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Project Summary

Compatibility of Grouts with Hazardous Wastes

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A study was conducted to determine existing information on the compatibility of grouts with different classes of chemicals. The data gathered can be used as a guide for testing and selecting grouts to be used at specific waste disposal sites with various leachates.

The 12 types of grouts used in this study were chosen because of their availability and use in waterproofing and soil consolidation projects. These grouts are bitumen, Portland cement Type I, Portland cement Types II and V, clay, clay-cement, silicate, acrylamide, phenolic, urethane, urea-formaldehyde, epoxy, and polyester. Sixteen general classes of organic and inorganic compounds are also identified as being the types most likely to be found in leachate from a hazardous waste disposal site. The known effects of each chemical class on the setting time and durability of each grout are identified and presented in a matrix. These data were based on a review of the available literature and contact with knowledgeable persons in industries, universities, and government agencies. The physical and chemical properties, reaction theory, and known chemical compatibility of each grout type are discussed.

Since compatibility data are not complete for each grout type, predictions are made where possible for the silicate and organic polymer grouts based on their reaction theory. These results are also presented in a matrix.

To establish the compatibility of chemicals with grouts, a series of laboratory tests should be performed. The two grout properties that must be addressed are permeability of the grouted soil and set time of the grout. No established testing procedures are identified in the literature for determining the effects of chemicals on these grout properties. Fixed-wall and triaxial permeameters, which are used for soil testing, can be used for measuring the effects of chemicals on permeability. No single procedure applies to all grout types for determining set time. Visual observation is the easiest method, though somewhat subjective.

The selection of a grout for a specific waste site depends on its injectability, durability, and strength. These factors relate site hydrology, geochemistry, and geology to grout physical and chemical properties.

This Project Summary was developed by EPA's Municipal Environmental Research Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The purposes of this project were to compile data on the compatibility and durability of grouts in the presence of hazardous wastes and leachates, and to summarize the test procedures available to measure grout durability. This report presents the basic information for selecting grouts based on their compatibility with chemicals; it does not specifically address stability of grouts with respect to ground conditions, other factors that affect durability, or grout-specific properties that ultimately influence the grout selection.

Grouting has been used for years by the construction industry as a technique for consolidating and sealing ground masses. The principal use for grouting has been for large dam and tunneling projects. Although grouting is still very much an art rather than an exact engineering discipline, much has been published on properties, applications, and testing of grouts. Nearly all of this information, however, has focused on the use of grout in construction rather than in remedial work at hazardous waste disposal sites. Adaptations to waste sites could include forming a tie-in between a slurry wall and highly fractured or weathered bedrock, or sealing leaks in aquitards resulting from exploratory bore holes or improperly installed wells.

The grouts used for soil consolidation and groundwater control are emulsions, polymers, and particle suspensions. These materials are generally water-based solutions of sufficiently low viscosity to penetrate rock and soil voids. Particulate grouts composed of cements or clays or both constitute approximately 95% of all grout used. The remaining 5% is primarily silicate grout, though bitumen and organic polymer grouts do have some limited use for water sealing. These proportions, however, may not be applicable to hazardous waste sites.

The testing procedures in current use are not yet standardized and do not deal directly with the grout's ability to set up in and withstand attack from hazardous wastes and leachates. Many of the available data regarding the chemical compatibility of grouts do not specify chemical concentrations, and when they do, they generally are listed as "dilute" or "high-strength," without actual concentrations. Furthermore, chemical compatibility data for chemical grouts often consisted of data for mortar or pipe-sealing applications in which the chemical concentrations would likely be higher than that for soil sealing.

A key aspect of this report is a series of matrices presenting the known and predicted effects of different chemical groups on set time and durability of the various grouts currently in use. The chemical grouts contain most of the compounds found in hazardous wastes and associated leachate. For our purposes, the compounds are all assumed to act independently.

This study centered on collecting or organizing and analyzing existing information on the compatibility and durability of grouts with various classes of chemicals. Where sufficient information was not available, compatibility determinations were based on chemistry and reaction theories of the various grouts and chemical classes.

Compatibility with Wastes

Through a detailed evaluation of available information on the effects of chemicals on grout performance, a series of matrices were developed that summarize and define the compatibilities of grouts with various chemical groups. The information was gathered through a detailed review of the published literature and through contacts with representatives from universities, industries, trade associations, and government agencies.

