

## Chapter 15

# Contaminant Leaching from Solidified-Stabilized Wastes

## Overview

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The current state-of-the-art of solidification/stabilization (S/S) technologies is reviewed. This includes the legal impetus and basis for use of solidification/stabilization for hazardous wastes or contaminated soils, the principles and chemistry of contaminant immobilization within the waste form matrix, leaching mechanisms, and environmental factors affecting leachability. It is shown that S/S processes can be very effective at immobilizing certain waste materials, but other wastes may not be amenable to these processes.

Stabilization/solidification (S/S) processes have been developed to concurrently eliminate land disposal of liquid wastes and minimize leaching of the resultant solid waste after disposal. These processes are also being used to remediate existing hazardous waste sites by markedly reducing the rate of leaching of pollutants from contaminated soils and debris.

Land disposal of wastes should not be the primary means of waste disposal if other alternatives are available. Waste reduction, recycle and reuse are much superior alternatives. Where this is not possible, destruction or detoxification options should be considered. There will always be some wastes, though, where these options are not viable. Most reuse or destruction operations will result in some residue which cannot be further reduced and which must be disposed of on land. This includes flyash and bottom ash from incineration processes, mixed metal sludges, foundry sands, heavy metal contaminated soils, etc. Direct land disposal of these wastes could lead to potentially serious consequences, however, if contaminants in the waste leach into ground or surface waters. Many of these wastes can be effectively treated by stabilization/solidification processes so as to minimize leaching to environmentally acceptable levels.

Much of the impetus for S/S of hazardous wastes has been provided by the Resource Conservation and Recovery Act (RCRA) of 1976, including the 1984 amendments, and the Comprehensive Environmental Response, Liability and Recovery Act (CERCLA) of 1980, later reauthorized in 1986 as the Superfund Amendments and Reauthorization Act (SARA). RCRA deals primarily with the generation, handling, treatment and disposal of hazardous wastes, while CERCLA and SARA established a massive remedial program for the cleanup of existing sites that threaten the environment.

In 1985, under RCRA authority, the U.S. EPA banned the disposal of bulk hazardous liquids into landfills, necessitating solidification of the waste. Stabilization/solidification technologies have been specified by EPA as "best demonstrated available technologies" for a number of waste streams, and some can be used as a basis for "delisting" a waste as hazardous under RCRA.

Under SARA provisions, permanent treatment of contaminated soil and debris is being emphasized rather than the use of nontreatment containment systems such as covers, grout walls and similar methods. A large number of Superfund sites are now using S/S treatment processes for soil treatment.

Stabilization/solidification technology refers to treatment processes that are designed to (1) improve the handling and physical characteristics of the waste (2) decrease the surface area of the waste mass across which transfer or loss of contaminants can occur, and/or (3) limit the solubility of any hazardous constituents of the waste such as by pH adjustment or sorption phenomena.

Stabilization processes attempt to reduce the solubility or chemical reactivity of a waste by changing its chemical state or by physical entrapment. The hazard potential of the waste is reduced by converting the contaminants to their least soluble, mobile or toxic form. Solidification refers to techniques that encapsulate the waste in a monolithic solid of high structural integrity. Solidification does not necessarily involve a chemical interaction between the wastes and the solidifying reagents, but may mechanically bind the waste into the monolith. Contaminant migration is restricted by vastly decreasing the surface area exposed to leaching and/or by isolating the wastes within a relatively impervious capsule [1].

The most important factor in determining whether a particular stabilization/solidification process is effective in treating a given waste is the reduction in the short- and long-term leachability of the waste [2]. Leaching can be defined as the process by which a component of waste is removed mechanically or chemically into solution from the solidified matrix by the passage of a solvent such as water. Resistance to leaching will depend on both the characteristics of the solidified/stabilized waste and on those of the leaching medium it will come into contact with.

This paper discusses the principles of contaminant immobilization in solidified/stabilized wastes, leaching mechanisms from these materials, factors which affect leaching, and models which can be used to predict short- and long-term leaching rates.

