, conversions to similar units must be made. The equations below convert both of the mg/L concentrations resulting from ICP analysis into concentrations of mg constituent per kg of sample (mg/kg).

(1) Calculation to convert TCLP concentration from mg/L to mg/kg

$$\text{TCLP conc. (mg/L)} * \frac{1.000\text{ L dilution}}{50.0\text{ g}} * \frac{1000\text{ g}}{1\text{ kg}} = \text{TCLP conc (mg/kg)}$$

(2) Calculation to convert Acid Digestion concentration from mg/L to mg/kg

$$\text{Acid conc. (mg/L)} * \frac{0.100\text{ L dilution}}{2.0\text{ g}} * \frac{1000\text{ g}}{1\text{ kg}} = \text{Acid conc (mg/kg)}$$

(3) Calculation to determine the % of leachable metal that leached during the TCLP

$$\%\text{ TCLP leached} = 100 * \left(\frac{\text{TCLP conc. (mg/kg)}}{\text{Acid conc. (mg/kg)}}\right)$$

3.10.1 SW-846 Method 3050

SW-846 Method 3050, "Acid Digestion of Sediments, Sludges, and Soils," subjects a 2.0-g (wet weight) sample of material to a series of several strong acids at an elevated temperature. This procedure uses hot plates to heat the samples as various reagents are added to the sample. A detailed procedure is provided in Appendix C.

3.10.2 SW-846 Method 3051

SW-846 Method 3051, "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils," uses a microwave to heat 0.5 g of material in 10 mL nitric acid for 10 minutes. The procedure results in a substantial time savings when compared to Method 3050, while "the precision and recovery of analyses using this technique are not substantially different from those obtained using" Method 3050 (Federal Register, September 11, 1992). A detailed procedure is provided in Appendix C.

3.11 Sequential Extraction Leaching Procedure

Sequential extractions were performed on solidified/stabilized products to further investigate the leaching behavior of the metals present. The procedure was based on the TCLP as it is described in Section 3.9. A 50g sample was extracted with 1L of extraction fluid. The leachate was analyzed for pH, metals concentration and alkalinity. The liquid was then filtered through Whatman GF/F filters, leaving behind the solid portion of the sample-leachate solution.

The solids remaining after filtration were then subjected to another extraction with 1 L of fresh extraction fluid, and the procedure was repeated for a total of seven extractions. Sequential extractions were performed on three mixes using TCLP extraction fluid #2, seawater, and freshwater.

3.12 ICP Method Detection Limits for Metals

Measurements of trace quantities of pollutants are often subject to detection difficulties which may arise from interference, instrument noise or analytical errors. Because of this, there exists a concentration at which lower concentrations cannot be measured within a certain confidence level. The method detection limit (MDL) is defined as the lowest concentration that an analysis procedure can reliably detect with 99% confidence that the concentration is greater than zero (Verner 1990).

The MDL is determined by analyzing seven or more replicates of one sample, and then multiplying the standard deviation of the concentrations by the t-distribution quantile at the 99% confidence level (Verner 1990):

$$\mathbf{MDL = StDev * t\text{-}dist_{99\%}}$$

where: **StDev** = the standard deviation of 7 or more (n) replicates
t-dist99% = t-statistic at the 99% confidence level for n-1 degrees of freedom.

For this research, ten samples of S/S mix SDT 300 were analyzed to determine the method detection limits for cadmium, chromium, lead and aluminum. The t-statistic value for ten replicates (thus, nine degrees of freedom) is 2.821. Table 3.23 shows the detection limits found for SDT 300. Concentrations that fall below these levels will be reported as < the corresponding detection limit for that metal.

Table 3.23: Method Detection Limits for Metals

Metal	Detection Limit
Chromium	0.07 mg/L
Cadmium	0.02 mg/L
Lead	0.07 mg/L
Aluminum	0.26 mg/L

For example, the ten chromium TCLP concentrations for mix SDT 300 averaged 0.595 mg/L with a standard deviation of 0.0264. Therefore, the method detection limit for chromium is:

$$\mathrm{MDL} = 0.0264\ \mathrm{mg/L} * 2.821 = 0.07\ \mathrm{mg/L}$$

The procedure for determining the method detection limits calls for the sample concentrations to be less than ten times the resulting detection limit (Verner 1990). In the case of mix SDT 300, this criterion was not met for aluminum. Therefore, the method detection limit used for aluminum was obtained by performing the TCLP on ten replicate samples of spent abrasive sand. The sand used was from Barrel #2 of the Odessa, Texas, material.

3.13 Quality Assurance/ Quality Control (QA/QC) Measures

The purpose of including QA/QC measures in laboratory experiments is to ensure that the data obtained from the experiments are verifiable, defensible, and of known quality (Verner 1990). Therefore, to guarantee the quality of the reported data, QA/QC measures such as matrix spikes, matrix blanks, and internal standards were used in this research.

The method of internal standards is a technique used to minimize the effects of machine fluctuations, of variations of injection volumes and of matrix differences. The internal standard should be an element that is not originally present in any of the samples or standards. That element is then added at the same ratio to each of the standards, the blank, and every sample analyzed. Scandium was used in this research. The method of internal standards provides normalized results that will compensate for the variations mentioned above. The method of internal standards is recommended for analysis when the standards will be used for a long time and where samples are sometimes not of the same matrix. A matrix is the major chemical composition of a solution. For instance, the matrix of extraction fluid #2 is acetic acid.

Matrix spikes were used to monitor the accuracy of the analytical methods used and to determine whether matrix interferences existed. For each set of samples analyzed, one of the sample replicates was spiked with 5.0 mg/L of chromium, cadmium, lead and aluminum. Thus, the analyses of the spiked samples could be compared to the analyses of the unspiked samples. Ideally, the concentration of the spiked sample should be 5.0 mg/L higher for each of the metals. This was not always the case, and, therefore, it was necessary to quantify the recovery of the spike. The equation below calculates the percent recovery of a spiked sample.

$$\% \textbf{ Recovery} = 100 * [(X_s - X_u)/ K]$$

where: X_s = Measured value for spiked sample
X_u= Measured value for unspiked sample
K = Known value of spike

The percent recovery enables the experimenter to quantify the accuracy of experimental results. The closer the percent recovery is to 100%, the higher the quality of the data. SW-846 recommends that percent recoveries should be between 75-125%. If the recoveries are outside this range, then measures should be taken to determine where the inaccuracies are occurring.

In addition to matrix spikes, matrix blanks were implemented for each set of extractions. The matrix blank went through the extraction process and the filtering process to determine if any contamination of the samples occurred throughout the experimental process. If metals entered the sample through the process, the matrix blank would show the quantity of contamination. The blank consisted solely of distilled deionized water.

In order to keep a high level of purity in the experimental procedures, all plastic and glassware used were acid washed with a 10 % nitric acid (HNO_3) solution. Following the acid washing, the materials were rinsed twice with warm water and then twice with deionized water.

3.14 Statistics

Statistics were used in this report to quantify the variability of data and to compare data sets.

3.14.1 Statistical Analysis of Data

Data presented in this report are the average of n samples with the number of replicate samples run for each analysis (n) indicated in the respective tables. The coefficient of variation (CV) for the n samples is given in parenthesis and was computed as follows (Skoog and Leary 1992):

$$\mathrm{CV} = \frac{\text{Standard Deviation}}{\text{Sample Mean}} \mathrm{x}100\%$$

The standard deviation (s) was computed as:

$$s = \sqrt{\frac{\sum_{i=1}^{N} (xi - \bar{x})^2}{N - 1}}$$

For example, in Table 3.3, five samples were analyzed (n=5) for total chromium, cadmium and lead content. The average chromium content was 92 mg/kg. The standard deviation for the five samples was 1.04 mg/kg. The coefficient of variation was calculated as:

$$\mathrm{CV} = \frac{1.04\mathrm{mg/kg}}{92\mathrm{mg/kg}} \mathrm{x}100\% = 1.12\%$$

3.14.2 Statistical Comparisons

The inclusion of statistical comparisons was used to determine if trends were statistically supported (i.e., to determine if the metal concentration of one mix was statistically different from that of another). The StatWorks statistical software package for Macintosh computers contained an unpaired Student's t-Test program (Rafferty et al 1985). For this analysis, it was assumed that the data were distributed normally, but this assumption was not verified. The unpaired student's t-test compares the means and standard deviations of two independently collected data sets. The hypothesis being tested is that the data sets are from the same population (they are not statistically different).

The program evaluates the data sets and provides a number called the significance. If the significance is less than the chosen significance level, then the data sets are said to be statistically different. If the significance is greater, then the data sets are said to be statistically the same. In this research, the significance level was chosen at 95% confidence. A level of 95% confidence means there is a 1 out of 20 probability that the hypothesis (the data sets are from the same population) will not hold true. Therefore, if the significance from the StatWorks unpaired Student's t-test of two data sets is less than 0.05, then the data sets are statistically different; otherwise, there is no statistical difference.

In this study, the data sets were the metal concentrations of the samples' TCLP extracts. The unpaired Student's t-test was used to determine if there was a statistical difference between the metal concentrations of the different S/S samples to determine whether trends were present.

CHAPTER 4: RESULTS

4.1 Introduction

This chapter presents the results of the physical and chemical tests conducted on concrete mortars resulting from the solidification/stabilization (S/S) of spent abrasive media. The leaching behavior of chromium, cadmium and lead from the S/S products was investigated, and the compressive strengths of the mixes were determined. Designations and proportions for the mixes studied are detailed in Appendix A. Appendix D contains TCLP concentrations, leachate pH data, and compressive strengths for each mix.

In this study, successful solidification/stabilization of the spent abrasive media was gauged by two objectives:

1) To immobilize the metals contained in the spent abrasive media.
2) To provide sufficient compressive strength for the concrete products to be used as non-structural concrete.

To achieve the first objective, TCLP concentrations cannot exceed the EPA limits for chromium, cadmium and lead, which are shown in Table 4.1. To achieve the second objective, the S/S products must have compressive strengths of at least 1,000 psi (6.9 MPa). This second criterion was set by the Texas Department of Transportation (TxDOT).

Table 4.1: EPA TCLP Concentration Limits for Chromium, Cadmium and Lead (40 CFR Part 261.24)

CHROMIUM	5.0 mg/L
CADMIUM	1.0 mg/L
LEAD	5.0 mg/L

4.2 Odessa Experiments

Two barrels of unseparated spent abrasive sand were obtained from TxDOT, and originated at a site in Odessa, Texas (Section 3.2.12). S/S mixes prepared with this material will be referred to as the Odessa mixes, and the subsequent studies of this material are referred to as the Odessa experiments. As each barrel contained sand with different total metal contents, the barrels were handled separately.

The first barrel (barrel #1) was used to prepare mixes designed to study the effects of silica fume, fly ash and calcium nitrite (DCI) on the leaching behavior and compressive strengths of S/S products. The second barrel (barrel #2) was used to prepare mixes equivalent to those prepared with barrel #1 sand. This allowed for a comparison of the leaching behavior and compressive strengths of S/S products made from spent abrasives with different metal contents.

4.2.1 Barrel #1

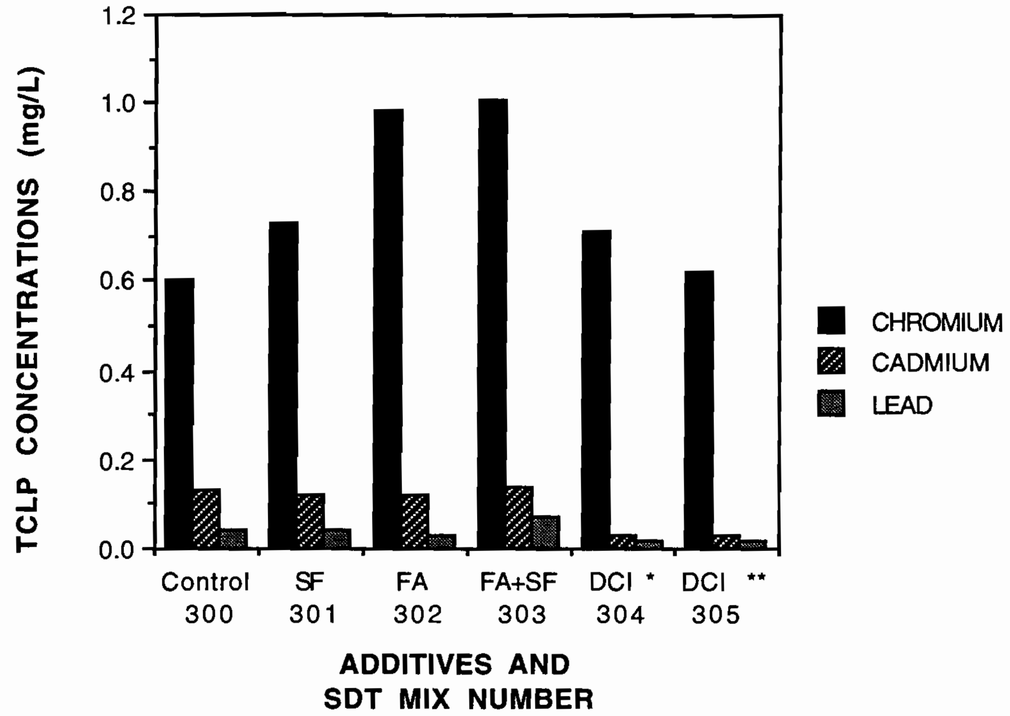
To study the effect of silica fume, fly ash and DCI on the leaching behavior and compressive strengths of S/S products, two series of mixes were designed. In each series there was a control containing only spent abrasive sand, one mix with silica fume, one mix with fly ash, and one mix with both silica fume and fly ash. Each series also contained two mixes with different dosages of DCI, but with no silica fume or fly ash. The only difference between the two series of mixes was the cement content. The SDT 300 series of mixes (SDT 300 - SDT 305) contained five sacks of portland cement, or 470 lb (212 kg). The SDT 400 series of mixes (SDT 400 - SDT 405) contained seven and a half sacks of portland cement, or 705 lb (317 kg). Fly ash was incorporated as a 30% by weight replacement of the portland cement. Mixes containing fly ash contained 70% of the corresponding cement content for that series of mixes, or 329 lb (149 kg) for five-sack mixes and 493.5 lb (224 kg) for seven-and-a-half-sack mixes. All mixes contained 1,100 lb (495 kg) of Barrel #1 unseparated spent abrasive sand. All mixes contained superplasticizer as necessary to ensure adequate workability of the mixes. A summary of the mix compositions for these experiments is given in Table 4.2.

Table 4.2: Composition Summary for Odessa Mixes Made with Unseparated Spent Abrasive Sand* (Barrel #1)

MIX #	CEMENT lb	FLY ASH lb	SILICA FUME lb	DCI† gal/yd^3	SUPER PLASTICIZER oz/cwt
SDT 300	470	0.0	0.0	0.0	12.0
SDT 301	470	0.0	56.4	0.0	16.2
SDT 302	329	141	0.0	0.0	12.0
SDT 303	329	141	56.4	0.0	17.7
SDT 304	470	0.0	0.0	2.0	23.2
SDT 305	470	0.0	0.0	4.0	28.9
SDT 400	705	0.0	0.0	0.0	12.0
SDT 401	705	0.0	84.6	0.0	16.5
SDT 402	493.5	211.5	0.0	0.0	5.2
SDT 403	493.5	211.5	84.6	0.0	12.0
SDT 404	705	0.0	0.0	2.0	12.0
SDT 405	705	0.0	0.0	4.0	12.0

*All mixes contained 1,100 lb unseparated spent abrasive sand
†Calcium-nitrite based corrosion inhibitor (Section 3.2.7)
1 lb = 0.45 kg
1 oz/cwt = 0.65 mL per kg of cement
1 gal = 3.785 L: 1 yd^3 = 0.77 m^3

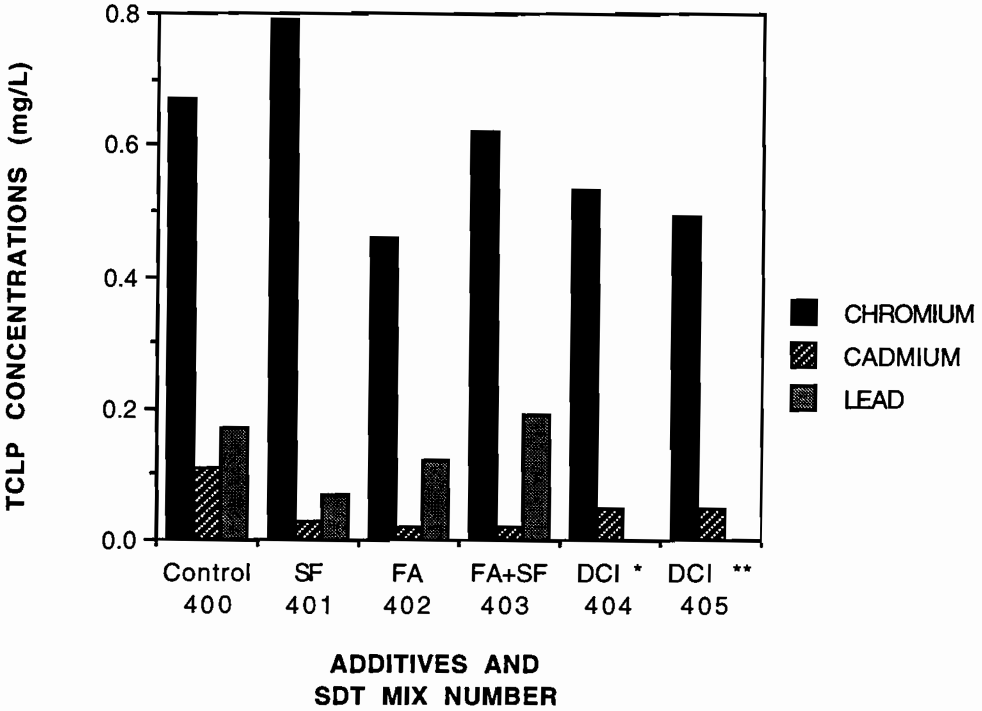
The TCLP chromium, cadmium and lead concentrations for extracts of the SDT 300 series mixes are presented in Figure 4.1. The TCLP chromium, cadmium and lead concentrations for the SDT 400 series mixes are presented in Figure 4.2. For all of these mixes, the TCLP lead concentrations were close to or below the lead detection limit of 0.07 mg/L. Cadmium concentrations were all very low with the highest equaling 0.14 mg/L. Chromium concentrations were somewhat higher, but none exceeded 1.0 mg/L.



DCI added in 2.0 (*) and 4.0 (**) gal dosages; 1 gal = 3.785 L

Figure 4.1: TCLP Concentrations for Odessa SDT 300 Series Mixes

TCLP lead concentrations were below detection limits for all of the SDT 300 series mixes. TCLP cadmium concentrations varied from 0.03 mg/L to 0.14 mg/L with no statistical trends present. TCLP chromium concentrations varied from 0.60 mg/L to 1.01 mg/L. While these levels are well below the EPA limits, some interesting trends are evident. All mixes except for the one with the higher calcium nitrate dosage had statistically chromium concentrations different from that of the control mix. The addition of silica fume and fly ash increased the TCLP chromium concentration with respect to the control. The addition of fly ash resulted in a larger increase in chromium concentration than did the addition of silica fume. The addition of calcium nitrite had no appreciable effect on TCLP concentrations.



DCI added in 2.0 (*) and 4.0 (**) gal dosages; 1 gal = 3.785 L

Figure 4.2: TCLP Concentrations for Odessa SDT 400 Series Mixes

The leaching behavior of the SDT 400 series mixes was not as distinct. The TCLP cadmium concentrations were again very low, ranging from below the 0.02 mg/L detection limit to 0.11 mg/L. No statistical trends were observed for the cadmium TCLP concentrations. While many of the TCLP lead concentrations were below detection limits, a few were increased to levels as high as 0.19 mg/L. The TCLP chromium concentrations were generally lower than those for the SDT 300 series, and trends were more difficult to observe than for the SDT 300 series mixes. The only mix that did not have a chromium concentration less than the control mix was the mix containing silica fume but no fly ash.

Twenty-eight-day compressive strength values for all Odessa Barrel #1 mixes are presented in Figure 4.3. The values varied greatly, but all were greater than 1,000 psi (6.9 MPa). For the lower cement content mixes

(SDT 300 series), silica fume increased the compressive strength, while fly ash decreased the compressive strength. For the higher cement content mixes (SDT 400 series mixes), these effects were less pronounced. The addition of calcium nitrite resulted in a dramatic increase in compressive strength.

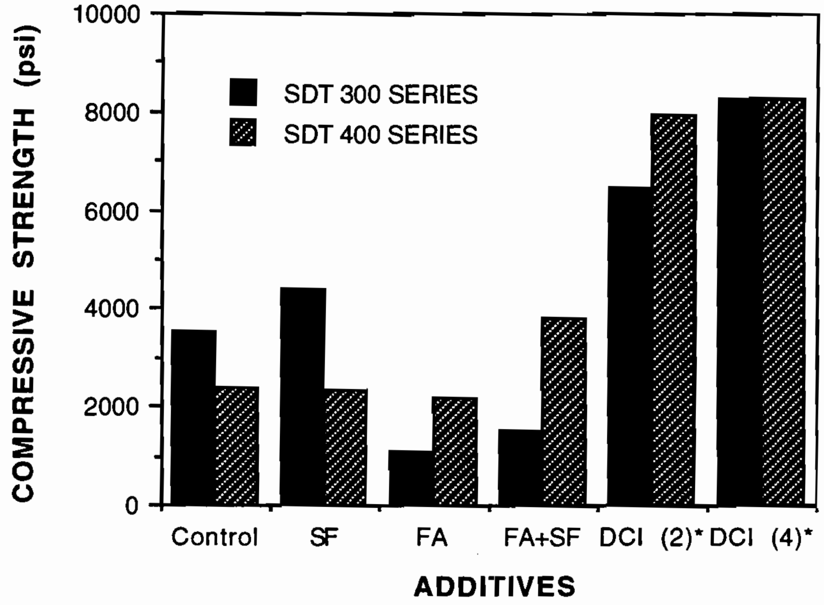
As discussed in Section 2.4.2, the presence of aluminum in concrete mixes can lead to the production of hydrogen gas which results in swelling and cracking of the concrete. As all Odessa Barrel #1 mixes set and provided compressive strengths of 1,000 psi (6.9 MPa), it appears that aluminum did not present a problem in these experiments. TCLP aluminum concentrations ranged from 30.30 mg/L to 39.92 mg/L, and no correlation could be made between TCLP aluminum concentration and compressive strength.

In summary, all the Odessa mixes prepared with Barrel #1 unseparated spent abrasive sand successfully achieved the objectives of this study. All of the SDT 300 and SDT 400 series mixes met the first objective of immobilizing the metals in spent abrasive media with respect to the EPA limits for TCLP leaching (Table 4.1), and a compressive strength of at least 1,000 psi (6.9 MPa) was achieved with every mix.

4.2.2 Barrel #2

To compare the leaching behavior and compressive strengths of S/S products made from spent abrasives with different metal contents, Barrel #2 sand was used to prepare mixes comparable in composition to the SDT 300 and SDT 400 series mixes. Again, two series of mixes were prepared. The SDT 500 series of mixes (SDT 500 - SDT 503) contained five sacks of portland cement, or 470 lb (211.5 kg). The SDT 600 series of mixes (SDT 600 - SDT 603) contained seven and a half sacks of portland cement, or 705 lb (317.25 kg). Fly ash was incorporated as a 30% by weight replacement of the portland cement. Mixes containing fly ash contain 70% of the corresponding cement content for that series of mixes, or 329 lb (149 kg) for five-sack mixes and 493.5 lb (224 kg) for seven-and-a-half-sack mixes. All mixes contained superplasticizer as necessary to ensure adequate workability of the mixes. DCI was not incorporated in any of these mixes. A summary of the mix compositions for the SDT 500 and SDT 600 series mixes is provided in Table 4.3. The total metal content for each barrel of media is shown in Table 4.4.

TCLP lead concentrations for both the SDT 500 and SDT 600 series mixes were near or below the lead detection limit of 0.07 mg/L. With the exception of one mix, SDT 603 (cadmium concentration of 0.34 mg/L), all TCLP cadmium concentrations were either 0.14 mg/L or 0.15 mg/L. TCLP chromium concentrations ranged from 0.61 mg/L to 1.10 mg/L with no statistical trends apparent. Again, TCLP chromium concentrations were slightly lower for the mixes with higher cement contents, the SDT 600 series. Upon comparison of the TCLP concentrations of the corresponding Barrel #1 and Barrel #2 mixes, no statistical trends were observed.



*DCI added in 2.0 and 4.0 gal dosages; 1 gal = 3.785 L

Figure 4.3: 28-Day Compressive Strengths for Odessa Barrel #1 Mixes

Therefore there was no discernible effect of the varied metal content of the Odessa abrasive media on the leaching behavior of these metals from the S/S products. Figure 4.4 compares TCLP chromium concentrations for the

Table 4.3: Composition Summary for Odessa Mixes Made with Unseparated Spent Abrasive Sand* (Barrel #2)

MIX #	CEMENT lb	FLY ASH lb	SILICA FUME lb	SUPER PLASTICIZER oz/cwt
SDT 500	470	0.0	0.0	17.7
SDT 501	470	0.0	56.4	23.4
SDT 502	329	141	0.0	6.9
SDT 503	329	141	56.4	17.7
SDT 600	705	0.0	0.0	5.2
SDT 601	705	0.0	84.6	14.2
SDT 602	493.5	211.5	0.0	5.1
SDT 603	493.5	211.5	84.6	8.6

*All mixes contained 1,100 lb unseparated spent abrasive sand
1 lb = 0.45 kg
1 oz/cwt = 0.65 mL per kg of cement
1 gal = 3.785 L: 1 yd^3 = 0.77 m^3

Table 4.4: Total Metal Content* for Unseparated Spent Abrasive Sand/Dust Used in This Study

Metal	Total Metal Content	(Based on wet weight)
	Barrel #1 (n=2)	Barrel #2 (n=2)
	Mean (CV)†	Mean (CV)†
Chromium	53 mg/kg (2.88 %)	80 mg/kg (13.8 %)
Cadmium	11 mg/kg (16.4 %)	7 mg/kg (16.0 %)
Lead	246 mg/kg (3.66 %)	184 mg/kg (3.66 %)

*As determined by procedure explained in Section 3.10.1
†CV = (standard deviation/mean)*100% (Section 3.14.1)

corresponding SDT 300 and SDT 500 series mixes. Figure 4.5 compares TCLP chromium concentrations for the corresponding SDT 400 and SDT 600 series mixes. These figures illustrate the lack of a discernible trend toward higher TCLP leaching concentrations with the higher chromium content of Barrel #2 unseparated spent abrasive sand.

4.3 Port Arthur Experiments

Spent abrasive media was obtained from TxDOT's Rainbow Bridge site in Port Arthur, Texas. The media consisted of vacuum separated spent abrasive sand and separated spent abrasive dust (Sections 3.2.10 and 3.2.11). The mixes prepared with this material are referred to as the Port Arthur mixes, and the subsequent studies of this material are referred to as the Port Arthur experiments. This material was used to study the effects of varying dust and calcium nitrite (DCI) content on the leaching behavior and compressive strengths of S/S products. Additional mixes were prepared by incorporating silica fume in an effort to improve the characteristics of the S/S products. All mixes contained superplasticizer as necessary to ensure adequate workability of the mixes. All mixes also contained 1,100 lb (499 kg) of separated spent abrasive sand.

To study the effects of increasing dust and DCI contents on S/S products, four series of mixes were prepared. Each series consisted of four mixes with increasing dust content. Dust was incorporated as a percent addition based on the amount of separated spent sand in the mix. Each series had mixes with no dust (control mix), 55 lb (25 kg), 165 lb (74 kg) and 275 lb (124 kg) of dust, or 0%, 5%, 15% and 25%, respectively.

Of the four series of mixes, two series (SDT 205 and SDT 206) contained 5 sacks (470 lb, 212 kg) of cement and two series (SDT 204 and SDT 207) contained 7.5 sacks (705 lb, 317 kg) of cement. One series of each cement content contained DCI (SDT 206 and SDT 207), while the remaining two series (SDT 204 and SDT 205) did not.

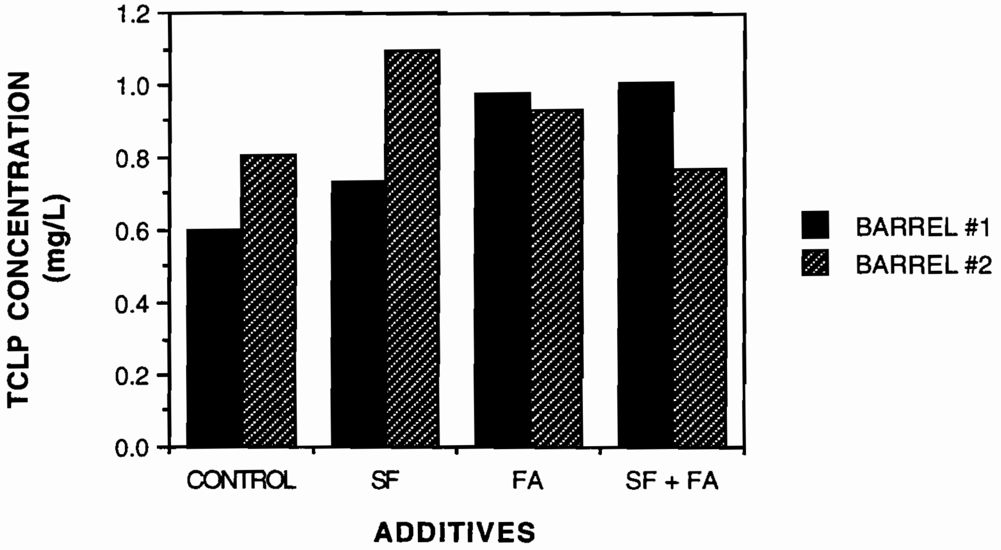


Figure 4.4: Comparison of TCLP Chromium Concentrations for Odessa Mixes Made With Barrel #1 (SDT 300 Series) and Barrel #2 (SDT 500 Series) Unseparated Spent Abrasive Sand

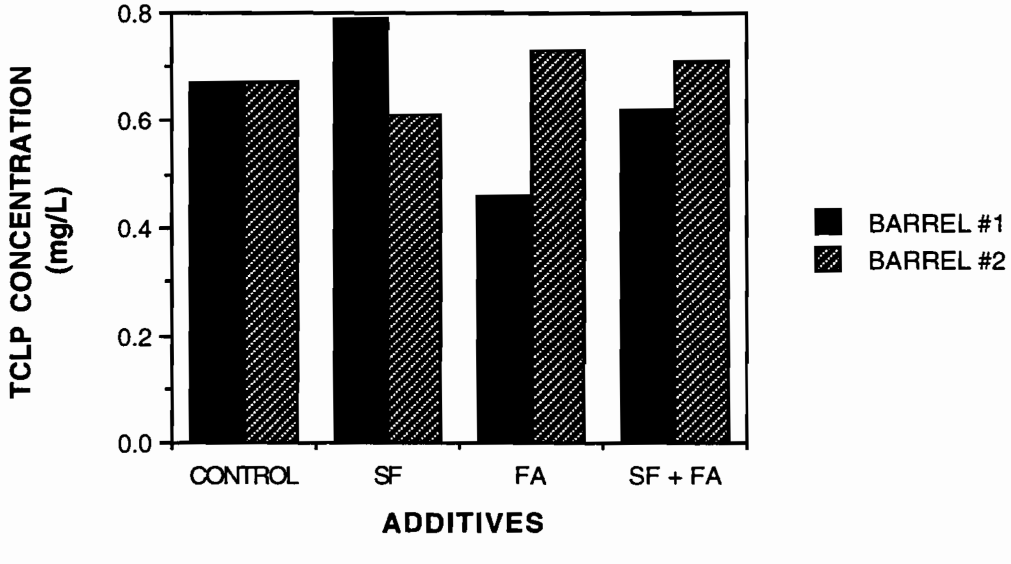


Figure 4.5: Comparison of TCLP Chromium Concentrations for Odessa Mixes Made With Barrel #1 (SDT 400 Series) and Barrel #2 (SDT 600 Series) Unseparated Spent Abrasive Sand

As many of the mixes prepared in the first four series of Port Arthur experiments did not set, two more series of mixes (SDT 208 and SDT 209) were prepared incorporating silica fume in an attempt to enhance the cementation process. These two series contained silica fume as a 12% addition based on the portland cement content, but did not contain DCI. A summary of the mix compositions for these experiments is given in Table 4.5.