From the information obtained, matrices were developed to summarize and define the compatibilities of grouts with chemical groups. The matrices provide a step-by-step analysis of the data, moving from general to specific information. Most of the information found detailed the effects of pure chemicals or did not specify concentration. Thus the data are assumed to be related to the effects of undiluted chemicals. While leachates generally contain low levels of compounds, there can be instances where grouts will come into contact with high concentrations of chemicals such as organic solvents.

Based on the information search, six grout categories were chosen for study: bitumen; Portland cement Types I, II, and V; clay (bentonite); clay-cement; silicates; organic polymers (including acrylamide, phenolic, urethane, urea-formaldehyde, epoxy, and polyester).

To simplify the matrix, the chemical universe was divided into 16 basic groups representing the types of compounds found in landfills. The organic categories were chosen by functional groups or structural characteristics. The inorganics were divided into acid, base, and silt categories. Grouping organics by functional groups is useful because although physical properties may differ, the interaction of any functional group with other groups remains essentially the same.

Two general characteristics of grouts are affected by the presence of chemicals: set time and durability. Numeric codes define the effects on set time as follows: 1) no significant effect, 2) increased set time, and 3) decreased set time. Alphabetic codes are used to convey the durability of the set grout in the presence of chemicals: a) no significant effect, b) increased durability, c) decreased durability in the short-term, and d) decreased durability in the long-term. The matrix codes only address changes in set time or durability as a result of exposure to chemicals. The codes do not address specific mechanisms that lead to the changes or mechanisms other than chemical action.

Table 1 presents a detailed matrix in which the chemical groups listed are the 16 mentioned above. The groups have been divided into organic and inorganic categories. The data contained in the matrix were derived from both general and specific information regarding classes of chemicals. Many of the available data refer to the effect of general classes and not specific chemicals. They were derived from available literature and conversations with persons knowledgeable about grouts. Table 1 does not contain any predictions or estimates of chemical/grout interactions.

To fill the data information gaps, a matrix was developed that contains predictions or estimates of grout/chemical interactions (Table 2). The predictions are based on the chemical structure, reaction theory, and estimated behavior of grouts in the presence of the various chemical groups. To make these estimates, the following assumptions were made:

- Typical landfill leachate has a high salt content, approximately 1% organic compounds concentration, less than 1% metal ions concentration, and a pH between 3 and 11.
- Groundwater is static (no turbulence) and is a multicomponent dilute solution in which interactions between components do not occur; interactions may occur between these components and the grout, however.
- Complete reactions between organic polymer grouts and their curing agents do not occur, and other unreacted constituents will remain.

Test Procedures

To establish the compatibility of grouts with the compounds contained in groundwater, a series of tests must be performed. The two properties that must be investigated are permeability of grouted mass and setting time in the contaminated environment. No established procedures exist for these tests, but the following methods have been identified as potential procedures.

To measure permeability and observe the limits established by Darcy's Law, constant-head and variable-head tests have been developed. Three basic categories of testing equipment are: fixed wall, triaxial, and consolidation permeameter cells.

The advantages of constant-head permeameters are the simplicity of data interpretation and reduced confusion resulting from the changing volume of air-filled voids when the soil is not saturated. The major advantage of variable-head testing is that small flows can be measured more easily. Disadvantages are longer times and gas bubbles that may develop in the sample when gas pressure is used to reduce testing time.