### Principles of Immobilization

Stabilization/solidification processes employ systems which both solidify the waste mass and eliminate free liquids; and stabilize the contaminant in their least soluble form. The overall objective is to minimize the rate of leaching of pollutants from the resulting waste form. These processes typically involve the addition of binders and other chemical reagents to the contaminated soil or sludge to physically solidify the waste and chemically bind the contaminants into the monolith.

Binder systems can be placed into two broad categories, inorganic or organic. Most inorganic binder systems in use include varying combinations of hydraulic cements, lime, flyash, pozzolans, gypsum and silicates. Organic binders used or experimented with include epoxy, polyesters, asphalt/bitumen, polyolefins (primarily polyethylene and polybutadiene) and urea formaldehyde. Combinations of inorganic and organic binder systems have also been used. These include diatomaceous earth with cement and polystyrene, polyurethane and cement, polymer gels with silicates, and lime cement with organic modified clays [2].

Most immobilization processes currently in use involve hydraulic cements, such as Portland cement, cement kiln dust, flyash, or other pozzolanic materials. Consequently, the focus of this review will be on cement-based processes.

The main components of cement are lime and silicates. Cementation of the mixture begins when water is added, either directly or as part of the waste being immobilized. First, a calcium-silicate-hydrate gel forms, followed by hardening of the material as thin, densely-packed, silicate fibrils grow and interlace. The hydration reactions form a variety of compounds as the cement paste sets, including calcium hydroxides and calcium silicate hydrates. The latter provides the cement's structural stability, while the former supplies large amounts of entrapped alkaline material.

The water-to-cement ratio (W/C) is very important to the properties of the final product. The volume of the cement approximately doubles upon hydration, creating a network of very small gel pores. The volume originally occupied by the added water forms a system of much larger capillary pores. As the water-cement ratio increases, the percentage of larger pores increases, substantially increasing the permeability of the waste form and increasing the potential for contaminant leaching. A W/C of 0.48 by weight will fully

hydrate the cement, leaving some free pore water, gel water and air voids. Above this W/C, permeability increases rapidly, which could lead to increased leaching rates. Because of economic reasons, though, these low water-binder ratios are usually not feasible for waste immobilization. Very low permeability is sacrificed for a decrease in the amount of binder required.

A number of factors affect the degree of immobilization, or fixation, of constituents in the waste. Major factors include solubility minimization through pH or redox potential control; chemical reaction to form carbonate, sulfide or silicate precipitates; adsorption, chemisorption; diadochy (substitution in the calcite crystal lattice); and encapsulation.

Not all wastes can be effectively treated by solidification/stabilization technologies. The major category of wastes for which immobilization is applicable are those which are essentially all inorganic. Cement and pozzolan-based waste forms rely heavily on pH control for pollutant containment. Cement-based waste forms typically have a pore water pH of 10-12 because of the excess lime present in the pores. These high pH values are usually desirable for heavy metal immobilization because most metal hydroxides have minimum solubility in the range of 7.5-11. Some metals, though, are amphoteric and have higher solubility at both low and high pH. These metals may be soluble at the high pH of the pore water (see Figure 1). Other contaminants, such as anions (arsenate, selenite, etc.), may be more soluble at high pH than low. Metals may also precipitate as carbonates, silicates or metal sulfides [3,4].

Metal immobilization is primarily dependent on the extent of solubilization of precipitated metals. This is governed by the solubility product,  $K_{s0}$ . Therefore, the solid metal concentration in the waste does not affect the concentration in the pore water which can leach out; only the pore water (leachant) composition will govern the amount of metal which will leach. It is only the solubilized fraction which can diffuse out of the waste form into the surrounding environment.

Cement and pozzolan-based systems rely heavily on hydroxide formation for metal containment, but other factors can come into play. Shively et al. [5] demonstrated that even after the alkali was leached from cement-based waste forms, lead and chromium leaching was much lower than would be expected from metal hydroxide solubilities. In this case, the metals were probably bound into the silica matrix itself. Cote [3] also found differences between calculated hydroxide solubility-pH curves and those determined empirically.

