HIGHWAY RESEARCH RECORD

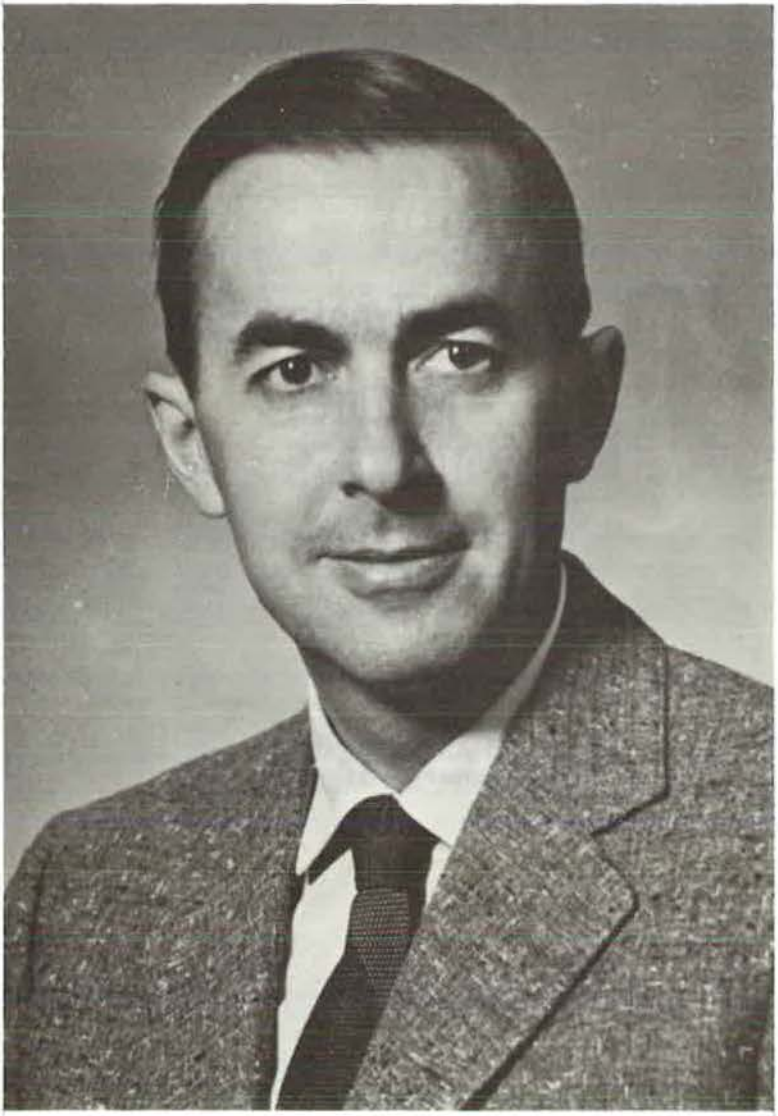
Number 36

Cement-Treated Soil Mixtures

10 Report

Presented at the
nd ANNUAL MEETI
January 7-11, 1963

HIGHWAY RESEARCH BOARD
of the
Division of Engineering and Industrial Research
National Academy of Sciences—
National Research Council
Washington, D. C.
1963



Dedication

The field of soil stabilization lost one of its ablest leaders when Donald Thomas Davidson died in October 1962. He had a wide interest in soil technology, and for five years was Chairman of the Highway Research Board Committee on Soil-Portland Cement Stabilization, a committee which he helped organize and headed until his death. His ability and intense interest in soil stabilization was an inspiration to many of his students and associates.

Mr. Davidson was born January 15, 1919, in Concord, New Hampshire. He received the degree of Bachelor of Science in Civil Engineering from the University of New Hampshire, and the degrees of Master of Science and Doctor of Philosophy in Soil Engineering from Iowa State University. He remained at Iowa State, where he was successively Instructor, Assistant Professor, Associate Professor, and Professor of Civil Engineering. At the time of his death, he was in charge of the teaching and research programs in Soil Engineering.

Mr. Davidson was very active in the general field of soil stabilization research and was author or co-author of more then 70 papers, bulletins, and journal articles for a wide range of technical publications. The scope of his work ranged from field and laboratory studies of materials from many different states to development of testing equipment and stabilization construction techniques. He was granted several patents on pieces of apparatus. He also served industry as special consultant on foundation engineering and soil stabilization.

Mr. Davidson was active in a number of technical societies, including the American Society of Civil Engineers, American Society for Testing and Materials, American Society for Engineering Education, Iowa Academy of Science, and Iowa Geological Society, in addition to the Highway Research Board. He was also a member of several honorary organizations, including Phi Kappa Phi and the Society of the Sigma Xi.

This publication is affectionately dedicated to Donald Thomas Davidson, who gave so much of himself to soil stabilization research.

HRB Committee on
Soil-Portland Cement Stabilization

Department of Soils, Geology and Foundations

Eldon J. Yoder, Chairman
Joint Highway Research Project, Purdue University
Lafayette, Indiana

COMMITTEE ON SOIL-PORTLAND CEMENT STABILIZATION

James M. Hoover, Chairman
Assistant Professor, Civil Engineering Department
Engineering Experiment Station Laboratory
Iowa State University, Ames

R. D. Bauman, Materials Engineer, Utah State Department of Highways, Salt Lake City
William A. Cordon, Department of Civil and Irrigation Engineering, Utah State University, Logan
George R. Kozan, Chief, Soils Stabilization Section, U. S. Corps of Engineers, Waterways Experiment Station, Vicksburg, Mississippi
D. J. Maclean, Head of Soils Section, Road Research Laboratory, Department of Scientific and Industrial Research, Harmondsworth, Middlesex, England
William H. Mills, Consulting Engineer, Atlanta, Georgia
C. E. Minor, Materials and Research Engineer, Washington Department of Highways, Highways-Licenses Building, Olympia
Radnor J. Paquette, Georgia Institute of Technology, Atlanta
J. F. Redus, Michael Baker, Jr., Inc., Consulting Engineers, Jackson, Mississippi
E. G. Robbins, Portland Cement Association, Chicago, Illinois

Contents

Standard Laboratory Tests for Soil-Cement—Development, Purpose And History of Use

L. T. NORLING, Senior Soil-Cement Engineer, Portland Cement Association

One of the reasons for the good service record of soil-cement has been that standardized laboratory tests have been available for determining control factors for construction. These test methods have been used with consistent success for a wide range of soils, climatic and subgrade conditions.

This opening paper of the symposium on soil-cement mix design reviews the philosophy of the standard tests, how they were developed and the refinements that have been made during their 27 years of use.

Valuable tools that can be used to reduce laboratory test work to a minimum such as the Department of Agriculture pedological soil identification system and the short-cut test method for granular soils are also reviewed.

The paper also describes recent basic research that will aid in defining more clearly the fundamental reactions that take place when soil, cement and water are mixed together and which may permit a more rational approach to mix design.

• DURING the past 27 years the use of soil-cement has grown from the first 20,000 sq yd scientifically-controlled job built near Johnsonville, S. C., in 1935, to an annual use in the United States of over 80,000,000 sq yd. It has also been used in over 30 other countries.

One of the reasons for the acceptance and the outstanding service record of soil-cement is that standardized laboratory tests have been available to determine the control factors for construction. These tests have been used with consistent success for a wide range of soils, climatic and subgrade conditions.

Soil-cement in the following discussion is defined as a tightly compacted mixture of pulverized soil, portland cement, and water which, as the cement hydrates, forms a hard, durable, structural material. The required cement content is determined by standard wet-dry and freeze-thaw tests with appropriate criteria or by methods correlated with the standard tests.

This paper reviews the philosophy of the standard tests, how they were developed, and the refinements that have been made during their 27 years of use. The paper also describes other test methods that have been developed by correlation with the standard tests. Also included is a description of the research that is under way to further improve the standard test methods and to permit a more rational approach to laboratory methods of proportioning materials.

STANDARD TESTS

During the 1920's and early 1930's (1, 2) several state highway departments experimented with mixtures of soil and cement for paving. Although results of these experiments were unpredictable they were encouraging and stimulated an extensive research

---

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

program by the Portland Cement Association in 1935. This research (3, 4, 5, 6) resulted in the development of dependable and predictable test methods that can be used to determine the factors needed for quality soil-cement. These tests[1] (the moisture-density, wet-dry and freeze-thaw) have since been adopted as standard by the AASHO, ASTM and ASA.

Development of Standard Tests

One of the first significant findings of the 1935 research was that the moisture-density relationship for soils as discovered in 1929 by Proctor was also valid for mixtures of cement and soil when compacted immediately after mixing and before cement hydration. It was found that optimum moisture content as determined by the moisture-density test not only produced the highest density for a particular compactive effort but also provided sufficient water for cement hydration and produced maximum strength. Thus the moisture-density test for soil-cement was developed at the same time the test was being developed for soil compaction. The soil-cement moisture-density test was adopted as a tentative standard by ASTM (D558) in 1940 and advanced to standard in 1944. It was adopted as standard by AASHO (T134) in 1945. The procedure was refined by both organizations in 1957 to include the use of plus No. 4 material in the sample.

The next step in the 1935 research program was to devise methods of measuring the effect that various cement contents, moisture contents and densities have on the physical properties of compacted soil-cement mixtures. Since the rate and amount of cement hydration would influence final results materially, specimens were permitted to remain undisturbed for 7 days in an atmosphere of high humidity before being tested. This permitted hydration of a significant portion of the cement.

In analyzing possible test methods that might be used for evaluating compacted and hydrated soil-cement mixtures, tests used in soil and concrete testing were analyzed. Consideration was given to various compression and tension tests that might be modified to simulate the internal forces of expansion and contraction produced by moisture changes in soil. It was considered that these tests were not applicable since they do not simulate the nature and magnitude of the desired forces. However, it was found that repeated wetting and drying and repeated freezing and thawing could induce internal forces similar to those induced by changes in moisture content.

Thus, the wet-dry and the freeze-thaw tests were evolved to reproduce in the laboratory the phenomenon of moisture changes in soils. The wet-dry test was designed primarily to simulate shrinkage forces. It was recognized that temperatures occurring in service would be below the drying temperature used in the test and that this relatively high temperature would accelerate cement hydration.

The freeze-thaw test was designed to simulate internal expansive forces produced by moisture change in the fine-grained soils. Also, this test avoided the accelerated cement hydration present in the wet-dry test.

Since moisture plays a predominant role in the strength of soils and road bases it was essential that water be permitted to play a predominant part in both the wet-dry and freeze-thaw tests. In the wet-dry test this is accomplished by submerging the specimens in water during the wetting portion of each cycle. In the freeze-thaw test, specimens are permitted to absorb water by capillarity during the thawing portion of each cycle.

The number of cycles of testing and their duration was evolved by exploratory tests on freezing temperatures, freezing time, drying temperatures, drying time, and soaking time. Twelve cycles for each test produced interpretable data and also met the requirements of a practical time limit.

Early in the development of the tests, volume, moisture, and weight measurements were made on the specimens at each test cycle to judge their condition. The weight

---

[1] Methods of Test for Moisture-Density Relations of Soil-Cement Mixtures, ASTM D558, AASHO T134, ASA 37.50. Methods of Wetting and Drying Test of Compacted Soil-Cement Mixtures, ASTM D559, AASHO T135, ASA 37.51. Methods of Freezing and Thawing Test of Compacted Soil-Cement Mixtures, ASTM D560, AASHO T136, ASA 37.52.

losses during the tests were erratic, particularly for specimens containing low cement contents. This problem was solved by developing a brushing procedure which removes the loosened material. This resulted in more consistent and reproducible results.

As part of the research program, data on soil gradation, surface area, physical test constants, compressive strength, organic content, pH, density, and cement-void ratio were correlated with cement requirement in a search for relationships that could be used to determine cement contents for construction (7). These studies, which gave erratic results, showed that physical test data alone do not reveal the factors governing the reaction of soil and cement and that surface chemistry of the soil plays a predominant role. The studies also showed the value of the wet-dry and freeze-thaw tests in evaluating the surface chemistry effects.

Thus the wet-dry and freeze-thaw tests were developed to determine the minimum cement content required to produce a structural material that will resist volume changes produced by changes in moisture. Although the primary intention was to measure the effects of moisture change, the tests also measure the effects of temperature change. Since moisture and temperature changes occur in varying degrees in all climates and geographic areas, use of both the wet-dry and freeze-thaw tests assure that a hardened, structural material is produced for any area. The durability of soil-cement mixtures designed using the standard tests and appropriate criteria for determining a minimum cement content to produce a structural material has been proved by the field service record.