Table 1. Interactions Between Grouts and Specific Chemical Groups

Chemical Group	*Bitumen*	*Type I*	*Type II and V*	*Clay (Bentonite)*	*Clay-Cement*	*Silicate*	*Acrylamide*	*Phenolic*	*Urethane*	*Urea-formaldehyde*	*Epoxy*	*Polyester*
		Portland Cement					*Polymers*					
Organic Compounds												
Alcohols and Glycols	?a	?d	?d	?d	?d	?	?d	?	3a	?	?a	?a
Aldehydes and Ketones	?d■	?	?	?d	?	?	?a	3a	?d	?	?	?
Aliphatic and Aromatic Hydrocarbons	?d	2a	2?	?d	?	?	?a	?d‡	?a	2a	?d	?d
Amides and Amines	?	?	?	?	?	?	?	?	3?	?	?	?
Chlorinated Hydrocarbons	?d	2d	2d	?	?	?	?a	?d	?a	2a	?d	?d
Ethers and Epoxides	?	?	?	?	?	?	?a	?	?a	?d	?	?
Heterocyclics	?	?	?	?d	?	?	?a	?	?	?	?	?
Nitriles	?	?	?	?	?	?	?	?	?	?	?	?
Organic Acids and Acid Chlorides	?a	1d	1d	?d	?d	?a	2a	?a	2a	1a	?d	?d
Organometallics	?	?	?	?	?	?	?	?	?	?	?	?
Phenols	?d	1d	?	?d	?d	?	?	2a	?c	?	?	?
Organic Esters	?	?	?	?	?	1a	?	?	?	?	?	?
Inorganic Compounds												
Heavy Metals Salts and Complexes	?d	2c	2a	?d	2c	3?	2?	?	?	?a	?	?
Inorganic Acids	?a§□	1d	1a	?c▶	?c	3a	2c	?a§	2c	1d	?a§	?a☆
Inorganic Bases	?a	1a	1a†	?c▶	?d	2c	3d	?d	?d	2c	?a	?d
Inorganic Salts	?d	2c	2a	2d	?d*	3?	3d	3a#	?d	?a	?a	?a

Grout Type

KEY: Compatibility Index

Effect on Set Time
1 No significant effect
2 Increase in set time (lengthen or prevent from setting)
3 Decrease in set time

Effect on Durability
a No significant effect
b Increase durability
c Decrease durability (destructive action begins within a short time period)
d Decrease durability (destructive action occurs over a long time period)

** Except sulfates, which are ?c*
† Except-KoH and NaOH, which are 1d
‡ Low molecular weight polymers only
§ Non-oxidizing
☆ Non-oxidizing, except HF
□ Except concentrated acids
■ Except aldehydes which are 1a
 # Except bleaches which are 3d
▶ For modified bentonites, ?d
? Data Unavailable

Fixed-wall permeameter cells are the simplest. In this permeameter, the sample is contained in a fixed-wall cylinder supported by a porous disc or screen. To prevent swelling, a plate can be clamped against the sample's upper surface. This apparatus can be used either as a constant- or variable-head system. The advantage of this technique is that the apparatus is readily available and easy to use. The disadvantage is possible leakage between the sample and the permeameter wall. Consolidation permeameter cells are similar to fixed-wall cells except that a load is placed on the top of the sample to create a seal between the sample and the cell walls.

In a triaxial permeameter cell, a cylindrical sample is confined in a rubber membrane and subjected to an external hydrostatic pressure during the permeability test. The advantages of using this type of permeameter are the reduced chances of liquid flow around the sample and the fact that it allows for complete saturation of the sample. The disadvantage is that it has a relatively complex procedure and requires expensive equipment.

Several potential sources of error are associated with all permeability tests: Incomplete saturation of the sample or accumulation of gas bubbles, leakage around the sides of the sample, and changes in temperature.

Grouts to be tested must first be mixed with a material that resembles soil. Sand is usually used for this purpose. Though no current method exists for preparing the grouted sand sample, an ASTM procedure has been proposed.

The moment at which a grout sets can be expressed as a specific point in the evaluation of a property characteristic. Unfortunately, no single property can be used for all grouts. The simplest method for determining set time is the interval of time after which the grout can no longer be transferred from one container to another. A number of devices have been used that will give quantitative measurements by measuring changes in viscosity with time.

To measure the effects of chemicals on setting time, the chemicals should be mixed with the grouts. In the case of pure grouts, the chemicals can be mixed