A number of agents which may be present in the waste may interfere with the binding systems and lead to decreased immobilization of hazardous constituents. Some of these are inorganic (certain metals, sulfates, etc.), while others are organic (oil, grease, HCB, TCE, phenol, etc.) [6,7]. For example,

some metals may temporarily inhibit setting of cement-based processes; chlorides may decrease durability; oils, greases and other nonpolar organics may inhibit setting and decrease long-term durability. Sulfates are a particular problem because they cause the expansive compound ettringite to form.

Long-term immobilization of a contaminant incorporated into a cement matrix depends primarily on the ability of the matrix to maintain its integrity. Durability refers to the resistance of the matrix to chemical and physical interactions in the environment. All compounds of cement hydration are relatively insoluble in neutral water with the exception of  $\text{Ca}(\text{OH})_2$ . Lime will leach easily, leaving a much more porous structure. Acids can dissolve the matrix of hydrated cement, releasing many of the bound metals. Freeze-thaw and wet-dry cycles can cause fracturing of the matrix, resulting in increased liquid-solid interfaces where leaching can occur. Little research has been conducted on the influence of these factors on long-term containment of heavy metals.

Very little research has been reported on immobilization of organics in waste forms, and what has been reported is often contradictory. Several researchers have reported chemical reactions between organic waste and binder, resulting in immobilization, but most researchers report that these positive results may actually be due to sorption effects, volatilization of organics or dilution by reagent chemicals. Much more research is needed on the stabilization/solidification of organics, because most principally inorganic wastes which are suited for stabilization/solidification also contain appreciable quantities of organics which may leach.

Organic constituents tend to retard cementitious reactions, inhibiting the formation of a solid monolithic mass. Also, the organic components may be easily leached from the waste form. Recently, research has been conducted into the use of organically modified clays in order to overcome these difficulties [8]. When these clays are mixed with cement-based stabilization agents, they reportedly adsorb and retain organic pollutants while solidifying organic wastes into a stable mass with low leaching potential.

The modified organophilic clays are made by mixing quaternary ammonium ions with the clay. The  $[\text{R}_4\text{N}]^+$  ions substitute for metal ions present between the layers of alumina and silica in the clay minerals. This yields clays that have both organic and inorganic properties. Introduction of these ions increases the interplanar distance between clay plates allowing organics to penetrate, and makes the polarity of the stationary phase more compatible to that of the organic waste to be stabilized [9,10]. Preliminary studies indicate that chemical bonding between the organophilic clay binder and certain organic wastes may occur, but it is too early to tell the long-term fate of these complexes. It is possible that a unique binding mixture may be required for each organic compound encountered.