The wet-dry and freeze-thaw tests were adopted as tentative standards by ASTM (D559 and D560) in 1940 and advanced to standard in 1944. The tests were adopted as standards by AASHO (T135 and T136) in 1945. Some refinements in the procedures were made in 1957.

Development of Test Criteria

Investigations were then undertaken to determine criteria that could be used with the wet-dry and freeze-thaw tests to determine cement contents required to produce a paving material that would have adequate durability and long life. Studies of laboratory test data, outdoor exposure of laboratory specimens, and studies of field performance were used in selecting the criteria.

The selected criteria[2] included requirements of volume change, maximum moisture content, soil-cement loss, and trend of compressive strength. The criterion of maximum volume increase of the specimens (not more than 2 percent) was chosen as an indication that the cement is holding the mass intact and preventing volume increases that would otherwise take place. The criterion of maximum moisture content (not more than that required to fill the voids) was selected as a further indication of resistance to disruptive volume changes. The criterion of maximum soil-cement loss from brushing was used as an indication that the forces of expansion and shrinkage resulting from the wetting and drying and freezing and thawing, that will disrupt and disintegrate soil specimens, had been resisted. The compressive strength criterion was used because increases in strength due to increases in time and cement content are evidence that the cement is functioning normally and that the soil is not interfering with hydration of the cement.

The validity of these criteria has been verified by the quarter-century of successful field performance of soil-cement projects in service.

---

[2] 1. Soil-cement losses during 12 cycles of either the wet-dry or freeze-thaw test shall conform to the following limits: Soil groups A-1, A-2-4, A-2-5 and A-3, not over 14 percent. Soil groups A-2-6, A-2-7, A-4 and A-5 not over 10 percent. Soil groups A-6 and A-7 not over 7 percent.
2. Maximum volume at any time during either test shall not exceed the volume at time of molding by more than 2 percent.
3. Maximum moisture content at any time during either test shall not exceed that quantity which will completely fill the voids of the specimen at time of molding.
4. Compressive strengths of soil-cement specimens soaked in water 1 to 4 hours prior to test shall increase both with age and with cement content at and above the cement content which produced results meeting requirements 1, 2 and 3.

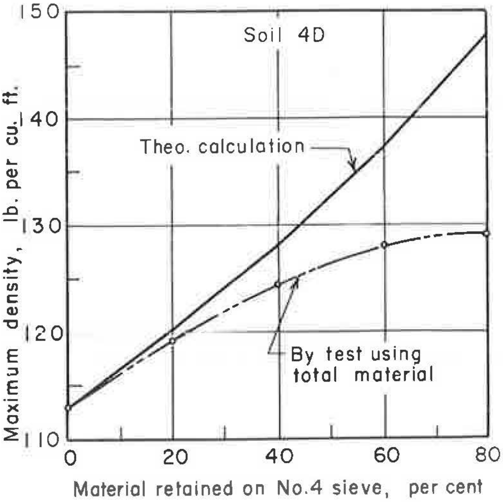


Figure 1. Effect of material retained on the No. 4 sieve on the maximum density, soil No. 4D.

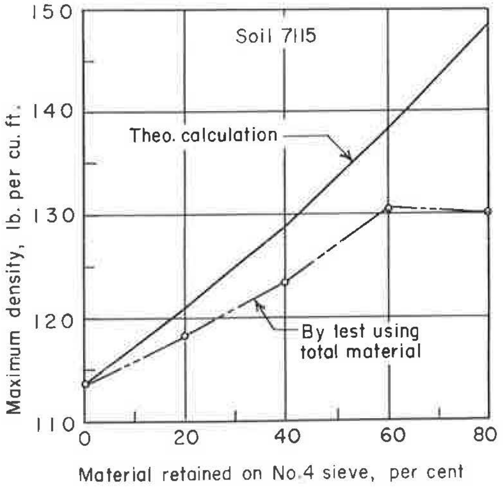


Figure 2. Effect of material retained on the No. 4 sieve on the maximum density, soil No. 7115.

As previously mentioned, the test methods were adopted as tentative standards in 1940 and advanced to standard in 1944 and 1945. As would be expected, some refinements in laboratory equipment and techniques were made through the years by various agencies engaged in soil-cement testing. When changes in the test methods were made by ASTM and AASHO in 1957, these refinements were included. This resulted in test methods which required less time, manpower and materials to perform.

Refinements of the Standard Tests

One of the refinements made in 1957 was the method of analyzing the plus No. 4 material. In previous practice the optimum moisture and maximum density of the total mixture was computed from data obtained on the minus No. 4 fraction by taking into account the amount, specific gravity, and absorption of the plus No. 4 material. The computations were made on the assumption that the addition of the plus No. 4 material increases the density of the mixture by displacing the minus No. 4 soil in equal volume. No allowance was made for possible increase in void space. As a result, the densities obtained in preparing laboratory test specimens that contain the plus No. 4 material were often considerably less than the theoretical calculated maximum density (8). The difference between the theoretical calculated density and the actual density obtained when using total material increases as the amount of plus No. 4 increases (Figs. 1 and 2).

The changes made in 1957, which specified the inclusion of the plus No. 4 material in the sample used for the moisture-density test, resulted in a procedure that gives maximum densities more readily reproduced in laboratory test specimens and in a mixture more representative of the total materials used in construction.

Changes in 1957 of the wet-dry and freeze-thaw tests permitted a reduction in the number of test specimens required and in the amount of data collected for each cycle of test when routine testing is involved. The more detailed procedures are used only for research investigations.

Thus the 1957 procedures incorporated for routine use the information and experience gained during 13 years of successful use of the tests. They reduced the time, manpower, and material required to perform the tests. The basic methods and philosophies of the tests, however, remained the same.

CORRELATED SHORT-CUT TEST METHODS

It is a natural desire of the testing engineer to reduce his work load of routine testing as much as possible. As a result, studies have been made of various methods of reducing this work. These studies have produced helpful tools for the testing engineer. The two methods described here have been correlated with the standard wet-dry and freeze-thaw tests. Both make use of optimum moisture and maximum density as determined by the standard moisture-density test.

Tests on Soils Identified by Soil Series

A most helpful tool for reducing soil-cement test work is the Department of Agriculture Soil Identification System (9, 10). In this system, soils are subdivided into groups called soil series. Soils of the same series have similar characteristics of subsoil (B horizon), parent material (C horizon), climate, age, vegetation, and relief. The importance of the use of soil series in soil-cement work is that soils of the same series, horizon, and texture require the same amount of cement for adequate hardening (11, 12, 13). Once the cement requirement of a given soil series and horizon has been determined by laboratory tests, only the identification of the particular soil series and horizon and a minimum of exploratory check tests are required. Thus, the need for conducting complete soil-cement tests can be sharply reduced or eliminated in those areas where detail soil survey maps are available.

Soil surveys have been made over a large portion of the United States and maps have been prepared by the Department of Agriculture. The Highway Research Board reports periodically on the status of this and other mapping through publications sponsored by the Committee on Surveying, Mapping and Classification (14). An increasing number of engineers are making use of this soil identification system to reduce their soil-cement testing work.

Short-Cut Test Method for Granular Soils

Another helpful tool for the testing engineer is the short-cut test method developed in 1952 for use with granular soils (8, 15, 16). The short-cut procedure is based on correlations of data obtained using the standard wet-dry and freeze-thaw tests. No new tests or laboratory equipment are required. Instead, data and charts developed from previous tests of similar soils are used. The only laboratory tests required are a grain size analysis, a moisture-density test, and compressive strength tests. Relatively small soil samples are needed and all tests can be completed in one day, except for results of 7-day compressive strength tests.

Although the short-cut test method does not always indicate the minimum cement factor that can be used, it provides a safe cement factor generally close to that indicated by standard ASTM-AASHO wet-dry and freeze-thaw tests. This procedure is finding wide application by engineers and builders.

OTHER TEST METHODS

As would be expected, a few agencies have modified the standard soil-cement test methods or have developed other procedures for routine testing. These modifications have been based on experience with soil-cement for particular local soil materials and climatic conditions. Some modifications have attempted to consider climatic area. For example, in some southern areas the freeze-thaw test is omitted and only the wet-dry test is run. It is recognized that the use of the wet-dry test only is less severe for most soils than using both the wet-dry and freeze-thaw tests. The drying temperature in the wet-dry test accelerates cement hydration and is above the temperatures that prevail in the field. Also, for the lower clay content soils the expansion caused by the temperature used for drying may actually cancel or reduce the shrinkage due to drying (17). Although these features of the wet-dry test oppose the objectives of a climatic simulation test for non-frost areas, the test has been used on a local basis with satisfactory results. Again, this modification is based on experience with local materials and local climatic conditions.

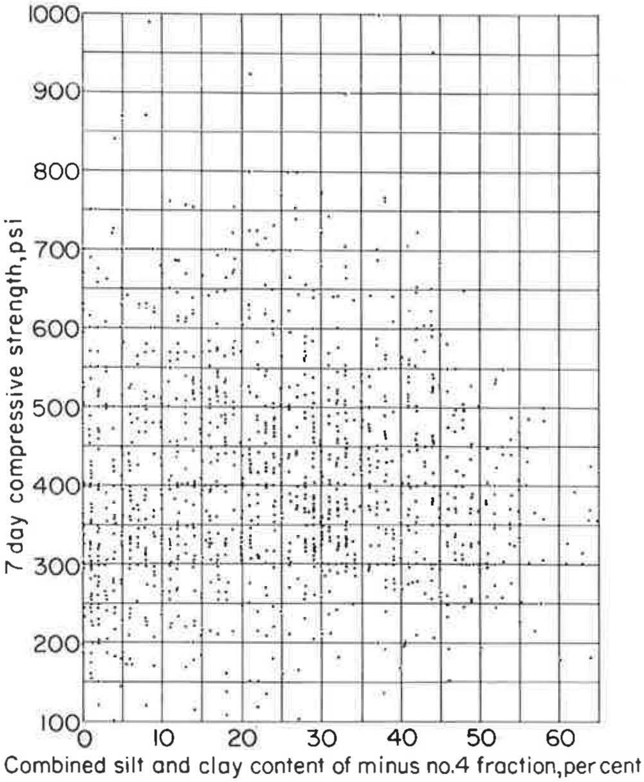


Figure 3. Seven-day compressive strengths at the cement content shown adequate by wet-dry and freeze-thaw tests.

Some other test methods involve the use of a strength test only. This approach requires the assignment for all soil types of a laboratory compressive strength that represents a durable base material which will maintain its strength properties during many years of field service. The compressive strength that will produce this durable base material varies with the physical and chemical properties of the soil (15). For example, Figure 3 gives 7-day compressive strengths at the cement content producing durable soil-cement as determined by the wet-dry and freeze-thaw tests with appropriate criteria plotted against percent of combined silt and clay. It is apparent that a compressive strength that would be adequate for all soils would of necessity be a value higher than needed for many of the soils.

The determination of a suitable compressive strength is simplified when materials meeting a rather narrow range of gradations and/or soil types are used. As a result some agencies have determined and used successfully a compressive strength requirement, generally based on results of wet-dry and freeze-thaw tests for a particular type of material.

The value of the standard wet-dry and freeze-thaw tests is that they determine for any type of soil material the amount of cement needed to hold the mass together and maintain stability under shrinkage and expansive forces which occur in the field. Any strength requirement then becomes of secondary importance and becomes more a matter of thickness design considering traffic loads and volumes.

To insure satisfactory results, test methods for determining cement requirements other than the standard methods should be correlated with results obtained from the standard tests which have a satisfactory service record. The validity of these test methods can also be determined by long-term field performance studies of test sections covering the range of soil and climatic conditions for which the proposed test method will be applied. Some test methods in use are based on such correlations and are giving satisfactory results.

RECENT RESEARCH

The rapidly expanding use of soil-cement has spurred interest in further basic research on the reactions that take place when cement and soil are mixed together in the presence of moisture. Studies are also under way to determine more about the engineering properties of soil-cement and on laboratory methods to measure the effects of freezing, thawing, wetting, and drying on these properties.

Soil and Cement Reactions

Research on the phenomenon of cement hydration as related to soil-cement mixtures and on the effect of the physico-chemical properties of the soil will more clearly define the fundamental reactions that take place when soil, cement, and water are mixed together.

Work of this type now under way at the Portland Cement Association's research laboratories includes determination of fundamental properties of soil and their relationship to the soil's reaction with cement. Included are such properties as surface area, base exchange capacity, solubility in alkali, dimensional stability, and various chemical properties.

