United States Environmental Protection Agency Hazardous Waste Engineering Research Laboratory Cincinnati OH 45268

Research and Development

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Project Summary

Precision and Reliability of Laboratory Permeability Measurements

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In this study, a limited set of laboratory test data on clay liner permeabilities was gathered to create a data bank suitable for a preliminary statistical analysis. The methods used to collect, organize and analyze the data are brifely described.

In the main part of the study, the findings from the data analysis are presented. In the analysis, consideration was given to the degree of variability found in replicated permeability tests, the question of sample equilibration with water and chemicals, and the effect of the magnitude of gradient on permeability. Permeability test results using flexible-wall and fixed-wall permeameters were also compared. Consideration was also given to the effects of parameters of sample preparation, such as the molding water content, which may be strongly related to permeability, and the amount of variability of permeabilities that may be found in location-to-location sampling from within a source of liner material. In addition, the statistical aspects of the design and analysis of permeability experiments were discussed and alternate test protocols were suggested.

This Project Summary was developed by EPA's Hazardous Waste Engineering 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

Clay liners and clay caps are important components of hazardous waste disposal facilities. In engineering practice the permeability of proposed clay liner material is customarily determined in geotechnical engineering laboratories, usually using water as the permeant. A few years ago, (1981) however, it became evident that certain chemical leachates have pronounced effects on clay liner permeabilities. Since then, the Land Pollution Control Division, Containment Branch, of the Hazardous Waste Engineering Research Laboratory of the U.S. EPA has sponsored extensive research on the permeabilities of clay liner materials with hazardous chemicals and leachates.

The investigations revealed that certain chemicals may indeed have dramatic effects on compacted clays. However, considerable variability was observed in the equilibrium permeabilities from the various laboratories and individual researchers. There may be several reasons for this. Some investigators used rigid-wall permeameters, while others used flexible-wall permeameters. Further sources of variability may have been the varying percentages of molding water contents, the individual testing techniques used, the methods of deairing the samples and the degree of water-saturation achieved before the introduction of chemicals, the type and concentration of chemicals used, and the various driving gradients applied to the test specimens.

Having access to a considerable quantity of accumulated data on clay liner permeabilities, the Hazardous Waste Engineering Research Laboratory was interested in establishing the overall reliability of the laboratory measurements of clay liner permeabilities and their variability. For this pur-

pose, the present study was undertaken with the following objectives:

- To collect available historic data on permeability measurements from both public and private sources and to identify the test methods and parameters used.
- b. To analyze the collected data in order to determine the degree of variability likely to be found among the results of permeability tests and to identify sources of errors in the testing procedures and estimate their significance.
- c. To make recommendations to enhance the applicability of statistical methods to the analysis of future clay liner permeability tests.

Methods and Procedures Data Collection and Organization

Permeability data were collected from laboratories that were active either in research or design of clay liners. They were selected based on established contacts, past and on-going research, data availability and publications. These included three EPA contract sources, one government laboratory, and two private consulting firms.

The typical permeability data received contained information on the clays, testing methods, types of permeameters, types of chemicals, etc. used, and a tabulation of permeability versus flow data for each test. The data were organized by filling out a "Permeability Test Specifications Sheet" and a "Clay Type Sheet" for each test and clay, respectively, and attaching them to the corresponding permeability test data sheets.

Data Base Limitations

It was known at the initiation of the project that the data base resulting from the data collection effort would be limited in certain respects:

- a. Data sources were identified based on the knowledge that they possessed relevant and available data on permeability testing. The selected sources were in no sense chosen randomly from any pool of available sources.
- For many of the tests obtained from private sources, concomitant information concerning the preparation of specimens and the physical characteristics of the clay soils was not as complete as would be desired.

- Also, there was virtually no replication of tests from within the same samples, and all private source tests used only water as a permeant.
- c. The data received from EPA contract sources was highly project specific in the sense that researchers in general used different types of equipment and test methods, investigated different claychemical combinations, and in some cases, no replications of tests on specific clay-permeant combinations were conducted.
- d. Criteria for test termination varied considerably, sometimes making the assignment of a terminal permeability rather subjective. Often, tests were terminated prior to the point in time where equilibrium was clearly established. This led to extreme complications in attempting to reasonably quantify the degree of variation between tests.
- e. As a general rule, precise information on the level at which tests were replicated was not available. In most cases it was not known if replicate specimens were prepared from a single moisturized batch of clay soil, or from separately moisturized batches. In no case were tests systematically replicated at both the within and between-batch levels, as would be required for a rigorous analysis of the components of variance in test results to be attributed to the operations of moisturization, compaction and testing.
- f. In general, there appeared to be little conscious effort to randomize experimentation with regard to either the assignment of experimental material to treatments or time sequence of testing.