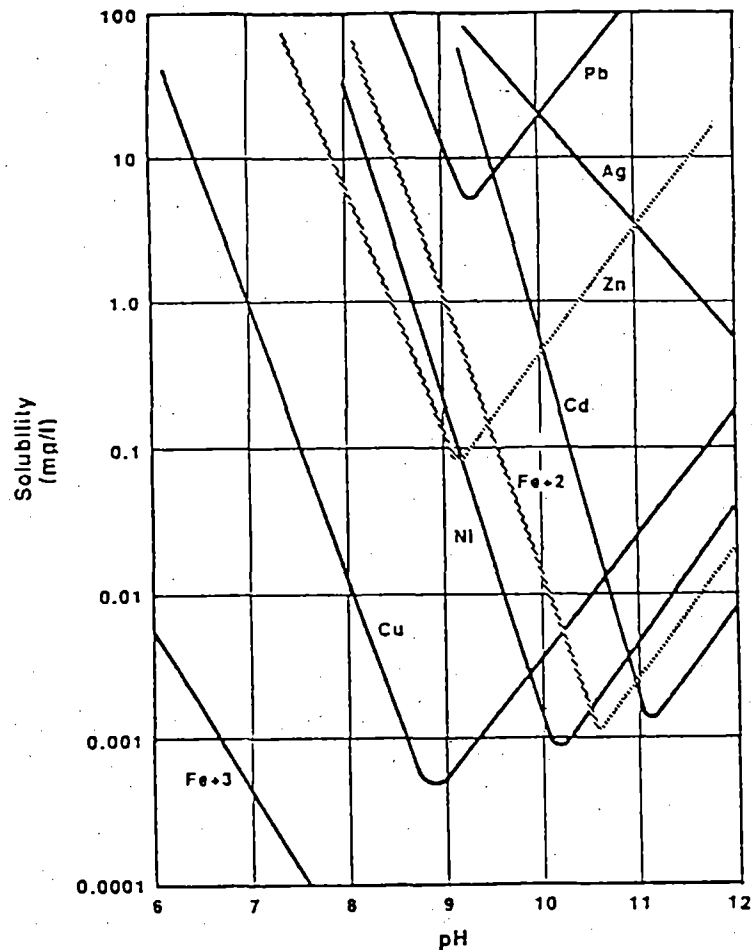


Figure 1. Solubilities of metal hydroxides as a function of pH.

### Leaching Mechanisms

A solidified waste is a porous solid at least partially saturated with water. The pore water in the solid is in chemical equilibrium with the solid phase. When the solid is exposed to leaching conditions, equilibrium is disturbed. The resulting difference in chemical potential between the solid and the leaching solution causes a mass flux between the solid surface and the leachant. This in turn causes concentration gradients that result in bulk diffusion through the solid [11,12]. Figure 2 depicts the leaching mechanisms in effect. Transport can either be by diffusion of metal ions from the solid matrix surface into the bulk aqueous phase, or by dissolution into the water in matrix pores and microfractures and then diffusion out. Consequently, the porosity and integrity of the waste form is of major importance.

For any constituent to leach, it must first dissolve in the pore water of the solid matrix. The amount of dissolution which occurs is dependent on the solubility of the constituent and the chemical makeup of the pore water, particularly its pH. Under neutral pH leaching conditions, the leaching rate is controlled by molecular diffusion of the solubilized species. Under acidic conditions, however, the rate will also be governed by the rate of penetration of hydrogen ions into the solid matrix, since this establishes the speciation and solubility of the contaminants present. Acid attacks pozzolanic-based paste through permeation of pore structure and dissolution of ions that must diffuse back through a chemically altered layer to enter solution. Acid consumes most of the calcium hydroxide in the leached layer and leaves a highly porous structure. Diffusion across this layer can be considered as a steady-state process since the leached layer provides little resistance to diffusion. At the leaching front, diffusion of hydrogen ions proceeds as if the unleached medium is infinite and dissolution reactions occur simultaneously in the pores. Proton transfer reactions are usually very fast with half-lives less than milliseconds. Hence, the dissolution reactions can be treated as diffusion-controlled fast reactions. The whole process then can be described as steady-state diffusion across the leached layer and unsteady-state diffusion controlled fast reactions in the porous leaching front [13].

Figure 3 depicts theoretical concentration gradients produced in the waste form during leaching [12]. The  $H^+$  ions in the penetrating leachant react with metal hydroxides in the waste form, solubilizing the metal and reducing the  $H^+$  concentration. The result is a leached layer where  $H^+$  is essentially totally consumed. The soluble metal concentration in the pore water peaks at the leaching front. There is a gradient for the metal to diffuse out to the surface, but it can also move further back into the matrix where it reacts with the excess alkalinity and reprecipitates. Thus a narrow zone behind the leaching front is denser than the bulk solid.