Work on the nature of soil and cement reactions has also recently been carried out at the Institute of Transportation and Traffic Engineering, University of California, Berkeley. A paper (18) on this work appears elsewhere in Highway Research Record 36. It presents a hypothesis of the reactions that take place when cement, soil, and water react to form a hardened soil-cement material.

Similar studies at Iowa State University and Massachusetts Institute of Technology have also been recently reported (19, 20).

Physical Properties

Research on the physical properties of soil-cement has been simplified by the development of laboratory tests and equipment for determining compressive, flexural, and shear strength and modulus of elasticity and Poisson's ratio (21, 22). Methods for molding and testing compressive and flexural strength specimens were adopted as tentative standards by ASTM[3] in 1957 and are now in the process of being advanced to standard.

As shown in Figures 4, 5, and 6, values for such properties as compressive strength, flexural strength and modulus of elasticity depend on the soil type, cement content, and time of curing (21, 22). As given in Table 1, soil 2 in these figures is an A-2-4 sandy soil requiring 4 percent cement and soil 4 is an A-4 silty soil requiring 8 percent cement. Of significance is the fact that the test values for any one soil generally vary linearly with cement factor. Thus, it is possible to interpolate and predict values for the various strength and elastic properties when incomplete test data are available. Furthermore, the interrelationships between the different structural properties vary in an orderly fashion making it possible to estimate one property when another is known. An example of the interrelationship between compressive strength and modulus of rupture is shown in Figure 7, which shows that the modulus of rupture is approximately 20 percent of the compressive strength.

Effects of Freezing, Thawing, Wetting and Drying

Another study under way at the Portland Cement Association's soil-cement laboratory is to determine the effects of freezing, thawing, wetting, and drying on the properties of

---

[3] Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory, ASTM D1632. Compressive Strength of Molded Soil-Cement Cylinders, ASTM D1633. Compressive Strength of Soil-Cement Using Portions of Beams Broken in Flexure, ASTM D1634. Flexural Strength of Soil-Cement Using Simple Beam with Third-Point Loading, ASTM D1635.

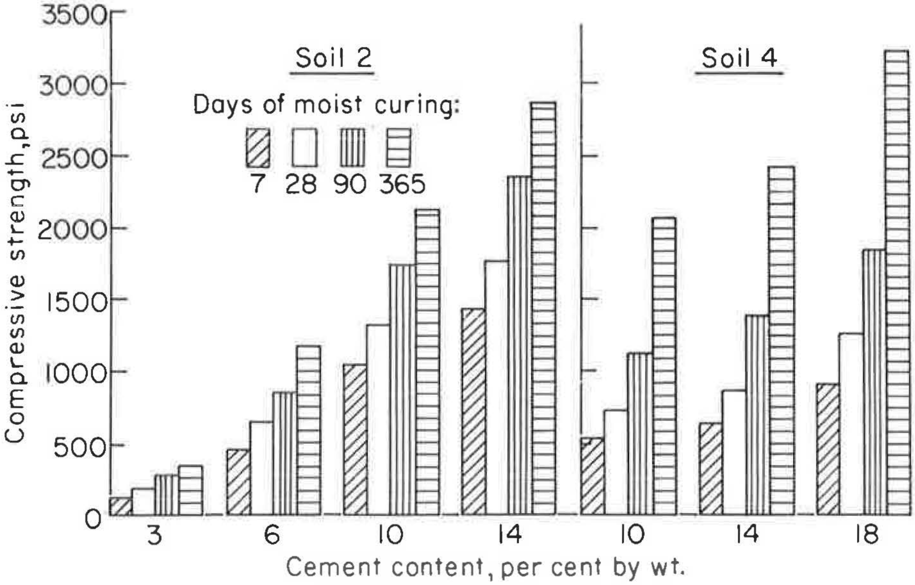


Figure 4. Effect of cement content, curing time and soil type on compressive strength.

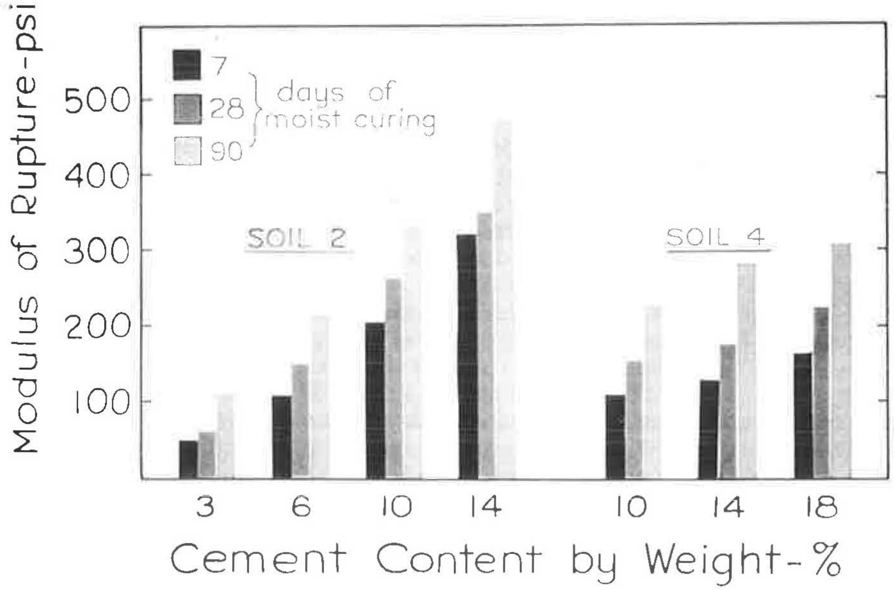


Figure 5. Effect of cement content, curing time and soil type on modulus of rupture.

soil-cement mixtures and to evaluate the techniques for measuring these effects. Techniques being studied, in addition to the manual brushing procedure, include the use of compressive strength before and after tests, pulse velocity measurements each cycle, and length change measurements during the test. The use of length change is particularly promising. This method has immediate sensitivity to deterioration of a specimen, the measurements are easy to make, and the results are accurate. These studies are also providing additional information on the effects of freezing, thawing, wetting, and

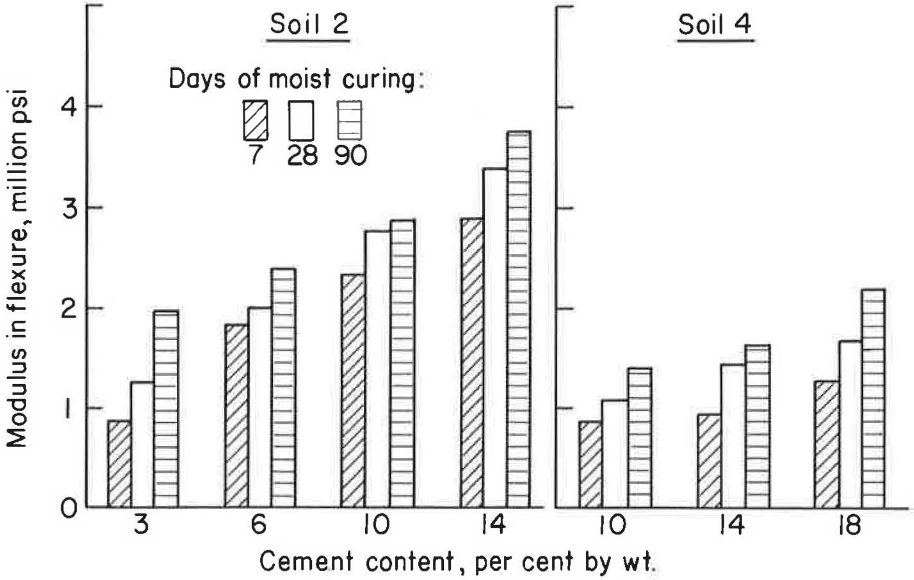


Figure 6. Effect of cement content, curing time and soil type on modulus of elasticity.

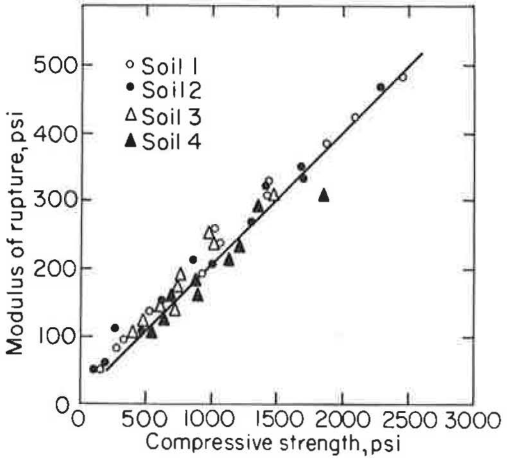


Figure 7. Relationship between modulus of rupture and compressive strength.

TABLE 1

GRADATION, LIQUID LIMIT AND PLASTICITY INDEX OF SOILS 2 AND 4

Item	Soil No. 2	Soil No. 4
Gravel, ¾ in. to No. 4	0	0
Gravel, No. 4 to No. 10	20	0
Sand, No. 10 to No. 40	22	1
Sand, No. 40 to No. 200	39	7
Silt and clay, minus No. 200	19	92
Liquid limit	17	26
Plasticity index	NP	7
AASHO soil group	A-2-4	A-4
Cement requirement, % by wt.	4	8

drying that will be helpful in further development of soil-cement testing procedures. An initial report on this work was previously presented (17). Results of further work are reported elsewhere in Highway Research Record 36 (23).

All of this research work on the phenomenon of cement hydration as related to soil-cement, on the physical properties of soil-cement, and on the evaluation of the effects of freezing, thawing, wetting, and drying will add to the knowledge of soil-cement mixtures and will make the standard laboratory tests even more useful. The data may also permit a more rational approach to laboratory mix design. Thus, soil-cement will continue to be an engineered pavement making use of the latest and most up-to-date technical information available.

REFERENCES

1. Mills, W. H., "Road Base Stabilization with Portland Cement." Engineering News-Record, 115:22, 751-3 (Nov. 28, 1935).
2. Mills, W. H., "Stabilizing Soils with Portland Cement, Experiments by South Carolina Highway Department." HRB Proc., 16:322-47 (1936).
3. Catton, Miles D., "Basic Principles of Soil-Cement Mixtures and Exploratory Laboratory Results." HRB Proc., 17:Part II, 7-31 (1937).
4. Catton, Miles D., "Soil-Cement Mixtures for Roads." HRB Proc., 18:Part II, 314-21 (1938).
5. Catton, Miles D., "Early Soil-Cement Research and Development." Jour. of the Highway Div., ASCE Proc., Vol. 85, No. HWI (Jan. 1959).
6. Catton, Miles D., "Soil-Cement Technology—A Resume." PCA Research Dept. Bull. 136 (1962).
7. Catton, Miles D., "Research on the Physical Relations of Soil and Soil-Cement Mixtures." HRB Proc., 20:821-55 (1940).
8. Norling, L. T., and Packard, R. G., "Expanded Short-Cut Test Methods for Determining Cement Factors for Sandy Soils." HRB Bull. 198, 20-31 (1958).
9. U. S. Department of Agriculture, Yearbook of Agriculture, Soils and Men (1938).
10. U. S. Department of Agriculture, Handbook No. 18, Soil Survey Manual (1951).
11. Hicks, L. D., "Soil-Cement Design in North Carolina." HRB Proc., 22:415-421 (1942).
12. Hicks, L. D., "Use of Agricultural Soil Maps in Making Soil Surveys." HRB Bull. 22, 108-116 (1949).
13. Leadabrand, J. A., Norling, L. T., and Hurless, A. C., "Soil Series as a Basis for Determining Cement Requirements for Soil-Cement Construction." HRB Bull. 148, 1-17 (1957).
14. "Agricultural Soil Maps." HRB Bull. 22-R, 109 pp. (1957).
15. Leadabrand, J. A., and Norling, L. T., "Soil-Cement Test Data Correlation in Determining Cement Factors for Sandy Soils." HRB Bull. 69, 29-46 (1953).
16. Portland Cement Association, Soil-Cement Laboratory Handbook, Chicago, Ill.
17. Packard, R. G., "Alternate Methods for Measuring Freeze-Thaw and Wet-Dry Resistance of Soil-Cement Mixtures." HRB Bull. 353, 8-41 (1962).
18. Herzog, A., and Mitchell, J. K., "Reactions Accompanying the Stabilization of Clay with Cement." Highway Research Record 36, 146-171 (1963).
19. Handy, R. L., "Cementation of Soil Minerals with Portland Cement or Alkalies." HRB Bull. 198, 55-64 (1958).
20. Lambe, T. W., Michaels, A. S., and Moh, Z. C., "Improvement of Soil-Cement with Alkali Metal Compounds." HRB Bull. 241, 67-108 (1960).
21. Felt, Earl J., and Abrams, Melvin S., "Strength and Elastic Properties of Compacted Soil-Cement Mixtures." ASTM Spec. Tech. Pub. No. 206 (1957).
22. Balmer, Glen C., "Shear Strength and Elastic Properties of Soil-Cement Mixtures Under Triaxial Loading." ASTM Proc., Vol. 58 (1958).
23. Packard, R. G., and Chapman, G. A., "Developments in Durability Testing of Soil-Cement Mixtures." Highway Research Record 36, 97-122 (1963).