Statistical Analysis Methods

All computations performed in the analyses described in the report were performed at the University of Cincinnati, Cincinnati, Ohio on either an Amdahl 470 V/7A or an IBM 3081D computer. Data transformations and summarizations were accomplished by use of SAS arithmetic capabilities, built-in functions or data summarization procedures. All statistical analyses performed or discussed in the report are available through SAS procedures or in the BMDP statistical software computer programs, with the exception of those

proposed for the confirmation of equilibrium of permeability tests.

Data Analysis Results and Conclusions

In the main part of the study, analyses of the variability among the results of laboratory permeability tests were made and the factors which appeared to contribute to that variability were investigated. The highlights of the results and the most important conclusions reached are:

- a. Given that the permeability test samples have achieved equilibrium, the variability of permeability test results using standard aqueous leachate, standardized compaction, and samples prepared from a uniformly moisturized batch of homogeneous clay soil appears to be reasonably small relative to other sources of error. The variability of tests using chemicals as permeants differed substantially, with some claychemical combinations yielding variability characteristics comparable to water permeation, while others resulted in extreme inconsistency.
- b. Certain series of tests in the collected data indicated that, even with water as the permeant liquid, reaching liquid flow equilibrium may be somewhat more difficult to establish than is commonly believed. Furthermore, the error in a permeability determination which may be caused by premature ter mination may not be small in com parison to the variability of the ini tial permeabilities of replicated tests. This implies that the length of testing time commonly found ir private sector tests in the collected data may be insufficient. Because the measured permeability of a test often undergoes a prolonged period of transitory behavior be fore leveling off at an equilibrium value, deciding when a test has ex hibited stable permeability long enough to conclude that equi librium has been achieved is no straightforward.
- c. The permeability of a soil samplis extremely sensitive to the parameters of its preparation such as the molding water content which it is compacted. This appears to be true not only for water as permeant but for chemical permeant but for chemical permeant.

- meants as well. Minor variations in these parameters can cause changes in permeability which may be large relative to the accuracy of the test itself.
- d. Several series of tests on which data have been provided appear to indicate that the gradient at which tests are run may have an effect on the resulting permeabilities in both rigid-wall and flexible-wall tests, the magnitude of which in some cases appears to be practically significant. Such a phenomenon could be caused by factors whose effects are opposite in nature. That is, increased gradient could cause sample consolidation in flexible-wall permeameters leading to decreased permeability. which appears to have occurred in several series of tests discussed in this study. In tests of chemical permeants in rigid-wall permeameters, on the other hand, there are data that suggest that higher gradients may promote channel formation and increased permeabil-
- e. Other variations in testing methods may be expected to affect test results as well. In one series of tests on a bentonite clay, using xylene as the permeant and rigidwall permeameters, the initial permeabilities in nonsaturated tests were four orders of magnitude greater than those found in corresponding tests which were presaturated by permeation of .01N CaSO₄ prior to addition of the xylene. Even after two or more pore volumes of flow, the permeabilities of the presaturated tests had not reached the level of the nonsaturated tests.
- f. There were almost no within-lab comparisons of flexible-wall and rigid-wall permeability tests with concentrated chemical permeants in the data sets. The data that were available, together with between-lab comparisons under admittedly dissimilar conditions, suggest that often quantitatively different results may be obtained by the two methods. They also suggest that flexible-wall test behavior may be somewhat less erratic in testing certain chemicals.
- g. It is important to assess the degree of uncertainty of permeability determinations due to the nature of

the test itself. It is equally important to estimate the variability of permeabilities that might occur in location-to-location sampling from within a proposed source of liner material in order to judge the amount of sampling required to ascertain the acceptability of the soil within the source. Limited data from the private sector suggest that permeabilities of samples taken from various positions within a site exhibit far more variability than do the replicate tests on uniform clays observed in the research setting, even when the samples are relatively homogeneous with respect to Unified Soil Classification, Atterberg limits and grain size information. Although it seems reasonable to ascribe this variability in large part to locationto-location heterogeneity of the soil, for a number of reasons discussed in the study this factor cannot be completely separated in the data from variability which has been caused by differences in sample preparation and testing.

Experimental Plans for Permeability Studies and Statistical Methods for the Analysis of Data

A number of experimental plans were discussed in the study which may be of use in proposed laboratory research studies designed to compare the effect of various factors on permeability tests. It is not suggested that a single protocol is appropriate for all studies which might be proposed, since the best approach in a particular situation is specific to the research objectives of interest, and also is a function of the degree to which the experimenter is capable of controlling such extraneous factors as soil heterogeneity, molding water content and compactive effort. The study describes the basic features of the potentially useful plans to compare their relative strengths and weaknesses, and to illustrate the concepts of randomization, replication and blocking. The features of completely randomized design, randomized complete block design, factorial experiments, randomized incomplete block design and splitplot designs were discussed. In addition, the construction of analysis of variance tables for the various experimental plans was discussed. Also, methods for the determination of adequate replication of experiments were given, and diagnostic checks of analysis assumptions were outlined.