SDT 204 and SDT 205 series mixes varied only in cement content. For the 7.5 sack mixes (SDT 204 series), only the control mix and the mix with a 5% addition of dust set. For the 5-sack mixes (SDT 205 series), only the control mix set. No SDT 205 series mixes containing separated spent abrasive dust set within seven days of casting.

Two series of mixes were prepared with DCI at a dosage of 2.0 gal (7.6 L). For the 7.5-sack mixes (SDT 207 series), once again, only the control mix and the mix with a 5% addition of dust set. For the 5-sack mixes (SDT 206 series), the control mix and the mix with a 5% addition of dust set.

The SDT 208 and SDT 209 series mixes contained silica fume as a 12% addition based on the portland cement content. For the 7.5-sack mixes (SDT 209 series), the control mix and the mixes containing 5%, 15% and 25% separated spent abrasive dust all set. For the 5-sack mixes (SDT 208 series), the control mix and the mixes containing 5% and 15% separated spent abrasive dust set, while the mix with 25% dust did not set.

TCLP chromium, cadmium and lead concentrations for extracts of the mixes containing no DCI or silica fume (SDT 204 and SDT 205 series) are presented in Figure 4.6. SDT 205 D1 had a lower cement content than SDT 204 D1 and D2. TCLP cadmium and lead concentrations were very low for each of these mixes. TCLP chromium concentrations ranged from 2.16 mg/L to 2.97 mg/L. Upon statistical comparison of TCLP values for each mix, no correlation could be made between increased dust content or varying cement content and TCLP chromium, cadmium and lead concentrations.

Table 4.5: Composition Summary for Port Arthur Mixes*

MIX #	CEMENT lb	SPENT DUST† lb	DCI‡ gal/yd^3	SUPER PLASTICIZER oz/cwt	SILICA FUME lb
SDT 206D1	470	0 (0%)	2.0	3.2	0.0
SDT 206D2	470	55 (5%)	2.0	12.0	0.0
SDT 206D3[x]	470	165 (15%)	2.0	30.6	0.0
SDT 206D4[x]	470	275 (25%)	2.0	44.3	0.0
SDT 207D1	705	0 (0%)	2.0	0.0	0.0
SDT 207D2	705	55 (5%)	2.0	2.6	0.0
SDT 207D3[x]	705	165 (15%)	2.0	8.4	0.0
SDT 207D4[x]	705	275 (25%)	2.0	17.1	0.0
SDT 205D1	470	0 (0%)	0.0	12.0	0.0
SDT 205D2[x]	470	55 (5%)	0.0	12.0	0.0
SDT 205D3[x]	470	165 (15%)	0.0	21.3	0.0
SDT 205D4[x]	470	275 (25%)	0.0	41.0	0.0
SDT 208D1	470	0 (0%)	0.0	20.7	56.4
SDT 208D2	470	55 (5%)	0.0	23.9	56.4
SDT 208D3	470	165 (15%)	0.0	30.6	56.4
SDT 208D4[x]	470	275 (25%)	0.0	34.6	56.4
SDT 204D1	705	0 (0%)	0.0	0	0.0
SDT 204D2	705	55 (5%)	0.0	18.9	0.0
SDT 204D3[x]	705	165 (15%)	0.0	39.1	0.0
SDT 204D4[x]	705	275 (25%)	0.0	60.7	0.0
SDT 209D1	705	0 (0%)	0.0	3.7	84.6
SDT 209D2	705	55 (5%)	0.0	9.7	84.6
SDT 209D3	705	165 (15%)	0.0	26.7	84.6
SDT 209D4	705	275 (25%)	0.0	32.3	84.6

*All mixes contained 1,100 lb separated spent abrasive sand

†Separated spent abrasive dust was incorporated as a percent addition based on the amount of separated spent abrasive sand in the mixes

‡Calcium-nitrite based corrosion inhibitor (Section 3.2.7)

[x] Mix did not set

1 lb = 0.45 kg

1 oz/cwt = 0.65 mL per kg of cement

1 gal = 3.785 L: 1 yd^3 = 0.77 m^3

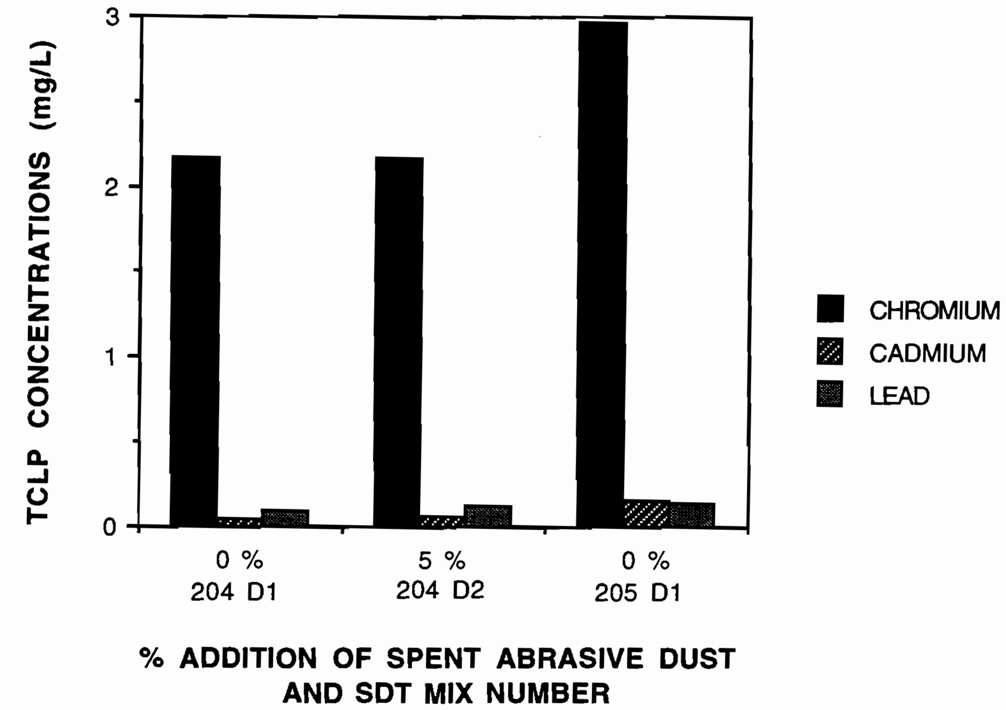


Figure 4.6: TCLP Concentrations for Port Arthur Mixes Containing No DCI or Silica Fume

TCLP chromium, cadmium and lead concentrations for extracts of the mixes containing DCI in a 2.0-gal (7.6-L) dose are presented in Figure 4.7. SDT 206 series mixes had a lower cement content than SDT 207 series mixes. TCLP lead concentrations were below the detection limit of 0.07 mg/L for the SDT 206 series mixes. SDT 207 D1 and D2 had TCLP lead concentrations of 0.18 mg/L and 0.19 mg/L, respectively. TCLP cadmium concentrations ranged from 0.14 mg/L to 0.19 mg/L. TCLP chromium concentrations were higher for the SDT 206 series mixes (2.56 mg/L and 2.47 mg/L) than for the SDT 207 series mixes (1.72 mg/L and 1.92 mg/L). A 5% addition of dust had no effect on TCLP chromium, cadmium or lead concentrations for the SDT 206 series, and had no effect on TCLP cadmium or lead concentrations for the SDT 207 series. The presence of dust (5%)

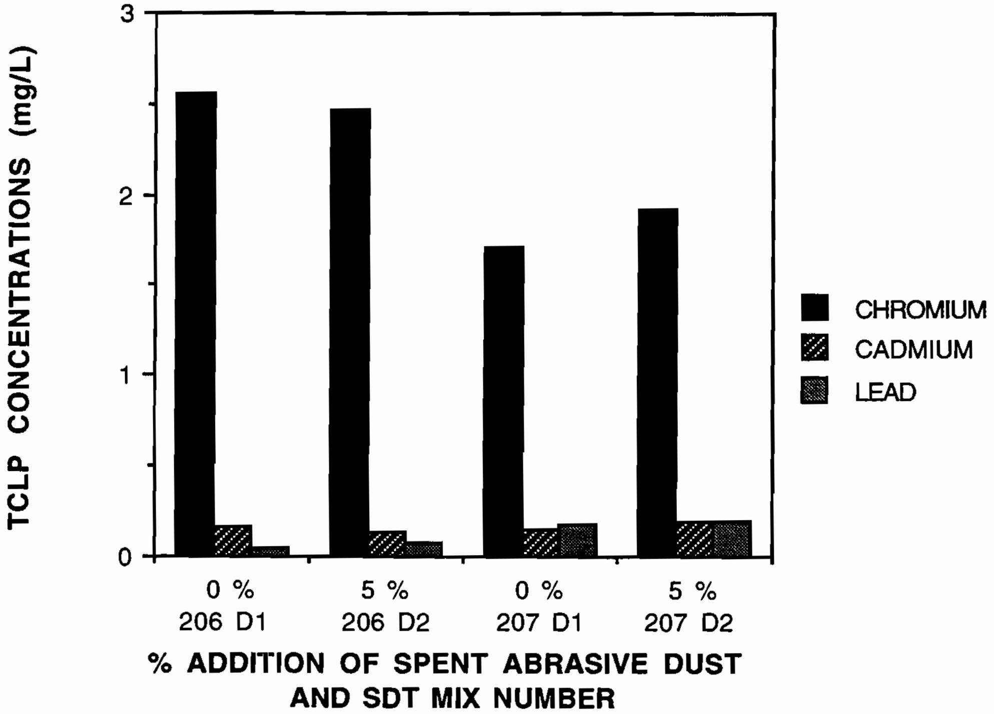


Figure 4.7: TCLP Concentrations for Port Arthur Mixes Containing DCI

did result in an increase in TCLP chromium concentration for the SDT 207 series.

TCLP chromium, cadmium and lead concentrations for extracts of the mixes containing silica fume (SDT 208 and SDT 209 series) are presented in Figure 4.8. SDT 208 series mixes had a lower cement content than SDT 209 series mixes. TCLP lead concentrations were near or below the detection limit of 0.07 mg/L for all SDT 208 and SDT 209 series mixes. TCLP cadmium concentrations were very low, ranging from 0.06 mg/L to 0.12 mg/L. TCLP chromium concentrations ranged from 1.77 mg/L to 2.65 mg/L. In general, increasing dust content had little effect on the TCLP concentrations for mixes containing silica fume. TCLP chromium concentrations were lower for the mixes with a higher cement content, the SDT 209 series.

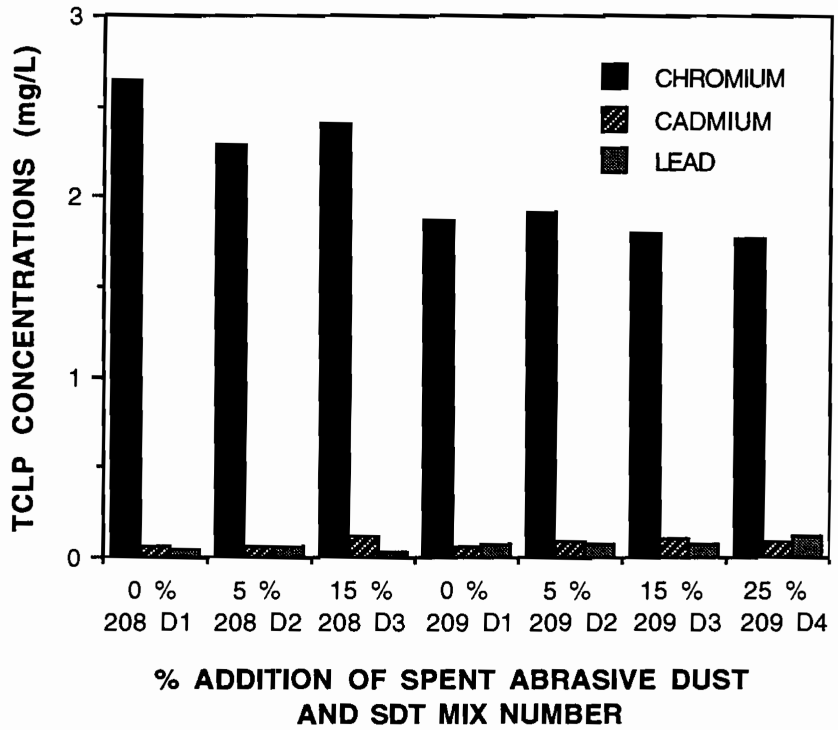


Figure 4.8: TCLP Concentrations for Port Arthur Mixes Containing Silica Fume

Twenty-eight-day compressive strength values for the Port Arthur mixes are presented in Figure 4.9. In general, increasing dust content led to lower compressive strength values. An exception occurred for the SDT 209 series mixes, where the compressive strength increased as the dust content increased from 0% to 15%. As in the Odessa experiments, no correlation could be made between TCLP aluminum concentration and compressive strength. The only mix that set and did not provide a compressive strength of at least 1,000 psi (6.9 MPa) was SDT 204 D2.

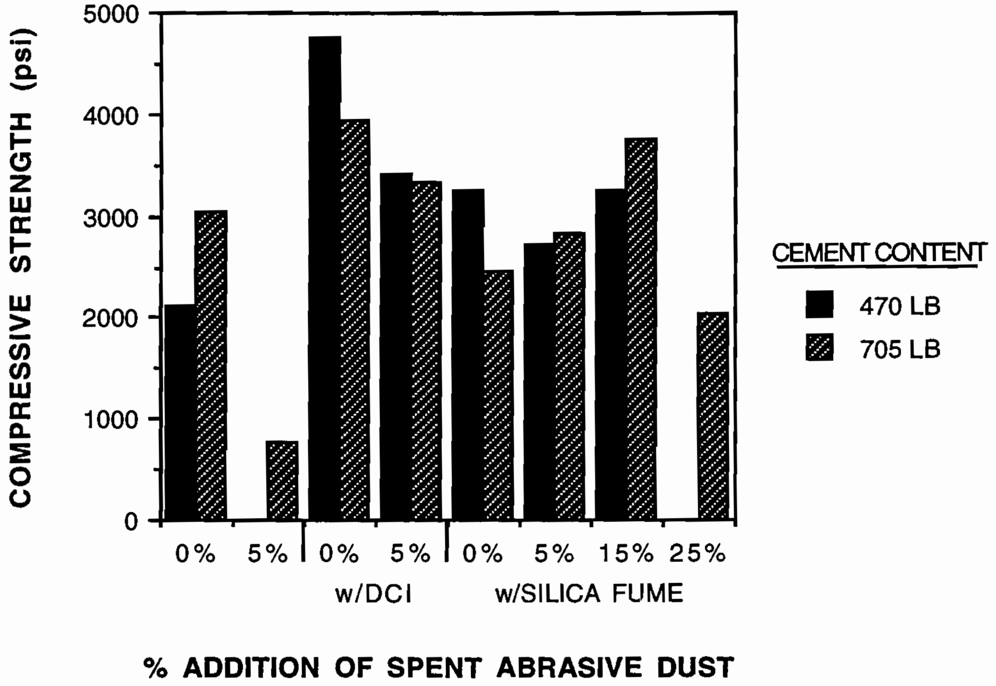


Figure 4.9: 28-Day Compressive Strengths for Port Arthur Mixes

In summary, TCLP extract lead and cadmium concentrations were very low for all Port Arthur mixes that set. TCLP chromium concentrations were as high as 2.97 mg/L, but none exceeded 5.0 mg/L. Therefore, all of the Port Arthur mixes which set met the first objective of immobilizing the metals in spent abrasive media with respect to the EPA limits for TCLP leaching (Table 4.1).

Increased separated spent abrasive dust content hindered concrete setting. For mixes with more than a 5% addition of dust, silica fume was required for the mix to set. For all but one of the mixes which did set, a compressive strength of at least 1,000 psi (6.9 MPa) was obtained.

4.4 Sodium Silicate Experiments

Three series of mixes were prepared to investigate the effect of sodium silicate content on the leaching behavior and compressive strength of S/S products. These are referred to as the sodium silicate mixes. The mixes contained 1,100 lb (495 kg) of concrete river sand with varying amounts of separated spent abrasive dust. Dust was incorporated as a percent addition based on the amount of concrete river sand in the mix. Each series had mixes with no dust, 55 lb (25 kg), 165 lb (74 kg) and 275 lb (124 kg) of dust, or 0%, 5%, 15% and 25%, respectively. All mixes contained superplasticizer as necessary to ensure adequate workability of the mixes.

The first series of mixes (CM #1 through CM #4) were the control mixes and contained no sodium silicate. The second series of mixes (Sili 1A through Sili 1D) contained 14.1 lb (6.4 kg) of sodium silicate. The third series of mixes (Sili 2A through Sili 2D) contained 28.2 lb (12.7 kg) of sodium silicate. A summary of the mix compositions for these experiments is given in Table 4.6.

TCLP lead concentrations for extracts of all sodium silicate mixes were below the detection limit of 0.07 mg/L. TCLP cadmium concentrations also were very low, ranging from 0.13 mg/L to 0.17 mg/L. TCLP chromium concentrations for all mixes are presented in Figure 4.10, and ranged from 0.20 mg/L to 0.80 mg/L. In general, TCLP chromium concentrations increased with increasing dust content. No correlation could be made between TCLP chromium concentration and sodium silicate content.

Twenty-eight-day compressive strength values for the sodium silicate mixes are shown in Figure 4.11. The control mixes (no sodium silicate) showed a sharp dropoff in compressive strength with increasing dust content. For the two series of mixes containing sodium silicate, those mixes containing separated spent abrasive dust had lower compressive strengths than those without dust. However, the amount of dust, 5%, 15% or 25%, had little effect on the compressive strength for the mixes containing sodium silicate. Mixes containing sodium silicate and separated spent abrasive dust had higher compressive strengths than the corresponding control mixes which contained dust but no sodium silicate. The dosage of sodium silicate had little effect on the 28-day compressive strength values (Salt 1993).

Table 4.6: Composition Summary for Sodium Silicate Mixes*

MIX #	CEMENT lb	SPENT DUST† lb	SODIUM SILICATE lb	SUPER PLASTICIZER oz/cwt
CM #1	705	0 (0%)	0.0	3.2
CM #2	705	55 (5%)	0.0	5.4
CM #3	705	165 (15%)	0.0	7.5
CM #4ˣ	705	275 (25%)	0.0	17.0
SILI 1A	705	0 (0%)	14.1	1.8
SILI 1B	705	55 (5%)	14.1	3.0
SILI 1C	705	165 (15%)	14.1	12.0
SILI 1D	705	275 (25%)	14.1	26.7
SILI 2A	705	0 (0%)	28.2	2.2
SILI 2B	705	55 (5%)	28.2	5.5
SILI 2C	705	165 (15%)	28.2	14.3
SILI 2D	705	275 (25%)	28.2	35.7

*All mixes contained 1,100 lb concrete river sand

†Separated spent abrasive dust was incorporated as a percent addition based on the amount of concrete river sand in the mixes

ˣMix did not set

1 lb = 0.45 kg

1 oz/cwt = 0.65 mL per kg of cement

In summary, all sodium silicate mixes achieved the objectives of this study as stated in Section 4.1. TCLP chromium, cadmium and lead concentrations were below the EPA limits (Table 4.1) for all mixes. A compressive strength of at least 1,000 psi (6.9 MPa) was achieved with every mix. In general, TCLP chromium concentrations increased with increasing dust content. TCLP cadmium and lead concentrations were very low and independent of dust content. The sodium silicate content had no effect on TCLP chromium, cadmium or lead concentrations. Sodium silicate was effective at increasing compressive strengths for mixes containing separated spent abrasive dust as shown in Figure 4.11 (Salt 1993).

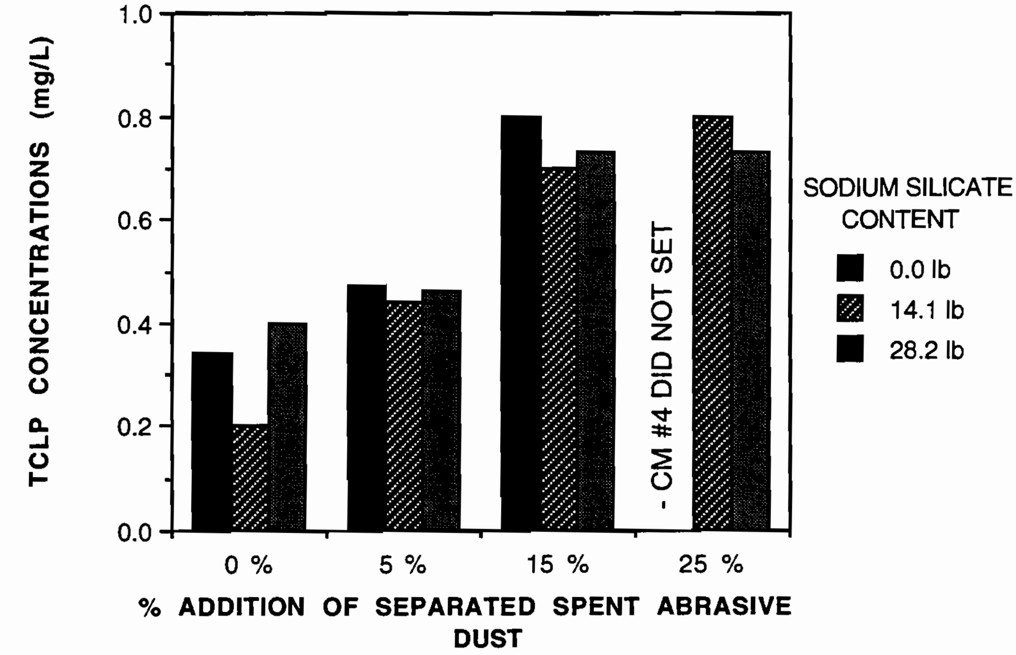


Figure 4.10: TCLP Chromium Concentrations for Sodium Silicate Mixes

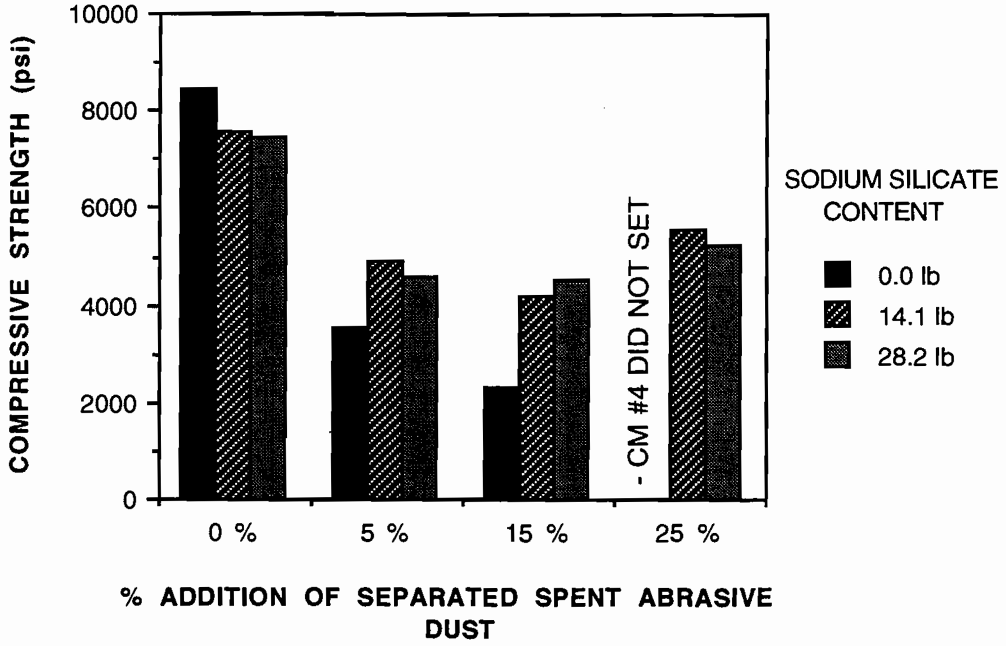


Figure 4.11: 28-Day Compressive Strengths for Sodium Silicate Mixes

4.5 Montopolis Bridge Experiments

Spent abrasive slag was obtained from TxDOT's Montopolis Bridge project in Austin, Texas (Section 3.2.9). Knowledge gained from earlier experiments was used to develop mixes which would achieve the objectives stated in Section 4.1. Because of time constraints, only those mixes thought to have the most effective compositions were subjected to the TCLP. Three mixes were chosen based on their set times and compressive strengths.

Each mix contained 705 lb (317 kg) of portland cement and 1,100 lb (495 kg) of spent abrasive slag. One mix contained silica fume, one sodium silicate and one both silica fume and sodium silicate. All mixes contained superplasticizer as necessary to ensure adequate workability of the mixes. A summary of the mix compositions for these experiments is given in Table 4.7.

Table 4.7: Composition Summary for Montopolis Mixes Made with Spent Abrasive Slag

MIX #	CEMENT lb	SPENT SLAG lb	SILICA FUME lb	SODIUM SILICATE lb	SUPER PLASTICIZER oz/cwt
MONT 3	705	1,100	84.6	0.0	12.0
MONT 4	705	1,100	0.0	14.1	5.3
MONT 11	705	1,100	84.6	28.2	16.6

1 lb = 0.45 kg
1 oz/cwt = 0.65 mL per kg of cement

The TCLP chromium and cadmium concentrations for extracts of the Montopolis mixes tested are shown in Figure 4.12. TCLP lead concentrations were below the lead detection limit of 0.07 mg/L for all three mixes. TCLP cadmium concentrations were 0.17 mg/L for the Mont 3 and Mont 4 mixes, and 0.14 mg/L for the Mont 11 mix. TCLP chromium concentrations were 2.38 mg/L, 1.60 mg/L and 1.42 mg/L for the Mont 3, Mont 4 and Mont 11 mixes, respectively. The TCLP chromium concentrations for these three mixes were found to be statistically different.

Twenty-eight-day compressive strength values for the three Montopolis mixes are presented in Figure 4.13. The mix containing both silica fume and sodium silicate, Mont 11, had the highest compressive strength at 4,620 psi (31,900 MPa). The mix containing only silica fume (Mont 3) had a higher compressive strength than the mix containing only sodium silicate (Mont 4).

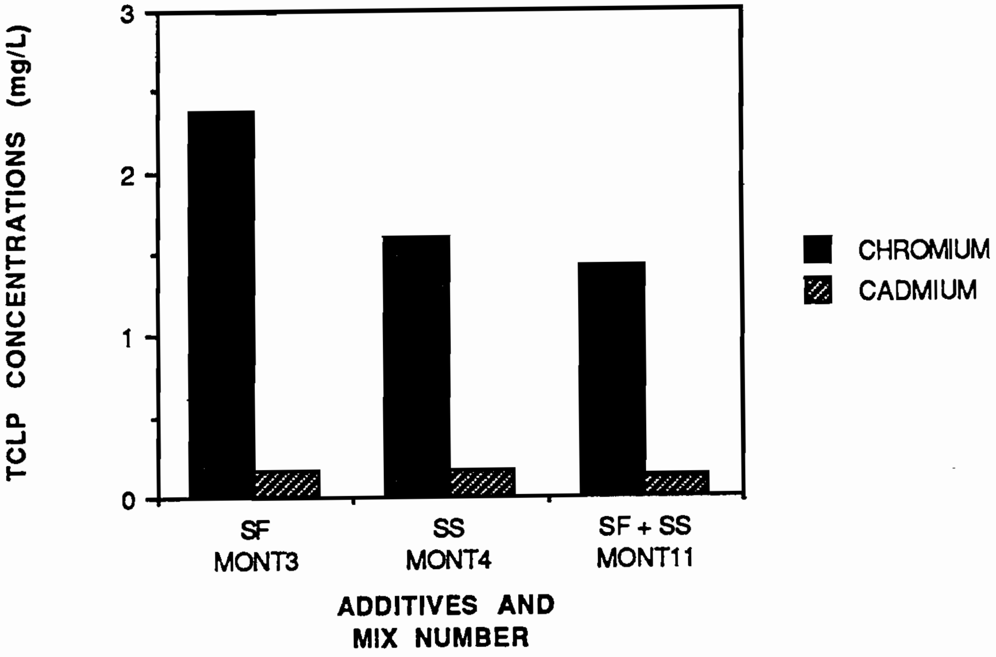


Figure 4.12: TCLP Concentrations for Montopolis Mixes

All three mixes tested achieved the objectives stated in Section 4.1. TCLP chromium, cadmium and lead concentrations were below the EPA limits given in Table 4.1. All three mixes also provided compressive strengths greater than 1,000 psi (6.9 MPa). In summary, the incorporation of sodium silicate alone (Mont 4) decreased the chromium TCLP concentration, while the addition of silica fume alone (Mont 3) resulted in a higher compressive strength. When both silica fume and sodium silicate were incorporated in the mix (Mont 11), the highest compressive strength was obtained and the lowest TCLP chromium and cadmium concentrations resulted.