California Mix Design for Cement-Treated Bases

F. N. HVEEM and E. ZUBE, respectively, Materials and Research Engineer, and Supervising Materials and Research Engineer, Materials and Research Department, California Division of Highways

The California Division of Highways has constructed extensive mileage of cement-treated bases since 1938. These bases have been used primarily under asphaltic concrete pavements carrying moderate to heavy traffic. Depending upon the traffic intensity, 2 to 5 in. of asphaltic concrete is placed as a wearing course over these treated bases. The increasing occurrence of pumping and step-offs in portland cement concrete (PCC) pavements as a result of a large increase in truck traffic led to the construction of so-called cement-treated subgrades under all newly placed concrete pavements beginning about 1945. The materials used in the earlier jobs included fine sand, disintegrated granite, sandstones and stream bed gravels. During the last few years, more emphasis has been placed on the use of granular materials, resulting in a decrease in cement content and a tendency to minimize shrinkage cracks.

In the design of the pavement structure, a reduction in the overall thickness is obtained by virtue of the cohesion or tensile strength of the cement-treated base. The mix design is based on a compressive strength requirement of 750 psi (on a 4- × 4-in. cylinder) after a 7-day curing period. A rapid cement control test for field use to determine the actual percentage of cement in the mixture has been developed.

•SCANTY records and hazy recollections do not warrant any definite statement about the time or place when portland cement was first mixed with soil to produce a hardened water-resistant support base for highway pavements. Once the process had become established however, at least one engineer was heard to remark that he "could have discovered it years before." He had observed that truck drivers hauling cement over a dirt road would occasionally scatter a few sacks in some of the mud holes in order to get through. These crude mixtures of soil and cement were apparently effective, but he did not take the hint.

According to available information, a cement-treated section was constructed in Pennsylvania before 1930. Apparently, this project was not publicized, and in fact, appears to have been carried out almost in an atmosphere of secrecy. However, regardless of accidental or scattered prior trials, South Carolina deserves the credit for first constructing and reporting successful soil-cement bases. The pioneer work in South Carolina was reported in 1936.

Although California had tried mixing portland cement with heavy clay soils as far back as 1921, there was no immediate follow-up or attempt to develop the process further. Following the reports from South Carolina, California in 1937 constructed

---

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

two sections each approximately one mile long using State forces. Additional projects, each some three miles in length, were constructed under contract in 1938 and 1939.

Up to 1940, all California work of this type employed the road-mix method. However, it was often difficult to secure uniform construction both in depth of material treated and in thorough distribution of the cement. Furthermore, road mixing with farm equipment (such as disc and harrow, then generally used) required considerable time and it was common to have a delay of 6 or 8 hours between the introduction of cement and water and final compaction which, of course, had a considerable adverse effect on the compressive strength.

Long experience in mixing asphaltic materials both by road-mix and plant-mix methods had indicated the superiority of plant mixing so far as speed and uniformity are concerned. It seemed logical that the process of mixing soil and cement would likewise be improved. Therefore, beginning in 1939 three projects were initiated requiring the mixing of cement with pit run gravels or granular materials in a central mixing plant.

Because the proposed materials were generally granular and not of the type that an agriculturist would ordinarily class as "soil," it was decided that the term cement-treated base was more appropriate. Also, the term "soil stabilization" was being widely appropriated by salesmen or advertising agencies and applied to a variety of treatments which have little or nothing in common. Since that time, all work of this sort in California has been called "cement-treated base" regardless of gradation of the soil, mineral aggregate, or the method of mixing. Another change from Eastern practice is in the method of expressing the cement content. Designating cement contents in terms of volume is sound from a theoretical standpoint, but it is less convenient in practical application. There are many more individuals engaged in construction control and inspection than there are in the laboratories; therefore, it was decided to specify the cement content in terms of percentage by weight of the aggregate. By this step, California has a uniform practice for indicating the amount of water in a soil, the amount of asphalt or road oil used in bituminous mixtures and the percentage of cement in cement-treated bases, all of which are now stated in terms of percentage by weight of the dry aggregate.

Some 30 miles of plant-mixed cement-treated base had been completed by 1941 and this mileage had increased to over 100 miles by the end of 1943 (1). During this period, laboratory work was under way to determine appropriate test procedures, methods of mixture design, specifications, etc. The establishment of test methods must, of course, require some knowledge of the essential properties, and it was taken for granted that cement-treated base mixtures should develop some appreciable compressive strength in line with the usual concepts concerning the properties of portland cement concrete. The question of what strengths were appropriate was not so self-evident and was much debated. As in all new developments, ideas are prone to be influenced by the evolutionary steps involved or by the background of the individuals. Those engineers who had long experience or indoctrination in the design and construction of concrete were inclined to favor high strengths for cement-treated bases. Therefore, these treated bases were regarded by some as a sort of inferior concrete while others thought of them as an improved aggregate base. The "concrete boys" were inclined to think that anything developing less than 3,000 psi must be of dubious quality. On the other hand, tests on specimens of the best crushed stone base aggregates "cemented" with natural fines gave only 75 psi, and as these crushed stone bases had proven to have the qualities necessary for supporting heavy loads, there seemed to be no reason for requiring extraordinarily high compressive strengths for cement-treated bases.

It was decided to follow the practice established by the Portland Cement Association for the size of specimen (4-in. diameter and 4-in. height). However, to permit ready compaction in the field and shipping of the specimens to a central laboratory a procedure was developed involving the use of specimen molds in the form of thin metal shells or sleeves which are made to order from tinned sheet (Appendix A). By the means adopted, it is possible to compact the test specimens, seal the containers, protect and hold the specimens without loss in moisture until time for testing. The tin sleeves are then opened along the soldered joint and the specimens soaked before testing to determine compressive strengths.

A curing period of 7 days was adopted to shorten the time of testing. A compressive strength requirement of 850 psi in 7 days, and 1,000 psi at 28 days, was agreed upon. In general, the compressive strength at age of 7 days is about 85 percent of that obtained at 28 days.

The next step was to establish a suitable compaction procedure which would simulate the compaction to be expected on the road. It will be noted that the compressive strength increases very rapidly with increased compaction. Cores were taken from finished bases and comparisons were made with laboratory compacted specimens for density and strength. The compaction procedure finally adopted was aimed at reproducing the results found in the core specimens.

It became evident that a rather wide variety of aggregate types and gradations could be successfully treated with cement, and it has been the general practice to write grading specifications which would permit the use of local materials with a minimum of waste or importation of expensive aggregates. However, the gradation of the aggregate can have some effect on the compressive strength.

It became evident that with the granular materials being used, substantial compressive strength values could be developed even with nominal amounts of cement ranging from 4 percent to 6 percent of the aggregate. Therefore, as previously mentioned, the mix design was initially based on a compressive strength requirement of 1,000 psi at the age of 28 days. Work was governed by this concept for two or three years. However, a number of engineers began to complain of the transverse shrinkage cracks which appeared in the bituminous surfaces over these bases and a quick survey of all jobs constructed seemed to indicate that cracks were more evident and often opened wider over the high-strength bases than over the sections where the strengths were low. This led to a revision in strength requirements, and four types of cement-treated bases were established (2), as follows:

Class	7-Day Comp. Strength	Typical Cement Content (% by dry wt. of agg.)
A	750 psi	3½ to 6
B	400 psi	2½ to 4½
C	R-value = 80+	2 to 3
D	-[a]	4 to 6

[a] A reconstruction method where an existing asphalt treated surfacing is scarified and mixed with an equal amount of existing base or imported gravel. No strength requirement specified.

The Portland Cement Association had recommended a test procedure involving alternate cycles of wetting and drying, after which the surface of the specimen is abraded by means of a wire brush. It was judged that this operation was subject to considerable variation, depending on the individual operator, and after some investigation, it was concluded that the same evidence of deterioration could be produced by making compressive strength tests after a series of wet and dry cycles (Appendix C). However, California has encountered only a few types of material where the wet and dry test procedure seemed to be warranted.

A freeze-thaw test, patterned after the wetting and drying method, has also been developed to determine the durability of cement-treated base mixtures where intensive frost conditions are anticipated (Appendix B). This test has not had much application in "sunny" California, however, since only a limited mileage in the high mountain regions requires consideration of severe winter conditions.

When the cement-treated bases were first being considered in California, recommendations from the Portland Cement Association favored the use of only a thin bituminous surface in the form of a seal coat or surface treatment. However, it was not felt that such a surface would be adequate for heavy traffic, and there were reports from other states that failures had occurred because of the slipping or peeling off of the thin surfaces. Observations on one or two early projects where the bituminous surfacing was varied from $\frac{3}{4}$ inch to $1\frac{1}{2}$ inch in thickness indicated that slippage failures were much more likely to occur with the thin bituminous surface layers. About 1940, it was decided that cement-treated materials should be regarded only as a base and should be protected by a substantial dense-graded asphalt surface course not less than 3 in. thick. With a few exceptions, this policy has been maintained, and experience seems to justify the practice of placing an adequate thickness of asphaltic surfacing over these treated bases.

With the completion of an intensive survey and an investigation of PCC pavements through the years 1944 to 1946, it was concluded that some means must be provided to prevent the loss of support caused by the pumping out of subgrade soil from beneath the concrete slabs. Several means were considered and undoubtedly several expedients would have been effective, but a cement-treated layer 4 in. thick seemed to be the most economical, and it appeared that such a base would resist erosion if protected by a heavy penetration application of cutback asphalt (3).

The first cement-treated subgrade to support a concrete pavement was constructed in 1946 and within the next year or so it became general practice throughout the state. Since 1950, all California concrete pavements have been placed over treated bases. In a few cases where the underlying soil was a relatively clean cohesionless sand, asphalt-treated subgrades were used in lieu of portland cement and for many years it appeared that the performance was about equal. However, it is becoming increasingly apparent that the asphalt-treated subgrades are less permanent, but the cement-treated subgrades (now called CTB Class B) can be considered as being successful in more than 95 percent of the projects built. Pumping has been eliminated and faulting at the joints is rarely perceptible as a result of this design, even though dowels or load transfer devices are not used. When used to prevent pumping and erosion of the subgrade support, cement-treated bases were usually constructed after the side forms were in place, the mixing being done by traveling mixing machines.

This extended use of road mixing for the cement-treated bases under concrete pavements led the construction forces to believe that road mixing would be more economical and equally satisfactory for the heavier bases required to support an asphalt pavement. With the passage of a few more years it became increasingly evident, however, that there were more failures and more evidence of generally poor results. An investigation led to the recommendation that the road mixing process be abandoned for all heavy-duty main-line highways and that its use should be confined to lightly-traveled roads or to the relatively thin treatment employed beneath concrete pavements. It also became evident that there was more evidence of distress in the Class C bases using the very low cement contents. Furthermore, the thin bases ranging from 5 in. to 6 in. have not given a satisfactory performance in many cases, and today it is standard practice for heavy-traffic roads to construct cement-treated bases no less than 8 in. in thickness to be covered with at least 3 in. or 4 in. of dense-graded asphaltic concrete.

In effect then, it is a composite pavement in which the general behavior and characteristics are very similar to the pavements constructed 50 to 60 years ago in which an asphalt wearing surface was supported by a lean concrete base. It may be pointed out, however, that for a given aggregate and cement content, a cement-treated base is superior to and more efficient than plastic concrete. The water-cement ratio law is still valid and these relatively dry mixtures rolled and compacted with heavy equipment are stronger than if enough water were added to permit placing as conventional concrete.