Generally, the information presented is directed toward the non-statistician who is involved in the development of experimental plans. However, it was suggested that an experienced statistician should be consulted as required, both during the experimental planning phase of the project and during the statistical analysis of the data.

Permeability Tests of Proposed Borrow Material

In this section of the study, general considerations concerning the design of a testing program for the evaluation of borrow material, which is under consideration for use in the construction of a liner, were discussed. In such a case the major goal of the investigation is the estimation of the permeability of the soil within the proposed site in order to determine whether it will meet specified performance criteria.

One consideration is that the soil contained within the proposed site may exhibit a good deal of heterogeneity in its chemical and physical properties. This may lead to considerable variation in permeability so that an estimate of the average permeability throughout the site may not be very relevant in judging its overall acceptability. That is, a source whose average permeability might be considered to be acceptably small for use as liner material might nevertheless contain a significant amount of material having substandard permeability characteristics. For this reason, it is suggested that the site be partitioned into smaller sub-sites, which will be referred to as "cells", in such a way that the characteristics of the soil found within any one cell are reasonably uniform. This could be done based on a preliminary determination of characteristics such as Atterberg limits, grain size distribution and water content-density relationships. Such cells should not be made up of noncontiguous parts, should include a single soil layer, and at a minimum should contain soils having the same Unified Soil Classification.

Once the site has been stratified into cells, replicated permeability tests can be made on samples drawn from different locations within each cell. This will allow an estimation of the permeability of the soil within each cell, which may be used to determine which, if any, of the cells are not suitable for use as liner material.

Methods to Confirm Equilibrium of Research Permeability Tests

Because the measured permeability of a test often undergoes a prolonged period of transitory behavior before leveling off at an equilibrium value, deciding when the test has exhibited stable permeability long enough to conclude that equilibrium has been achieved is not straightforward. Development of objective criteria, which may be useful in confirming the experimenter's judgment in this decision, is a topic which deserves considerable attention in future permeability research. In this section of the study, two approaches were described which are currently under investigation. The first is an adaptation of a test for trend originally proposed by Mann, which has been heuristically modified for use as a test termination rule. The second approach is based on a Bayesian analysis in which the posterior probability that equilibrium has been achieved is updated after each new observation, until this posterior probability first falls below a specified bound.

In the authors' opinion, the proposed methods represent reasonable approaches to the test equilibrium problem which are deserving of additional consideration and empirical testing. The methods described will probably be of practical value only in situations where permeability tests approach their equilibrium levels in a relatively continuous manner. They are not intended to be able to predict the behavior of tests which are subject to sudden breakthroughs leading to episodic increases in permeability.

Recommendations

Based on the analysis results and conclusions reached, the following recommendations are made:

- a. In comparative tests on chemical permeants, the use of identically prepared and concurrently run water control tests is recommended in order to reduce ambiguity in distinguishing between the effects of equilibration and chemical.
- b. In some situations in which chemical permeants have strong effects on the clay samples and lead to crack formation and channeling, which in turn lead to massive increases in conductivity, it may be that the concept of specimen equilibration is less useful than meas-

- ures based on the notion of time to failure. If this is the case, then there is a need for the development of practically meaningful failure criteria for use in research studies.
- c. The permeability of some soil samples is very sensitive to the conditions of their preparation, such as molding water content and compaction effort. Therefore, methods of statistical experimental design, such as randomization and blocking, should be applied in comparative permeability research experiments to control the effects of the above factors.
- d. The sensitivity of permeability to factors such as molding water content and compaction effort also indicates a need for research which would estimate the bounds within which these factors can be maintained during the actual construction of a liner. If these factors vary substantially during liner construction, the results of tests performed only at their target values may give little indication of the future performance of the liner.
- e. It was observed in the limited data from the private sector that permeabilities of samples from various locations within a site exhibit far more variability than do replicate tests on uniform clays in the research laboratory. Although it is reasonable to attribute most of this variability to the location-to-location heterogeneity of the soil deposit, this cannot be completely separated from the variability caused by sample preparation and testing. Additional research in this area is indicated.
- f. Since deciding when a permeability test has achieved equilibrium is not straightforward, the development of objective criteria, which may be useful in confirming the experimenter's judgment in this decision, is a topic which deserves considerable attention in future permeability research. Two approaches under consideration as decribed in the study, need further investigation.

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Jonathan G. Herrmann is the EPA Project Officer (see below).
The complete report, entitled "Precision and Reliability of Laboratory Permeability Measurements," (Order No. PB 87-113 791/AS; Cost: \$18.95, subject to change) will be available only from:

National Technical Information Service 5285 Port Royal Road Springfield, VA 22161 Telephone: 703-487-4650

The EPA Project Officer can be contacted at:

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