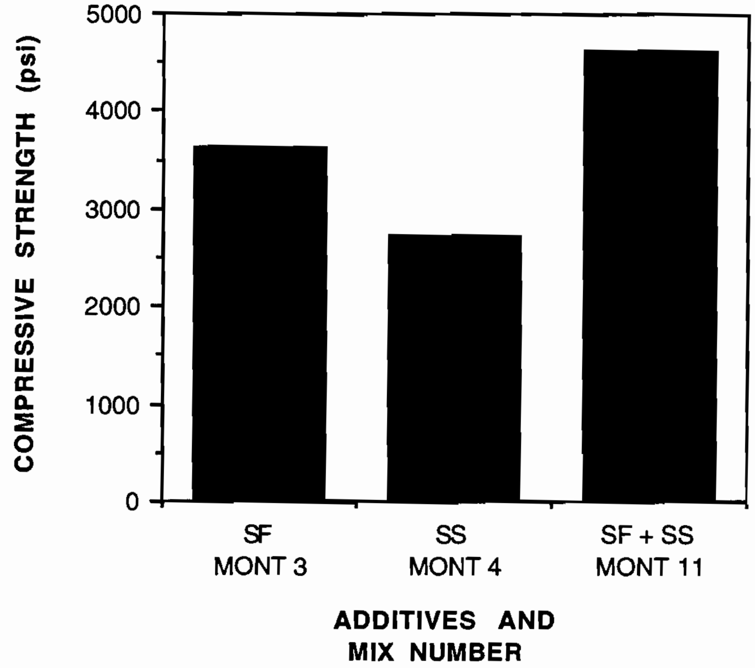


Figure 4.13: 28-Day Compressive Strengths for Montopolis Mixes

4.6 Field Application at Rainbow Bridge, Port Arthur, Texas

From the results of the work done by Brabrand (1992), a mix design was chosen for use at TxDOT's Rainbow Bridge site in Port Arthur, Texas. Three forms of spent abrasive media resulted from the repainting operations at Rainbow Bridge: separated spent abrasive sand, separated spent abrasive dust, and unseparated spent abrasive sand/dust. The spent abrasive media was incorporated into approximately 50,000 12-inch-square by 6-inch-thick (30.5-cm-square by 15.2-cm-thick) concrete blocks. The blocks are to be used as filler material in dolphins, which are used to protect the bridge piers from damage due to ship collisions (Salt 1993).

The mix proportions used are given in Table 4.8. The 1,100 lb (495 kg) of spent abrasive media was either all unseparated spent abrasive sand/dust, or 917 lb (kg) separated spent abrasive sand with 183 lb (kg) separated spent abrasive dust.

Table 4.8: Mix Proportions Used in Field Application at Rainbow Bridge

Ingredient	Quantity
Portland Cement	705 lb
Spent Abrasive Media*	1,100 lb
Superplasticizer	21.8 oz/cwt
Silica Fume	84.6 lb
Water/Cement Ratio	0.35

*1,100 lb of unseparated spent abrasive sand/dust or 917 lb of separated spent abrasive sand with 183 lb of separated spent abrasive dust

1 lb = 0.45 kg

1 oz/cwt = 0.65 mL per kg of cement

Approximately one year after casting, three of the concrete blocks were chosen at random and sent to The University of Texas at Austin for testing. As the blocks were chosen at random, it was not known whether they contained unseparated spent abrasive sand/dust or separated spent abrasive sand with separated spent abrasive dust.

TCLP lead concentrations were at or below the detection limit of 0.07 mg/L for each block. TCLP cadmium concentrations were 0.20 mg/L for blocks #1 and #3, and 0.21 mg/L for block #2. TCLP chromium concentrations for the three blocks are shown in Figure 4.14. TCLP chromium concentrations were 2.27 mg/L, 0.70 mg/L and 0.49 mg/L for blocks #1, #2 and #3, respectively.

Compressive strength values for the three blocks are shown in Figure 4.15. Block #1 had a compressive strength of 360 psi, while blocks #2 and #3 had compressive strengths of 5,970 psi and 2,320 psi, respectively.

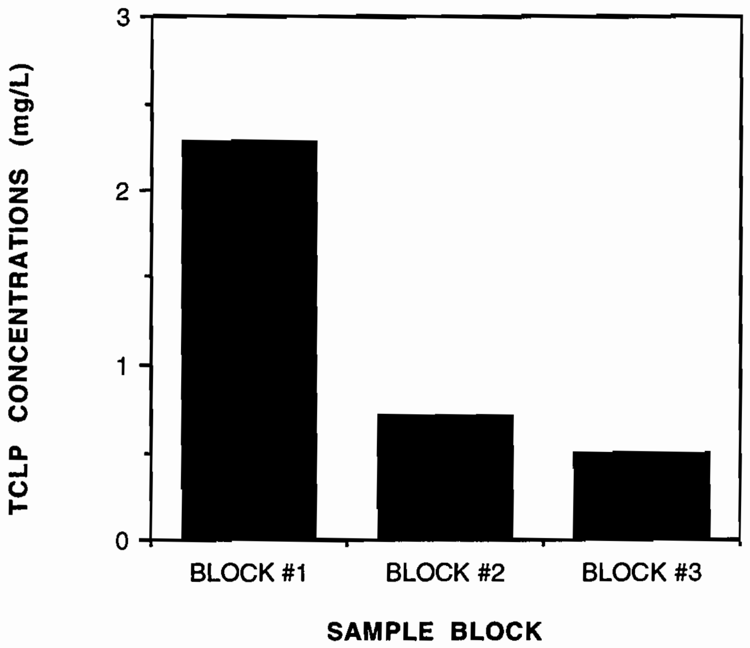


Figure 4.14: TCLP Chromium Concentration for Rainbow Bridge Field Application Sample

In summary, all Rainbow Bridge field application samples met the first objective of this study. TCLP chromium, cadmium and lead concentrations were below the EPA limits (Table 4.1) for all three blocks. Compressive strength values exceeded 1,000 psi (6.9 MPa) for two of the blocks, but block #1 had a compressive strength of only 360 psi (2,480 MPa).

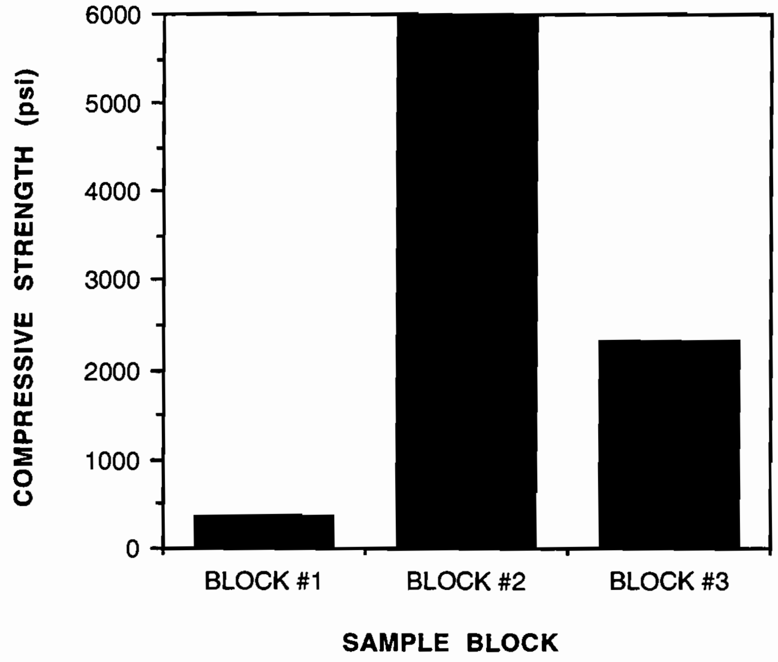


Figure 4.15: Compressive Strengths for Rainbow Bridge Field Application Samples

4.7 Sequential Extractions

Sequential extraction leaching tests were carried out on three S/S mixes prepared for the Port Arthur experiments. These tests were conducted to further investigate the leaching behavior of chromium, cadmium and lead from S/S products. As discussed in Section 2.5, pH control has been determined to be an integral part of effective metal stabilization in concrete systems. During single batch extractions, such as the TCLP, leaching of the alkalinity present in concrete leads to high leachate pH values. At these high pH values, metal complexes (such as hydroxides) are relatively insoluble. By subjecting S/S products to sequential extractions, long-term leaching profiles may be observed. It is possible that, over time, the alkalinity of the concrete may be leached to the extent that the pH drops to levels at which metal complexes are more soluble. This may lead to the leaching of metals at substantial levels.

The procedure used was explained in Section 3.11. To reiterate, a 50g sample was extracted with 1L of extraction fluid. The leachate was analyzed for chromium, cadmium and lead concentrations and pH. The solids were then separated from the leachate and subjected to extraction with a fresh liter of extraction fluid. This procedure was repeated for a total of seven extractions, using fresh leachant for each extraction. Tests were conducted using three different leachants: TCLP extraction fluid #2, seawater and freshwater.

The three mixes used for the sequential extraction tests were SDT 204 D1, SDT 204 D2 and SDT 205 D1. A summary of the mix composition for these three mixes is given in Table 4.9. Appendix F contains leachate chromium, cadmium and lead concentrations and pH data for these tests.

Table 4.9: Composition Summary for Port Arthur Mixes* Used in Sequential Extraction Tests

MIX #	CEMENT lb	SPENT SAND lb	SPENT DUST† lb	SUPER PLASTICIZER oz/cwt
SDT 204 D1	705	1,100	0 (0%)	0.0
SDT 204 D2	705	1,100	55 (5%)	18.9
SDT 205 D1	470	1,100	0 (0%)	12.0

†Separated spent abrasive dust was incorporated as a percent addition based on the amount of separated spent abrasive sand in the mixes
1 lb = 0.45 kg
1 oz/cwt = 0.65 mL per kg of cement

4.7.1 Acidic Sequential Extractions

Acidic sequential extractions were conducted using TCLP extraction fluid #2 as the leachant. Extraction fluid #2 is comprised of 5.7 mL of glacial acetic acid diluted to 1 L with distilled deionized water. The pH of the solution is 2.88.

Figure 4.16 shows the leachate chromium concentration for each mix for the seven sequential extractions. For SDT 204 D1, the leachate chromium concentration peaked at the third extraction at a value of 3.79 mg/L. It then decreased with each extraction to a value of 0.50 mg/L at extraction seven. For SDT 204 D2, the leachate chromium concentration rose slightly to a value of 2.42 mg/L at extraction two, and then steadily decreased to a value of 0.43 mg/L at extraction seven. For SDT 205 D1, the leachate chromium concentration was 3.45 mg/L for the first extraction and then steadily decreased. Although it appears that the value for the fourth extraction is greater than the value for the third extraction, no statistical difference was found between these concentrations.

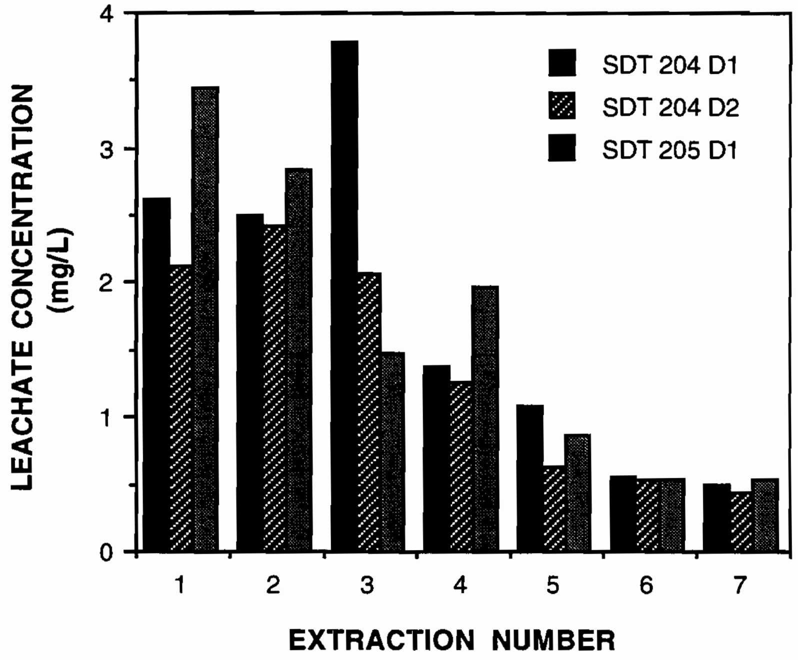


Figure 4.16: Leachate Chromium Concentrations for Acidic Sequential Extractions

The general trend for chromium leaching during acidic sequential extractions showed increasing concentration to a peak value in the first few extractions, followed by a gradual decline in leachate chromium concentration for each subsequent extraction. SDT 204 D1 and SDT 204 D2 followed very similar patterns, with a slight increase over the first few extractions before reaching a peak. SDT 205 D1 varied slightly from this behavior in that the peak leachate chromium concentration occurred in the first extraction.

Figure 4.17 shows the leachate cadmium concentration for each mix for the seven sequential extractions. For SDT 204 D1, the leachate cadmium concentration rose dramatically at extraction three, reaching a concentration of 1.39 mg/L. The concentration then decreased to 0.39 mg/L at extraction four and gradually decreased to 0.07 mg/L at

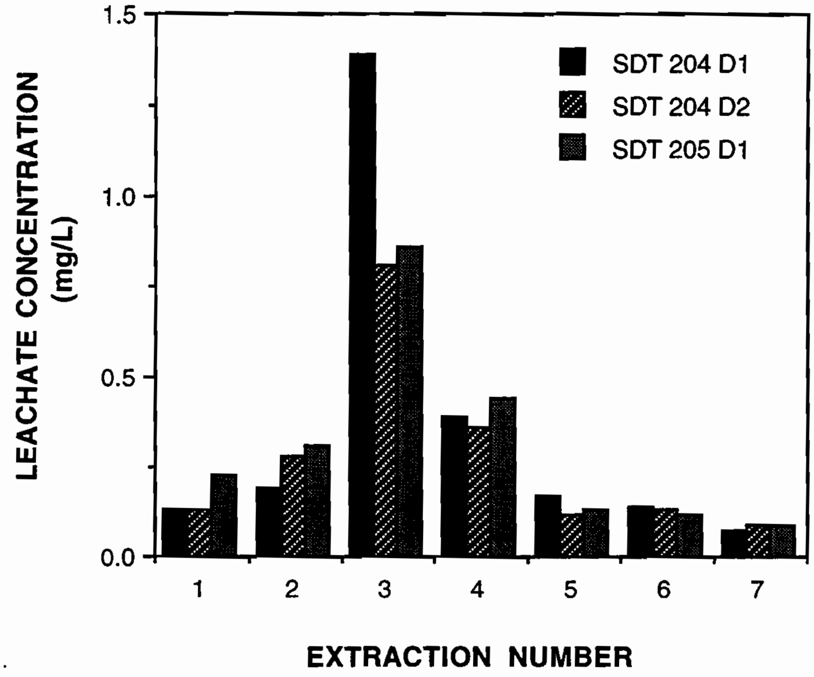


Figure 4.17: Leachate Cadmium Concentrations for Acidic Sequential Extractions

extraction seven. For SDT 204 D2, the leachate cadmium concentration followed a similar pattern, with a maximum concentration of 0.81 mg/L at extraction three. Results for SDT 205 D1 were also similar, with a maximum value of 0.86 mg/L reached at extraction three.

The general trend for cadmium leaching during acidic sequential extractions showed a slow rise in leachate cadmium concentration, followed by a dramatic peak value, and then a slow decline in leachate cadmium concentration. All three mixes followed this trend in a similar manner.

Figure 4.18 shows the leachate lead concentration for each mix for the seven sequential extractions. For SDT 204 D1, the leachate lead concentration was below the detection limit of 0.07 mg/L for the first extraction. The leachate lead concentration then rose gradually for each subsequent extraction, reaching a value of 5.39 mg/L at extraction seven. Similarly, for SDT 204 D2, the leachate lead concentration was below the detection limit of 0.07 mg/L for the first extraction and then rose gradually for each subsequent extraction, reaching a value of 6.03 mg/L at extractions six and seven. For SDT 205 D1, the leachate lead concentration was below the detection limit of 0.07 mg/L for the first extraction and then rose quickly, reaching a value of 11.21 mg/L at extraction six.

The general trend for lead leaching during acidic sequential extractions showed very low leachate lead concentrations for the first few extractions, followed by large increases in leachate lead concentrations. The highest values observed occurred at extraction six and seven. Upon statistical comparison, there was no difference between the leachate lead concentrations at extraction six and those at extraction seven for any of the three mixes. It is difficult to determine whether the leachate lead concentration would have continued to increase or would have decreased had more sequential extractions been conducted on the mixes.

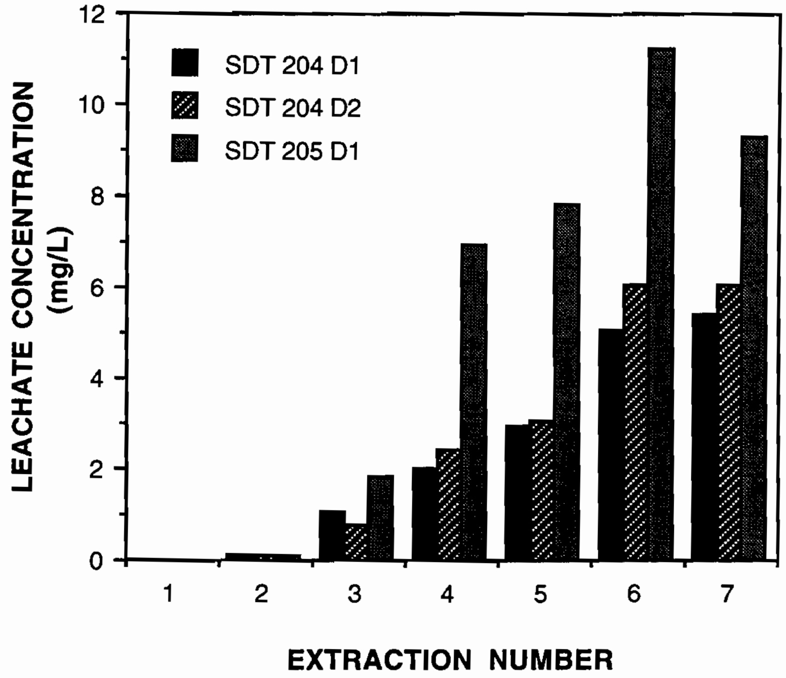


Figure 4.18: Leachate Lead Concentrations for Acidic Sequential Extractions

Figure 4.19 shows the leachate pH for each mix for the seven sequential extractions. As fresh leachant was used for each extraction, the pH of the leachant was the same at the beginning of each extraction. Leachate pH values were very similar for each of the three mixes at each extraction. Leachate pH values ranged from 11.3 to 11.4 for the first extraction, then decreased rapidly through extraction four. For extractions five through seven, the leachate pH value for each mix decreased gradually, with values ranging from 3.3 to 3.4 at extraction seven.

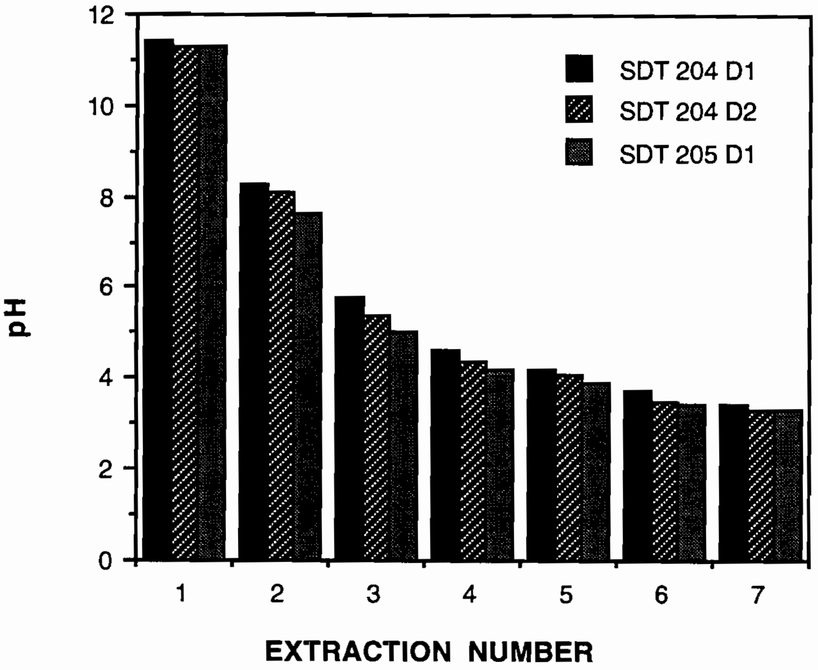


Figure 4.19: Leachate pH for Acidic Sequential Extractions

4.7.2 Seawater Sequential Extractions

Sequential extractions were conducted using seawater as the leachant. Seawater was used in sequential extraction leaching tests to better simulate the actual conditions S/S products made from spent abrasive media may encounter in tidal areas of Texas and the Gulf Coast. The seawater used in this research was obtained from the Gulf of Mexico near Corpus Christi, Texas, and was described in Section 3.2.13.

Figure 4.20 shows the leachate chromium concentration for each mix for the seven extractions. For SDT 204 D1, the leachate chromium concentration was 2.41 mg/L for the first extraction. It then decreased to a value of 0.32 mg/L at extraction five, followed by a mild increase to 0.57 mg/L at extraction seven. For SDT 204 D2, the leachate chromium concentration was 4.91 mg/L for the first extraction and decreased for each subsequent extraction, reaching a value of 0.27 mg/L at extraction seven. For SDT 205 D1, the leachate chromium concentration was 6.54 mg/L for the first extraction and decreased to 0.38 mg/L at extraction five. There was no statistical difference between the leachate chromium concentrations for extractions five, six and seven for SDT 205 D1.

The general trend for chromium leaching during seawater sequential extractions showed initially high leachate chromium concentrations, followed by a quick decrease from extraction two to extraction three, and finally a gradual decrease from extraction three to extraction seven. SDT 204 D1, SDT 204 D2 and SDT 205 D1 all followed this pattern, with some minor exceptions at the later extractions, when concentrations were relatively low.

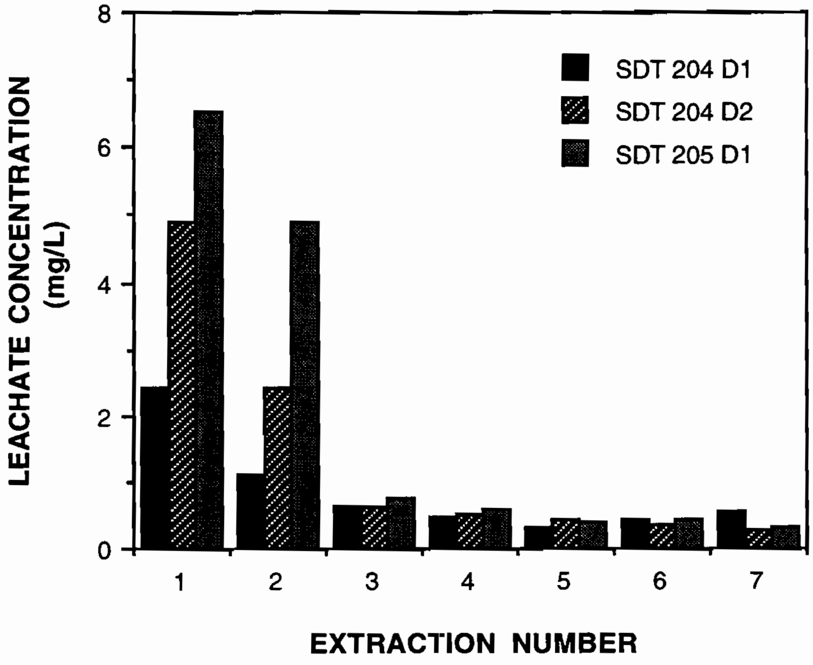


Figure 4.20: Leachate Chromium Concentrations for Seawater Sequential Extractions

Figure 4.21 shows the leachate cadmium concentration for each mix for the seven extractions. Leachate cadmium concentrations were very low for each mix at every extraction. The highest value observed was 0.16 mg/L for SDT 205 D1 for the first extraction. For SDT 204 D1, the leachate cadmium concentration ranged from below the detection limit of 0.02 mg/L to 0.12 mg/L. For SDT 204 D2, the leachate cadmium concentration ranged from 0.05 mg/L to 0.14 mg/L. For SDT 205 D1, the leachate cadmium concentration ranged from 0.04 mg/L to 0.16 mg/L. No statistically supported trends could be observed for leachate cadmium concentrations for the seven seawater sequential extractions.

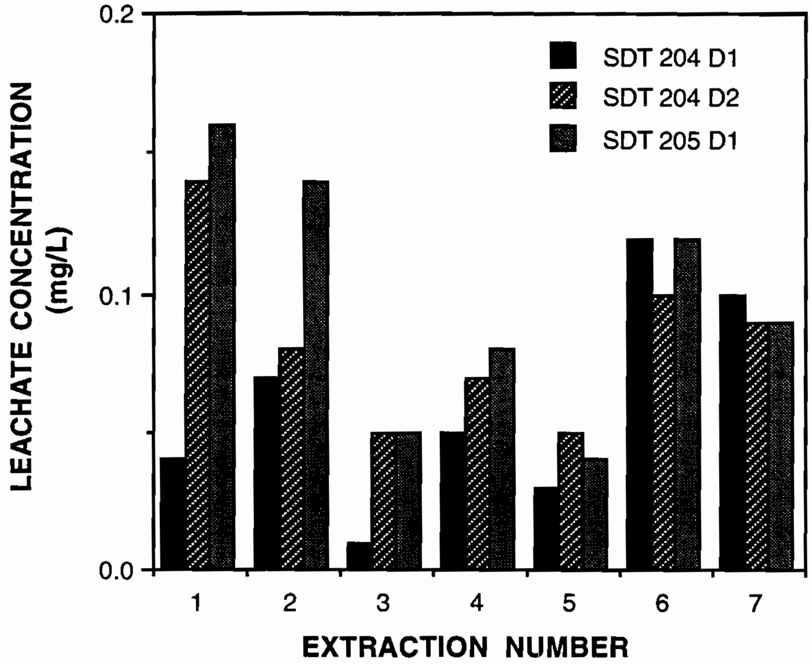


Figure 4.21: Leachate Cadmium Concentrations for Seawater Sequential Extractions

Leachate lead concentrations for the seawater sequential extractions were very low for all three mixes. Many were below the detection limit of 0.07 mg/L. The highest leachate lead concentration found was 0.12 mg/L.

Figure 4.22 shows the leachate pH for each mix for the seven sequential extractions. Leachate pH values ranged from 10.4 to 11.6 for the first extraction. For the second extraction, the pH dropped to values ranging from 6.5 to 8.6. For extractions two through seven, the differences in pH from one extraction to the next were not statistically significant. The only exception was that for SDT 204 D1. The leachate pH at extraction five (4.7) was statistically lower than the pH at extractions four (9.1) and six (8.6). With this one exception, the leachate pH ranged from 5.9 to 9.1, a range of roughly three pH units, after the initial drop in pH from extraction one to extraction two.

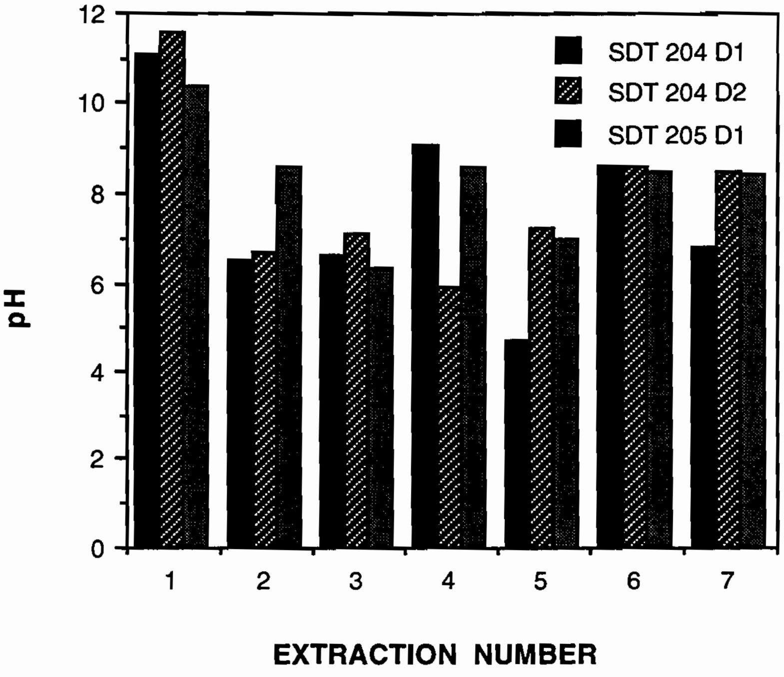


Figure 4.22: Leachate pH for Seawater Sequential Extractions

4.7.3 Freshwater Sequential Extractions

Sequential extractions were attempted using freshwater as the leachant. Freshwater was to be used in sequential extraction leaching tests to better simulate the actual conditions S/S products made from spent abrasive media may encounter in upland streams and lakes. The freshwater used was obtained from the Town Lake portion of the Colorado River in Austin, Texas (Section 3.2.14).

Problems were encountered upon attempting concentration analyses on the freshwater sequential extraction leachates. Reliable results could not be obtained upon introduction of leachate samples to the ICP-AES (Section 3.9.4) for concentration analysis. Reasons for the complications were not discerned.

The ICP-AES makes two replicate readings for each sample analyzed. The average of these two replicates is then reported. For the freshwater sequential extraction leachates, the two replicate readings were highly variable for each sample. In many instances, concentration readings would as much as double from one replicate to the next. Many attempts were made to remedy the problem, including changes in ICP-AES parameters and sample introduction methods. None of the measures taken resulted in reliable concentration data.

4.8 Summary of Results

This chapter presented the results of the physical and chemical tests conducted on concrete mortars resulting from the solidification/stabilization (S/S) of spent abrasive media. S/S mixes were prepared incorporating varying amounts of spent abrasive media in four forms: separated spent abrasive sand, separated spent abrasive dust, unseparated spent abrasive sand/dust, and spent abrasive slag. Additives used consisted of silica fume, fly ash, superplasticizer, calcium nitrite and sodium silicate.

As stated in Section 4.1, successful solidification/stabilization of the spent abrasive media was gauged by two objectives:

1) To immobilize the metals contained in the spent abrasive media.
2) To provide sufficient compressive strength for the concrete products to be used as non-structural concrete.

For all of the solidification/stabilization products tested, TCLP chromium, cadmium and lead concentrations were below the EPA limits shown in Table 4.1. With the exception of one mix, all S/S products provided a compressive strength of 1,000 psi (6.9 MPa), as required by TxDOT. The effects of mix composition on the leaching behavior and compressive strength of the S/S products made in these experiments are discussed in Chapter 5.

Field application of S/S technology for the reuse of spent abrasive media was initiated by TxDOT, and three field samples were acquired for TCLP and compressive strength testing. All of the samples tested met the first objective of immobilizing the metals present in spent abrasive media with respect to the EPA limits for TCLP leaching. One of the three samples, however, did not provide a compressive strength of at least 1,000 psi (6.9 MPa). A discussion of these results appears in Chapter 5.

Sequential extraction tests were performed on three S/S mixes to further investigate the leaching of chromium, cadmium and lead from S/S products made with spent abrasive media. The relationships between leachate concentrations, pH and type of leachant are investigated in Chapter 5.

CHAPTER 5: DISCUSSION

5.1 Introduction

As previously stated, the objectives of this research were to:

1) Immobilize the metals in spent abrasive media using portland cement.
2) Provide sufficient compressive strength for these concrete products to be used as non-structural concrete.
3) Examine the leaching behavior of metals in concrete products and elucidate the possible mechanisms that control the leaching of the metals.
4) Compare the leaching behavior of concrete products made from spent abrasive media with varying characteristics.
5) Compare the leaching behavior of these concrete products under the influence of different leachants.

To achieve the first objective, TCLP concentrations cannot exceed the EPA limits for chromium, cadmium and lead, which were shown in Table 4.1. For all the mixes tested in this study, TCLP chromium, cadmium and lead concentrations were below the EPA limits. The results of this study show that solidification/stabilization is an effective means of immobilizing the metals in spent blasting abrasives with respect to TCLP leaching.