CEMENT-TREATED BASE MIX DESIGN METHOD

The design of Classes A and B cement-treated base (CTB) mixtures largely involves determining, by laboratory tests, the amount of cement and water necessary to meet the minimum 7-day compressive strength requirements with a given source of aggregates. California specifications require that aggregate for cement treatment must have a sand equivalent of not less than 25 and conform to the following requirements unless special gradings are provided in the special provisions for the particular project.

Sieve Size	Percent Passing
1 in.	100
¾ in.	90-100
No. 4	35-55
No. 30	10-30
No. 200	3-9

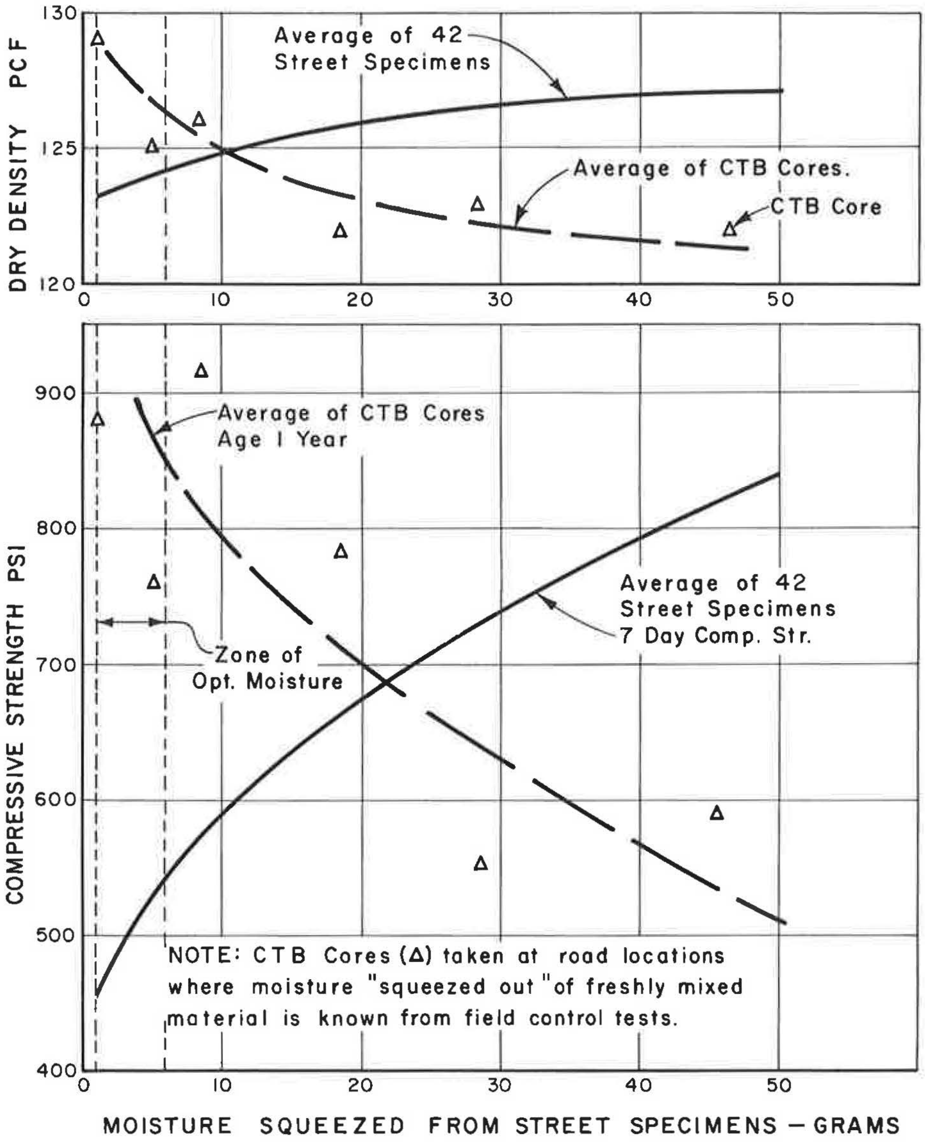


Figure 1. Comparison of fabricated street CTB specimens with cores taken from the road on the basis of the degree of saturation of CTB material during construction.

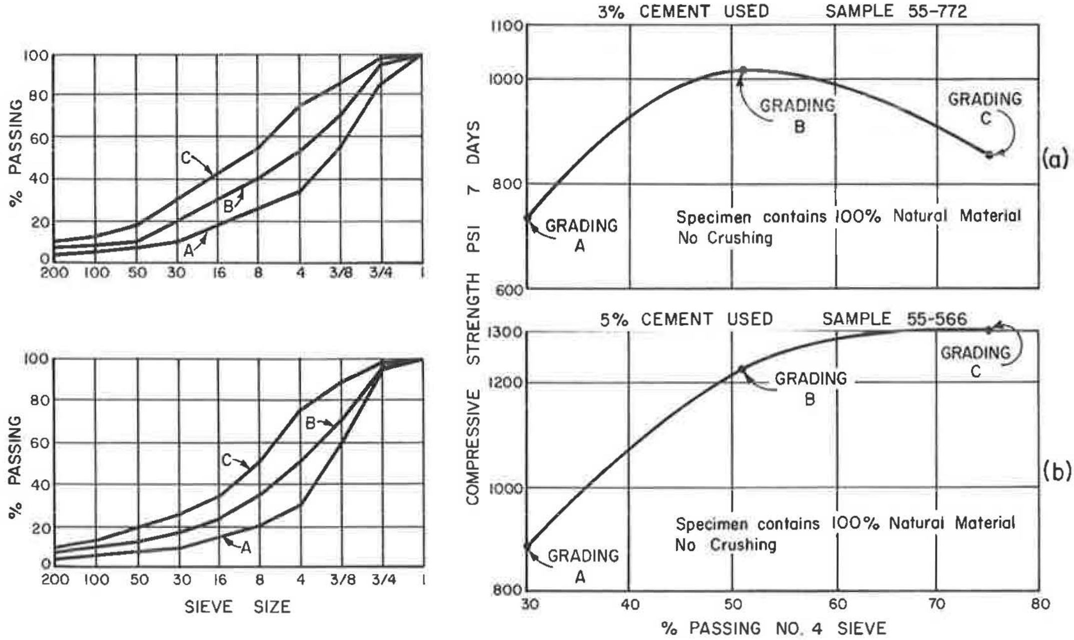


Figure 2. Relationship between percent passing No. 4 sieve and compressive strength.

This grading is identical to Class 2 aggregate base. It should be pointed out, however, that cement-treated bases have been constructed using a wide variety of aggregate gradings, particularly when local deposits are utilized.

Optimum Moisture Determination

The first step in the laboratory testing involves the determination of the optimum moisture at which maximum density and consequently the highest compressive strength

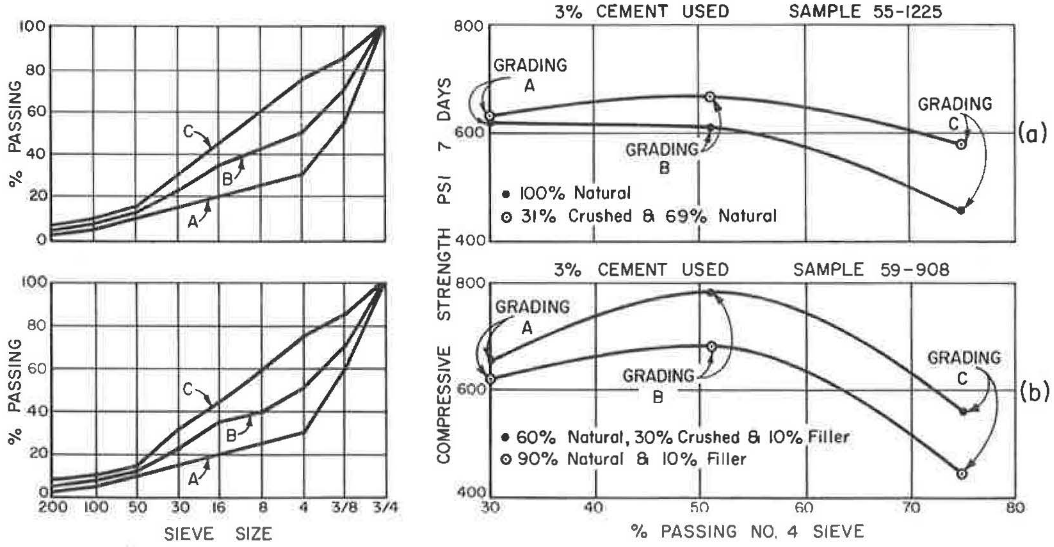


Figure 3. Relationship between percent passing No. 4 sieve and compressive strength.

will occur. Briefly, the procedure consists of fabricating three 4-in. diameter × 4-in. high test specimens containing different percentages of moisture but the same amount of cement, typically 3 or 4 percent (the exact amount of cement is not important to this phase of the test). The mixtures of cement, aggregate and water are hand rodded (mechanical kneading compaction can be used as an alternative) into molds containing tin sleeves and then loaded in a press (or hydraulic jack in frame), for a 2½-min period, to 25,000 lb or about 2,000 psi. During the process of static loading, notation is made of any moisture squeezed out of the specimen. The amount of such moisture loss is determined by weighing immediately before and after application of the static load. The amount of moisture added to the mixtures, during the fabrication process, is set high enough so that some moisture will be exuded from at least two of the three specimens. One of the specimens should be prepared at the point of saturation which

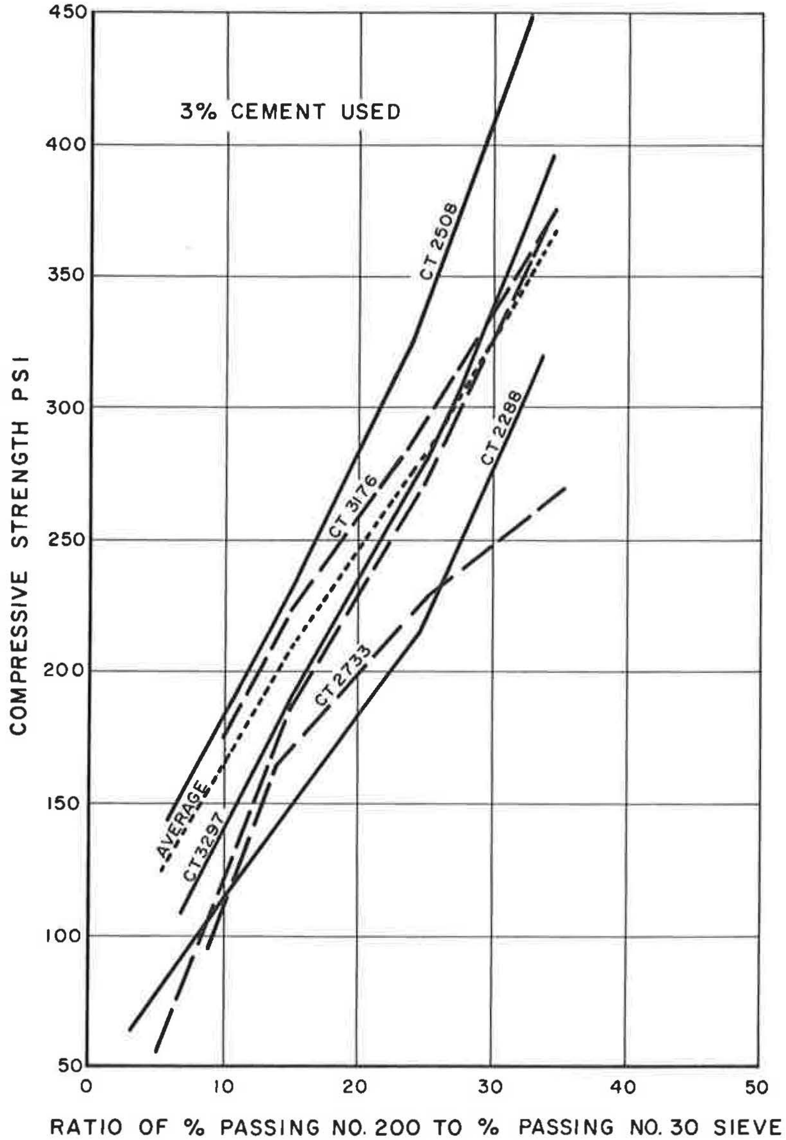


Figure 4. Relationship between compressive strength and ratio of percent passing No. 200 to percent passing No. 30 sieve.

is normally considered to be attained when 1 to 6 g of water are squeezed from the specimen. The other two should be slightly above and below (this is usually ± or 2% moisture) saturation, respectively. The optimum moisture and density are calculated from the saturated specimen which exuded the 1 to 6 g of water. All three specimens are then cured and tested for 7-day compressive strength for supplemental information. While the optimum moisture determination is necessary to the second phase of the laboratory test, it is also reported for ultimate use in field construction control. In this case, the moisture content is usually expressed as a range which extends from the point of saturation to approximately 1 percent below this point. The test data (including compressive strength) from the specimens fabricated above and below the saturation serve as a guide in establishing this range.