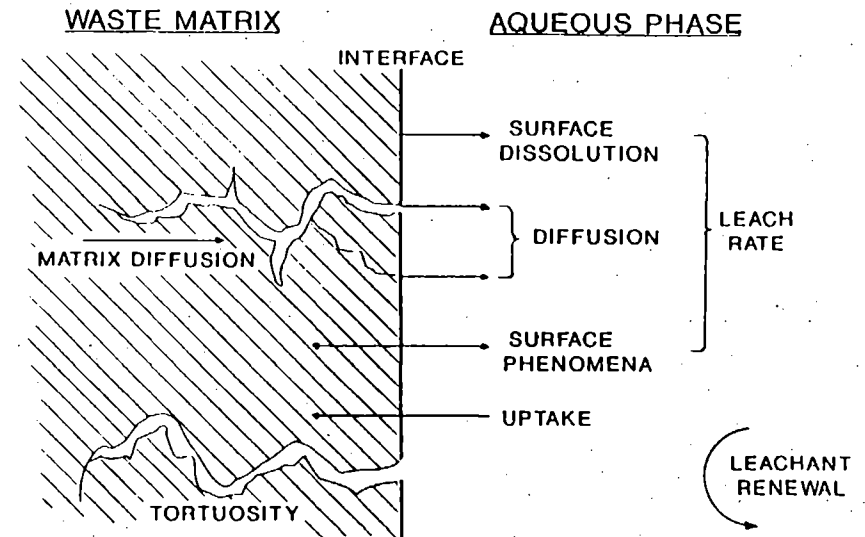


Figure 2. Schematic showing leaching mechanisms from a waste product. (Reprinted with permission from ref. 12. Copyright 1989 American Society for Testing and Materials.)

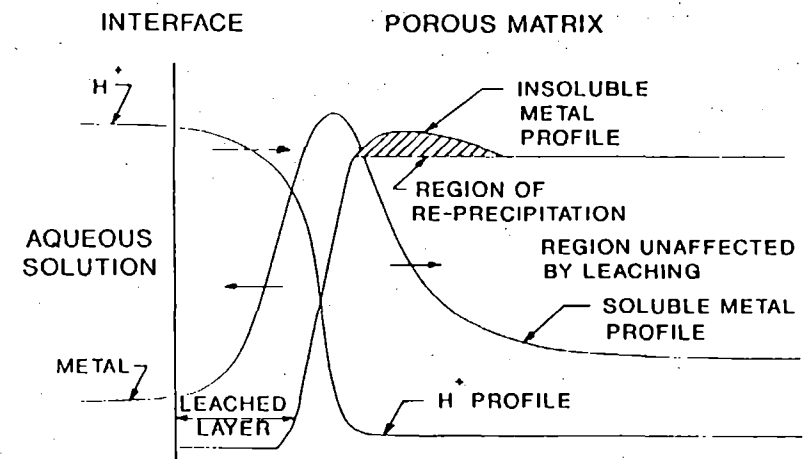


Figure 3. Concentration gradients during leaching. (Reprinted with permission from ref. 12. Copyright 1989 American Society for Testing and Materials.)

Recent research in our laboratories confirms these findings [14]. Measurements of pH and heavy metals across the profile at various stages of leaching demonstrate the leached layer, leaching zone and reprecipitation zone. It has been shown that the leaching zone is very narrow, usually less than 10 $\mu$ m deep. There is little or no change in pH from that of the leachant all the way through the leached zone to the leaching front, indicating that all available alkalinity is leached from the leaching zone as the leaching front progresses.

Once solubilized, the constituent is transported from the solid matrix through the leached zone to the leaching solution by molecular diffusion. The flux of the constituent within the solid can be described by Fick's first law:

$$J = -D \frac{dC}{dz} \quad (1)$$

where:

- C = concentration of the constituent
- D = diffusion coefficient
- J = flux
- z = distance

A semi-infinite medium diffusion model with uniform initial concentration and zero surface concentration can be used to interpret the kinetic data generated from serial batch leaching tests [15]. The equation takes the form

$$\frac{\sum a_n}{A_0} \frac{V}{S} = 2 \left( \frac{D_e}{\pi} \right)^{0.5} t_n^{0.5} \quad (2)$$

- where
- $a_n$  = contaminant loss during leaching period n (mg)
  - $A_0$  = initial amount of contaminant present in the specimen (mg)
  - V = volume of specimen (cm<sup>3</sup>)
  - S = surface area of specimen (cm<sup>2</sup>)
  - $t_n$  = time to end of leaching period n (sec)
  - $D_e$  = effective diffusion coefficient (cm<sup>2</sup>/sec)

The American Nuclear Society recommends use of a series of seven batch leaching tests in order to determine the effective diffusion coefficient in the Godbee and Joy model [16]. They suggest that the results be presented

as a "leachability index", LX, equal to the average negative logarithm of  $D_e$ .

$$LX = \frac{1}{7} \log \sum_1^7 \left( \frac{1}{D_e} \right) \quad (3)$$

This index can be used to compare the relative mobility of different contaminants on a uniform scale that varies from about 5 ( $D_e = 10^{-5}$  cm<sup>2</sup>/s, very mobile) to 15 ( $D_e = 10^{-15}$  cm<sup>2</sup>/s, immobile) [17].