To achieve the second objective, the S/S products must have compressive strengths of at least 1,000 psi (6.9 MPa), as required by the Texas Department of Transportation (TxDOT). With one exception (SDT 204 D2), this objective was met for all of the mixes tested in this study. Thus, with this exception, the S/S products tested provided sufficient compressive strengths for the material to be used for non-structural concrete purposes.

The remaining three objectives deal with the examination of the leaching behavior of metals from S/S products. The effect of mix composition on the leaching of metals from S/S products was investigated using the results of the Odessa, Port Arthur, Sodium Silicate and Montopolis Bridge experiments. To further investigate the leaching of chromium, cadmium and lead from S/S products made with spent abrasive media, sequential extraction tests were performed on three S/S mixes. The relationships between leachate concentrations, pH and type of leachant are investigated in this chapter.

5.2 Effect of Mix Composition on Metals Leaching and Compressive Strength

This section examines the effect of mix composition on TCLP chromium, cadmium and lead concentration. The effect of mix composition on compressive strength has also been considered. A detailed discussion on the effects of mix composition on the structural properties of the S/S products tested in this study appears in the M.S. thesis of Bryan Salt (Salt 1993).

5.2.1 Effect of Abrasive Media Type and Contamination Level

Four types of spent abrasive media were used in this study: unseparated spent abrasive sand/dust (Section 3.2.12), separated spent abrasive sand (Section 3.2.10), separated spent abrasive dust (Section 3.2.11) and spent abrasive slag (Section 3.2.9). Each of these types of media had different total chromium, cadmium and lead contents. These metal contents were given in Chapter 3 and are summarized in Table 5.1. In the case of unseparated spent abrasive sand/dust, two barrels were used, each with different total metal contents.

Statistical comparisons were made between TCLP chromium, cadmium and lead concentrations for extracts of mixes prepared with different media types. The unpaired Student's t-test with a 95% confidence level was used for the statistical comparisons reported in this chapter

(Section 3.14.2). A summary of the mix composition for the mixes compared is shown in Table 5.2.

Table 5.1: Total Metal Contents* for Spent Abrasive Media Used in This Study

Media	Total Metal Content (Based on wet weight)		
	Chromium	Cadmium	Lead
Odessa Unseparated Spent Abrasive Sand - Barrel #1	53 mg/kg	11 mg/kg	246 mg/kg
Odessa Unseparated Spent Abrasive Sand - Barrel #2	80 mg/kg	7 mg/kg	184 mg/kg
Port Arthur Separated Spent Abrasive Sand	462 mg/kg	27 mg/kg	1,580 mg/kg
Port Arthur Separated Spent Abrasive Dust	968 mg/kg	85 mg/kg	6,610 mg/kg
Montopolis Bridge Spent Abrasive Slag	564 mg/kg	61 mg/kg	1,941 mg/kg

*As determined by procedure explained in Section 3.10

The first three mixes in Table 5.2, SDT 400, SDT 600 and SDT 204 D1, differed only in the type of spent abrasive sand used in the mix. For SDT 400 and SDT 600, unseparated spent abrasive sand from Odessa Barrel #1 and Barrel #2, respectively, was used. For SDT 204 D1, Port Arthur separated spent abrasive sand was used. The Port Arthur separated spent abrasive sand had much higher chromium and lead contents and a slightly higher cadmium concentration than the unseparated spent abrasive sand from either of the Odessa barrels.

Table 5.2: Composition Summary for Mixes Used in Media Comparison

MIX #	CEMENT lb	SPENT MEDIA lb	TYPE OF MEDIA	SILICA FUME lb
SDT 400	705	1,100	Odessa Sand Barrel #1	0.0
SDT 600	705	1,100	Odessa Sand Barrel #2	0.0
SDT 204 D1	705	1,100	Port Arthur Separated Sand	0.0
SDT 401	705	1,100	Odessa Sand Barrel #1	84.6
SDT 601	705	1,100	Odessa Sand Barrel #2	84.6
SDT 209 D1	705	1,100	Port Arthur Separated Sand	84.6
MONT 3	705	1,100	Spent Slag	84.6

1 lb = 0.45 kg

TCLP concentrations for these three mixes are shown in Figure 5.1. Statistical comparisons were conducted on the TCLP chromium, cadmium and lead concentrations for extracts of the SDT 400 and SDT 204 D1 mixes. The TCLP chromium concentrations were statistically different. The TCLP chromium concentration was higher for the SDT 204 D1 mix which was made with spent abrasive sand containing a greater amount of lead. TCLP cadmium and lead concentrations were not statistically different despite the much larger concentrations of these metals in the separated spent abrasive sand used in the SDT 204 D1 mix. No statistical differences were found in TCLP concentrations between SDT 400 and SDT 600.

Statistical comparisons of the TCLP chromium, cadmium and lead concentrations for extracts of three of the mixes containing silica fume (SDT 401, SDT 209 D1 and MONT 3) were conducted. TCLP concentrations for these three mixes are shown in Figure 5.2. TCLP chromium and cadmium concentrations were higher for MONT 3 than for

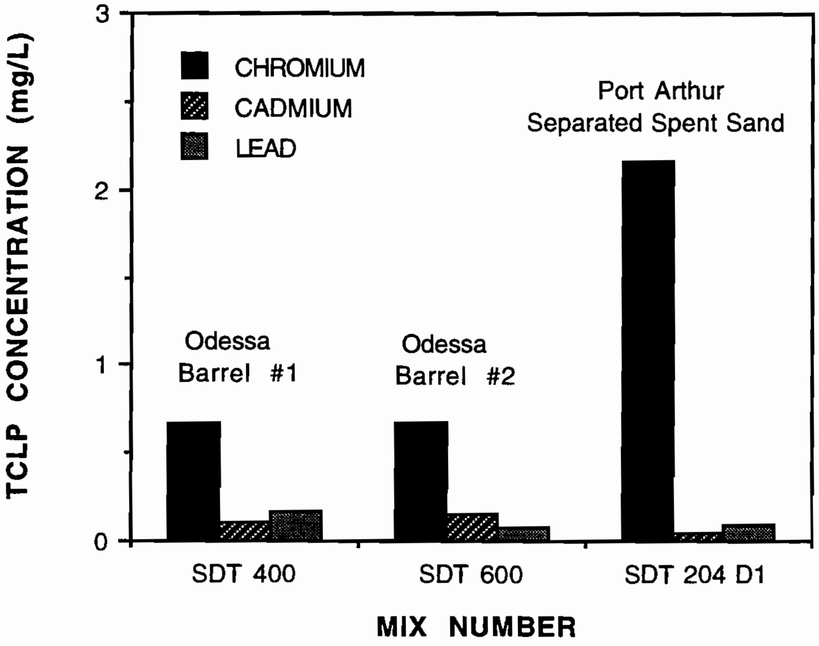


Figure 5.1: TCLP Concentrations for Media Comparison

either SDT 401 or SDT 209 D1. MONT 3 was made with spent abrasive slag containing higher amounts of chromium and cadmium than either the unseparated spent abrasive sand or the separated spent abrasive sand. TCLP lead concentrations were not statistically different, even though the lead content of the slag was higher as well. Upon a comparison of SDT 401 to SDT 209 D1, the TCLP chromium concentration was higher for SDT 209 D1, while no statistical differences existed between the TCLP cadmium and lead concentrations for these two mixes.

In the Odessa experiments, two barrels of unseparated spent abrasive media were used to prepare mixes of comparable composition, such as the SDT 400 and SDT 600 mixes considered above. As noted in Section 4.2.2, no statistical trends were observed upon comparing the TCLP concentrations of the corresponding Barrel #1 and Barrel #2 mixes.

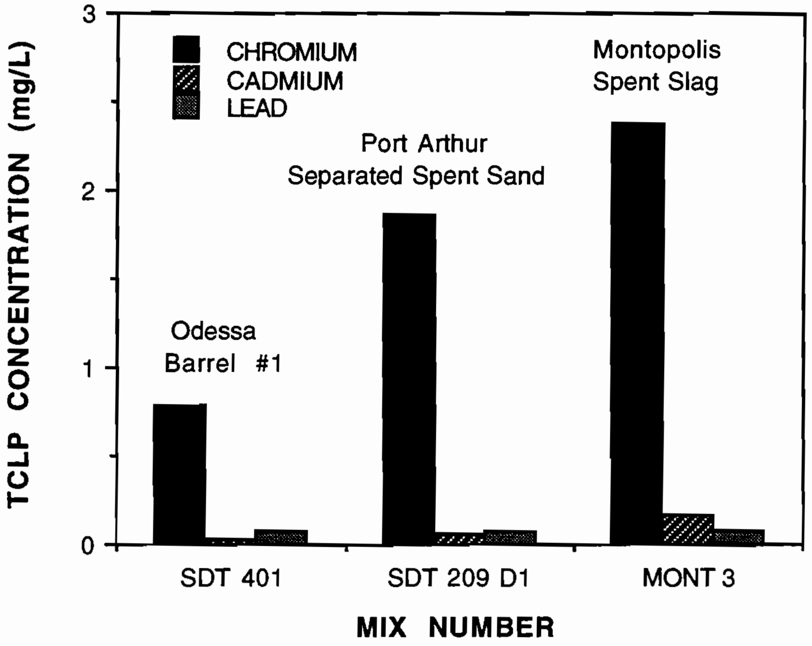


Figure 5.2: TCLP Concentrations for Media Comparison; Mixes Containing Silica Fume

The results of these analyses led to a few conclusions concerning the effect of varied metal content of spent abrasive media on the TCLP chromium, cadmium and lead concentrations. For large increases in media chromium content, TCLP chromium concentrations were increased. Examples of this behavior were the increased TCLP chromium concentrations as the media chromium content increased from the unseparated spent abrasive sand (53 mg/kg) to the separated spent abrasive sand (462 mg/kg) to the spent abrasive slag (564 mg/kg). For smaller increases in media chromium content, such as between Odessa Barrel #1 (53 mg/kg) and Barrel #2 (80 mg/kg), no appreciable effect on TCLP chromium concentration was noticed.

The TCLP cadmium concentration was highest for the mix prepared with spent abrasive slag, which had a much higher chromium content (61 mg/kg) than the other media (11 mg/kg for the unseparated sand and 27 mg/kg for the separated sand). No difference in TCLP cadmium concentration was found for the mixes prepared with separated and unseparated spent abrasive sand, where a smaller difference in media chromium concentration existed.

The TCLP lead concentrations were found to be independent of media type. No statistical differences in TCLP lead concentration were found for mixes made from media with total lead contents ranging from 246 mg/kg (unseparated spent abrasive media) to 1,941 mg/kg (spent abrasive slag).

Table 5.3 shows the ranges of TCLP concentrations for each set of experiments and the type of media used in those experiments. TCLP cadmium and lead concentrations were very low for all of the mixes tested. The type of media and extent of media contamination had little effect on TCLP cadmium and lead leaching. TCLP chromium concentrations were more dependent on the media contamination level than were the TCLP cadmium and lead concentrations. While the TCLP chromium concentrations changed little with small increases in media chromium

Table 5.3: Comparison of TCLP Concentration Ranges for Different Media Types

		TCLP Concentration Range (mg/L)		
Experiment	Media Type	Chromium	Cadmium	Lead
Odessa	Unseparated*	0.49 - 1.10	<0.02 - 0.34	<0.07 - 0.19
Port Arthur	Separated†	1.72 - 2.97	0.04 - 0.19	<0.07 - 0.19
Montopolis	Slag	1.42 - 2.38	0.14 - 0.17	all <0.07
Sodium Silicate	Separated Dustˣ	0.20 - 0.80	0.13 - 0.17	all <0.07

*Unseparated spent abrasive sand/dust

†Separated spent abrasive sand and separated spent abrasive dust

ˣConcrete river sand and separated spent abrasive dust content, large increases in media chromium content led to more substantial increases in TCLP chromium concentration.

This section has focused on the effects of media type and extent of contamination on the leaching of chromium, cadmium and lead from the S/S mixes prepared in this study. The types of media examined were unseparated spent abrasive sand/dust, spent abrasive slag and separated spent abrasive sand. Separated spent abrasive dust was also incorporated into many of the mixes studied. A discussion of the effect of separated spent abrasive dust content on the leaching of chromium, cadmium and lead from the S/S products studied appears in the next section.

5.2.2 Effect of Separated Spent Abrasive Dust Content

Separated spent abrasive dust was incorporated into the S/S mixes as a percent addition based on the amount of sand in the mix. The Port Arthur and sodium silicate mixes contained separated spent abrasive dust. Mix compositions and TCLP results were given in Sections 4.3 and 4.4, respectively. Mixes were prepared with 0% (control mixes), 5%, 15% and 25% additions of separated spent abrasive dust.

For the Port Arthur mixes, no statistical correlation was found between the separated spent abrasive dust content and the TCLP chromium concentration. The only instance of increased TCLP chromium concentration with increased dust content occurred in the SDT 207 series mixes. With the addition of 5% dust, the TCLP chromium concentration increased with respect to the control mix (no dust). Even for the SDT 208 and SDT 209 series where 15% and 25% additions of dust were achieved, there was no statistical difference in chromium concentration between mixes within these series.

Similar results were observed for TCLP cadmium and lead concentrations. No statistical trends were observed that would indicate a relationship between dust content and TCLP concentration. Increasing the dust content affected the physical properties (compressive strength and set time) of the S/S product more than the chemical properties (TCLP leaching) (Section 4.3).

Increased dust content inhibited the cementation process and increased set times. As previously stated (Section 4.3), for the SDT 204, 205, 206 and 207 series mixes, only a 5% addition of dust allowed for a set time of less than 7 days. The addition of silica fume in the SDT 208 and SDT 209 series mixes allowed for a 15% and 25% addition of dust, respectively.

In most instances, increased dust content led to lower compressive strengths. A few exceptions did occur in which mixes with higher dust contents had higher compressive strengths than the control mix or mixes with lower dust contents (Salt 1993).

For the sodium silicate mixes, TCLP chromium concentrations did increase with increased dust content. With two exceptions, the TCLP chromium concentration was statistically higher for mixes with higher dust contents. TCLP chromium concentrations increased as the dust content was increased from 0% (control mix) to 5%, 15% and 25%. The difference in behavior for the sodium silicate mixes compared to the Port Arthur mixes could be attributed to the type of sand used in each experiment.

The Port Arthur mixes used separated spent abrasive sand, while the sodium silicate mixes used uncontaminated concrete river sand. As a result, the Port Arthur mixes contained much higher amounts of chromium, cadmium and lead. TCLP concentrations for the Port Arthur mixes were most likely controlled by the spent abrasive sand. For the sodium silicate mixes, TCLP concentrations were a direct result of the dust content, as the separated spent abrasive dust was the only source of chromium, cadmium and lead in these mixes. The TCLP chromium concentrations for the Port Arthur mixes were much higher than those for the sodium silicate mixes. The effects of increased dust content on TCLP leaching were dampened by the relatively high concentrations resulting from the spent sand in the mixes.

TCLP cadmium and lead concentrations were independent of dust content for the sodium silicate mixes. For each series of the sodium silicate mixes, the compressive strengths of mixes containing dust (CM #2, CM #3, CM #4, Sili 1B, 1C, 1D, 2B, 2C and 2D) were lower than the compressive strengths of the mixes with no dust (CM #1, Sili 1A and Sili 2A). In most instances, an increase in dust content from a 5% addition to 15% and 25% additions led to decreased compressive strengths, although a few exceptions occurred (Salt 1993).

5.2.3 Effect of Cement Content

Of all the mixes that set, 16 pairs of mixes differed only in cement content. In other words, the mix composition for each mix in the pair was identical, with the exception of the amount of cement in the mix. Mixes were based either on a five-sack cement content or a seven-and-a-half-sack cement content.

Table 5.4: Comparison of TCLP Chromium Concentrations for Mixes with Different Cement Contents

	Five-Sack Mix		Seven-and-a-Half-Sack Mix	
Pair	Mix Number	TCLP Chromium Concentration	Mix Number	TCLP Chromium Concentration
1	SDT 302	0.98 mg/L	SDT 402	0.46 mg/L
2	SDT 303	1.01 mg/L	SDT 403	0.62 mg/L
3	SDT 304	0.71 mg/L	SDT 404	0.53 mg/L
4	SDT 501	1.10 mg/L	SDT 601	0.61 mg/L
5	SDT 502	0.93 mg/L	SDT 602	0.73 mg/L
6	SDT 503	0.77 mg/L	SDT 603	0.71 mg/L
7	SDT 206 D2	2.47 mg/L	SDT 207 D2	1.92 mg/L
8	SDT 208 D1	2.65 mg/L	SDT 209 D1	1.87 mg/L
9	SDT 208 D3	2.41 mg/L	SDT 209 D3	1.79 mg/L

Statistical comparisons were made of the TCLP chromium concentrations for each pair of mixes. For each pair, the TCLP chromium concentration of the lower cement content mix was compared to that of the higher cement content mix. Of the 16 pairs, 9 had mixes with statistically different TCLP chromium concentrations. Table 5.4 shows these mixes and their TCLP chromium concentrations. Mix compositions were summarized in Chapter 4. In each case, the TCLP chromium concentration was higher for the mix with the lower cement content. Figure 5.3 shows the TCLP chromium concentration comparisons for each of these nine pairs of mixes.

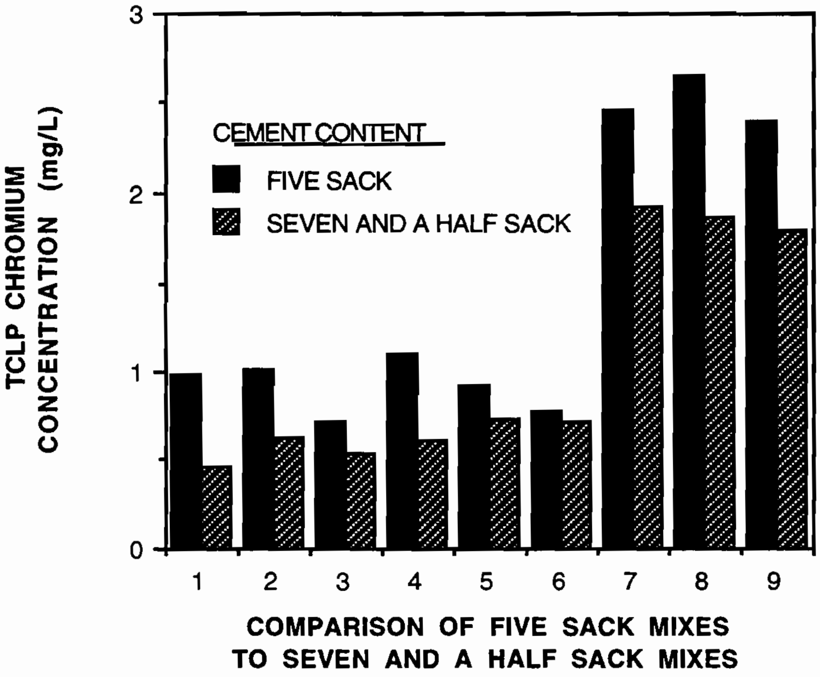


Figure 5.3: TCLP Chromium Concentration Comparisons for Mixes With Different Cement Contents

Statistical comparisons were also made of the TCLP cadmium concentrations for each pair of mixes. For each pair, the TCLP cadmium concentration of the lower cement content mix was compared to that of the higher cement content mix. Of the 16 pairs, 6 had mixes with statistically different TCLP cadmium concentrations. Table 5.5 shows these mixes and their TCLP cadmium concentrations. For 4 of these 6 cases, the TCLP chromium concentration was higher for the mix with the lower cement content. The exceptions were SDT 304 vs. SDT 404 and SDT 206D2 vs. SDT 207D2. Figure 5.4 shows the TCLP cadmium concentration comparisons for these six pairs of mixes.

Table 5.5: Comparison of TCLP Cadmium Concentrations for Mixes with Different Cement Contents

	Five-Sack Mix		Seven-and-a-Half-Sack Mix	
Pair	Mix Number	TCLP Cadmium Concentration	Mix Number	TCLP Cadmium Concentration
1	SDT 301	0.12 mg/L	SDT 401	0.03 mg/L
2	SDT 302	0.12 mg/L	SDT 402	0.02 mg/L
3	SDT 303	0.14 mg/L	SDT 403	0.02 mg/L
4	SDT 304	0.03 mg/L	SDT 404	0.05 mg/L
5	SDT 205 D1	0.15 mg/L	SDT 204 D1	0.04 mg/L
6	SDT 206 D2	0.14 mg/L	SDT 207 D2	0.19 mg/L

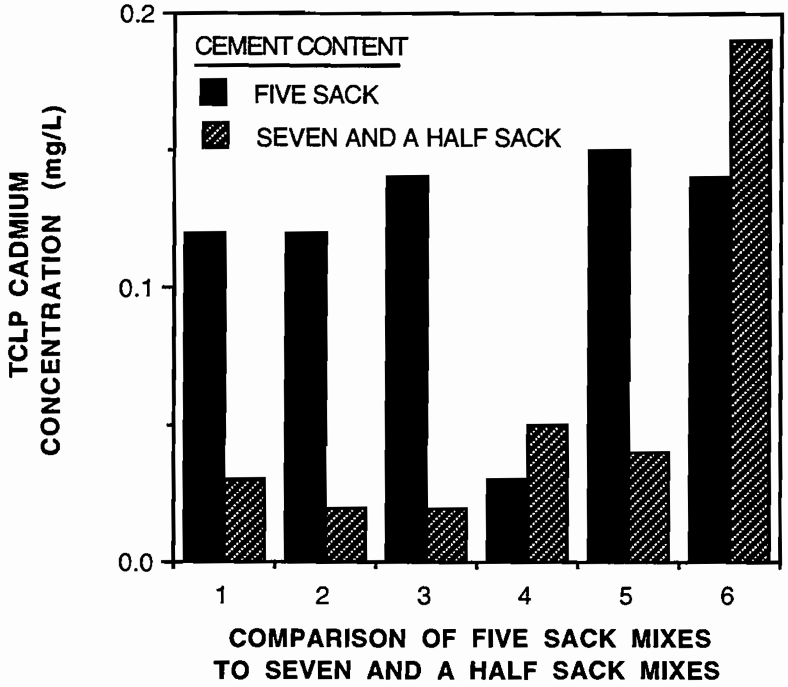


Figure 5.4: TCLP Cadmium Concentration Comparisons for Mixes With Different Cement Contents

Statistical comparisons were conducted on the TCLP lead concentrations for each pair of mixes. For each pair, the TCLP lead concentration of the lower cement content mix was compared to that of the higher cement content mix. Of the 16 pairs, 11 had mixes with statistically different TCLP lead concentrations. Table 5.6 shows these mixes and their TCLP lead concentrations. For the Odessa Barrel #1 mixes (SDT 300 and SDT 400 series) and the Port Arthur mixes (SDT 204 through SDT 209 mixes), the mixes with the lower cement content had lower TCLP lead concentrations than the mixes with the higher cement content. The five-sack mixes had TCLP lead concentrations below the detection limit of 0.07 mg/L. For the Odessa Barrel #2 mixes (SDT 500 and SDT 600 series), the mixes with the lower cement content had higher TCLP lead concentrations.

Table 5.6: Comparison of TCLP Lead Concentrations for Mixes with Different Cement Contents

Five-Sack Mix		Seven-and-a-Half-Sack Mix	
Mix Number	TCLP Lead Concentration	Mix Number	TCLP Lead Concentration
SDT 300	<0.07 mg/L	SDT 400	0.17 mg/L
SDT 302	<0.07 mg/L	SDT 402	0.12 mg/L
SDT 303	<0.07 mg/L	SDT 403	0.19 mg/L
SDT 500	0.07 mg/L	SDT 600	<0.07 mg/L
SDT 501	0.11 mg/L	SDT 601	<0.07 mg/L
SDT 502	0.09 mg/L	SDT 602	<0.07 mg/L
SDT 503	0.08 mg/L	SDT 603	<0.07 mg/L
SDT 206 D1	<0.07 mg/L	SDT 207 D1	0.18 mg/L
SDT 206 D2	<0.07 mg/L	SDT 207 D2	0.19 mg/L
SDT 208 D1	<0.07 mg/L	SDT 209 D1	0.08 mg/L
SDT 208 D3	<0.07 mg/L	SDT 209 D3	0.08 mg/L

The seven-and-a-half-sack mixes (SDT 600 series) had TCLP lead concentrations below the detection limit of 0.07 mg/L.

TCLP cadmium and lead concentrations were very low for all of the mixes studied. The effects of cement content on the leaching of cadmium and lead from these S/S products were less evident than the effects of cement content on the leaching of chromium. These effects were also less important given the low TCLP cadmium and lead concentrations. TCLP chromium concentrations were generally lower for mixes with higher cement contents. For both the Odessa and the Port Arthur experiments, the compressive strength increased as the cement content increased (Salt 1993). Figure 5.5 compares the compressive strengths of a few of the pairs of mixes with different cement contents.

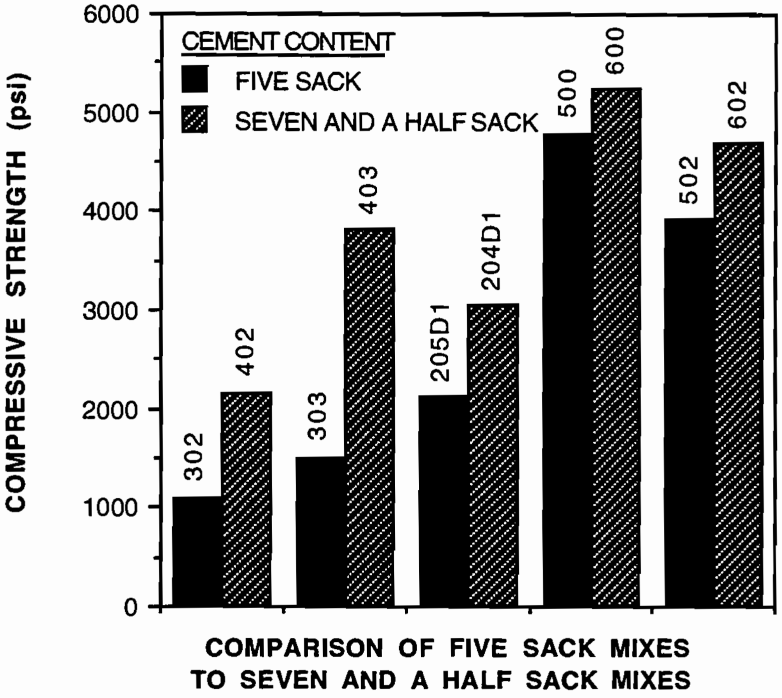


Figure 5.5: Comparison of Compressive Strengths for Mixes With Different Cement Contents (SDT Mix Numbers are shown)

5.2.4 Effect of Silica Fume

Silica fume (Section 3.2.4) was incorporated into some of the Odessa, Port Arthur and Montopolis Bridge mixes. In the Odessa (Section 4.2) and Port Arthur (Section 4.3) experiments, seven pairs of mixes varied only in silica fume content. For each pair, one mix contained silica fume while one did not. Silica fume was added as a 12% addition based on the portland cement content.

In only one case did the presence of silica fume in a mix (SDT 301) lead to a statistically increased TCLP chromium concentration with respect to the mix containing no silica fume (SDT 300). The TCLP chromium concentration was increased from 0.6 mg/L (SDT 300) to 0.73 mg/L (SDT 301). No correlation was found between TCLP cadmium or lead concentration and silica fume content. Silica fume was effective at increasing the compressive strength of mixes, and was necessary for mixes with more than a 5% addition of dust to achieve a set time of less than 7 days (Salt 1993).

For the Montopolis Bridge mixes studied, one mix contained silica fume (Mont 3), one contained sodium silicate (Mont 4) and one contained both silica fume and sodium silicate (Mont 11). A higher compressive strength was obtained for Mont 3 than for Mont 4. The TCLP chromium concentration was also higher for Mont 3, however. For Mont 11, the TCLP chromium concentration was the lowest, and the compressive strength was the highest. It appears that in the case of the Montopolis Bridge mixes, silica fume was more effective than sodium silicate at increasing the compressive strength of the mixes (Salt 1993), while sodium silicate was more effective at reducing the TCLP chromium concentration. No correlation was found between TCLP cadmium or lead concentration and silica fume content for these three mixes.

5.2.5 Effect of DCI

DCI corrosion inhibitor (Section 3.2.7), a calcium nitrite-based liquid, was used in some of the mixes. Dosages of 2.0 gallons (7.6L) and 4.0 gallons (15.2 L) per cubic yard (0.77 m^3) of concrete were used. DCI was used in Odessa mixes and Port Arthur mixes.

For the Odessa Barrel #1 experiments, mixes were prepared that varied from the control mix only in DCI content. One mix in each of the SDT 300 and SDT 400 series mixes contained 2.0 gallons (7.6L) and one contained 4.0 gallons (15.2 L). TCLP chromium concentrations for these mixes were compared to the TCLP chromium concentrations of the control mix, which contained no DCI. For the SDT 300 series, the lower DCI content mix (SDT 304) had a statistically higher TCLP chromium concentration than the control mix (SDT 300). The TCLP chromium concentration of the mix with the higher DCI content (SDT 305) was not statistically different than that of the control mix. For the SDT 400 series, the TCLP chromium concentration was progressively lowered as the DCI content was raised from 0 (SDT 400) to 2.0 gallons (SDT 404) to 4.0 gallons (SDT 405).

TCLP cadmium concentrations for the mixes containing DCI were also compared to those of the control mixes. No correlation was found between TCLP cadmium concentrations and DCI content. The TCLP lead concentrations were also compared. For the SDT 300 series mixes, no correlation was found between TCLP lead concentrations and DCI content. For the SDT 400 series mixes, the TCLP lead concentration was lowered from 0.17 mg/L for the control mix with no DCI (SDT 400) to below the detection limit of 0.07 mg/L for each of the mixes containing DCI (SDT 404 and SDT 405).

DCI was effective at increasing the compressive strengths of S/S products made from spent abrasive media (Salt 1993). For the Odessa mixes, the effect was substantial, as can be seen in Figure 4.3. Compressive strengths were also increased for the Port Arthur mixes containing DCI. DCI was more effective than silica fume at increasing the compressive strengths of these mixes. As stated earlier, however, silica fume was required for mixes containing more than a 5% addition of dust to set within 7 days (Salt 1993).

5.2.6 Effect of Sodium Silicate

Sodium silicate (Section 3.2.8) was used in the sodium silicate mixes and some of the Montopolis Bridge mixes. Doses were based on the sodium silicate to portland cement ratio. Values of 0.02 and 0.04 based on weight were used.

Statistical comparisons were conducted on the mixes that varied only in sodium silicate content (e.g. CM #1, Sili 1A and Sili 2A). The sodium silicate content had no effect on TCLP chromium, cadmium or lead concentrations. Sodium silicate was effective at increasing compressive strengths for mixes containing separated spent abrasive dust, as shown in Figure 4.11 (Salt 1993). As stated in Section 5.2.4, sodium silicate was more effective than silica fume at lowering TCLP chromium concentrations for the Montopolis mixes. Silica fume was more effective at increasing the compressive strengths of these mixes.