At this point, it might be well to digress for a moment and discuss the background which forms the basis for the mode of optimum moisture control of CTB's. In the

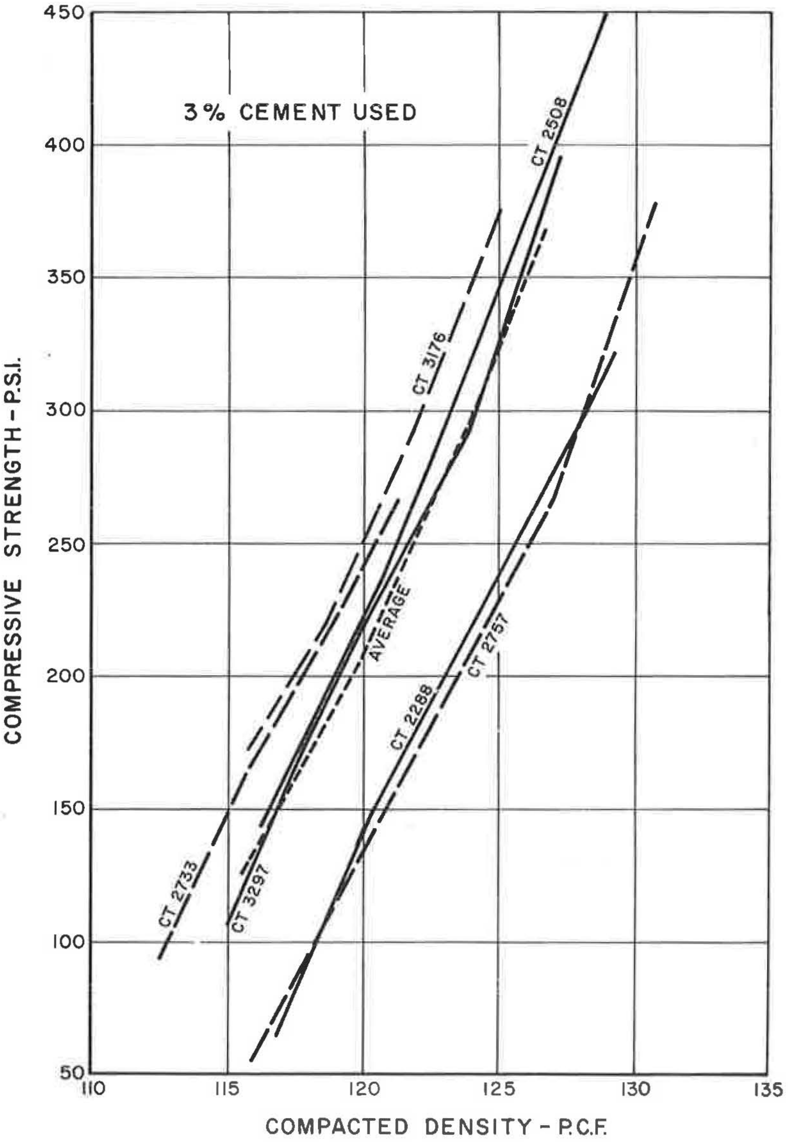


Figure 5. Relationship between compressive strength and density.

original field studies, it was found that the optimum moisture condition that produced the highest density and strength under normal construction compaction, occurred most frequently when a small excess of moisture was present in the mixture. On the basis of this observation, the compaction procedure for laboratory specimens was developed which indicated a reasonable correlation of physical properties between field cores and specimens fabricated at the saturation point. It is often found in the laboratory that continued increases in the moisture at compaction, beyond the saturation point of the specimens, will result in further increases in density and resulting higher strength. Experience indicates, however, that these higher laboratory values are usually excessive, in relation to the physical properties actually attained under normal field compaction, if "over-saturation" of the material occurs during construction.

This concept is illustrated in Figure 1, with test data from an actual project constructed in Santa Barbara County. During construction numerous "street" specimens were fabricated by laboratory methods using freshly-mixed CTB sampled from the grade. Both the densities and compressive strengths of the laboratory compacted street specimens (solid lines) continue to increase in magnitude, even when the moisture content of the material is so high that 40 or 50 g of water are squeezed out during fabrication.

About one year after construction, a number of 4-in. diameter CTB cores were cut from this road at specific locations where the moisture condition of the material at time of construction was known. The density and compressive strength data from the cores are plotted (Fig. 1) against the amount of moisture squeezed from street samples previously obtained at the same respective road locations where the cores were taken. Although there is a scattering of core data, due to the influence of factors other than moisture (e.g., cement distribution and variations in construction compaction), it is still possible to define the trend by plotting an average of the values obtained (dashed lines). Contrary to the laboratory tests, the highest density and strength is attained in the road when the moisture level is in the area where saturation of the compacted

TABLE 1

EFFECT ON COMPRESSIVE STRENGTH, DENSITY AND VOIDS OF COMPACTED CEMENT-TREATED MATERIAL[a] WHEN THE 200-MESH/30-MESH RATIO IS INCREASED BY ADDITION OF FINE BLENDING MATERIAL

Sample No.	Material Type	% Blending Material	% Passing No. 30 Sieve	% Passing No. 200 Sieve	Ratio No. 200 to No. 30	Compr. Strength[b] (psi)	Compacted (pcf)	% Voids
CT 2288	Sandy gravel from	0	38	1	3	65	116.5	29.5
	gravel bar	5	41	6	15	150	120.2	27.3
	Feather River,	10	44	11	25	220	124.0	24.9
	Dist. III	16	48	16	34	325	129.3	21.7
CT 2508	Sandy soil	0	46	3	6	145	116.2	30.9
	from SD Co.,	5	49	8	16	240	120.7	28.3
	Dist. XI	10	51	12	24	325	124.1	26.2
		17	55	19	35	475	129.8	22.9
CT 2525	Sandy soil	0	80	4	5	235	115.5	32.9
	from SD Co.,	8	82	12	14	315	120.1	30.0
	Dist. XI	18	84	21	25	340	122.7	28.5
		28	86	30	35	385	124.7	27.3
CT 2733	Sandy soil,	0	97	9	9	95	112.3	35.2
	borrow pit,	6	97	14	14	165	115.4	33.4
	Austin Rd. and	17	97	24	25	230	119.2	30.7
	Rt. 66, Dist. X	29	98	34	35	270	121.3	29.2
CT 2757	Sand	0	44	2	5	55	115.7	30.2
	Reliance Pit,	5	47	7	15	185	122.5	26.1
	Dist. XI	11	50	12.5	25	270	126.9	23.5
		18	54	19	35	380	130.7	21.5
CT 3176	Sandy soil,	0	92	9	10	175	115.8	30.9
	United Pipe	6	92	13.5	15	225	118.7	29.2
	pit No. 2,	16	93	23	25	295	121.8	27.4
	Dist. X	27	94	33	35	380	125.0	25.5
CT 3297	Sandy soil,	0	42	3	7	110	114.8	31.5
	Jenkins Pit,	4	45	7	15	190	118.1	29.6
	Dist. VI	10	48	13	27	295	123.7	26.2
		16	51	18	35	400	127.2	24.1

[a] 7-day curing period.
[b] 3 percent cement.

material just occurs (normally occurring when 1 to 6 g of moisture are squeezed out of fabricated specimens) and that "over-saturation" causes a reduction in these properties.

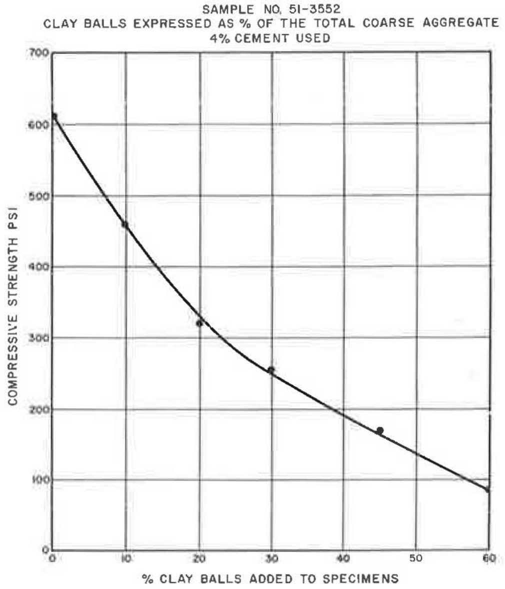


Figure 6. Effect of adding varying percentages of hard balls of clayey materials to a well-graded material.

An explanation of this paradox lies primarily in behavior of CTB materials under "static" load conditions in the laboratory process as compared to what usually happens in construction compaction. When a moisture content above saturation is encountered, the laboratory method causes the excess water to be permanently removed from the specimen. Any resulting higher density (as compared to a saturated condition) is presumably due to the added lubrication provided by the excess moisture and the fact that the volume of the void spaces in the specimen is reduced during the 2½-min static compaction period in proportion to the amount of the moisture squeezed out. However, in the case of construction compaction on the road, the surplus moisture generally remains in the material for two reasons: (a) the application and release of load, by the passage of a compactor wheel (or roller) at any given point on the CTB, is almost instantaneous and does not normally allow sufficient time for the movement of the water through the pore spaces and out of the material; and (b) the compacted underlying subbase or basement soil, which is usually fairly impermeable, forms a highly resistant barrier to the complete escape of the excess moisture. The moisture retention in the CTB layer not only affects the density of the material, but causes a higher water-cement ratio (and a corresponding lowering in strength) than if the water could escape as it does in test specimen fabrication.

In the final analysis, the optimum moisture-density relationship, determined from the test at the point of saturation, is most nearly related to the behavior of the same material when subjected to the proper construction compaction and does not necessarily relate to the absolute maximum compaction that is possible to attain in the laboratory test.

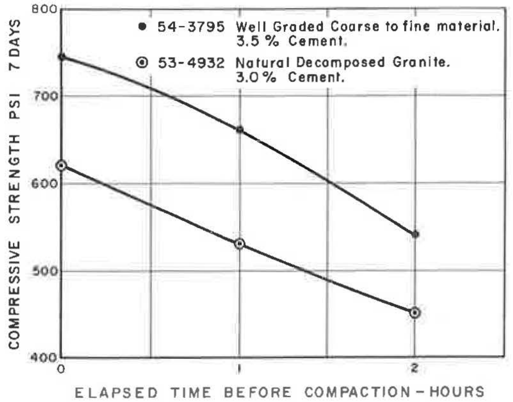


Figure 7. Relative compressive strengths of cement-treated specimens when mixtures were allowed to stand for different intervals before compacting.

Compressive Strength Determination

The next step in the mix design concerns the fabrication of a test series for compressive strength determinations. Again, three or more specimens are prepared, but in this case the moisture content is held constant at the optimum moisture level determined from the previous test series. Each specimen is fabricated at a different cement content which is normally arranged in approximately 1 or 2 percent increments to cover the anticipated range for the class of cement treatment under consideration.

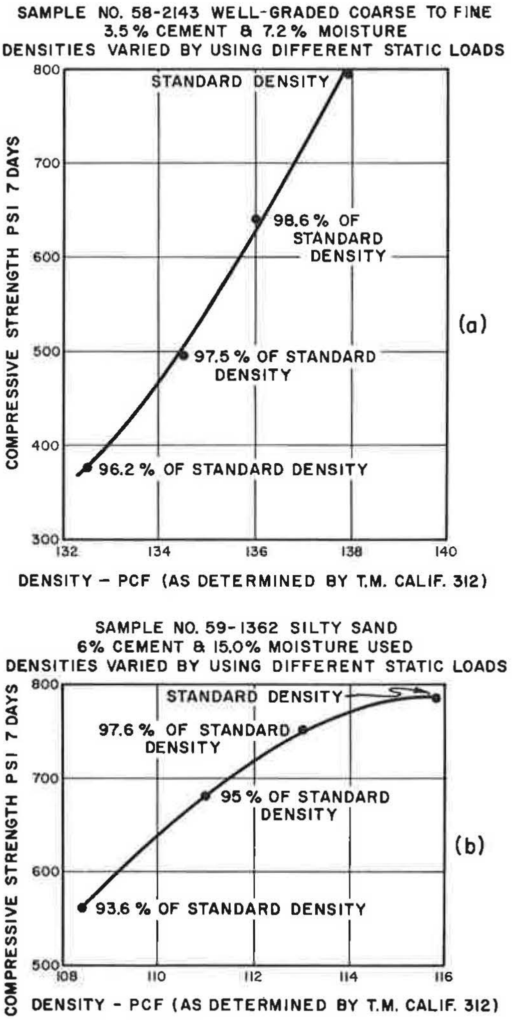


Figure 8. Relationship between compressive strength and densities of CTB test specimens.