Leachate generation is an extremely complex process. The free alkalinity present in the pozzolanic-based paste maintains a high pH environment and limits the metal leachability of fixed wastes. Calcium hydroxide, which is produced by the hydration reactions of the binder, provides most of the buffering capacity. The leaching model shown above, however, does not include the factor of acid strength of the leachant and cannot describe the rate of movement of leaching front into the waste solid.

Cheng and Bishop [13] have shown that the rate of advance of the leaching front can be expressed as steady-state diffusion across the leached layer. Figure 4 depicts the cumulative amount of calcium leached and Figure 5 shows the penetration distance into the waste matrix versus the square root of time for two leachant acetic acid strengths (5 and 15 meq/g solids). The plots are linear, as would be expected for diffusion-based processes. The leachant strengths differ by a factor of 3.0, but the ratios of the amount of leaching and the penetration distances differ by factors of 1.65 and 1.95, respectively. This can possibly be explained by comparing the free hydrogen ions arising from dissociation of the diffusing acetic acid. A 15 meq/g acetic acid leachant contains approximately 1.73 times more free hydrogen ions than the 5 meq/g leachant. This is a very close agreement, considering the complex, heterogeneous nature of the waste matrix. Thus, leachant acid strength may be very important in determining the rate and extent of leaching of solidified/stabilized wastes. This concept is currently not included in any leaching models, however.

#### Factors Affecting Leachability

There are a number of factors which can affect the leachability of a particular solidified/stabilized waste form. Table 1 presents a summary of some of the more important ones.

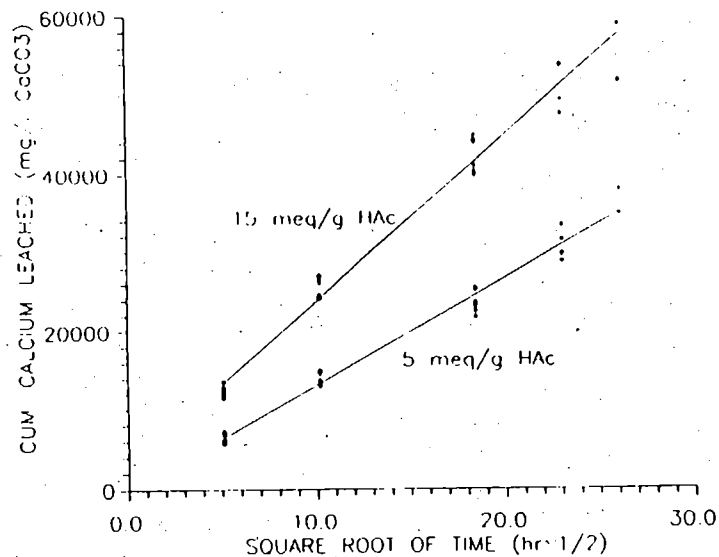


Figure 4. Cumulative calcium hardness leached versus square root of time during leaching of portland cement solidified/stabilized wastes using two strengths of acetic acid leachant.