5.2.7 Summary of Effect of Mix Composition on TCLP Leaching and Compressive Strength

TCLP cadmium and lead concentrations were very low for all mixes tested. No consistent trends were observed to indicate any effect of mix composition on the TCLP cadmium and lead concentrations. Mix composition had the most pronounced effect on TCLP chromium concentrations and mix compressive strengths.

The TCLP chromium concentration increased as the total chromium content of the media increased. The only exception was for the Port Arthur experiments, where increased dust contents had no effect on TCLP chromium concentrations. This was believed to be a result of the dominating influence of the separated spent abrasive sand present in these mixes. TCLP chromium concentrations were decreased with increased cement content, as was seen from the results of the Odessa and Port Arthur experiments. Silica fume and sodium silicate were found to have no effect on TCLP chromium concentrations. Increased DCI contents resulted in decreased TCLP chromium concentrations for the SDT 400 series mixes, but had no effect on the TCLP concentrations for the SDT 300 series mixes.

The compressive strengths of the mixes studied were dependent on mix composition. The results of the Odessa and Port Arthur experiments showed that higher cement contents yield higher compressive strengths. Increasing the separated spent abrasive dust content resulted in decreased compressive strengths and inhibited mix setting. The inclusion of silica fume in mixes containing greater than a 5% addition of dust was necessary to achieve a set time of less than 7 days. DCI was more effective than silica fume at increasing mix compressive strengths. Sodium silicate was effective at increasing the compressive strengths of mixes containing separated spent abrasive dust.

5.3 Field Application at Rainbow Bridge, Port Arthur, Texas

As stated in Section 4.6, three blocks of concrete were obtained from TxDOT's Rainbow Bridge site in Port Arthur, Texas, approximately 1 year after casting. The mixes contained either unseparated spent abrasive sand or a mixture of separated spent abrasive sand and separated spent abrasive dust. As the blocks were chosen at random, the type of media used to prepare each block was not known.

Samples were taken from each block and subjected to the TCLP and compressive strength testing. TCLP concentrations were below the EPA limits for each block. The TCLP chromium concentration for Block #1 was substantially higher than those for Blocks #2 and #3 (Figure 4.14). Also, Block #1 provided a compressive strength of only 360 psi (2.5 MPa). Blocks #2 and #3 provided compressive strengths greater than 1,000 psi (6.9 MPa) (Salt 1993).

A possible explanation for the low compressive strength of Block #1 could be a high aluminum content. As discussed in Section 2.4.2, the presence of aluminum in concrete mixes can lead to the production of hydrogen gas, which results in swelling and cracking of the concrete. Acid digestions for total chromium, cadmium, lead and aluminum content were conducted on the three concrete blocks. The results are given in Table 5.7.

The total aluminum content of Block #1 was between the total aluminum contents of Blocks #2 and #3. Therefore, a higher aluminum content was not the reason for the low compressive strength of Block #1. The total chromium and lead contents for Block #1 were substantially higher than those for Blocks #2 and #3. This large disparity in metal concentration may have been the reason for the large variation in compressive strength and TCLP chromium concentration.

The difference in total chromium and lead contents may have been due to the use of different types of spent abrasive media in the blocks. Block #1 may have contained separated spent abrasive dust. Three observations support this assertion. First, the results of the Port Arthur

Table 5.7: Total Metal Contents* for Rainbow Bridge Field Application Blocks

Sample	Total Metal Content in mg/kg (Based on wet weight) (n=3)			
	Mean (CV)†			
	Chromium	Cadmium	Lead	Aluminum
BLOCK #1	250 (8.7%)	33 (24%)	1,315 (2.6%)	8,140 (5.8%)
BLOCK #2	73 (11%)	19 (40%)	300 (8.0%)	7,220 (7.3%)
BLOCK #3	76 (7.3%)	21 (30%)	398 (4.8%)	9,930 (5.6%)

*As determined by procedure explained in Section 3.10.2

†CV = (standard deviation/mean)*100% (Section 3.14.1)

experiments (Section 4.3) showed that the incorporation of separated spent abrasive dust decreases the compressive strength of S/S products.

Second, the Port Arthur separated spent abrasive sand and separated spent abrasive dust had significantly higher total metal contents than the Odessa unseparated spent abrasive sand (Table 5.1). This may have been the result of higher metal contents in the bridge coatings at the Port Arthur site. More likely, the metals were concentrated in the Port Arthur separated spent abrasive sand and dust as the media was reused.

The purpose of conducting the separation processes which result in separated spent abrasive sand and dust is to reclaim some of the abrasive sand for reuse. As it is unlikely that all of a metal (such as chromium or lead) present in the media will be concentrated in the dust in the separation process, metals may accumulate in the separated sand. This will result in higher metal concentrations in both the separated sand and the separated dust than in unseparated sand that is not reused.

The third observation supporting the assertion that Block #1 contained separated spent abrasive sand and separated spent abrasive dust pertains to the TCLP concentration ranges observed in this study. Table 5.3 compared the TCLP concentration ranges observed for experiments conducted on S/S products made with different media types. The TCLP chromium concentrations for Blocks #2 (0.70 mg/L) and #3 (0.49 mg/L) fell within the range of TCLP chromium concentrations observed in the Odessa experiments, where unseparated spent abrasive sand was used. The TCLP chromium concentration for Block #1 (2.27 mg/L) fell within the range of TCLP chromium concentrations observed in the Port Arthur experiments, where unseparated spent abrasive sand and dust were used. No differences in TCLP cadmium and lead concentrations were observed for the three blocks. This was expected, as, throughout this study, TCLP cadmium and lead concentrations were very low for S/S mixes made from all types of media.

In summary, it appears that Block #1 was made with separated spent abrasive sand and separated spent abrasive dust, while Blocks #2 and #3 were made with unseparated spent abrasive sand. This would explain both the very low compressive strength and relatively high TCLP chromium concentration of Block #1.

In the mix design used in this field application (Table 4.8), separated spent abrasive dust was incorporated as a 20% addition based on the separated spent abrasive sand content. The results of the Port Arthur experiments indicated that such high levels of dust could lead to high set times and lowered compressive strengths. While the incorporation of silica fume in the mixes alleviated the problems with set time in the Port Arthur experiments, it may be advisable to incorporate lower amounts of separated spent abrasive dust in the design of mixes to be used for field applications.

5.4 Discussion of Leaching Mechanisms

5.4.1 Introduction

TCLP chromium, cadmium and lead concentrations were below the EPA limits (Table 4.1) for all of the mixes tested. TCLP cadmium and lead concentrations were extremely low for all of the mixes tested. The low leachate metals concentrations may be attributable to pH control. TCLP leachate pH values ranged from 9.1 to 11.7.

As discussed in Section 2.3, the TCLP is an acidic extraction procedure carried out on a ground-up sample of the material being tested. For concrete systems, a single batch extraction results in the leaching of large amounts of the alkalinity present in the concrete. This results in high leachate pH values. The metals of concern in most S/S systems are relatively insoluble at these high pH values. It is possible that the alkalinity content of the cement may be leached away over time, leading to lower leachate pH levels. The metals bound in the concrete matrix may then be more soluble. The concrete matrix may also be degraded as the calcium content is removed through the leaching of alkalinity.

Sequential extractions were conducted on three of the S/S mixes prepared in this study (SDT 204D1, SDT 204D2 and SDT 205D1) to investigate the long-term leaching behavior of chromium, cadmium and lead from the concrete matrix. The procedure was described in Section 3.11 and the results of these leaching tests were presented in Section 4.7. TCLP extraction fluid #2 (pH = 2.88) was used for the acidic sequential extractions. As it is unlikely that S/S products will be subjected to acidic leachants in real-world applications, sequential extractions were also conducted on the same three mixes using seawater (Section 3.2.13) as the leachant. A discussion of the results of these sequential extraction leaching tests follows.

5.4.2 Leachate Metal Concentrations as a Function of pH

Acidic Sequential Extractions

Leachate chromium concentrations were plotted as a function of pH for the acidic sequential extractions. During the sequential extraction procedure, the leachate pH decreased from one extraction to the next. Therefore, the chronological order for these figures is from right to left. Results for SDT 204D1, SDT 204D2 and SDT 205D1 are shown in Figures 5.6, 5.7 and 5.8, respectively.

The lines were drawn by eye to illustrate the leaching trends. For each mix, the leachate chromium concentration was highest for the first few extractions, when the pH was relatively high. As the pH dropped below 6, the leachate chromium concentration dropped as well.

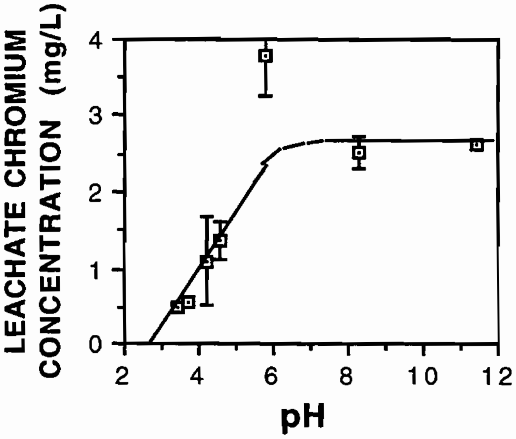


Figure 5.6: Leachate Chromium Concentration vs pH for Acidic Sequential Extractions: SDT 204D1 (shown with standard deviation bars)

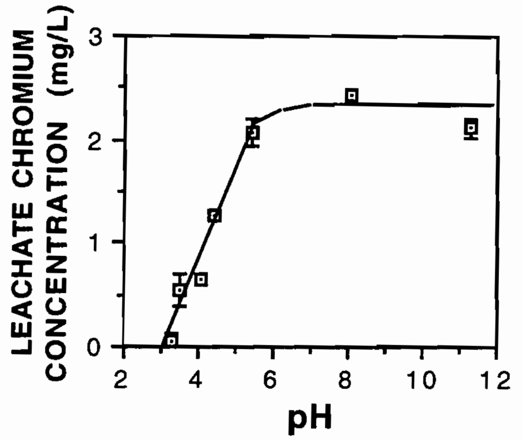


Figure 5.7: Leachate Chromium Concentration vs pH for Acidic Sequential Extractions: SDT 204D2 (shown with standard deviation bars)

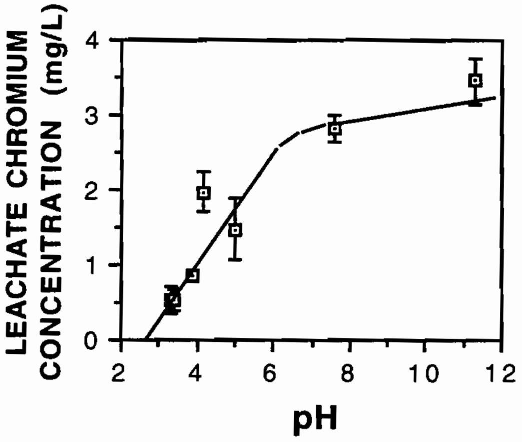


Figure 5.8: Leachate Chromium Concentration vs pH for Acidic Sequential Extractions: SDT 205D1 (shown with standard deviation bars)

Leachate cadmium concentrations also were plotted as a function of pH for the acidic sequential extractions. Results for SDT 204D1, SDT 204D2 and SDT 205D1 are shown in Figures 5.9, 5.10 and 5.11, respectively. For each mix, the pH was high and leachate cadmium concentrations were low for the first few extractions. As the pH fell below 8, leachate cadmium concentrations increased to a peak and then decreased to very low levels.

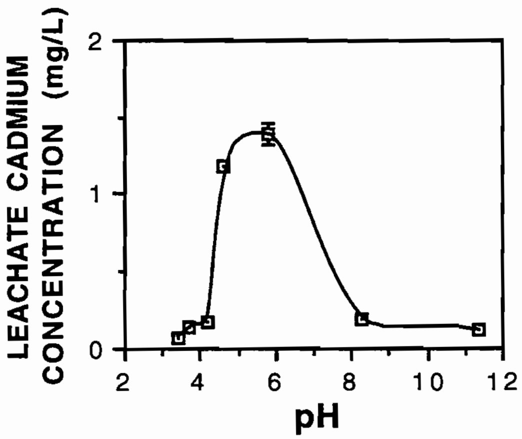


Figure 5.9: Leachate Cadmium Concentration vs pH for Acidic Sequential Extractions: SDT 204D1 (shown with standard deviation bars)

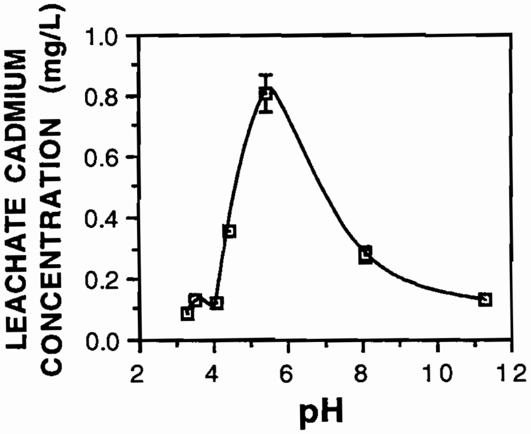


Figure 5.10: Leachate Cadmium Concentration vs pH for Acidic Sequential Extractions: SDT 204D2 (shown with standard deviation bars)

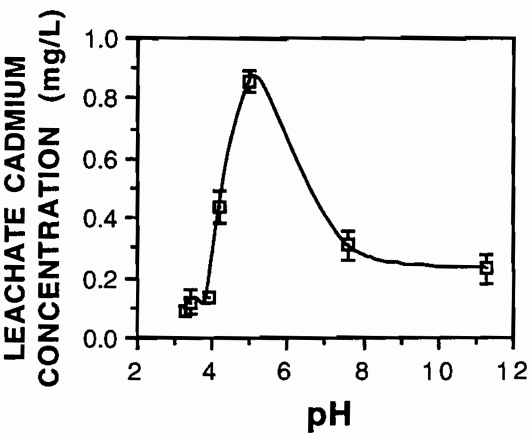


Figure 5.11: Leachate Cadmium Concentration vs pH for Acidic Sequential Extractions: SDT 205D1 (shown with standard deviation bars)

Leachate lead concentrations were plotted as a function of pH for the acidic sequential extractions. Results for SDT 204D1, SDT 204D2 and SDT 205D1 are shown in Figures 5.12, 5.13 and 5.14, respectively. For each mix, pH values were high and the leachate lead concentrations were below the detection limit of 0.07 mg/L for the first few extractions. As the pH fell below 6, a dramatic increase in leachate lead concentration occurred. For the seventh extraction, the pH had been lowered to 3.3 (SDT 205D1) and 3.4 (SDT 204D1 and SDT 204D2), and leachate lead concentrations were increasing.

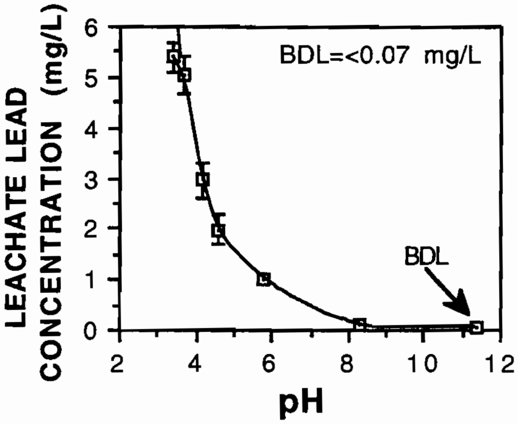


Figure 5.12: Leachate Lead Concentration vs pH for Acidic Sequential Extractions: SDT 204D1 (shown with standard deviation bars)

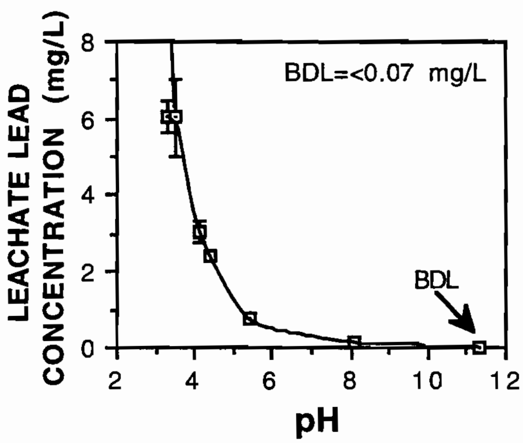


Figure 5.13: Leachate Lead Concentration vs pH for Acidic Sequential Extractions: SDT 204D2 (shown with standard deviation bars)

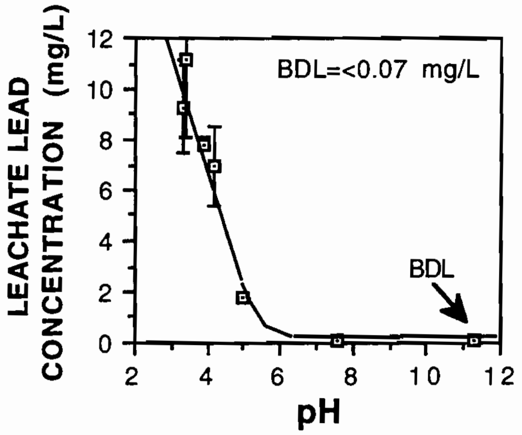


Figure 5.14: Leachate Lead Concentration vs pH for Acidic Sequential Extractions: SDT 205D1 (shown with standard deviation bars)

Before the effect of pH on the leachate chromium, cadmium and lead concentrations could be determined, it was necessary to determine whether the amount of metal present in the concrete was limiting the leachate concentration. Mass balance calculations were conducted to determine how much of each metal had leached out of the concrete matrix throughout the seven extractions. The leachate concentrations were summed for the seven extractions and converted to units of mg metal per kg cement as follows (0.05 kg of concrete were extracted in 1L of extraction fluid):

$$\text{Conc. (mg / L) x } \frac{1.0 \text{ L leachate}}{0.05 \text{ kg concrete}} = \text{Conc. (mg / kg).}$$

The total amount leached was then compared to the total metal contents of each mix. The total metal content and cumulative metal leached for chromium, cadmium and lead for the acidic sequential extractions are given in Table 5.8.

For each mix, the cumulative amounts of chromium and lead leached were close to or greater than the total metal content of the mix.

The cumulative amount of lead leached was much less than the total lead content for each mix. It appears that chromium and cadmium leaching became limited by the amount of these metals in the concrete. Most of the chromium and cadmium in the mixes had leached out by the seventh extraction. Lead leaching was not limited by the amount of lead present in the concrete. The percentage of the total lead in the mix that leached during the acidic sequential extraction was 29%, 31% and 50% for SDT 204D1, SDT 204D2 and SDT 205D1, respectively.

Table 5.8: Total Metal Contents[*] and Cumulative Amount Leached for the Acidic Sequential Extractions

Mix	Total Metal Content in mg/kg (Based on wet weight) (n=5)		
	Mean (CV)[†]		
	Chromium	Cadmium	Lead
SDT 204D1	218 (7.8 %)	22 (6.8 %)	1143 (21 %)
SDT 204D2	212 (13 %)	19 (17 %)	1185 (30 %)
SDT 205D1	225 (6.2 %)	29 (3.8 %)	1500 (60 %)
	Cumulative Amount Leached[x] in mg/kg (n=3)		
SDT 204D1	250	50	330
SDT 204D2	190	39	370
SDT 205D1	230	44	740

[*] As determined by procedure explained in Section 3.10.2

[†] CV = (standard deviation/mean)*100% (Section 3.14.1)

[x] Concentrations are the average of three replicate samples

The effect of pH on metal hydroxide solubilities was discussed in Section 2.5. As the amount of chromium and cadmium available limited the leaching of these metals, it is not possible to compare leachate concentrations to the theoretical hydroxide solubilities. The leachate lead concentrations were much lower than what would be predicted by the theoretical lead hydroxide solubility. The theoretical lead hydroxide solubility, as well as data from the acidic sequential extractions on SDT 205D1, was plotted in Figure 5.15. SDT 205D1 was chosen, as it had the highest leachate lead concentrations of the three mixes. If the leachate lead concentrations for this mix were below the theoretical solubility, the leachate lead concentrations of the other two mixes would be as well. The minimum solubility for lead hydroxide is approximately 10 mg/L, which occurs at a pH of 10. For pH values above and below 10, the solubility increases. Leachate lead concentrations for SDT 205D1 were several orders of magnitude lower than the theoretical hydroxide solubility for all of the pH values encountered. Therefore, the lead (+2) hydroxide solubility does not explain the lead leaching behavior observed in the acidic sequential extractions.

Adsorption/desorption processes are also a function of pH. In Section 2.5, adsorption was discussed as a potential metal stabilization mechanism in S/S systems. Desorption of chromium, cadmium and lead from amorphous silica gel is believed to occur as the pH drops below values of approximately 5.5, 8.5 and 6.5, respectively (Table 2.2). These pH values were termed the sorption edge for each metal. As the leachate chromium concentration was maximum for the highest pH values encountered (which were greater than 11), no evidence was found to support the claim that chromium leaching was controlled by adsorption processes.

Although leachate cadmium concentrations decreased at low pH values, after having reached a peak, an initial rise in concentration was observed for each mix at a pH value near 8 (Figures 5.9 through 5.11). It is possible that cadmium was adsorbed in the concrete matrix until the pH

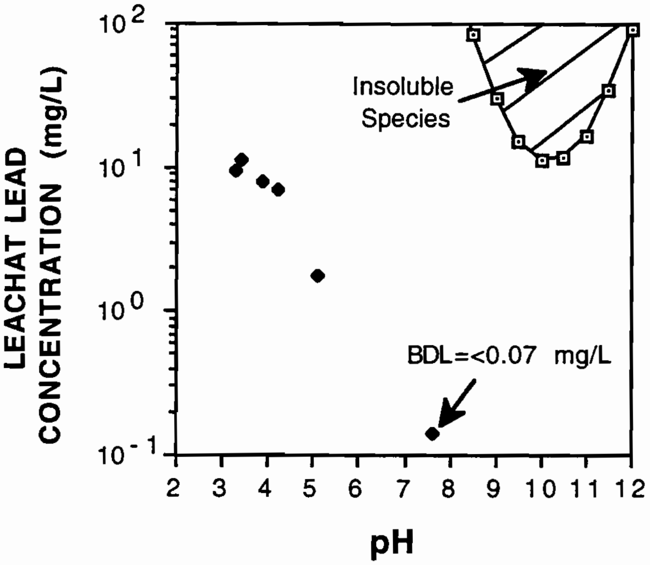


Figure 5.15: Leachate Lead Concentrations for Acidic Extractions on SDT 205D1 Compared to the Theoretical Lead(+2) Hydroxide Solubility

was decreased below the sorption edge. Leachate cadmium concentrations then increased due to desorption until mass limitations caused them to decrease.

For each mix, a substantial increase in leachate lead concentration was observed as the pH decreased below a value slightly higher than 6 (Figures 5.12 through 5.14). The pH range of the sorption edge for lead was given as 5.0 to 6.5 (Table 2.2). Lead may have been adsorbed in the concrete matrix until the pH was decreased below 6.5, at which point desorption occurred and leachate lead concentrations increased.

Seawater Sequential Extractions

Leachate concentrations also were plotted as a function of pH for the seawater sequential extractions. Leachate chromium concentrations for SDT 204D1, SDT 204D2 and SDT 205D2 are shown in Figures 5.16, 5.17 and 5.18, respectively. The lines were drawn by eye to illustrate the leaching trends. For each mix, the leachate chromium concentration and pH was the highest for the first extraction. Leachate chromium concentrations then decreased to below 1 mg/L for pH values less than 10.

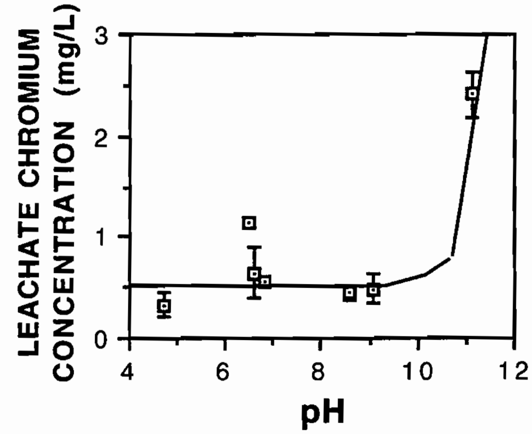


Figure 5.16: Leachate Chromium Concentration vs pH for Seawater Sequential Extractions: SDT 204D1 (shown with standard deviation bars)

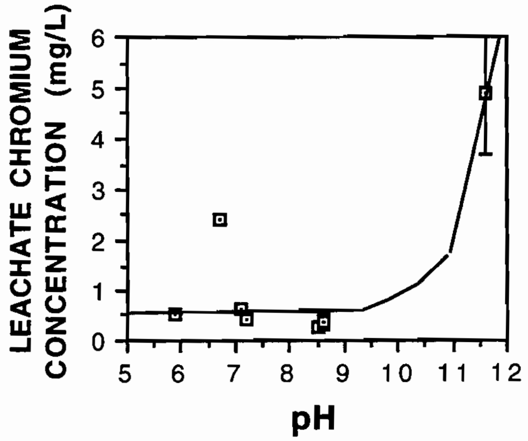


Figure 5.17: Leachate Chromium Concentration vs pH for Seawater Sequential Extractions: SDT 204D2 (shown with standard deviation bars)

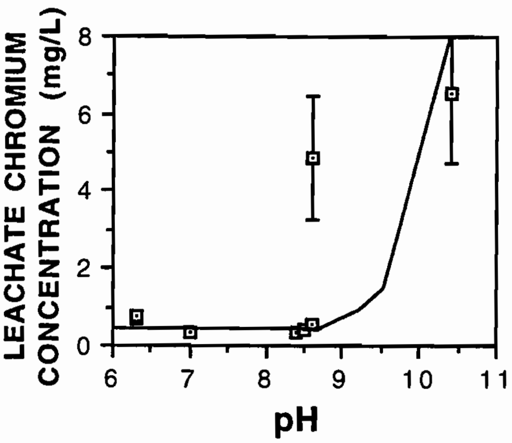


Figure 5.18: Leachate Chromium Concentration vs pH for Seawater Sequential Extractions: SDT 205D1 (shown with standard deviation bars)

Leachate cadmium concentrations were plotted as a function of pH for the seawater sequential extractions. Results for SDT 204D1, 204D2 and 205 D1 are shown in Figures 5.19, 5.20 and 5.21, respectively. For SDT 204D1, the data was scattered with no clear relationship between pH and leachate cadmium concentration. For SDT 204D2 and SDT 205D1, the leachate cadmium concentration decreased with decreasing pH. Leachate cadmium concentrations were very low for each mix throughout the seven extractions. The maximum leachate cadmium concentration encountered for the seawater sequential extractions was 0.16 mg/L.

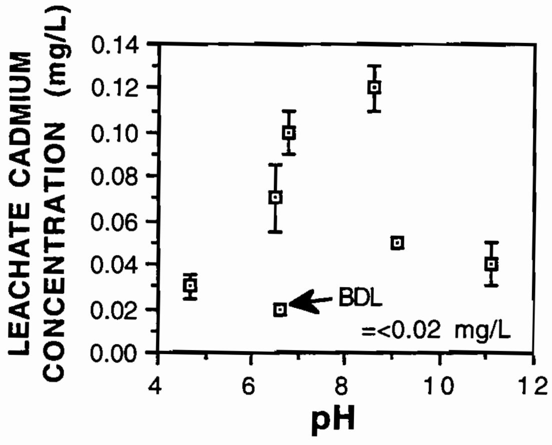


Figure 5.19: Leachate Cadmium Concentration vs pH for Seawater Sequential Extractions: SDT 204D1 (shown with standard deviation bars)

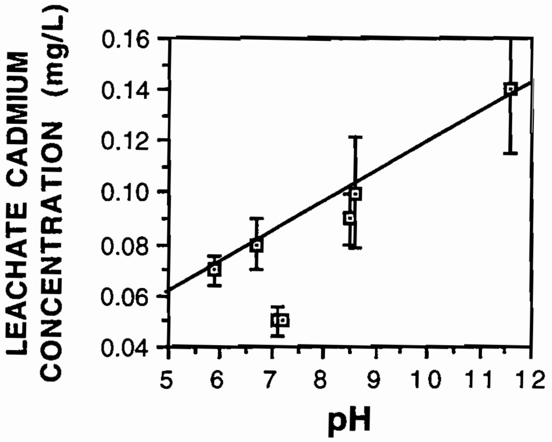


Figure 5.20: Leachate Cadmium Concentration vs pH for Seawater Sequential Extractions: SDT 204D2 (shown with standard deviation bars)

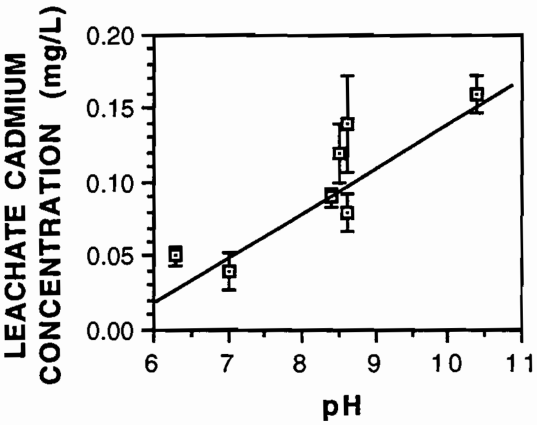


Figure 5.21: Leachate Cadmium Concentration vs pH for Seawater Sequential Extractions: SDT 205D1 (shown with standard deviation bars)

Leachate lead concentrations were very low for each mix throughout the seven extractions. The highest leachate lead concentration observed for any of the three mixes was 0.12 mg/L. No correlation could be made between pH and leachate lead concentration for the seawater sequential extractions.

Once again, mass balance calculations were made to determine whether the amount of chromium, cadmium and lead present in the concrete limited the leaching of the metals. The total metal content and cumulative metal leached for chromium, cadmium and lead for the seawater sequential extractions are given in Table 5.9. To check the mass balance calculations, acid digestions for total chromium, cadmium and lead content were conducted on the solid residue remaining after seven extractions. These results are given in Table 5.10.

Table 5.9: Total Metal Contents[*] and Cumulative Amount Leached for the Seawater Sequential Extractions

Mix	Total Metal Content in mg/kg (Based on wet weight) (n=5)		
	Mean (CV)[†]		
	Chromium	Cadmium	Lead
SDT 204D1	218 (7.8 %)	22 (6.8 %)	1,143 (21 %)
SDT 204D2	212 (13 %)	19 (17 %)	1,185 (30 %)
SDT 205D1	225 (6.2 %)	29 (3.8 %)	1,500 (60 %)
	Cumulative Amount Leached[x] in mg/kg		
SDT 204D1	120	8.1	3.0
SDT 204D2	190	12	2.2
SDT 205D1	280	14	12

[*] As determined by procedure explained in Section 3.10.2

[†] CV = (standard deviation/mean)*100% (Section 3.14.1)

[x] Concentrations are the average of three replicate samples

Mass balance differences were encountered for the seawater sequential extractions. The difference in the total metal content of the unleached mix (Table 5.9) and the sequential extraction residue (Table 5.10) was compared to the cumulative amount of metal leached (Table 5.10) for each metal and each mix. For chromium, more mass leached out of the concrete than was indicated by the difference in total metal contents. For lead, the opposite occurred. Almost no lead leached during the seawater sequential extractions, yet the total metal content of the residue was substantially less than that of the unleached concrete. Cadmium mass balance differences were less significant than those for chromium and lead.