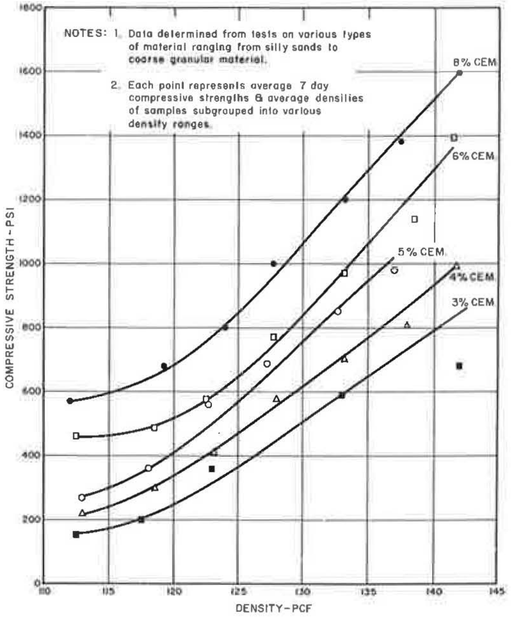


Figure 9. Relationship between density and compressive strength of CTB specimens.

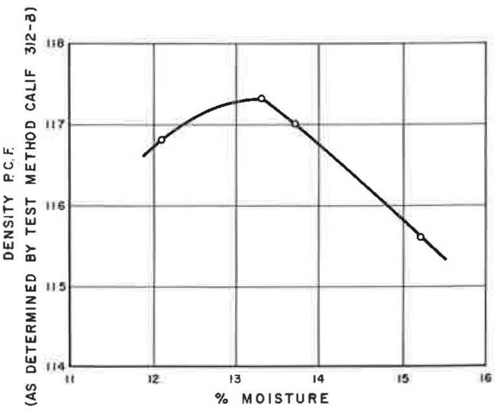


Figure 10. Moisture-density curve (sample No. 59-1362, silty sand, IV-CC-75, 107-susp.).

The specimens are molded and compacted, in the same manner as previously described for the optimum moisture series, and then subjected to a curing period. This is accomplished by either storing in a moist cabinet or sealing the specimens by taping metal covers on the ends of the tin sleeves. After curing in this manner for 6 days, the sleeves are removed and the specimens are submerged in water for one more day. At the conclusion of the 7-day curing, the specimens are capped with plaster of Paris and tested for ultimate compressive strength in a testing machine. Interpolation of the compressive strength data thus obtained is used to determine the amount of cement required to meet the specified strength under optimum moisture conditions. Recommendations of cement content for field application usually include an additional ½ percent cement to cover the normal variation of cement distribution in the mixture characteristic of field mixing operations. This provides some assurance that most of the mixture

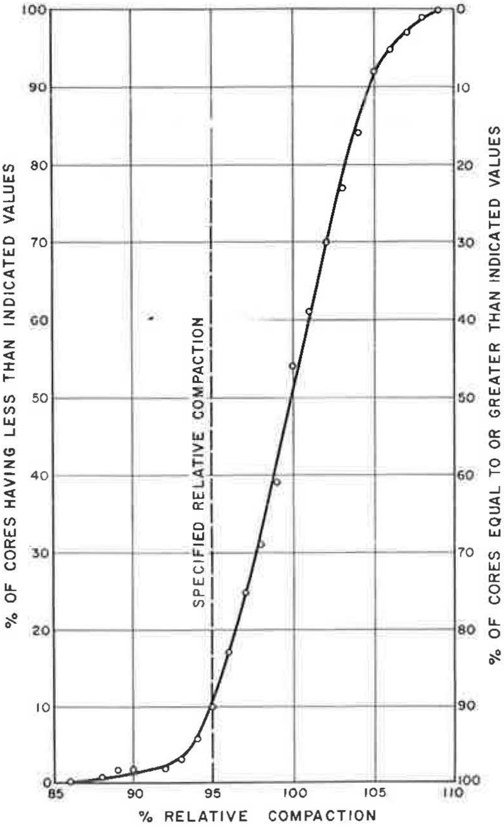


Figure 11. Comparison of construction audit cement-treated base cores with laboratory specimens.

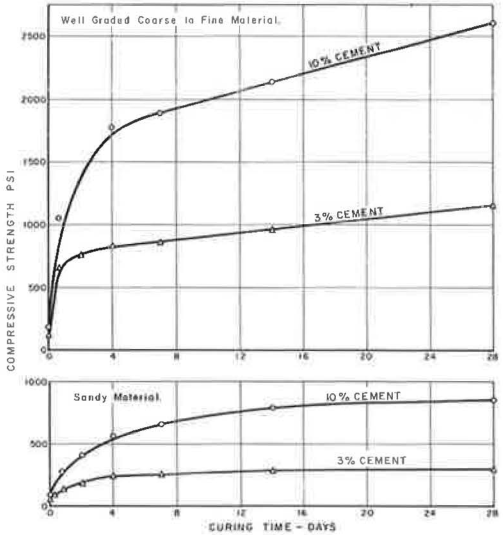


Figure 12. Relationship between curing time and compressive strength of CTB specimen.

will contain at least the minimum amount of cement required by the design, if the construction operations are carried out properly.

Effect of Grading

As previously mentioned, satisfactory CTB's have been constructed using many types of gradings. Materials uniformly graded from coarse to fine require less cement, whereas materials lacking either in coarse aggregate or fines require higher cement contents to meet equal specification requirements. Figures 2 and 3 show the relation between percent passing the No. 4 sieve and compressive strength. Figure 3 shows that there is a small increase in compressive strength if the aggregate is crushed.

Ratio of Percent Passing No. 200 Sieve to Percent Passing No. 30 Sieve

To obtain an indication of the compressive strength of sands or sandy soils from sieve analysis only, work was performed some years ago by varying the relationship between the amount of materials passing the No. 30 and No. 200 sieves and some interesting data were obtained.

Recognizing that this class of material develops the most trouble in terms of compressive strength and field compaction, when relatively low cement contents are used, a series of tests was conducted using a fixed cement content of 3 percent and varying the ratio of the passing No. 30 and No. 200 sieves on various sandy materials by means of adding a filler.

Figures 4 and 5 and Table 1 indicate that in order to obtain compressive strengths ranging from 200 to 400 psi on the normal sandy material using 3.0 percent cement, the ratio of percentage material passing the No. 200 to percentage material passing No. 30 sieve should not be less than 15 percent and possibly not more than 40 percent. The density or weight per cubic foot of the compacted material should range from 120 to approximately 130 pcf.

These experimental combinations were tested to provide some indication of the compressive strength that could be assumed from an inspection of the sieve analysis. Due to the necessity of employing blending operations to obtain the proper ratio it may not, in many cases, be practicable in actual construction practice to vary or adjust the proportion of fine material. It appears, however, that these relationships may be useful to field engineers in trying to determine reasons for any sudden reduction in strength.

Effect of Clay Balls

On one project, hardened clay balls were present among the coarse aggregate. To determine the effect on the compressive strength a series of tests was performed in which the percentage of hard balls of clayey material was varied. Figure 6 shows the results. A 10 percent addition of clay balls to the sound aggregate reduced the strength about 25 percent.

Compaction and Curing

Figure 7 shows the effect of delay in compacting the materials on the road and the resulting reduction in compressive strength for two different materials. To guard against this California's standard specifications specify that "not more than 2 hours shall elapse between the time water is added to the aggregate and cement, and the time of completion of final compaction after trimming." Figures 8 and 9 show the relationship between density of the specimens and the compressive strength. Figure 10 shows the effect of moisture on the density of cement-treated specimens.

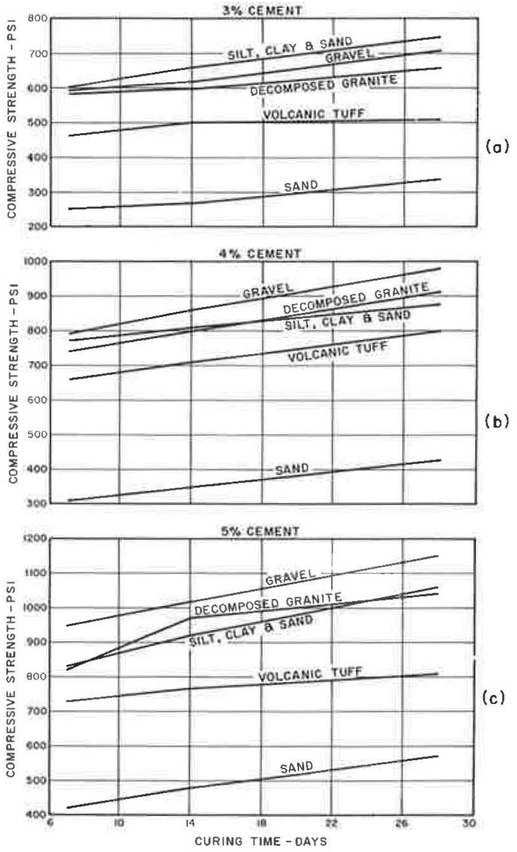


Figure 13. Relationship between curing time and compressive strength of CTB specimens.

In connection with the USBPR record sampling program, field compaction densities on 261 cores from 19 different projects have been obtained and compared with laboratory compacted specimens. Specifications require that the CTB material shall be compacted to at least 95 percent of the laboratory compacted specimen. The Ogive curve (Fig. 11) indicates that 40 percent of the cores showed compaction between 95 and 100 percent of the laboratory compaction. Fifty percent of the cores showed in excess of 100 percent laboratory compaction and ten percent were compacted to less than 95 percent. However, only 1½ percent were compacted to less than 90 percent and none to less than 86 percent of relative compaction.

Figure 12 indicates the gain in strength with increasing curing time (after compaction) for two different materials, a well graded (from coarse to fine) aggregate and a sandy material. As a rule, clean sandy materials are not too well suited for cement treatment as they require a relatively high cement content to bind the individual sand grains together. Blending with a fine filler material to reduce the size of the voids will reduce the required cement content markedly.

Figures 13c and 14 show the increase in compressive strength with curing time for miscellaneous aggregates and soil types when treated with 3, 4, 5, 6 and 8 percent of cement. Well-graded gravelly materials, when treated with the higher cement content,

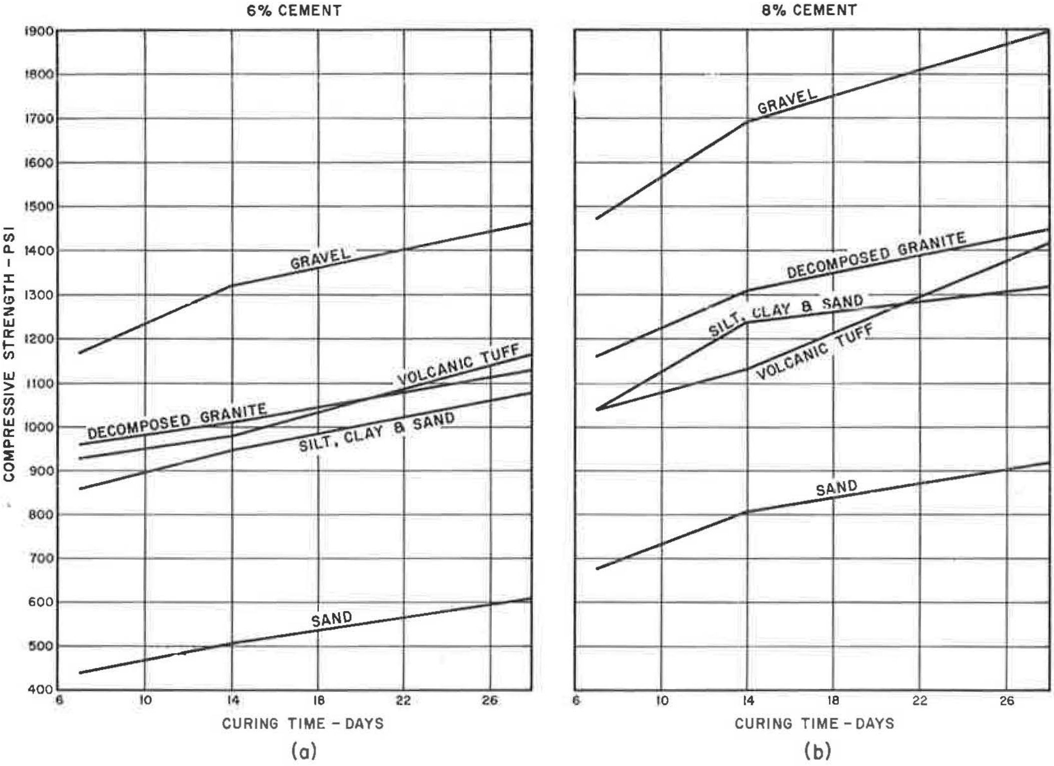


Figure 14. Relationship between curing time and compressive strength of CTB specimens.

develop strengths comparable to weak concrete. All materials show gains in strength with an increase of curing time.