Table 2. Predicted Grout Compatibilities

	Grout Type						
		Polymers					
Chemical Group	*Silicate*	*Acrylamide*	*Phenolic*	*Urethane*	*Urea-formaldehyde*	*Epoxy*	*Polyester*
Organic Compounds							
Alcohols and Glycols	*1a*	*1—*	*3b*	*—*	*1?*	*1—*	*1—*
Aldehydes and Ketones	*1a*	*?—*	*—*	*1—*	*?*	*1a*	*1a*
Aliphatic and Aromatic Hydrocarbons	*1d*	*1—*	*?—*	*1—*	*—*	*1—*	*1—*
Amides and Amines	*3a*	*3d*	*3b*	*—a*	*1a*	*1a*	*3a*
Chlorinated Hydrocarbons	*1d*	*1—*	*?—*	*1—*	*—*	*1—*	*1—*
Ethers and Epoxides	*1a*	*1—*	*1a*	*1—*	*?—*	*1a*	*1a*
Heterocyclics	*1d*	*1—*	*1a*	*1a*	*1a*	*1a*	*1a*
Nitriles	*1a*	*3?*	*1a*	*1a*	*1a*	*1a*	*1a*
Organic Acids and Acid Chlorides	*1—*	*—*	*3—*	*2—*	*—*	*?—*	*1—*
Organometallics	*1a*	*3a*	*?*	*—*	*1a*	*1a*	*3▶?*
Phenols	*1a*	*1a*	*—*	*2—*	*1a*	*1a*	*1?*
Organic Esters	*—*	*?*	*?*	*?*	*?*	*?*	*1d*
Inorganic Compounds							
Heavy Metal Salts and Complexes	*—a*	*—?*	*?*	*3▶?*	*?—*	*3?*	*3?*
Inorganic Acids	*—*	*—*	*2—*	*—*	*—*	*1—*	*1—*
Inorganic Bases	*—*	*—*	*3—*	*?—*	*—*	*?—*	*1—*
Inorganic Salts	*—d*	*—*	*—*	*?—*	*?—*	*?—*	*3*—*

KEY: Compatibility Index

Effect on Set Time
1 No significant effect
2 Increase in set time (lengthen or prevent from setting)
3 Decrease in set time
Effect on Durability
a No significant effect
b Increase durability
c Decrease durability (destructive action begins within a short time period)
d Decrease durability (destructive action occurs over a long time period)
** If metal salts are accelerators*
▶ If metal is capable of acting as an accelerator
? Determination of compatibility could not be made based on available information
— Data available

directly into the grout. If the grout is injected into sand, the sand can be saturated with the chemical. By comparing the effects of mixing the grout with pure water or the chemical, the chemical effect on set time can be determined.

Grout Selection

The success of a grouting operation depends on the selection of the proper grouting materials for the specific area to be treated. Thus the properties of the grout must be matched with the hydrogeological and geological properties of the area to be grouted. This task can be accomplished with a step-by-step analysis of three basic grout properties: injectability, strength, and durability. In addition to these properties, other factors not directly related to the geological setting (cost and toxicity, for example) must also be considered and may be more important than the properties.

By comparing each property to the conditions present in the geological structure, the proper type of grout can be selected. But the selection of a specific formulation for field application requires the assistance of experts in the grouting field.

The injectability of a grout is controlled either by its viscosity or particle size. This property will dictate the grout's ability to penetrate a soil/rock structure. The lower the viscosity, the finer the voids that can be penetrated. Also, the smaller the particle size in suspension grouts, the smaller the voids that can be penetrated.

Grouts with a viscosity of less than 2 centipoise (cP), such as many of the chemical grouts, can penetrate strata with permeabilities of less than 10^{-5} cm/sec. Higher-viscosity grouts, like particulate and some chemical grouts with a viscosity greater than 10 cP, can only penetrate coarse strata with permeabilities greater than 10^{-2} cm/sec.

For suspension grouts, the particle size also influences the ability to penetrate voids. A general rule of thumb sometimes used for determining a grout's penetration ability equates grain size of the particles within the grout to soil particles within the stratum. This relationship is:

$$\frac{D_{15}}{D_{85}}$$

where: D_{15} = diameter of grains in the stratum where 15% of the soil mass is finer

D_{85} = diameter of particles within in the grout where 85% of the particles are finer

This ratio should be at least 19 and preferably greater than 24 to ensure adequate penetration of grout into soil voids.

Once a grout has set in the voids in the ground, it must be able to resist hydrostatic forces in the pores that would tend to displace it. This ability will depend on the mechanical strength of the grout and can be estimated by the grout's shear strength.

The shear strength of a grout will depend not only on its class, but also on its formulation. Thus a class of grouts such as silicates can possess a wide range of mechanical strengths depending on the concentration and type of chemicals used in its formulation. The strength of the gel, then, can be adjusted within limits to the specific situation.

For permanent control of groundwater or leachate movement, the grout must not deteriorate because of the influence of the soil or groundwater chemistry. Thus in the selection process, the short- and long-term durability of the grout must be evaluated.

Deterioration of a grouted area over time can occur through several physical/chemical mechanisms. The grout can be dissolved or structurally changed by water or chemical action. Also, removal of water from the grout through desiccation or syneresis can shrink it. These factors can weaken a grout, leading to increased permeability.