Table 5.10: Total Metal Contents* for Residue of the Seawater Sequential Extractions

Mix	Total Metal Content in mg/kg (Based on wet weight) (n=5)		
	Mean (CV)†		
	Chromium	Cadmium	Lead
SDT 204D1	117 (37 %)	40 (17 %)	776 (14 %)
SDT 204D2	81 (1.2 %)	18 (0.9 %)	755 (6.9 %)
SDT 205D1	106 (15%)	19 (12 %)	913 (17 %)

*As determined by procedure explained in Section 3.10.2

This result may have been due to the relatively low total cadmium content of the mixes.

These mass balance differences were due either to experimental error or to the extremely variable contamination levels found in the media and the S/S products. For such large losses of lead to have occurred during the seawater sequential extraction procedure, as much as 20 g out of the initial 50 g sample would have had to have been lost. A loss of such a large fraction of the total solids undergoing extraction would have been easily noticed, and no such loss was observed. Most likely, the mass balance differences were due to the highly variable contamination levels of the concrete. As can be seen in Table 5.9, the variation in total lead content for the three mixes ranged from 21% to 60%. Large variations in the total metal contents of the sequential extraction residue also can be seen in Table

5.10. This alone could account for much of the mass balance differences encountered.

The effect of pH on leachate concentrations was less pronounced during the seawater sequential extractions than for the acidic sequential extractions. Leachate cadmium and lead concentrations were very low for all of the seawater sequential extractions, regardless of pH. The chromium leaching behavior during the seawater extractions was similar to that for the acidic extractions. Leachate chromium concentrations and pH values were high for the first few extractions. Both decreased over subsequent extractions. The total chromium content of the sequential extraction residue seemed to indicate that chromium leaching was not limited by the total amount of chromium present.

The concept of a sorption edge below which metals desorb was not supported by the results of the seawater sequential extractions. As in the acidic extractions, chromium concentrations were highest at the high pH values. Leachate cadmium concentrations did not increase as the pH fell below the cadmium sorption edge pH value of 8.5. The pH for desorption of lead is approximately 6.5. Leachate lead concentrations did not increase for the few extractions in which the pH fell below this level.

In summary, for the acidic sequential extractions, chromium and cadmium leaching were limited by the amount of chromium and cadmium present in the concrete. Leachate chromium and cadmium concentrations were very low for the latter extractions even when the pH was decreased to low levels. Much of the chromium present leached during the seven acidic sequential extractions, but appreciable amounts of chromium remained in the residue from the seawater sequential extractions. Leachate cadmium concentrations were much lower for the seawater sequential extractions than for the acidic sequential extractions.

The lead leaching behavior was significantly different for the acidic and seawater sequential extractions. For the acidic sequential extractions, the leachate lead concentration began below the detection limit of 0.07 mg/L at high pH levels, and rose dramatically as the pH fell below 8.

Leachate lead concentrations as high as 11.2 mg/L were observed as the pH fell below 4. For the seawater sequential extractions, leachate lead concentrations were extremely low. The maximum leachate lead concentration observed was 0.12 mg/L. Leachate pH values rarely fell much below 7 for the seawater sequential extractions.

Leachate pH values alone could not fully explain the leaching behavior of the metals present, especially for the seawater sequential extractions. Leaching concentrations did not follow theoretical hydroxide solubilities. Some evidence was found to support the claim that cadmium and lead leaching may be controlled by adsorption/desorption processes. This was true only for the acidic sequential extractions, however. The effect of the type of leachant on the leaching behavior of chromium, cadmium and lead during the sequential extractions is discussed in the following section.

5.4.3 Effect of Leachant Type on Leaching Behavior

The cumulative chromium and cadmium leached during the acidic and sequential extractions for SDT 204D1 is compared in Figures 5.22 and 5.23. The noted leaching behavior was essentially the same for each mix tested during the sequential extractions . The following analysis is limited to SDT 204D1, as the results were similar for each of the three mixes.

The effect of the type of leachant on the amount of chromium leached and on the amount of cadmium leached during the sequential extractions was similar. The total amount of chromium leached for the acidic sequential extractions was approximately twice that for the seawater sequential extractions. The total amount of cadmium leached during the acidic sequential extractions was more than six times greater than the total amount of cadmium leached during the seawater sequential extractions. As can be seen by the slopes of the curves, the amount of chromium and cadmium leached was not substantially different for the first extraction or two or for the last few extractions. A large difference in the amount of chromium and cadmium leached was observed for the middle extractions.

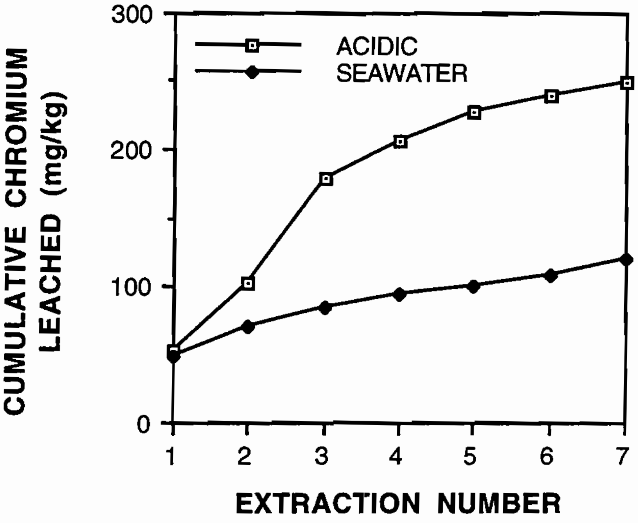


Figure 5.22: Cumulative Chromium Leached During Acidic and Seawater Sequential Extractions for SDT 204D1

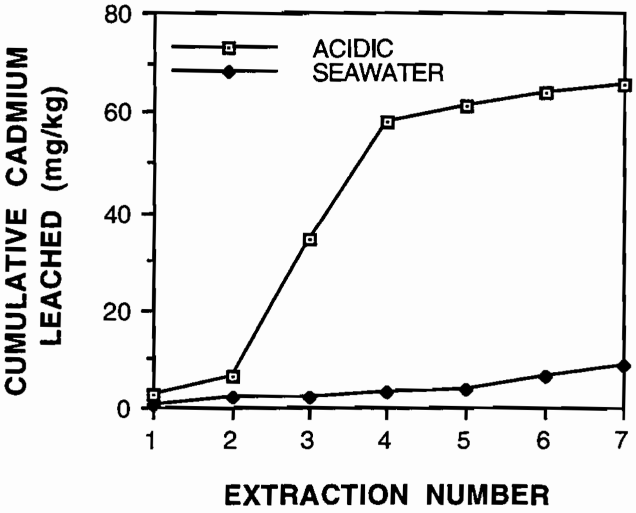


Figure 5.23: Cumulative Cadmium Leached During Acidic and Seawater Sequential Extractions for SDT 204D1

The cumulative lead leached during the acidic sequential extractions was plotted in Figure 5.24. As the leachate lead concentrations were below the detection limit of 0.07 mg/L for many of the seawater sequential extractions, and the highest leachate lead concentration measured was 0.12 mg/L, it was assumed that the cumulative lead leached in the seawater sequential extractions was essentially zero. The cumulative lead leached through seven acidic extractions was greater than 300 mg/kg. The majority of the lead leaching occurred in the later extractions.

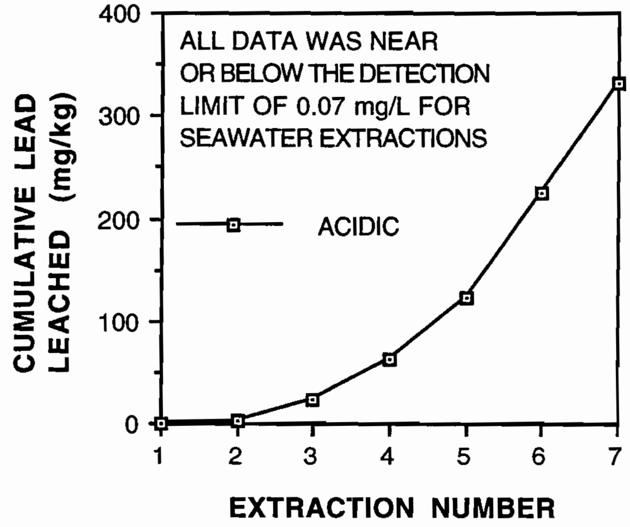


Figure 5.24: Cumulative Lead Leached During Acidic Sequential Extractions for SDT 204D1

To understand the leaching behavior of chromium, cadmium and lead under the influence of the two different leachants, it is necessary to consider the physical leaching mechanisms that may control this behavior. As the concrete samples were subjected to acidic attack, the alkalinity present in the concrete was leached out. The cumulative alkalinity leached for the acidic and seawater sequential extractions is shown in Figure 5.25.

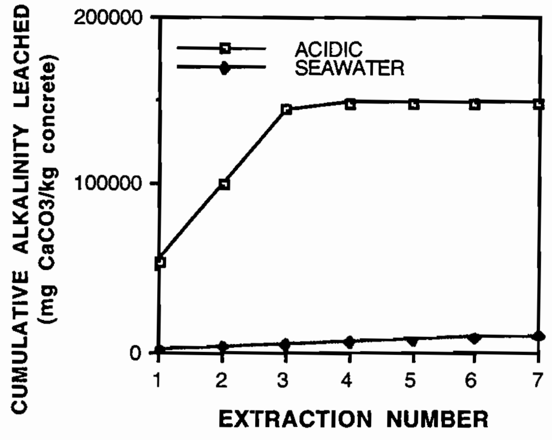


Figure 5.25: Cumulative Alkalinity Leached During Acidic and Seawater Sequential Extractions for SDT 204D1

All of the alkalinity present in the concrete was leached in the first four extractions when the acidic leachant was used. For the seawater sequential extractions, only small amounts of alkalinity were leached for each extraction; the total amount of alkalinity leached was ten times less than the total for the acidic sequential extractions. This was expected, as the pH of the acidic extraction fluid was 2.88 and the pH of the seawater was 8.3. The severe conditions of the acidic extractions led to the depletion of the alkalinity present in the concrete, followed by decreasing pH values. Leachate pH values fell below 4 for the acidic sequential extractions but were rarely less than 7 for the seawater sequential extractions.

The leaching behavior of alkalinity may help explain the leaching trends of chromium, cadmium and lead described above. As the leaching of the metals was relatively independent of leachant type for the first extraction or two, the presence of alkalinity in the cement may have been the controlling factor in leaching. However, as discussed above, leachate pH alone cannot explain the metals' leaching behavior. The leaching of large amounts of alkalinity indicates that the calcium-silicate-hydrate gel of the concrete matrix was being disintegrated as calcium was leached away in the form of alkalinity. Therefore, the effect of acidic attack on the leaching of the metals from the concrete matrix may have been as much a physical effect, due to the disintegration of the gel, as a chemical effect.

As the amount of cadmium and lead leached during the sequential extractions was dependent on the type of leachant used, it is possible that the stabilization of these metals involves the incorporation of the metals and metal complexes into the calcium structure of the concrete matrix. Leaching of these metals would then be dependent on the physical integrity of the concrete matrix.

As chromium leaching was relatively independent of leachant type, chromium may be more loosely bound in the concrete, perhaps being trapped in the pore spaces. Chromium leaching would then be limited only by mass transport through the solid which is controlled by diffusion (Cheng 1991), as discussed in Section 2.5. Mass transport limitations may be enough to account for the observed deviations from the theoretical solubility.

5.4.4 Summary of Investigation of Leaching Mechanisms

While pH control has widely been recognized as an integral part of the stabilization of metals in concrete systems, the results of this study indicate that pH control alone does not adequately explain the leaching behavior of chromium, cadmium and lead from S/S products subjected to sequential extractions. The leaching behavior of chromium, cadmium and lead did not follow the theoretical hydroxide solubilities of these metals. The leaching of cadmium and lead during the acidic sequential extractions seemed to support the claim that the metals were adsorbed onto the silica structure, as discussed in Section 2.5 (Cheng and Bishop 1992). This was not the case for the seawater sequential extractions, however.

The severe environment created during the acidic sequential extractions resulted in the leaching away of all of the alkalinity present in the mix. This phenomenon may have had both physical and chemical effects on the leaching of chromium, cadmium and lead. Leachate pH levels dropped below 4, where metals are highly soluble and not readily adsorbed. Also, the leaching of all the alkalinity from the concrete implies the disintegration of the calcium structure within the concrete matrix. This would reduce any physical barriers to leaching, such as those that may limit mass transport of leachant and metals through the bulk solid.

Sequential extractions using seawater as the leachant did not result in large quantities of cadmium and lead being leached from the concrete. Chromium leached in amounts that were just slightly lower than for the acidic sequential extractions. While higher leachate pH values occurred during the seawater sequential extractions, this does not appear to be able to account for the differences in leaching behavior compared to those observed during the acidic extractions. As only one-tenth of the alkalinity that leached in the acidic extractions (which was all of the alkalinity present for those samples) leached in the seawater extractions, the integrity of the calcium matrix within the concrete seemed to play a role in the successful stabilization of cadmium and lead.

Regardless of the exact mechanisms involved, leachate cadmium and lead concentrations were substantially less for the seawater extractions than for the acidic extractions. Leachate chromium concentrations were slightly lower for the seawater extractions. The most dramatic difference occurred for lead. Almost no lead leached during the seawater sequential extractions while leachate lead concentrations as high as 11.2 mg/L were encountered for the acidic extractions.

Therefore, acidic leaching tests provide for much more severe conditions than S/S products are likely to encounter in real-world applications. It should also be noted that the leaching tests were conducted on ground-up samples of the S/S products. This increases the surface area subject to leachant attack. In real-world applications where the S/S products may be used in monolithic form, the physical barriers to leaching will be increased greatly compared to those for the ground form used in these studies.

The laboratory acidic sequential extraction leaching tests performed in this study provided an extremely severe leaching environment. The use of an acidic leachant, coupled with the grinding up of the concrete sample prior to extraction, resulted in a worst-case scenario for the leaching of metals from concrete. Comparisons of metals leaching under the influence of acidic and seawater leachants have shown (Figures 5.22 through 5.24) that concrete in contact with seawater will leach substantially smaller amounts of metals than concrete in contact with acidic leachants. The seawater sequential extractions were also conducted on ground-up samples. Therefore, the leaching of metals from monolithic concrete in the presence of leachants that may be encountered in the environment (such as seawater), should be expected to be substantially less than was observed in the laboratory seawater sequential extractions conducted in this study.

5.5 Approach to Management of Spent Abrasive Media

The issue of spent abrasive media management was discussed in Section 2.4. The current regulatory environment has inhibited the use of traditional forms of land disposal that were formerly used for spent abrasive media management if the media is identified as a hazardous waste. The potential for the material to be classified as a hazardous waste has made disposal by such methods extremely costly. This section outlines a decision-making approach to the management of spent abrasive media and provides a set of guidelines for the recycling of spent abrasive media using portland cement.

5.5.1 Decision-Making Process

Upon generation of spent abrasive media, a decision needs to be made as to whether the material will be recycled or managed as a waste. Material that is recycled is not considered a waste and therefore is exempt from the status of a solid waste and the regulations that govern the handling of solid wastes, RCRA (Section 2.2). Therefore, recycling provides an attractive alternative to disposal of the material as a waste.

The decision-making logic is illustrated in Figure 5.26 and is discussed in the following paragraphs.

The results of this study have shown that spent abrasive media can be recycled as a component in portland cement based concrete. Mix proportions have been developed for many different media types and contamination levels. While not required, TCLP analyses can be conducted to verify that the material no longer poses a threat to human health and the environment and that the material containing the spent media is not a hazardous waste. Throughout this study, TCLP chromium, cadmium and lead concentrations of extracts of the concrete products made with spent abrasive media were below the EPA limits used to define a characteristic using the toxicity characteristic (TC) criteria. Recycling of the spent abrasive media in portland cement-based concrete provides for the beneficial reuse of a material that would otherwise be wasted.

If the material is to be handled as a solid waste, a determination needs to be made as to whether the material should be classified as a hazardous waste, as much different regulations apply to hazardous and non-hazardous waste. First, it must be determined if the spent abrasive media is a listed hazardous waste. As discussed in Section 2.2, spent abrasive media is not a listed waste. Then it is necessary to determine if the spent abrasive media is a characteristic waste. If the media is not reactive, ignitable or corrosive, the only other concern is whether the media is a toxicity characteristic (TC) waste. TCLP analysis is required to determine if the material is a TC hazardous waste. If the TCLP results so indicate, the material must be handled as a hazardous waste and the treatment, storage and disposal of the material is governed by RCRA Subtitle C. Treatment and disposal would most likely entail solidification/stabilization (S/S) followed by disposal in a hazardous waste landfill. The results of this study can also be applied to the treatment of spent abrasive media using S/S technologies.

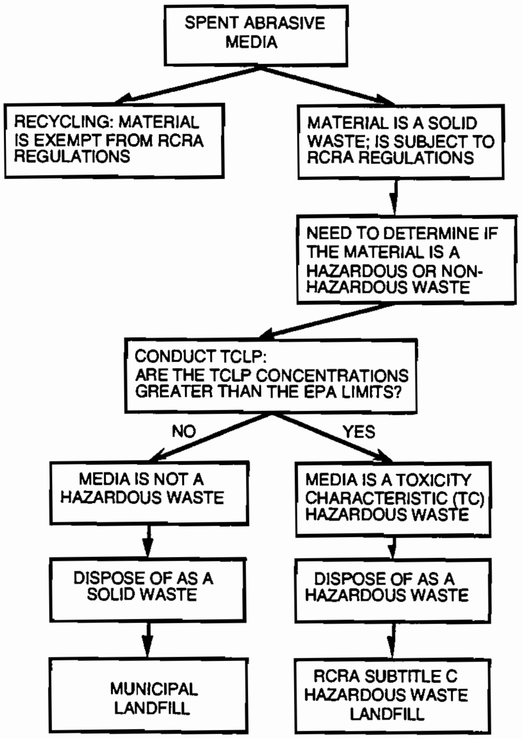


Figure 5.26: Decision-Making Process for Management of Spent Abrasive Media

If the spent abrasive media is not TC hazardous (the TCLP concentrations are below the EPA limits), the material can be handled as a non-hazardous solid waste and disposed of according to RCRA Subtitle D regulations. The material would most likely be disposed of in a municipal or an industrial landfill.

5.5.2 Recycling of Spent Abrasive Media Using Portland Cement

The results of this study have led to the development of guidelines for the recycling of spent abrasive media in portland cement-based concrete. A more detailed development of this approach appears in the M.S. thesis by Bryan Salt (Salt 1993). The following steps are recommended as an approach to the recycling of spent abrasive media:

1) Conduct the TCLP analyses on the spent abrasive media to determine whether it is a hazardous waste.

2) Conduct total metal analyses on the spent abrasive media to determine the extent of contained metals.

3) Conduct total metal analyses on the portland cement and mix additives to be used in the portland cement mortars. As some of these materials contain chromium, cadmium and lead, they could contribute to the TCLP leaching concentrations of the mixes.

4) Establish the desired physical properties for the concrete product such as compressive strength and set time.

5) Determine mix proportions through trial batches.
    a) Trial batch to reduce set times.
    b) Adjust mix proportions to achieve desired physical properties such as the set time and the compressive strength.
    c) Conduct the TCLP to determine the extent of metals leaching from the concrete product.
    d) If the TCLP analyses indicate that the concrete product is considered a hazardous waste, adjust the mix proportions (e.g., increase cement content, decrease spent abrasive media content) to avoid excessive metal leaching.

6) Define the field mix proportions to use the spent abrasive media as a component in the produced concrete.

7) Establish a field testing program to monitor the physical and chemical properties of the concrete produced during a project.

5.6 Engineering Significance

Bridge repainting operations result in spent abrasive media which contains metals such as chromium, cadmium and lead. The results of this study have shown that recycling this media in concrete provides an effective means of immobilizing the metals that exist in spent abrasive media with respect to the EPA limits for TCLP leaching. The resultant portland cement-based concrete products can provide sufficient compressive strengths for the material to be used for non-structural concrete purposes. The recycling of the potentially hazardous spent abrasive media also eliminates the need for costly land disposal while providing for the beneficial reuse of an otherwise wasted material.

The Texas Department of Transportation (TxDOT) has begun recycling spent abrasive media in portland cement-based concrete. This study involved the development of mixes for bridge repainting projects throughout Texas. Projects included the Rainbow Bridge in Port Arthur, Texas; the Montopolis Bridge in Austin, Texas; and a four-bridge project along IH 35 in Austin. The recycling of spent abrasive media has resulted in substantial cost savings for TxDOT. This study has resulted in the development of a set of guidelines for recycling of spent abrasive media in portland cement-based concrete that can be used wherever such use in considered.

Sequential extraction leaching tests, using both an acidic and a seawater leachant, provided insight into the potential mechanisms that control the leaching of metals from the concrete products. The results of these tests indicated that acidic leaching tests provide a much more severe environment than concrete products are likely to encounter in real world applications, such as use in the coastal areas of Texas. The results also indicated that the extent of metals leaching from concrete in contact with seawater could be expected to be less than that observed in these laboratory seawater sequential extractions, as these tests were performed on ground-up samples.

5.7 Future Research

The following topics are recommended for future research:

1) Conduct sequential extraction leaching tests using a freshwater leachant and compare the leaching behavior to that observed in the acidic and seawater sequential extractions.

2) Conduct leaching tests on monolithic concrete S/S products and compare the leaching behavior to that of crushed samples.

CHAPTER 6: CONCLUSIONS

The objectives of this research were to:

1) Immobilize the metals in spent abrasive media using portland cement.
2) Provide sufficient compressive strength for these concrete products to be used as non-structural concrete.
3) Examine the leaching behavior of metals in these concrete products and elucidate the possible mechanisms that control the leaching of the metals.
4) Compare the leaching behavior of concrete products made from spent abrasive media with varying characteristics.
5) Compare the leaching behavior of these concrete products under the influence of different leachants.

The conclusions of this research were:

1) Portland cement can successfully immobilize the metals present in spent abrasive media with respect to the EPA limits for TCLP leaching.

2) Concrete products made of spent abrasive media and portland cement provided sufficient compressive strength to be used for non-structural concrete purposes. With one exception, all of the mixes tested provided compressive strengths of 1,000 psi (6.9 MPa) as required by the Texas Department of Transportation.

3) TCLP cadmium and lead concentrations were very low for all concrete mixes tested. The TCLP chromium concentration increased as the total chromium content of the media increased.

4) Higher cement contents yielded higher compressive strengths. Increasing the separated spent abrasive dust content resulted in decreased compressive strengths and inhibited mix setting. DCI, a calcium nitrite-based corrosion inhibitor, was more effective than silica fume at increasing mix compressive strengths. Sodium silicate was effective at increasing the compressive strengths of mixes containing separated spent abrasive dust.

5) The conclusions of the sequential extraction leaching tests using acidic and seawater leachants were:

a) Leachate pH alone could not fully explain the leaching behavior of the metals present in the concrete products subjected to sequential extraction leaching tests. Leachate concentrations did not follow the theoretical hydroxide solubilities. Some evidence was found to support the claim that cadmium and lead leaching may be controlled by adsorption/desorption processes.

b) The severe environment created during the acidic sequential extractions resulted in the leaching away of all of the alkalinity present in the mix. Leachate pH levels dropped below 4, where metals are highly soluble and not readily adsorbed. Also, the disintegration of the calcium structure within the concrete matrix would reduce any physical barriers to leaching.

c) Leachate cadmium and lead concentrations were substantially less for the seawater extractions than for the acidic extractions. Leachate chromium concentrations were slightly lower for the seawater extractions. As only one-tenth of the alkalinity that leached in the acidic extractions leached in the seawater extractions, the integrity of the calcium matrix within the concrete seemed to play a role in the successful stabilization of cadmium and lead.

d) Acidic leaching tests provide for much more severe conditions than the concrete products are likely to encounter in real-world applications. Also, the extent of metals leaching from concrete in contact with seawater could be expected to be less than that observed in the seawater sequential extractions, as these tests were performed on ground-up samples.

6) This study has shown that spent abrasive media can be successfully recycled as a component in portland cement-based concrete. Recycling provides an attractive alternative to disposal of a material that is potentially a hazardous waste. The Texas Department of Transportation has begun to recycle spent abrasive media using portland cement, resulting in substantial cost savings.

7) A set of guidelines for the recycling of spent abrasive media in portland cement-based concrete has been developed.

APPENDIX A

SUMMARY OF S/S MIXES: DESIGNATIONS AND PROPORTIONS

APPENDIX A: SUMMARY OF S/S MIXES

MIX	CEMENT (lb)	SPENT SAND (lb)	SPENT DUST (lb)	SPENT SAND & DUST (lb)	SUPER PLASTIC. (oz/cwt)*	FLY ASH (lb)	SILICA FUME (lb)	DCI (gal/yd)	w/c# RATIO
ODESSA MIXES:									
MADE WITH BARREL #1									
SDT 300	470	0	0	1100	12.0	0	0	0	0.35
SDT 301	470	0	0	1100	16.2	0	56.4	0	0.35
SDT 302	329	0	0	1100	12.0	141	0	0	0.35
SDT 303	329	0	0	1100	17.7	141	56.4	0	0.35
SDT 304	470	0	0	1100	23.2	0	0	2	0.35
SDT 305	470	0	0	1100	28.9	0	0	4	0.35
SDT 400	705	0	0	1100	12.0	0	0	0	0.35
SDT 401	705	0	0	1100	16.5	0	84.6	0	0.35
SDT 402	493.5	0	0	1100	5.2	211.5	0	0	0.35
SDT 403	493.5	0	0	1100	12.0	211.5	84.6	0	0.35
SDT 404	705	0	0	1100	12.0	0	0	2	0.35
SDT 405	705	0	0	1100	12.0	0	0	4	0.35
MADE WITH BARREL #2									
SDT 500	470	0	0	1100	17.7	0	0	0	0.35
SDT 501	470	0	0	1100	23.4	0	56.4	0	0.35
SDT 502	329	0	0	1100	6.9	141	0	0	0.35
SDT 503	329	0	0	1100	17.7	141	56.4	0	0.35
SDT 600	705	0	0	1100	5.2	0	0	0	0.35
SDT 601	705	0	0	1100	14.2	0	84.6	0	0.35
SDT 602	493.5	0	0	1100	5.1	211.5	0	0	0.35
SDT 603	493.5	0	0	1100	8.6	211.5	84.6	0	0.35

*Ounces of superplasticizer per hundred pounds of cement
#Water to cement ratio
1 lb = 0.45 kg
1 oz/cwt = 0.65 mL/kg of cement
1 gal = 3.785 L; 1 cu.yd. = 0.77 m3

MIX	CEMENT (lb)	SPENT SAND (lb)	SPENT DUST (lb)	SPENT SAND & DUST (lb)	SUPER PLASTIC. (oz/cwt)*	FLY ASH (lb)	SILICA FUME (lb)	DCI (gal/yd)	w/c RATIO
PORT ARTHUR MIXES:									
SDT 204 D1	705	1100	0 (0%)	0	0	0	0	0	0.35
SDT 204 D2	705	1100	55 (5%)	0	18.9	0	0	0	0.35
SDT 204 D3	705	1100	165 (15%)	0	39.1	0	0	0	0.35
SDT 204 D4	705	1100	275 (25%)	0	60.7	0	0	0	0.35
SDT 205 D1	470	1100	0 (0%)	0	12	0	0	0	0.35
SDT 205 D2	470	1100	55 (5%)	0	12	0	0	0	0.35
SDT 205 D3	470	1100	165 (15%)	0	21.3	0	0	0	0.35
SDT 205 D4	470	1100	275 (25%)	0	41	0	0	0	0.35
SDT 206 D1	470	1100	0 (0%)	0	3.2	0	0	2	0.35
SDT 206 D2	470	1100	55 (5%)	0	12	0	0	2	0.35
SDT 206 D3	470	1100	165 (15%)	0	30.6	0	0	2	0.35
SDT 206 D4	470	1100	275 (25%)	0	44.3	0	0	2	0.35
SDT 207 D1	705	1100	0 (0%)	0	0	0	0	2	0.35
SDT 207 D2	705	1100	55 (5%)	0	2.6	0	0	2	0.35
SDT 207 D3	705	1100	165 (15%)	0	8.4	0	0	2	0.35
SDT 207 D4	705	1100	275 (25%)	0	17.1	0	0	2	0.35
SDT 208 D1	470	1100	0 (0%)	0	20.7	0	56.4	0	0.35
SDT 208 D2	470	1100	55 (5%)	0	23.9	0	56.4	0	0.35
SDT 208 D3	470	1100	165 (15%)	0	30.6	0	56.4	0	0.51
SDT 208 D4	470	1100	275 (25%)	0	34.6	0	56.4	0	0.48
SDT 209 D1	705	1100	0 (0%)	0	3.7	0	84.6	0	0.35
SDT 209 D2	705	1100	55 (5%)	0	9.7	0	84.6	0	0.35
SDT 209 D3	705	1100	165 (15%)	0	26.7	0	84.6	0	0.35
SDT 209 D4	705	1100	275 (25%)	0	32.3	0	84.6	0	0.35

*Ounces of superplasticizer per hundred pounds of cement

1 lb = 0.45 kg

1 oz/cwt = 0.65 mL/kg of cement

1 gal = 3.785 L; 1 cu.yd. = 0.77 m3

MIX	CEMENT (lb)	CLEAN SAND (lb)	SPENT DUST (lb)	SUPER PLASTIC. (oz/cwt)*	SODIUM SILICATE (lb)	w/c# RATIO
SODIUM SILICATE MIXES:						
CM #1	705	1100	0	3.2	0.0	0.35
CM #2	705	1100	55	5.4	0.0	0.35
CM #3	705	1100	165	7.5	0.0	0.35
CM #4	705	1100	275	17.0	0.0	0.35
SILI 1A	705	1100	0	1.8	14.1	0.35
SILI 1B	705	1100	55	3.0	14.1	0.35
SILI 1C	705	1100	165	12.0	14.1	0.35
SILI 1D	705	1100	275	26.7	14.1	0.35
SILI 2A	705	1100	0	2.2	28.2	0.35
SILI 2B	705	1100	55	5.5	28.2	0.35
SILI 2C	705	1100	165	14.3	28.2	0.35
SILI 2D	705	1100	275	35.7	28.2	0.35

*Ounces of superplasticizer per hundred pounds of cement
#Water to cement ratio
1 lb = 0.45 kg
1 oz/cwt = 0.65 mL/kg of cement

MIX	CEMENT (lb)	SPENT SLAG (lb)	SUPER PLASTIC. (oz/cwt)*	SILICA FUME (lb)	SODIUM SILICATE (lb)	w/c# RATIO
MONTOPOLIS BRIDGE MIXES:						
MONT 3	705	1100	12.0	84.6	0.0	0.35
MONT 4	705	1100	5.3	0.0	14.1	0.35
MONT 11	705	1100	16.6	84.6	28.2	0.35

*Ounces of superplasticizer per hundred pounds of cement
#Water to cement ratio
1 lb = 0.45 kg
1 oz/cwt = 0.65 mL/kg of cement

APPENDIX B

PROCEDURE FOR DETERMINING TCLP EXTRACTION FLUID

APPENDIX B: Procedure for Determining TCLP Extraction Fluid (Taken from 40 CFR Part 261 App.II)

1. Weigh out a small sub-sample of the waste, reduce if necessary to a particle size approximately 1mm in diameter or less, and transfer a 5.0 gram portion to a 500 ml beaker.