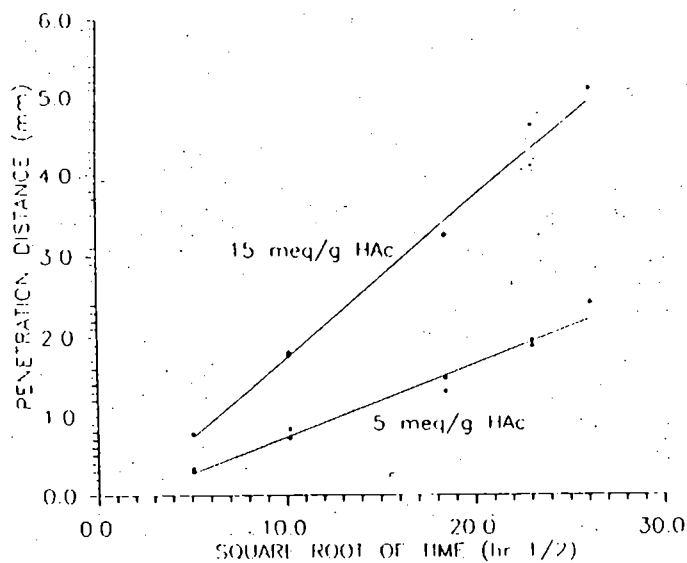


Figure 5. Acid penetration distance versus square root of time during leaching of portland cement solidified/stabilized wastes using two strengths of acetic acid leachant.

Table 1. Factors That Affect Leaching from Solidified/Stabilized Wastes

#### Waste Form Factors

- Contaminant binding mechanisms
- Alkalinity
- Surface-to-volume ratio
- Porosity and pore tortuosity
- Durability

#### Leachant Factors

- Composition (pH, acidity,  $E_h$ , chelating potential, etc.)
- Leachant volume to waste form surface area ratio
- Flow rate
- Temperature

Obviously, the composition of the waste form determines the physicochemical properties and the leaching mechanisms. Every effort should be made to minimize the potential for leaching by improving the quality of the waste form. Alkalinity is needed in the final product to maintain metals in their most insoluble form and to buffer against acid dissolution. One of the principle factors governing diffusion of soluble metals from the waste form is the solid surface-to-volume ratio (see equation 3). The larger the monolith, the smaller the surface-to-volume ratio and the smaller the potential for leaching. The role of internal fractures in determination of the applicable surface-to-volume ratio to use has not as yet been determined. Porosity and tortuosity, which is a measure of the path length for a diffusing substance to reach the surface of the waste product through winding and convoluted pores, governs to a large extent the rate of diffusion to be expected. A highly porous matrix will have a higher effective diffusion coefficient for a particular contaminant than a less porous one, while wastes with a large tortuosity factor will have reduced  $D_e$ . The "effective" diffusion coefficient in equation 3 modifies the true diffusion coefficient for the contaminant to account for these variations in porosity and tortuosity. Waste durability is very important because if an initially intact and acceptable monolith weathers poorly over time, the porosity and surface-to-volume ratios will increase markedly, resulting in increased leachability.

The leachant composition determines the reactions which will occur within the waste form. Acid dissolution, oxidation-reduction reactions and metal complexation can all occur, depending on the chemical composition of the leachant. Increased leachant volume to surface area ratios and flow rates will increase leaching because diffusing substances will be removed from the monolith surface more rapidly and concentration gradients in the solid will be greater. Temperature is not normally a factor for buried waste forms once the exothermic heat of reaction of the cement has dissipated. Increases in temperature result in increases in all reaction rates, including those involved in leaching, which can be described by the Arrhenius equation.

### Summary

Stabilization/solidification processes are being used to minimize the potential for groundwater pollution from land disposal of hazardous wastes. Many variations are used, but most rely on pozzolanic reactions to chemically stabilize and physically solidify the waste. Portland cement alone or in combination with fly ash, cement kiln dust, lime or other ingredients is the principal solidifying agent used.

Stabilization/solidification processes are very effective at immobilizing most heavy metals present in sludges, contaminated soils and other wastes. They are not as effective at immobilizing toxic organic materials. Organically modified clays are now being evaluated as an additive to S/S processes in order to adsorb and retain these organic pollutants in the solidified waste form.

The environmental acceptability of stabilization/solidification processes will depend on the long-term ability of the waste form to retain contaminants. This will be governed by the chemical binding mechanisms involved and by the durability of the waste form. Many S/S processes have been developed which can pass regulatory leaching tests, but these tests do not indicate the potential for leaching after long-term environmental exposure. Wide spread acceptance of stabilization/solidification processes will be hampered until the long-term durability of the waste form can be demonstrated.

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