Short-term deterioration of the grout can be caused by rapid chemical degradation or by an incorrect setting time. The effect on setting time can be caused by a miscalculation of the grout formulation, dilution of the grout by groundwater, or changes caused by chemicals contained within the grouted strata.

The effect that groundwater will have on grout stability depends on the class and formulation of the material. For areas that have large groundwater flow rates, the grout must be able to quickly set before it is diluted or washed away. The set time is often a controllable parameter. Water can also redissolve some of the grout constituents because of the reversibility of many of the polymerization or gelation reactions.

The actual durability of a grout in a specific geological setting should be determined from laboratory testing. Grout selection could also be based on the results of field applications in similar geologic settings.

Another grout selection factor that might be considered is the toxicity of the solidified grout and its components. This factor will be important if the aquifer with which the grout comes into contact is a potential drinking water source. The oral toxicity of most of the compounds used in grouts have been determined, as have many of the values for the set grout. The specific grout application and the amount of unreacted material must also be considered.

The cost of the grouting operation is also a selection factor. Material costs and injection costs should both be considered. The expense of chemical grouts is offset to some degree by the fact that particulate grouts may be three to five times more costly to pump into the ground.

Conclusions

Little actual chemical compatibility testing has been performed on grouts. Many of the data presented here have been taken from related uses of similar materials and not specifically from the testing of grouts in contaminated soils. Significant data were collected, for example, from research in waste solidification and encapsulation techniques. Few documented cases exist of the use of grouts and grouting technology for hazardous waste site remediation.

Laboratory testing of grout/chemical compatibility centers on evaluating two general factors: the effect of chemicals on grout setting time and how long the grout will remain effective after prolonged exposure to the chemicals. Protocols for conducting these particular tests have yet to be developed.

Selection of a grout for a particular purpose depends primarily on site characteristics and the material's injectability, strength, permeability, and durability. In addition, costs and toxicities of grouting materials are major factors.

Suspension grouts (cement, clay, and cement/clay) are the most common, accounting for approximately 95 % of all grout used. Silicate grouts are the most commonly used chemical grouts, followed by acrylamides and urethanes. Other minor grout types are used in less than 1% of grout applications.

Recommendations

During the course of this study, several areas were identified that lacked available information but are important in determining the usefulness of grouts at hazardous waste disposal sites. Areas recommended for further research include:

- Grout specifications and applications
- Compatibility of grouts with chemicals
- Long-term stability of grout
- Compatibility testing procedures.

Very few data were found on the formulation of currently used or potentially usable grouts and their specific areas of application. Information on the chemical make-up and specific area of application of each type of grout must be known to select the grouts and testing procedures to be included in a laboratory evaluation program. The specific areas that should be investigated include:

- Areas of potential and actual grout application at waste disposal sites
- Information on actual grout formulations currently used

Very limited information exists on the effects of chemicals on grouts used at disposal sites. Moreover, most compatibility information deals with the effects of high concentrations of simple chemicals. To evaluate the effects of leachates on grouts, information on grout compatibility with low chemical concentrations and chemical mixtures must be known. Thus areas for further research including a pilot-scale program are:

- Effects of dilute chemicals on grouts
- Effects of chemical mixtures on grouts
- Effects of actual leachates on grouts

Information is needed on the ability of grout to withstand leachate, water, hydrostatic pressure, and biodegradation once it is in the ground. Such data are very limited in the literature and are essential to obtaining a permanent seal. Thus the environmental effects on the structural integrity of grouts should be further researched in both a laboratory and pilot-scale program.

Currently no established compatibility test procedures exist for grouts, but the potential exists for using the same types of test procedures developed for evaluating soils, bentonite slurries, and cement. Permeability measurement techniques have been developed in all of these areas, and they are directly applicable to grout compatibility evaluations. Setting time measurements, on the other hand, are a little more difficult to apply because of the varying nature of the set grouts. For both types of measurements, different laboratory techniques must be used for testing the different grout types because of variation in physical and chemical properties. Further research areas include:

- Evaluation and selection of test procedures for permeability
- Evaluation and selection of set time test procedures

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The complete report, entitled "Compatibility of Grouts with Hazardous Wastes," (Order No. PB 84-139 732; Cost: $16.00, subject to change) will be available only from:
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