2. Add 96.5 ml of distilled deionized water (ASTM Type 2), cover with watchglass, and stir vigorously for 5 minutes. Measure and record the pH. If the pH ≤ 5.0, <u>EXTRACTION FLUID #1</u> is used.

3. If the pH > 5.0, add 3.5 ml of 1.0 N HCl, slurry for 30 seconds, cover with a watchglass, and heat to 50° C and hold for 15 minutes.

4. Let the solution cool to room temperature and then measure and record pH. If the pH is ≤ 5.0, use <u>EXTRACTION FLUID #1</u>. If the pH is > 5.0, <u>EXTRACTION FLUID #2</u> is used.

APPENDIX C

ACID DIGESTION PROCEDURE FOR SEDIMENTS, SLUDGES, AND SOILS

(Taken from SW-846 Methods 3050 and 3051)

METHOD 3050

ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare sediments, sludges and soil samples for anlysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also Step 2.1):

FLAA		GFAA
Aluminum	Magnesium	Arsenic
Barium	Manganese	Beryllium
Beryllium	Molybdenum	Cadmium
Cadmium	Nickel	Chromium
Calcium	Osmium	Cobalt
Chromium	Potassium	Iron
Cobalt	Silver	Lead
Copper	Sodium	Molybdenum
Iron	Thallium	Selenium
Lead	Vanadium	Thallium
	Zinc	Vanadium

Note: See Method 7760 for FLAA preparation for Silver

2.0 SUMMARY OF METHOD

2.1 A representative 1- to 2- g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se, and (2) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Os, Pb, Tl, V, and Zn. Dilute nitric acid is employed as the final dilution acid for the furnace AA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, Tl, and V. The diluted samples have an approximate acid concentration of 5.0% (v/v). A separate sample shall be dried for a total solids determination.

3.0 INTERFERENCES

3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether Method 3050 is applicable to a given waste.

4.0 APPARATUS AND MATERIALS

4.1 Conical Phillips beakers - 250 mL.

4.2 Watch glasses.

4.3 Drying Ovens - That can be maintained at 30°C.

4.4 Thermometer - That covers range of 0-200°C.

4.5 Filter Paper - Whatman No. 41 or equivalent.

4.6 Centrifuge and centrifuge tubes.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.3 Nitric acid (concentrated), HNO_3. Acid should be analyzed to determine level of impurities. If method blank is < MDL, the acid can be used.

5.4 Hydrochloric acid (concentrated), HCl. Acid should be analyzed to determine level of impurities. If method blank is < MDL, the acid can be used.

5.5 Hydrogen peroxide (30%), H_2O_2. Oxidant should be analyzed to determine level of impurities.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and water. Plastic and glass containers are both suitable. See Chapter Three, Step 3.1.3, for further information.

6.3 Nonaqueous samples shall be refrigerated upon receipt and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Mix the sample thoroughly to achieve homogeneity. for each digestion procedure, weigh to the nearest 0.01g and transfer to a conical beaker 1.00 - 2.00 g of sample.

7.2 Add 10 mL of 1:1 HNO_3, mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 to 15 minutes without boiling. allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Repeat this last step to ensure complete oxidation. Using a ribbed watch glass, allow the solution to evaporate to 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

7.3 After Step 7.2 has been completed and the sample has cooled, add 2 mL of water and 3 mL of 30% H_2O_2. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

7.4 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

NOTE: Do not add more than a total of 10 ML 30% H2O2.

7.5 If the sample is being prepared for (a) the ICP analysis of JAs, and Se, or (b) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Os, Pb, Tl, V, and Zn, then add 5 mL of concentrated HCl aand 10 mL of water, return the covered beaker to the hot plate, and reflux for an additional 15 minutes without boiling. After cooling, dilute to 100 mL with water. Particulates in the digestate that may clog the nebulizer should be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.5.1 Filtration - Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 mL with water.

7.5.2 Centrifugation - Centrifugation at 2,000-3,000 rpm for 10 minutes is usually sufficient to clear the supernatant.

7.5.3 The diluted sample has an approximate acid concentration of 5.0% (v/v) HCl and 5.0% (v/v) HNO3. The sample is now ready for analysis.

7.6 If the sample is being prepared for the furnace analysis of As, Be, Cd, Co, Cr, Fe, Mo,Pb, Se, Tl, and V, cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL. After cooling, dilute to 100 mL with water. Prticulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.6.1 Filtration - Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 mL with water.

7.6.2 Centrifugation - Centrifugation at 2,000-3,000 rpm for 10 minutes is usually sufficient to clear the supernatant.

7.6.3 The diluted digestate solution contains approximately 5% (v/v) HNO3. For analysis, withdraw aliquots of appropriate volume and add any required reagent or matrix modifier. The sample is now ready for analysis.

7.7 Calculations

7.7.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

7.7.2 If percent solids is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample.

8.0 QUALITY CONTROL

8.1 All quality control measures described in Chapter One should be followed.

8.2 For each group of samples processed, preparation blanks (water and reagent) should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

8.3 Replicate samples should be processed on a routine basis. Replicate samples will be used to determine precision. The sample load will dictate the frequency, but 20% is recommended.

8.4 Spiked samples or standard reference materials must be employed to determine accuracy. A spiked sample should be included with each group of samples processed and whenever a new sample matrix is being analyzed.

8.5 The concentration of all calibration standards should be verified against a quality control chech sample obtained from an outside source.

9.0 METHOD PERFORMANCE

9.1 No data provided.

METHOD 3051

MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS AND OILS

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the microwave assisted acid digestion of sludges, sediments, soils and oils for the following elements:

Aluminum
Antimony
Arsenic
Boron
Barium
Beryllium
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganes
Mercury
Molybdenum
Nickel
Potassium
Selenium
Silver
Sodium
Strontium
Thaliium
Vanadium
Zinc

1.2 This method is provided as an alternative to Method 3050A. It is intended to provide a rapid multielement acid leach digestion prior to analysis so that decisions can be made about site cleanup levels, the need for TCLP testing of a waste and whether a BDAT process is providing acceptable performance. If a decomposition including hydrochloric acid is required for certain elements, it is recommended that Mehod 3050A be used. Digests produced by the method are suitable for analysis by flame atomic absorption (FLAA), graphite furnace atomic absorption (GFAA), inductively coupled plasma emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS).

2.0 SUMMARY OF METHOD

2.1 A representative sample of up to 0.5 g is digested in 10 mL of concentrated nitric acid for 10 min using microwave heating with a suitable laboratory microwave unit. The sample is placed in a Teflon PFA vessel with 10 mL of concentrated nitric acid. The vessel is capped and heated in the microwave unit. After cooling, the vessel contents are diluted to volume and analyzed by the appropriate SW-846 method (Ref. 1).

3.0 INTERFERENCES

3.1 Very reactive or volatile materials that may create high pressures when heated may cause venting of the vessels with potential loss of sample and analytes. The complete decomposition of either carbonates, or carbon based samples, may cause enough pressure to vent the vessel if the sample size is greater than 0.25 g when used in the 120 mL vessels with a pressure relief device that has an upper limit of 7.5 ± 0.7 atm (110 ± 10 psi).

4.0 APPARATUS AND MATERIALS

4.1 Microwave apparatus requirements

4.1.1 The microwave unit provides programmable power with a minimum of 574 W and can be programmed to within ± 10 W of the required power.

4.1.2 The microwave unit cavity is corrosion resistant as well as ventilated.

4.1.3 All electronics are protected against corrosion for safe operation.

4.1.4 The system requires Teflon PFA digestion vessels (120 mL capacity) capable of withstanding pressures of up to 7.5 ± 0.7 atm (110 ± 10 psi) and capable of controlled pressure relief at pressures exceeding 7.5 ± 0.7 atm (110 ± 10 psi).

4.1.5 A rotating turntable is employed to insure homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 3 rpm.

4.1.6 Those laboratories now using or contemplating the use of kitchen type microwave ovens for this method should be aware of several significant safety issues. First, when an acid such as nitric is used to assist sample digestion in microwave units in open vessels, or sealed vessels equipped with venting features, there is the potential for the acid gases released to corrode the safety devices that prevent the microwave magnetron from shutting off when the door is opened. This can result in operator exposure to microwave energy. Use of a unit with corrosion resistant safety devices prevents this from occurring.

The second safety concern relates to the use of sealed containers without pressure relief valves in the unit. Temperature is the important variable controlling the reaction. Pressure is needed to attain elevated temperatures but must be safely contained. However, many digestion vessels constructed from certain Teflons may crack, burst, or explode in the unit under certain pressures. Only unlined PFA Teflon containers with pressure relief mechanisms are considered acceptable at present.

Users are therefore advised not to use kitchen type microwave ovens or to use sealed containers without pressure relief valves for microwave acid digestions by this method. Use of laboratory-grade microwave equipment is required to prevent safety hazards. For further details consult reference 2.

4.2 Polymeric volumetric ware in plastic (Teflon or polyethylene) 50 or 100 mL capacity.

4.3 Whatman No. 41 filter paper (or equivalent).

4.4 Disposable polypropylene filter funnel.

4.5 Analytical balance, 300 g capacity, and minimum ± 0.001 g.

5.0 REAGENTS

5.1 All acids should be sub-boiling distilled where possible to minimize the blank levels due to metallic contamination. Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

5.1.1 Concentrated nitric acid, HNO_3. Acid should be analyzed to determine levels of impurity.

5.2 Reagent Water. Reagent water shall be interference free. All references to water in the method refer to reagent water unless otherwise specified (Ref. 3).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids and water. Plastic and glass containers are both suitable. See Chapter Three, Step 3.1.3 of this manual, for further information.

6.3 Samples must be refrigerated upon receipt and anlayzed as soon as possible.

7.0 PROCEURE

7.1 Calibration of Microwave Equipment

7.1.1 Measurement of the available power for heating is evaluated so that absolute power in watts may be transferred from one microwave unit to another. for cavity type microwave equipment, this is accomplished by measuring the temperature rise in 1 kg of water exposed to microwave radiation for a fixed period of time. The analyst can relate power in watts to the partial power setting of the unit. The calibration format required for laboratory microwave uits depends on the type of electronic system used by the manufacturer to provide partial microwave power. Few units have an accurate and precise linear relationship between percent power settings and absorbed power. Where linear circuits have been utilized, the calibration curve can be determined by a three-point calibration method (7.1.3), otherwise, the analyst must use the multiple point calibration method (7.1.2).

7.1.2 The multiple point calibration involves the measurement of absorbed power over a large range of power settings. Typically, for a 600 W unit, the following power settings are measured; 100,99,98,97,95,90,80,70,60,50, and 40% using the procedure described in section 7.1.4. This data is clustered about the customary working power ranges. Nonlinearity has been commonly encountered at the upper end of the calibration. If the unit's electronics are known to have nonlinear deviations in any region of proportional power control, it will be necessary to make a set of measurements that bracket the power to be used. The final calibration point should be at the partial power setting that will be used in the test. This setting should be checked periodically to evaluate the integrity of the calibration. If a significant change is detected (± 10 W), then the entire calibration should be reevaluated.

7.1.3 The three-point calibration involves the measurement of absorbed power at three different power settings. Measure the power at 100% and 50% using the procedure described in section 7.1.4. From the 2-point line calculte the power setting corresponding to the required power in watts specified in the procedure. Measure the absorbed power at that partial power setting. If the measured absorbed power does not correspond to the specified power within +10 W, use the multiple point calibration in 7.1.2. This point should also be used to periodically verify the integrity of the calibration.

7.1.4 Equilibrate a large volume of water to room temperature (23 ± 2 °C). One kg of reagent water is weighed (1,000.0 g + 0.1g) into a Teflon beaker or a beaker made of some other material that does not significantly absorb microwave energy (glass absorbs microwave energy and is not recommended). The initial temperature of the water should be 23 ± 2 °C measured to ± 0.05 °C. The covered beaker is circulated continuously (in the normal sample path) through the microwave field for 2 minutes at the desired partial power setting with the unit's exhaust fan on maximum (as it will be during normal operation). The beaker is removed and the water vigorously stirred. Use a magnetic stirring bar inserted immediately after microwave irradiation and record the maximum temperature within the first 30 seconds to ± 0.05 °C. Use a new sample for each additional measurement. If the water is reused both the water and the beaker must have returned to 23 ± 2 °C. Three measurements at each power setting should be made.

The absorbed power is determined by the following relationship

$$P = \frac{(K)\,(Cp)\,(m)\,(\Delta T)}{t} \qquad \text{Eq.1}$$

P = the apparent power absorbed by the sample in watts (W). (W = joule sec^{-1})

K = the conversion factor for thermochemical calories sec^{-1} to watts (=4.184)

Cp = the heat capacity, thermal capacity, or specific heat (cal $g^{-1}{}^{\circ}C^{-1}$), of water. m = the mass of the water sample in grams.

ΔT = the final temperature minus the initial temperature (°C), and

t = the time in seconds (s).

Using the experimental conditions of 2 minutes and 1 kg of distilled water (heat capacity at 25 °C is 0.9997 cal $g^{-1}{}^{\circ}C^{-1}$) the calibration equation simplifies to :

Eq.2 $$P = (\Delta T)\ (34.85)$$

NOTE: Stable line voltage is necessary for accurate and reproducible calibration and operation. The line voltage should be within manufacturer's specification, and during measurement and operation should not vary by more than ± 2 V. A constant power supply may be necessary for microwave use if the source of the line voltage is unstable.

Electronic components in most microwave units are matched to the unit's function and output. When any part of the high voltage circuit, power source, or control components in the unit have been serviced or replaced, it will be necessary to recheck the unit's calibration. If the power output has changed significantly ($\pm$10W), then the entire calibration should be reevaluated.

7.2 All digestion vessels and volumetric ware must be carefully acid washed and rinsed with reagent water. When switching between high concentration samples and low concentration samples, all digestion vessels should be cleaned by leaching with hot (1:1) hydrochloric acid for a minimum of two hours followed with hot (1:1) nitric acid for a minimum of two hours and rinsed with reagent water and dried in a clean environment. This cleaning procedure should also be used whenever the prior use of the digestion vessels is unknown or cross contamination from vessels is suspected. Polymeric volumetric ware and storage containers should be cleaned by leaching with more dilute acids appropriate for the specific plastics used and then rinsed with reagent water and dried in a clean environment.

7.3 Sample Digestion

7.3.1 Weigh the Teflon PFA digestion vessel, valve and cap assembly to 0.001 g prior to use.

7.3.2 Weigh a well-mixed sample to the nearest 0.001 g into the Teflon PFA sample vessel equipped with a single-ported cap and a pressure relief valve. For soils, sediments, and sludges use no more than 0.500 g. For oils use no more than 0.250 g.

7.3.3 Add 10 ± 0.1 mL concentrated nitric acid in a fume hood. If a vigorous reaction occurs, allow the reaction to stop before capping the vessel. Cap the vessel and torque the cap to 12 ft-lb (16 N-m) according to the unit manufacturer's directions. The sample vessel may be connected to an overflow vessel using Teflon PFA connecting tubes. Weigh the vessels to the nearest 0.001 g. Place the vessels in the microwave carousel. Connect the overflow vessels to the center well of the unit.

CAUTION: When digesting samples containing volatile or easily oxidized organic compounds, initially weigh no more than 0.10 g and observe the reaction before capping the vessel. If a vigorous reaction occurs, allow the reaction to cease before capping the vessel. If no appreciable reaction occurs, a sample weight up to 0.25 g can be used.

7.3.4 Place the vessels evenly distributed in the turntable of the microwave unit using groups of 2 sample vessels or 6 samples vessels. Any vessels containing 10 mL of nitric acid for analytical blank purposes are counted as sample vessels. When fewer than the recommended number of samples are to be digested, i.e., 3 samples plus 1 blank, the remaining vessels should be filled with 10 mL of nitric acid to achieve the full complement of vessels. This provides an energy balance since the microwave power absorbed is proportional to the total mass in the cavity (Ref. 4). Irradiate each group of 2 sample vessels at 344W for 10 minutes and each group of 6 sample vessels at 574 W for 10 minutes. The temperature of each sample should rise to 175 °C in less than 5.5 minutes and remain between 170 - 180 °C for the balance of teh 10 minute irradiation period. The pressure should peak at less than 6 atm for most soil, sludge and sediment samples (Ref. 5). The pressure will exceed these limits in the case of high concentrations of carbonate of organic compounds. In these cases the pressure will be limited by the relief pressure of the vessel to 7.5 ± 0.7 atm (110 ± 10 psi).

7.3.4.1 Newer microwave units may be capable of higher power (W) that permits the digestion of a larger number of samples per batch. If the analyst wishes to digest other than two or six samples at a time, the analyst may use different values of power as long as they results in the same time and temperature conditions defined in 7.3.4. That is, any sequence of power that brings the samples to 175 °C in 5.5 minutes and permits a slow rise to 175 - 180 °C during the remaining 4.5 minutes (Ref. 5).

Issues of safety, structural integrity (both temperature and pressure limitations), heat loss, chemical compatibility, microwave absorption of vessel material, and energy transport will be considerations made in choosing alternative vessels. If all of the considerations are met and the appropriate power settings provided to reproduce the reaction conditions defined in 7.3.4, then these alternative vessels may be used (Ref 1,2).

7.3.5 At the end of the microwave program, allow the vessel to cool for a mimimum of 5 minutes before removing them from the microwave unit. When the vessels have cooled to room temperature, weigh and record the weight of each vessel assembly. If the weight of acid plus sample has decreased by more than 10 percent from the original weight, discard the sample. Determine the reason for the weight loss. These are typically attributed to loss of vessel seal integrity, use of a digestion time longer than 10 minutes too large a sample, or improper heating conditions. Once the source of the loss has been corrected, prepare a new sample or set of samples for digestion beginning at 7.3.1.

7.3.6 Complete the preparation of the sample by carefully uncapping and venting each vessel in a fume hood. Transfer the sample to an acid-cleaned polyethylene bottle. If the digested sample contains particulates which may clog nebulizers or interfere with injection of the sample into the instrument, the sample may be centrifuged, allowed to settle, or filtered.

7.3.6.1 Centrifugation: Centrifugation at 2,000-3,000 rpm for 10 minutes is usually sufficient to clear the supernatant.

7.3.6.2 Settling: Allow the sample to stand until the supernatant is clear. Allowing a sample to stand overnight will usually accompolish this. If it does not, centrifuge or filter the sample.

7.3.6.3 Filtering: The filtering apparatus must be thoroughly cleaned and prerinsed with dilute nitric acid. Filter the sample through quantitative filter paper into a second acid-cleaned container.

7.3.7 The diluted digest has an approximate acid concentrations of 20 percent (v/v) HNO_3. The digest is now ready for analysis for elements of interest using the appropriate SW-846 method.

7.4 Calculations: The concentrations determined are to be reported on the basis of the actual weight of the original sample.

8.0 QUALITY CONTROL

8.1 All quality control data must be maintained and available for reference or inspection for a period of three years. This method is restricted to use by, of under supervision of, experienced analysts. Refer to the appropriate section of Chapter One for additional quality control requirements.

8.2 Replicate samples should be processed on a routine basis. A replicate sample is a sample brought through the whole sample preparation and analytical process. A replicate sample should be processed with each analytical batch for every 20 samples, whichever is the greatest number. A replicate sample should be prepared for each matrix type (i.e., soil, sludge, etc.).

8.3 Spiked samples or standard reference materials should be included with each group of samples processed or every 20 samples, whichever is the greater number. A spiked sample should also be included whenever a new sample matrix is being analyzed.

9.0 METHOD PERFORMANCE

9.1 Precision: The precision of Method 3051, as determined by the statistical examination of interlaboratory test results is as follows:

9.2 Repeatability: If successive results are obtained by the same analyst with the same apparatus under constant operating conditions on identical test material, then the difference between these successive results will not, with 95% probability, exceed the repeatability value. For example, in the case of lead, an average of only 1 case in 20 would exceed

$$0.206\,x$$

in the long run, where x is one result in g/g (Ref. 6).

9.3 Reproducibility: If two successive measurements are made independently by each of two different analysts working in different laboratories on identical test material, then the difference between the average results for each analyst will not, with 95% probability, exceed the reproducibility value. For example, in the case of lead, an average of only 1 case in 20 would exceed

$$0.303\,x$$

in the long run, where x is the average of two successive measurements in g/g (Ref. 2).

As can be seen in Table 1, repeatability and reproducibility differ between elements, and usually depend on that element's concentration. Table 2 provides an example of how users of the method can determine expected values for repeatability and reproducibility; nominal values of lead have been used for this model (Ref.6).

9.4 Bias: In the case of SRM 1085 - Wear Metals in Oil, the basis of this test method is different for each element. An estimate of bias, as shown in Table 3, is:

Bias = Amount found - Amount expected.

However, the bias estimate inherits both the uncertainty in the measurements made using Method 3051 and the uncertainty on the certificate, so whether the bias is real or only due to measurement error must also be considered. The concentrations found for Al, Cr, and Cu using Method 3051 fall within their certified ranges on SRM 1085, and 95% confidence intervals for Fe and Ni overlap with their respective certified ranges: therefore, the observed biases for these elements are probably due to chance and should be considered insignificant. Biases should not be estimated at all for Ag and Pb because these elements were not certified. Therefore, the only two elements considered in this table for which the bias estimates are significant are Mg and Mo.

APPENDIX D

TCLP, COMPRESSIVE STRENGTH, AND pH RAW DATA FOR S/S MIXES

RAW DATA FOR ODESSA MIXES:
(Bolded underlined values are the averages of the replicate values)

	TCLP CONCENTRATIONS					
MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	Al (mg/L)	pH	Compressive Strength* (psi)
SDT 300	**0.60**	**0.13**	**<0.07**	**33.60**	**11.1**	**3540**
	0.58	0.15	<0.07	34.59	11.2	
	0.61	0.10	<0.07	32.60	10.9	
SDT 301	**0.73**	**0.12**	**<0.07**	**32.05**	**9.1**	**4420**
	0.72	0.12	<0.07	32.19	7.4	
	0.74	0.12	<0.07	31.91	10.7	
SDT 302	**0.98**	**0.12**	**<0.07**	**31.33**	**10.8**	**1100**
	0.96	0.12	<0.07	32.08	10.8	
	1.00	0.11	<0.07	30.58	10.7	
SDT 303	**1.01**	**0.14**	**<0.07**	**32.52**	**10.3**	**1500**
	1.07	0.13	<0.07	32.91	10.4	
	0.95	0.14	0.08	32.12	10.2	
SDT 304	**0.71**	**0.03**	**<0.07**	**31.21**	**11.1**	**6480**
	0.68	0.03	<0.07	31.56	11.1	
	0.74	0.03	<0.07	30.86	11.1	
SDT 305	**0.62**	**0.03**	**<0.07**	**30.30**	**11.1**	**8240**
	0.65	0.03	<0.07	30.47	11.1	
	0.58	0.02	<0.07	30.13	11.1	
SDT 400	**0.67**	**0.11**	**0.17**	**39.92**	**11.4**	**2370**
	0.65	0.09	0.18	37.02	11.3	
	0.68	0.13	0.16	42.82	11.5	
SDT 401	**0.79**	**0.03**	**<0.07**	**33.32**	**11.1**	**2330**
	0.85	0.03	<0.07	34.30	11.1	
	0.73	0.02	0.10	32.34	11.1	
SDT 402	**0.46**	**0.02**	**0.12**	**32.42**	**11.2**	**2150**
	0.45	0.02	0.13	32.61	11.2	
	0.46	0.02	0.10	32.22	11.2	
SDT 403	**0.62**	**<0.02**	**0.19**	**31.95**	**11.1**	**3820**
	0.65	<0.02	0.16	31.66	11.1	
	0.58	0.03	0.21	32.23	11.1	
SDT 404	**0.53**	**0.05**	**<0.07**	**30.83**	**11.3**	**7910**
	0.55	0.05	<0.07	31.22	11.3	
	0.50	0.05	<0.07	30.44	11.3	
SDT 405	**0.49**	**0.05**	**<0.07**	**30.78**	**11.3**	**8250**
	0.48	0.05	<0.07	31.24	11.3	
	0.50	0.04	<0.07	30.32	11.2	

	TCLP CONCENTRATIONS					
	Cr	Cd	Pb	Al	pH	Compressive Strength[*]
MIX	(mg/L)	(mg/L)	(mg/l)	(mg/L)		(psi)
SDT 500	0.81	0.15	0.07	36.39	11.4	4770
	0.95	0.11	0.07	40.00	11.2	
	0.46	0.21	<0.07	29.04	11.7	
	1.01	0.12	0.09	40.14	11.3	
SDT 501	1.10	0.15	0.11	42.58	9.9	5850
	1.12	0.15	0.10	42.99	9.6	
	1.10	0.15	0.08	42.12	10.3	
	1.08	0.16	0.14	42.63	9.9	
SDT 502	0.93	0.14	0.09	40.57	10.1	3920
	0.94	0.12	0.07	39.32	10.0	
	0.92	0.15	0.08	40.86	9.8	
	0.94	0.14	0.11	41.52	10.5	
SDT 503	0.77	0.15	0.08	34.73	9.7	5260
	0.91	0.19	0.07	45.69	7.5	
	0.33	0.09	<0.07	14.27	11.7	
	1.07	0.17	0.12	44.23	9.7	
SDT 600	0.67	0.15	≤0.07	24.83	11.3	5250
	0.67	0.14	<0.05	24.56	11.4	
	0.69	0.16	0.10	25.42	11.2	
	0.65	0.15	<0.04	24.52	11.3	
SDT 601	0.61	0.14	≤0.07	24.54	11.0	6520
	0.63	0.15	<0.07	24.79	10.9	
	0.56	0.15	<0.07	24.38	11.0	
	0.63	0.13	<0.07	24.46	11.1	
SDT 602	0.73	0.15	≤0.07	24.88	11.1	4690
	0.74	0.15	<0.07	24.83	11.1	
	0.72	0.16	<0.07	24.98	11.0	
	0.74	0.15	<0.07	24.82	11.2	
SDT 603	0.71	0.34	≤0.07	25.59	11.0	4750
	0.67	0.66	<0.07	25.72	11.0	
	0.77	0.22	0.07	28.84	10.8	
	0.68	0.15	<0.07	25.22	11.1	

[*] 28-day Compressive Strength; numbers are averages of two replicates (Salt 1993)
1000 psi = 6.895 MPa

RAW DATA FOR PORT ARTHUR MIXES:
(Bolded underlined values are the averages of the replicate values)

	TCLP CONCENTRATIONS					
MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	Al (mg/L)	pH	Compressive Strength[*] (psi)
SDT 204 D1	**2.17**	**0.04**	**0.09**	**37.76**	**11.6**	**3050**
	2.18	0.05	0.13	38.83	11.6	
	2.15	0.03	<0.07	37.19	11.6	
SDT 204 D2	**2.16**	**0.07**	**0.12**	**40.17**	**11.6**	**770**
	2.10	0.07	0.18	40.31	11.6	
	2.22	0.07	<0.05	40.03	11.6	
SDT 204 D3[†]	–	–	–	–	–	–
SDT 204 D4[†]	–	–	–	–	–	–
SDT 205 D1	**2.97**	**0.15**	**0.14**	**68.30**	**11.4**	**2120**
	3.65	0.14	0.16	67.67	11.3	
	2.29	0.15	0.11	68.94	11.5	
SDT 205 D2[†]	–	–	–	–	–	–
SDT 205 D3[†]	–	–	–	–	–	–
SDT 205 D4[†]	–	–	–	–	–	–
SDT 206 D1	**2.56**	**0.16**	**<0.07**	**70.60**	**11.7**	**4760**
	2.35	0.15	<0.07	68.48	11.7	
	2.77	0.17	0.10	72.71	11.7	
SDT 206 D2	**2.47**	**0.14**	**<0.07**	**68.91**	**11.7**	**3410**
	2.54	0.14	<0.07	67.69	11.7	
	2.40	0.14	0.15	70.13	11.7	
SDT 206 D3[†]	–	–	–	–	–	–
SDT 206 D4[†]	–	–	–	–	–	–
SDT 207 D1	**1.72**	**0.15**	**0.18**	**67.51**	**11.7**	**3930**
	1.68	0.14	0.15	67.06	11.6	
	1.75	0.16	0.20	67.96	11.7	
SDT 207 D2	**1.92**	**0.19**	**0.19**	**72.53**	**11.7**	**3340**
	1.92	0.19	0.18	72.68	11.7	
	1.91	0.19	0.20	72.37	11.7	
SDT 207 D3[†]	–	–	–	–	–	–
SDT 207 D4[†]	–	–	–	–	–	–
SDT 208 D1	**2.65**	**0.06**	**<0.07**	**35.81**	**11.1**	**3260**
	2.70	0.05	0.09	35.63	11.0	
	2.59	0.06	<0.07	35.98	11.1	
SDT 208 D2	**2.29**	**0.06**	**<0.07**	**37.09**	**11.3**	**2720**
	2.96	0.06	<0.07	37.13	11.2	
	1.61	0.06	0.07	37.05	11.3	
SDT 208 D3	**2.41**	**0.12**	**<0.07**	**38.55**	**10.4**	**3260**
	2.42	0.12	<0.07	38.87	10.4	
	2.40	0.11	<0.07	38.22	10.4	

	TCLP CONCENTRATIONS					Compressive
MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	Al (mg/L)	pH	Strength* (psi)
SDT 209 D1	1.87	0.06	0.08	36.13	11.5	2450
	1.71	0.05	0.07	35.71	11.5	
	2.02	0.07	0.09	36.54	11.5	
SDT 209 D2	1.92	0.09	≤0.07	38.00	11.5	2820
	1.98	0.08	<0.07	37.05	11.5	
	1.86	0.10	0.08	38.95	11.5	
SDT 209 D3	1.79	0.10	0.08	37.51	11.0	3750
	1.78	0.09	<0.07	37.39	11.0	
	1.79	0.10	0.10	37.63	11.0	
SDT 209 D4	1.77	0.09	0.12	35.58	11.2	2040
	1.73	0.08	0.16	35.11	11.1	
	1.81	0.09	0.07	36.04	11.2	

*28-day Compressive Strength; numbers are averages of two replicates (Salt 1993)
†Mix did not set
1000 psi = 6.895 MPa

RAW DATA FOR SODIUM SILICATE MIXES
(Bolded underlined values are the averages of the replicate values)

	TCLP CONCENTRATIONS					
MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	Al (mg/L)	pH	Compressive Strength[*] (psi)
CM #1	**0.34**	**0.16**	**≤0.07**	**30.76**	**11.1**	**8450**
	0.35	0.17	<0.07	31.29	11.1	
	0.33	0.16	<0.07	30.57	11.1	
	0.34	0.16	<0.07	30.41	11.0	
CM #2	**0.47**	**0.16**	**≤0.07**	**30.36**	**11.4**	**3510**
	0.47	0.16	<0.07	30.33	11.3	
	0.46	0.16	<0.07	30.43	11.4	
	0.47	0.16	0.10	30.33	11.4	
CM #3	**0.80**	**0.17**	**≤0.07**	**31.05**	**11.3**	**2340**
	0.79	0.16	<0.07	30.47	11.2	
	0.83	0.18	<0.07	32.16	11.3	
	0.78	0.16	<0.07	30.53	11.4	
CM #4[†]	–	–	–	–	–	–
SILI 1A	**0.20**	**0.13**	**≤0.07**	**30.01**	**11.1**	**7550**
	0.30	0.14	<0.07	30.33	10.9	
	0.29	0.12	<0.07	29.61	11.1	
	0.30	0.13	<0.07	30.08	11.2	
SILI 1B	**0.44**	**0.13**	**≤0.07**	**29.86**	**11.3**	**4900**
	0.43	0.12	<0.07	29.68	11.4	
	0.45	0.14	<0.07	30.08	11.4	
	0.45	0.13	<0.07	29.83	11.2	
SILI 1C	**0.70**	**0.14**	**≤0.07**	**30.30**	**11.2**	**4180**
	0.70	0.14	<0.07	30.15	11.2	
	0.70	0.14	<0.07	30.55	11.2	
	0.69	0.14	<0.07	30.21	11.3	
SILI 1D	**0.80**	**0.14**	**≤0.07**	**30.52**	**11.2**	**5560**
	0.84	0.14	<0.07	30.65	11.2	
	0.78	0.14	<0.07	30.42	11.2	
	0.78	0.14	<0.07	30.49	11.2	
SILI 2A	**0.40**	**0.13**	**≤0.07**	**29.11**	**11.3**	**7470**
	0.30	0.12	<0.07	29.12	11.2	
	0.30	0.13	<0.07	29.39	11.3	
	0.61	0.13	<0.07	28.83	11.4	
SILI 2B	**0.46**	**0.13**	**≤0.07**	**29.40**	**11.3**	**4600**
	0.45	0.13	<0.07	29.24	11.4	
	0.46	0.12	<0.07	29.29	11.2	
	0.46	0.13	<0.07	29.68	11.3	

	TCLP CONCENTRATIONS					
MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	Al (mg/L)	pH	Compressive Strength* (psi)
SILI 2C	**0.73**	**0.13**	**≤0.07**	**29.76**	**11.3**	**4510**
	0.73	0.13	<0.07	29.83	11.3	
	0.73	0.13	<0.07	29.61	11.2	
	0.73	0.14	<0.07	29.84	11.3	
SILI 2D	**0.73**	**0.14**	**≤0.07**	**29.73**	**10.9**	**5250**
	0.76	0.14	<0.07	29.99	11.1	
	0.73	0.13	<0.07	29.59	11.1	
	0.69	0.14	<0.07	29.62	10.6	

*28-day Compressive Strength; numbers are averages of two replicates (Salt 1993)
†Mix did not set
1000 psi = 6.895 MPa

RAW DATA FOR MONTOPOLIS BRIDGE MIXES
(Bolded underlined values are the averages of the replicate values)

	TCLP CONCENTRATIONS					
MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	Al (mg/L)	pH	Compressive Strength* (psi)
MONT 3	**2.38**	**0.17**	**≤0.07**	**33.28**	**11.1**	**3640**
	2.40	0.16	<0.07	33.13	11.2	
	2.40	0.17	<0.07	33.15	11.1	
	2.34	0.18	0.07	33.57	11.1	
MONT 4	**1.60**	**0.17**	**≤0.07**	**33.49**	**11.3**	**2710**
	1.64	0.17	<0.07	33.15	11.3	
	1.63	0.18	<0.07	34.10	11.3	
	1.52	0.17	<0.07	33.23	11.3	
MONT 11	**1.42**	**0.14**	**≤0.07**	**28.56**	**11.2**	**4620**
	1.44	0.15	<0.03	28.85	11.3	
	1.41	0.14	0.07	28.53	11.2	
	1.40	0.14	0.07	28.31	11.2	

*28-day Compressive Strength; numbers are averages of two replicates (Salt 1993)
†Mix did not set
1000 psi = 6.895 MPa

APPENDIX E

STATISTICAL COMPARISONS OF TCLP CHROMIUM AND CADMIUM CONCENTRATIONS FOR S/S MIXES

The unpaired Student's t-test was used to compare the leachate concentrations from one mix to the leachate concentrations of another mix. The test compares the means and standard deviations of the data sets of the two mixes and determines if they are from the same population. If they are from the same population, the concentrations are considered to be statistically the same. If they are not from the same population, the concentrations are considered to be statistically different (Section 3.14.2).

ODESSA BARREL #1 MIXES (470 lb Cement Content) CHROMIUM

	SDT 300	SDT 301	SDT 302	SDT 303	SDT 304	SDT 305
SDT 305	*	*	•	•	*	
SDT 304	•	*	•	•		
SDT 303	•	•	*			
SDT 302	•	•				
SDT 301	•					
SDT 300						

ODESSA BARREL #1 MIXES (470 lb Cement Content) CADMIUM

	SDT 300	SDT 301	SDT 302	SDT 303	SDT 304	SDT 305
SDT 305	*	•	•	•	*	
SDT 304	*	•	•	•		
SDT 303	*	*	*			
SDT 302	*	*				
SDT 301	*					
SDT 300						

Based on unpaired student's t statistics (95% confidence level)

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

ODESSA BARREL #1 MIXES (705 lb Cement Content) CHROMIUM

	SDT 400	SDT 401	SDT 402	SDT 403	SDT 404	SDT 405
SDT 405	•	•	*	*	*	
SDT 404	•	*	*	*		
SDT 403	*	*	•			
SDT402	•	•				
SDT 401	*					
SDT 400						

ODESSA BARREL #1 MIXES (705 lb Cement Content) CADMIUM

	SDT 400	SDT 401	SDT 402	SDT 403	SDT 404	SDT 405
SDT 405	*	*	•		*	
SDT 404	*	•	•			
SDT 403						
SDT 402	•	*				
SDT 401	*					
SDT 400						

Based on unpaired student's t statistics (95% confidence level)

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

ODESSA BARREL #2 MIXES (470 lb Cement Content) CHROMIUM

	SDT 500	SDT 501	SDT 502	SDT 503
SDT 503	*	*	*	
SDT 502	*	•		
SDT 501	*			
SDT 500				

ODESSA BARREL #2 MIXES (470 lb Cement Content) CADMIUM

	SDT 500	SDT 501	SDT 502	SDT 503
SDT 503	*	*	*	
SDT 502	*	*		
SDT 501	*			
SDT 500				

ODESSA BARREL #2 MIXES (705 lb Cement Content) CHROMIUM

	SDT 600	SDT 601	SDT 602	SDT 603
SDT 603	*	*	*	
SDT 602	•	•		
SDT 601	*			
SDT 600				

Based on unpaired student's t statistics (95% confidence level)

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

ODESSA BARREL #2 MIXES (705 lb Cement Content) CADMIUM

	SDT 600	SDT 601	SDT 602	SDT 603
SDT 603	*	*	*	
SDT 602	*	*		
SDT 601	*			
SDT 600				

SODIUM SILICATE MIXES: CHROMIUM

	CM #1	CM #2	CM #3
CM #3	●	●	●
CM #2	●	●	
CM #1	●		

SODIUM SILICATE MIXES: CHROMIUM

	SILI 1A	SILI 1B	SILI 1C	SILI 1D
SILI 1D	●	●	●	
SILI 1C	●	●		
SILI 1B	●			
SILI 1A				

Based on unpaired student's t statistics (95% confidence level)

* Denotes no statistical difference between samples

● Denotes statistical difference between samples

SODIUM SILICATE MIXES: CHROMIUM

	SILI 2A	SILI 2B	SILI 2C	SILI 2D
SILI 2D	●	●	*	
SILI 2C	●	●		
SILI 2B	*			
SILI 2A				

Based on unpaired student's t statistics (95% confidence level)

* Denotes no statistical difference between samples

● Denotes statistical difference between samples

Results of Statistical Comparisons of Mix "A" TCLP Chromium Concentrations to Mix "B" TCLP Chromium Concentrations using unpaired Student's t-test (95% confidence level)

Comparisons of Mixes made with 470 lb of cement to those made with 705 lb of cement

MIX A	MIX B	RESULT	MIX A	MIX B	RESULT
470 lb	705 lb		470 lb	705 lb	
SDT 300	SDT 400	*	SDT 204 D1	SDT 205 D1	*
SDT 301	SDT 401	*	SDT 206 D1	SDT 207 D1	*
SDT 302	SDT 402	•	SDT 206 D2	SDT 207 D2	•
SDT 303	SDT 403	•	SDT 208 D1	SDT 209 D1	•
SDT 304	SDT 404	•	SDT 208 D2	SDT 209 D2	*
SDT 305	SDT 405	*	SDT 208 D3	SDT 209 D3	•
SDT 500	SDT 600	*			
SDT 501	SDT 601	•			
SDT 502	SDT 602	•			
SDT 503	SDT 603	*			

1lb = 0.45 kg

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

Comparison of Odessa Mixes made with Barrel #1 Unseparated Spent Abrasive Sand to those made with Barrel #2 Unseparated Spent Abrasive Sand

Barrel #1	Barrel #2	Result	Barrel #1	Barrel #2	Result
SDT 300	SDT 500	*	SDT 400	SDT 600	*
SDT 301	SDT 501	•	SDT 401	SDT 601	•
SDT 302	SDT 502	*	SDT 402	SDT 602	•
SDT 303	SDT 503	*	SDT 403	SDT 603	*

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

Comparison of Mixes with Different Separated Spent Abrasive Dust Content

MIX A	MIX B	RESULT	MIX A	MIX B	RESULT
SDT 204D1	SDT204 D2	*	CM #1	CM #2	•
SDT 206D1	SDT206 D2	*	SILI 1A	SILI 1B	•
SDT 207D1	SDT 207D2	•	SILI 2A	SILI 2B	*
SDT 208D1	SDT 208D2	*	CM #1	CM #3	•
SDT 209D1	SDT 209D2	•	SILI 1A	SILI 1C	•
SDT 208D1	SDT 208D3	*	SILI 2A	SILI 2C	•
SDT 209D1	SDT 209D3	*	SILI 1A	SILI 1D	•
SDT 209D1	SDT 209D4	*	SILI 2A	SILI 2D	•
SDT 208D2	SDT 208D3	*	CM #2	CM #3	•
SDT 209D2	SDT 209D3	*	SILI 1B	SILI 1C	•
SDT 209D3	SDT 209D4	*	SILI 2B	SILI 2C	•
SILI 1B	SILI 1D	•	SILI 1C	SILI 1D	•
SILI 2B	SILI 2D	•	SILI 2C	SILI 2D	*

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

Comparison of Mixes with Different Silica Fume Content

MIX A	MIX B	RESULT	MIX A	MIX B	RESULT
SDT 300	SDT 301	•	SDT 204D1	SDT 209D1	*
SDT 400	SDT 401	*	SDT 204D2	SDT 209D2	*
SDT 500	SDT 501	*	SDT 205D1	SDT 208D1	*
SDT 600	SDT 601	*	MONT 4	MONT 11	•
MONT 4	MONT 3	•			

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

Comparison of Mixes with Different Fly Ash Content

MIX A	MIX B	RESULT	MIX A	MIX B	RESULT
SDT 300	SDT 302	•	SDT 500	SDT 502	•
SDT 400	SDT 402	•	SDT 600	SDT 602	•

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

Comparison of Mixes with Different DCI content

MIX A	MIX B	RESULT	MIX A	MIX B	RESULT
SDT 300	SDT 304	•	SDT 205D1	SDT 206D1	*
SDT 300	SDT 305	*	SDT 204D1	SDT 207D1	•
SDT 400	SDT 404	•	SDT 204D2	SDT 207D2	*
SDT 400	SDT 405	•			

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

APPENDIX F

LEACHATE CHROMIUM, CADMIUM AND LEAD CONCENTRATIONS AND pH DATA FOR SEQUENTIAL EXTRACTIONS

RAW DATA FOR ACIDIC SEQUENTIAL EXTRACTIONS:

(Bolded underlined values are the averages of the replicate values)

		TCLP CONCENTRATIONS			
Extraction Number	MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	pH
1	**SDT 204 D1**	**2.62**	**0.13**	**≤0.07**	**11.4**
		2.56	0.14	<0.07	11.4
		2.64	0.13	<0.07	11.4
		2.67	0.13	<0.07	11.4
	SDT 204 D2	**2.12**	**0.13**	**≤0.07**	**11.3**
		2.22	0.12	<0.07	11.3
		2.08	0.13	<0.07	11.3
		2.05	0.15	<0.07	11.3
	SDT 205 D1	**3.45**	**0.23**	**≤0.07**	**11.3**
		3.32	0.19	<0.07	11.2
		3.23	0.21	<0.07	11.4
		3.81	0.28	<0.07	11.4
2	**SDT 204 D1**	**2.51**	**0.19**	**0.12**	**8.3**
		2.29	0.19	0.13	7.1
		2.53	0.20	0.11	8.7
		2.70	0.19	0.12	9.0
	SDT 204 D2	**2.42**	**0.28**	**0.12**	**8.1**
		2.50	0.26	0.07	8.4
		2.35	0.28	0.13	8.0
		2.42	0.31	0.15	7.8
	SDT 205 D1	**2.83**	**0.31**	**0.14**	**7.6**
		3.02	0.36	0.14	7.0
		2.75	0.29	0.09	7.8
		2.72	0.27	0.18	8.1
3	**SDT 204 D1**	**3.79**	**1.39**	**1.03**	**5.8**
		3.28	1.32	1.09	5.7
		3.77	1.40	1.07	5.8
		4.31	1.46	0.93	6.0
	SDT 204 D2	**2.07**	**0.81**	**0.74**	**5.4**
		2.01	0.85	0.71	5.5
		1.96	0.74	0.75	5.4
		2.24	0.83	0.76	5.4
	SDT 205 D1	**1.47**	**0.86**	**1.80**	**5.1**
		1.37	0.85	1.75	4.9
		1.13	0.83	1.86	5.0
		1.92	0.90	1.80	5.1
4	**SDT 204 D1**	**1.37**	**0.39**	**1.99**	**4.5**
		1.20	0.36	2.11	4.5
		1.27	0.39	2.23	4.6
		1.64	0.43	1.64	4.5

		TCLP CONCENTRATIONS			
Extraction Number	MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	pH
4	204 D2	1.27	0.36	2.43	4.4
		1.22	0.37	2.38	4.5
		1.26	0.36	2.40	4.4
		1.34	0.35	2.51	4.4
	205 D1	1.98	0.44	6.95	4.2
		2.29	0.39	7.20	4.2
		1.92	0.50	8.38	4.2
		1.73	0.44	5.26	4.2
5	SDT 204 D1	1.09	0.17	2.96	4.2
		0.71	0.16	3.29	4.2
		1.76	0.17	3.01	4.2
		0.80	0.17	2.59	4.2
	SDT 204 D2	0.64	0.12	3.08	4.1
		0.63	0.11	3.29	4.0
		0.64	0.12	2.77	4.1
		0.65	0.12	3.18	4.1
	SDT 205 D1	0.87	0.13	7.81	3.9
		0.88	0.12	8.08	3.9
		0.89	0.13	7.75	3.9
		0.84	0.13	7.59	3.9
6	SDT 204 D1	0.55	0.14	5.05	3.7
		0.50	0.12	4.70	3.6
		0.60	0.14	5.44	3.7
		0.55	0.16	5.01	3.7
	SDT 204 D2	0.54	0.13	6.03	3.5
		0.45	0.14	6.20	3.4
		0.45	0.13	4.94	3.5
		0.71	0.13	6.95	3.5
	SDT 205 D1	0.53	0.12	11.21	3.4
		0.60	0.14	13.30	3.4
		0.36	0.07	7.65	3.3
		0.62	0.14	12.69	3.4
7	SDT 204 D1	0.50	0.07	5.39	3.4
		0.48	0.06	5.57	3.4
		0.50	0.07	5.52	3.4
		0.51	0.09	5.08	3.5
	SDT 204 D2	0.43	0.09	6.03	3.4
		0.48	0.10	6.29	3.4
		0.49	0.10	6.24	3.4
		0.33	0.08	5.55	3.3
	SDT 205 D1	0.54	0.09	9.30	3.3
		0.36	0.08	7.75	3.2
		0.56	0.09	8.78	3.3
		0.69	0.09	11.38	3.3

RAW DATA FOR SEAWATER SEQUENTIAL EXTRACTIONS:
(Bolded underlined values are the averages of the replicate values)

		TCLP CONCENTRATIONS			
Extraction Number	**MIX**	**Cr (mg/L)**	**Cd (mg/L)**	**Pb (mg/l)**	**pH**
1	**SDT 204 D1**	**2.41**	**0.04**	**<0.07**	**11.1**
		2.16	0.06	<0.07	11.2
		2.61	0.04	<0.07	11.4
		2.47	0.03	0.07	10.7
	SDT 204 D2	**4.91**	**0.14**	**<0.07**	**11.6**
		3.62	0.12	0.08	11.6
		5.14	0.14	<0.07	11.5
		5.96	0.17	0.08	11.6
	SDT 205 D1	**6.54**	**0.16**	**0.07**	**10.4**
		7.46	0.17	0.10	10.3
		7.72	0.17	<0.07	11.1
		4.45	0.15	0.07	9.9
2	**SDT 204 D1**	**1.13**	**0.07**	**<0.07**	**6.5**
		1.05	0.07	<0.07	5.8
		1.04	0.07	<0.07	8.1
		1.30	0.07	<0.07	5.5
	SDT 204 D2	**2.43**	**0.08**	**<0.07**	**6.7**
		2.29	0.06	<0.07	4.5
		2.58	0.09	<0.07	9.4
		2.42	0.10	<0.07	6.1
	SDT 205 D1	**4.88**	**0.14**	**0.07**	**8.6**
		3.36	0.12	<0.07	9.4
		4.69	0.13	<0.07	7.7
		6.60	0.18	0.22	8.7
3	**SDT 204 D1**	**0.65**	**<0.02**	**<0.07**	**6.6**
		0.57	0.02	<0.07	3.7
		0.60	<0.02	<0.07	9.0
		0.79	<0.02	<0.07	7.0
	SDT 204 D2	**0.65**	**0.05**	**<0.07**	**7.1**
		0.67	0.06	<0.07	3.5
		0.63	0.05	<0.07	8.6
		0.64	0.05	<0.07	9.3
	SDT 205 D1	**0.75**	**0.05**	**0.10**	**6.3**
		0.62	0.05	<0.07	9.0
		0.67	0.06	0.09	3.6
		0.96	0.05	0.21	6.1
4	**SDT 204 D1**	**0.49**	**0.05**	**<0.07**	**9.1**
		0.42	0.06	<0.07	9.1
		0.49	0.05	<0.07	9.3
		0.56	0.04	<0.07	9.0

		TCLP CONCENTRATIONS			
Extraction Number	MIX	Cr (mg/L)	Cd (mg/L)	Pb (mg/l)	pH
4	SDT 204 D2	0.50	0.07	0.10	5.9
		0.45	0.06	0.09	3.8
		0.53	0.08	0.20	8.1
		0.52	0.07	<0.07	5.7
	SDT 205 D1	0.58	0.08	0.12	8.6
		0.59	0.09	0.14	8.7
		0.49	0.07	0.08	8.0
		0.65	0.07	0.14	9.0
5	SDT 204 D1	0.32	0.03	<0.07	4.7
		0.33	0.03	<0.07	4.2
		0.35	0.03	<0.07	4.4
		0.27	0.02	<0.07	5.6
	SDT 204 D2	0.42	0.05	<0.07	7.2
		0.42	0.05	<0.07	7.2
		0.41	0.04	<0.07	9.0
		0.41	0.06	<0.07	5.4
	SDT 205 D1	0.38	0.04	<0.07	7.0
		0.45	0.05	<0.07	8.7
		0.31	0.03	<0.07	3.3
		0.39	0.03	<0.07	9.1
6	SDT 204 D1	0.45	0.12	0.08	8.6
		0.39	0.11	0.10	8.6
		0.49	0.12	0.08	8.6
		0.46	0.13	0.07	8.6
	SDT 204 D2	0.37	0.10	0.10	8.6
		0.35	0.10	0.07	8.5
		0.36	0.10	0.10	8.8
		0.39	0.10	0.12	8.4
	SDT 205 D1	0.44	0.12	0.12	8.5
		0.39	0.10	0.12	8.6
		0.45	0.14	0.10	8.3
		0.49	0.12	0.13	8.6
7	SDT 204 D1	0.57	0.10	0.07	6.8
		0.42	0.09	0.06	8.8
		0.84	0.10	0.07	3.0
		0.44	0.11	0.08	8.6
	SDT 204 D2	0.27	0.09	0.11	8.5
		0.24	0.08	0.07	8.0
		0.26	0.09	0.13	9.2
		0.30	0.09	0.14	8.5
	SDT 205 D1	0.33	0.09	0.10	8.4
		0.30	0.08	0.11	7.5
		0.29	0.09	0.09	9.0
		0.41	0.09	0.09	8.8

APPENDIX G

STATISTICAL COMPARISONS OF LEACHATE CHROMIUM, CADMIUM AND LEAD CONCENTRATIONS FOR SEQUENTIAL EXTRACTIONS

The unpaired Student's t-test was used to compare the leachate concentrations from one extraction to the leachate concentrations of next extraction to establish the existing leaching trends. The test compares the means and standard deviations of the data sets of the two extractions and determines if they are from the same population. If they are from the same population, the concentrations are considered to be statistically the same. If they are not from the same population, the concentrations are considered to be statistically different (Section 3.14.2).

ACIDIC SEQUENTIAL EXTRACTIONS

Results of Statistical Comparisons of Leachate Concentrations for Extraction "X" to Extraction "X+1" using unpaired Student's t-test (95% confidence level) (CR= chromium, CD = cadmium, PB = lead)

SDT 204 D1

Extraction X	Extraction X+1	RESULT	Extraction X	Extraction X+1	RESULT
CR 1	2	*	PB 1	2	–
2	3	•	2	3	•
3	4	•	3	4	•
4	5	*	4	5	•
5	6	*	5	6	•
6	7	*	6	7	*
CD 1	2	•			
2	3	•			
3	4	•			
4	5	•			
5	6	*			
6	7	*			

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

SDT 204 D2

Extraction X	Extraction X+1	RESULT	Extraction X	Extraction X+1	RESULT
CR 1	2	•	PB 1	2	–
2	3	•	2	3	•
3	4	•	3	4	•
4	5	•	4	5	•
5	6	*	5	6	•
6	7	*	6	7	*
CD 1	2	•			
2	3	•			
3	4	•			
4	5	•			
5	6	*			
6	7	*			

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

SDT 205 D1

Extraction X	Extraction X+1	RESULT	Extraction X	Extraction X+1	RESULT
CR 1	2	•	PB 1	2	—
2	3	•	2	3	•
3	4	*	3	4	•
4	5	•	4	5	*
5	6	•	5	6	*
6	7	*	6	7	*
CD 1	2	*			
2	3	•			
3	4	•			
4	5	•			
5	6	*			
6	7	*			

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

SEAWATER SEQUENTIAL EXTRACTIONS

Results of Statistical Comparisons of Leachate Concentrations for Extraction "X" to Extraction "X+1" using unpaired Student's t-test (95% confidence level)(CR= chromium, CD = cadmium)

SDT 204 D1

Extraction X	Extraction X+1	RESULT	Extraction X	Extraction X+1	RESULT
CR 1	2	•	CD 1	2	•
2	3	•	2	3	—
3	4	*	3	4	—
4	5	•	4	5	•
5	6	•	5	6	•
6	7	*	6	7	*

* Denotes no statistical difference between samples

• Denotes statistical difference between samples

SDT 204 D2

Extraction X	Extraction X+1	RESULT	Extraction X	Extraction X+1	RESULT
CR 1	2	•	CD 1	2	•
2	3	•	2	3	*
3	4	•	3	4	*
4	5	•	4	5	*
5	6	•	5	6	•
6	7	•	6	7	•

* Denotes no statistical difference between samples
• Denotes statistical difference between samples

SDT 205 D1

Extraction X	Extraction X+1	RESULT	Extraction X	Extraction X+1	RESULT
CR 1	2	*	CD 1	2	*
2	3	•	2	3	•
3	4	*	3	4	•
4	5	•	4	5	•
5	6	*	5	6	•
6	7	*	6	7	•

* Denotes no statistical difference between samples
• Denotes statistical difference between samples

BIBLIOGRAPHY

American Society of Testing and Materials, 1987 Annual Book of ASTM Standards, Section4, Construction, Vol. 04.02 Concrete and Aggregates, American Society of Testing and Materials, Philadelphia, (1987).

American Society of Testing and Materials, "Standard Test Method for Compressive Strength of Cylindrical Specimens," ASTM C39-79, Philadelphia, (1979).

American Society of Testing and Materials, "Standard Method of Load Verification Of Test Machines," ASTM E4-79, Philadelphia, (1979).

American Society of Testing and Materials, "Standard Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes ," ASTM C511-85, Philadelphia, (1985).

American Society of Testing and Materials, "Standard Specification for Portland Cement," ASTM C150-86, Philadelphia, (1986).

American Society of Testing and Materials, "Standard Specification for Ready-Mixed Concrete," ASTM C150-86b, Philadelphia, (1986).

American Society of Testing and Materials, "Standard Test Method for Compressive Strength of Hydraulic Cement Mortars," ASTM C109-87, Philadelphia, (1987).

American Society of Testing and Materials, "Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory," ASTM C198-88, Philadelphia, (1988).

Bishop, Paul L., "Leaching of Inorganic Hazardous Constituents from Stabiliized/Solidified Hazardous Wastes," Hazardous Waste & Hazardous Materials, 5(2):129-143 (1988).

Boss, Charles B. and Fredeen, Kenneth J., Concepts, Instrumentation, and Techniques in Inductively Coupled Plasma Atomic Emission Spectrometry, Perkin Elmer, (1989).

Brabrand, Douglas, Solidification/Stabilization of Spent Blasting Abrasives with Portland Cement For Non-Structural Concrete Purposes, M.S. Thesis, University of Texas at Austin, (1992).

Brabrand, Douglas J. and Raymond C. Loehr, "Solidification/Stabilization of Spent Abrasives and Use as Nonstructural Concrete," Waste Management, 13:333-339 (1993).

Bricka, Mark, Theresa Holmes and John Cullinane, "Project Summary: A Conparative Evaluation of Two Extraction.Procedures: The TCLP and the EP," EPA Risk Reduction Engineering Laboratory, Cincinnati, EPA #/600/S2-91/049, (1992).

Cheng, Kuang Ye, "Controlling Mechanisms of Metals Release From Cement-Based Waste Form in Acetic Acid Solution,"Ph.D. Dissertation, The University of Cincinnati, (1991).

Cheng K.Y. and Bishop P., "Metals Distribution in Solidified/Stabilized Waste Forms after Leaching," Hazardous Waste and Hazardous Materials, 9(2):163-171 (1992).

Chin, D. A Calcium Nitrite-Based, Non-Corrosive, Non-Chloride Accelerator. "Corrosion, Concrete, and Chlorides; Steel Corrosion in Concrete: Causes and Restraints", ACI SP-102. American Concrete Insitute, Detroit, 1987, pp.49-77.

Conner, Jesse R., Chemical Fixation and Solidification of Hazardous Wastes, Van Nostrand Reinhold, New York, N.Y., (1990).

Cote, P., Contaminant Leaching from Cement-Based Waste Forms Under Acidic Conditions, Ph.D Dissertation, McMaster Univ., Hamilton, Ontario, (1986).

"Eliminating the Costs of Solids Ash Disposal," Water Environment Technology, p. 69, January 1993.

Jackman, Alan P. and Powell, Robert L., Hazardous Waste Treatment Technologies, Noyes Publications, Park Ridge, New Jersey, (1991).

Journal of Protective Coatings & Linings, "News From the Field: Maine DOT Considers Treating Hazardous Waste With Iron Filings," 9(7):78, (1992).

Khosla, Paul N., and Leming, Michael L., "Recycling of Lead-Contaminated Blasting Sand in Construction Materials," Steel Structures Painting Council Lead Paint Removal- Procedings of the SSPC Symposium, Feb.20-Mar. 1, Steel Structures Painting Council, Pittsburg, (1988).

Kosmatka, Steven H., and William Panarese, Design and Control of Concrete Mixtures, Thirteenth Edition, Portland Cement Association, Skokie,IL, (1990)

Nehring, Karl W. and Brauning, Susan E., "S/S Process Converts Hazwaste For Reuse," Environmental Protection, 3(3):14-16(38), (1992).

Ovenden, Tom, "The Land Disposal Restrictions Program and the Paint and Coatings Industry," Journal of Protective Coatings and Linings, Vol. 7, No.8, pp. 35-58, (1990).

Pankow, James F., Aquatic Chemistry Concepts, Lewis Publishers, Chelsea, Michigan, (1991).

"PennDot Tries Total Containment on Bridge Repainting Project," Civil Engineering, pp. 20-22, November 1993.

Rafferty, James, Richard Norling, Robert Tamaru, Charles McMath, and David Morganstein, StatWorks: Statistics with Graphics for the Macintosh, Version 1.2, Cricket Graph Software, Philadelphia, (1985).

Roy, Amitava, Harvill C. Eaton, Frank K. Cartledge, and Marty E. Tittlebaum, "Solidification/Stabilization of Hazardous Waste: Evidence of Physical Encapsulation," Environmental Science and Technology, 26(7):1349-1353, (1992).

Salt, Bryan K. Recycling Contaminated Spent Blasting Abrasives In Portland Cement Mortars Using Solidification/Stabilization Technology, M.S. Thesis, University of Texas at Austin, (1993).

Shively, William, Bishop, Paul, and Brown Todd, "Leaching Tests of Heavy Metals Stabilized with Portland Cement," Water Pollution Control Federation Journal, 58(3):234-241 (1986).

Skoog, Douglas A., and James J. Leary, Principles of Instrumental Analysis Third Edition, Saunders College Publishing, Philadelphia, (1985).

"Solidification/Stabilization Using Portland Cement," The Hazardous Waste Consultant, pp. 1.6-1.11, July/August 1992.

Stumm, Werner, and James J. Morgan, Aquatic Chemistry: An Introduction Emphsizing Chemical Equilibria in Natural Waters, 2nd Edition , John Wiley and Sons, New York, (1981).

Transportation Research Board, Bridge Paint: Removal, Containment, and Disposal, , National Cooperative Highway Research Prgram Synthesis of Highway Practice, 176, February, (1992).

U.S. EPA, RCRA Orientation Manual, EPA 530-sw-86-001, (1986).

U.S. EPA, 40 CFR Part 261-267, July 1, (1990).

U.S. EPA, Test Methods for Evaluating Solid Waste, SW-846, Office of Solid Waste and Emergency Response,Washington, DC, Revision 1, December, (1987).

U.S. Federal Register, "Guidelines EstablishingTest Procedures for the Analysis of Pollutants; Microwave Digestion; Final Rule," September 11, 1992.

U.S. Federal Register, "Land Disposal Restrictions for Newly Identified and Listed Hazardous Wastes and Hazardous Soil; Proposed Rule," September 14, 1993.

Verner, Sidney, Handbook for Preparing Quality Assurance Project Plans for Environmental Measurements, Rockville, MD (1990).