Figure 15 shows the effect of curing temperature for two different aggregates. The higher temperatures expedite the gain in strength as is well known from concrete experience. Therefore, a cement-treated base constructed during the warm summer months will have no difficulty in attaining the specification requirement in 7 days. However, with the same aggregate and amount of cement, but with construction in the late fall or at higher altitudes where air temperatures are low, strength will develop slowly and if subjected to heavy construction traffic, cracks may develop and thus greatly reduce the anticipated slab strength.

Effect of Additives

A few years ago a short research project was conducted to determine the effect of some commercial additives on the compressive strength of cement-treated aggregates. Figure 16 shows the results obtained. The effect on the silty and sandy silt materials was not very pronounced. Some noteworthy variations, however, were apparent in the sand-gravel material.

Some years ago, the addition of asphaltic emulsion to cement-treated aggregates was proposed. One reason being that some Class D CTB, where an old asphaltic surface was pulverized and mixed with the untreated underlying base material and cement treated, had shown excellent service records. Figure 17 shows the results obtained after various methods of curing. Although the addition of emulsion seems to be slightly beneficial on some materials subjected to special curing periods, the additional handling of another material and the more complicated construction operations are factors that increase cost and may add to construction difficulties. The benefits of admixture with asphalt are not necessarily reflected by strength tests.

Cement-Treated Bases Subject to Sulfate Exposure

Up to the present time, the destructive action of sulfates on cement-treated bases has not been encountered frequently enough to be considered a serious problem in California highway construction. Although there are some localized areas in the central valleys and interior desert regions which abound with "alkali soils" (primarily containing minerals in the form of sodium and magnesium

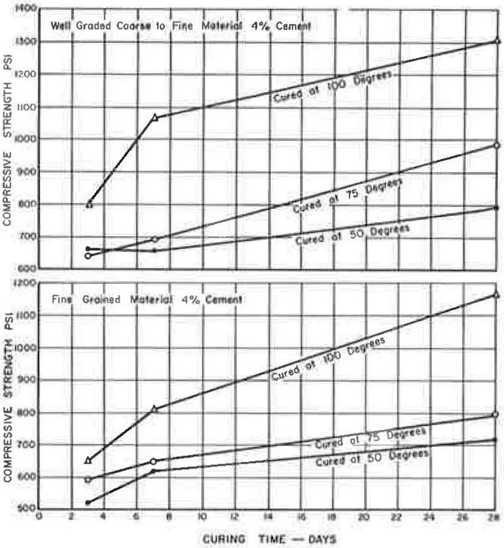


Figure 15. Comparison of compressive strengths of CTB specimens cured for 3, 7 and 28 days at 50, 75 and 100 deg.

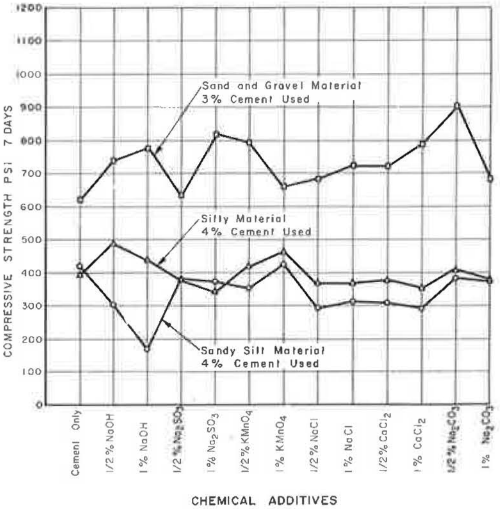


Figure 16. Compressive strength on specimens fabricated with chemical additives.

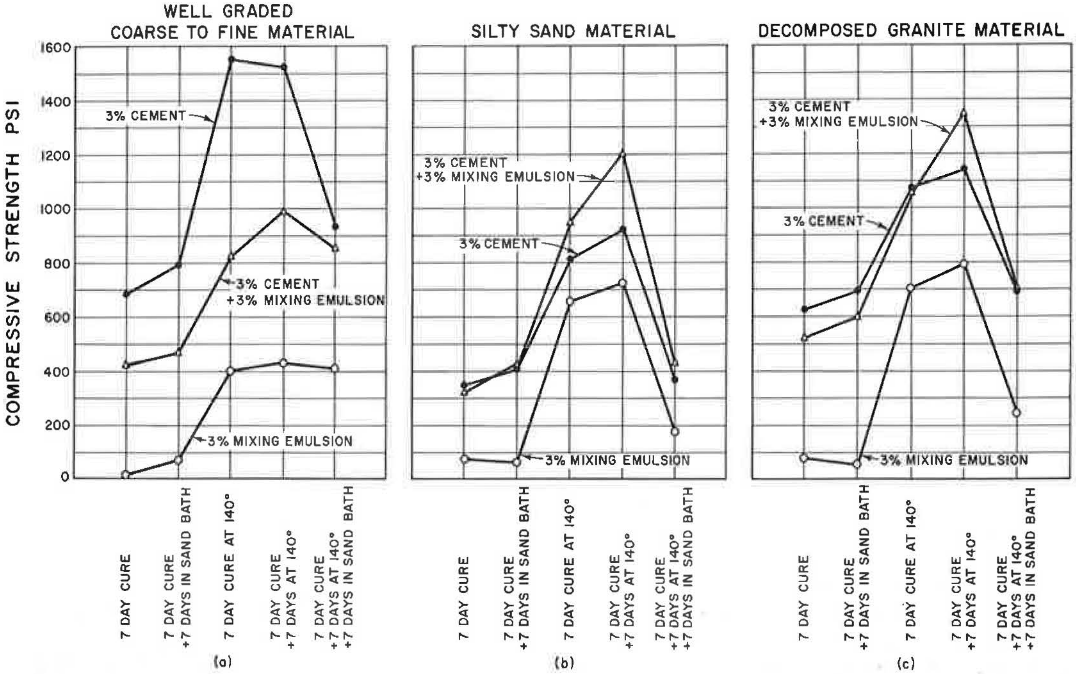


Figure 17. Effect on compressive strength of test specimens.

sulfate), these localities are generally isolated and are, for the most part, traversed by roads which carry only light to medium traffic. As a consequence only a few CTB projects have been constructed in areas where the base would be subjected to the most severe alkali attack.

However, some of these CTB projects have shown evidence of distress as a result of alkali attack. Thorough field investigations along with laboratory experiments have resulted in the determination of several important criteria which serve as guidelines for constructing more highly sulfate-resistant CTB's in the future.

First, it is most important to use a well-graded coarse granular aggregate for cement treatment. Fine-grained CTB's are fundamentally more susceptible to alkali attack. Second, Class A cement-treated bases should be used and it is preferable to increase the cement content at least 1 percent above that required in normal mix design. Sulfate resistance increases rapidly with higher cement contents. Third, a Type II or Type V, cement if readily available, should be used. However, experience indicates that this measure is not nearly as effective in CTB as it is in portland cement concrete for minimizing sulfate attack. Finally, construction compaction should be undertaken to provide the densest mixture possible in order to minimize water permeability. Also, care must be exercised during construction to prevent drainage conditions which would promote the leaching of alkalis from surrounding soils with the consequent concentration in the structural section.

Curing Seal of CTB

The present California standard specifications specify that the curing seal be applied as soon as possible, but not later than 8 hours after the completion of final rolling. The curing seal shall consist of MC-2 (now MC-250) and be applied at a rate between 0.15 and 0.25 gal per sq yd of surface.

Previous specifications permitted the use of asphaltic emulsion, either penetration or mixing type. From the construction standpoint, it appears that asphaltic emulsion is preferable as it can be applied cold and at frequent intervals whenever a certain stretch of road has been compacted. The MC-2 has to be heated for proper spray application and this can occasionally present a problem. The advantage of the MC-2,

Figure 18. Difference in penetration between asphaltic emulsion and MC-2 on cement-treated base specimens composed of coarse and fine aggregates.

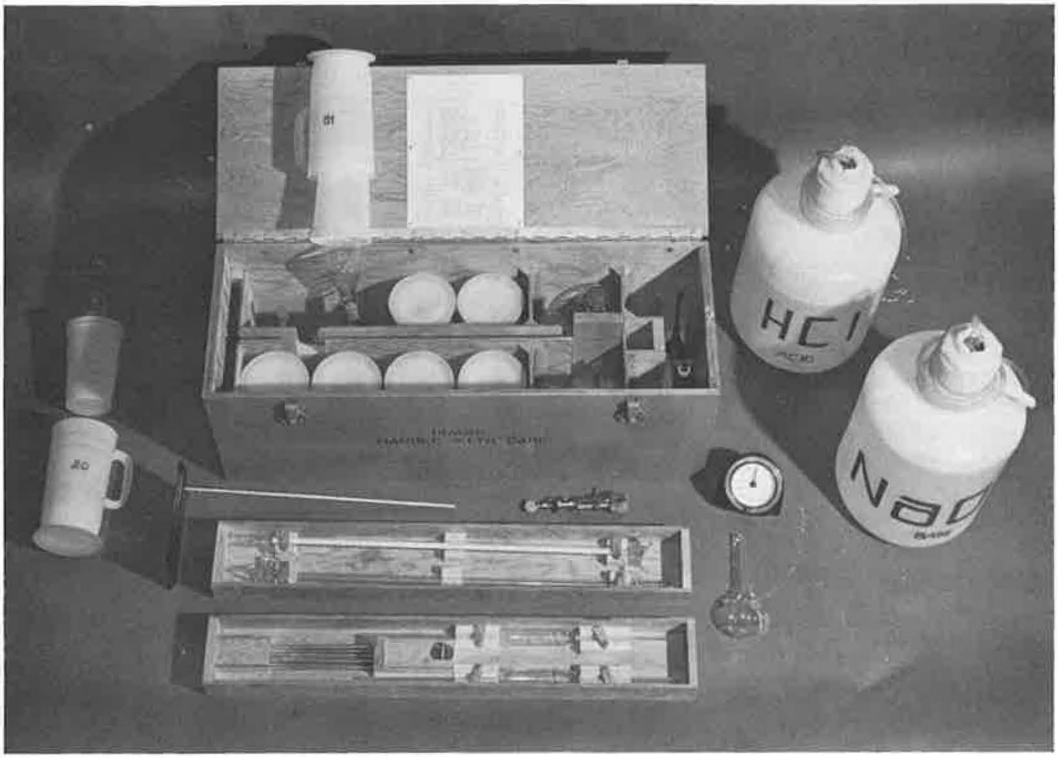


Figure 19. Field kit for titration test.

however, is that some penetration into the CTB occurs, without softening of the top crust. The emulsion, as a rule, does not penetrate into the compacted CTB and more or less covers the surface as a sheet (Fig. 18). If traffic has to be carried through the job or construction equipment is permitted to travel on the CTB, the emulsion layer will often stick to rubber tires and peel off the base. This usually is not the case with an MC-2 curing seal. In the emulsion's case, it may be necessary to apply a light sand cover to protect the curing seal. All loose sand, of course, should be removed before any surfacing is placed.

In the case of portland cement concrete pavements, any layer of asphalt placed on the cement-treated subgrade and then covered with a concrete pavement has a strong tendency to adhere tenaciously to the underside of the superimposed concrete slabs. When this happens the asphalt film will be pulled upward and leave the cement-treated subgrade without protection when the concrete slabs curl upward at the ends, as invariably occurs at some season of the year or at some time of the day.

Laboratory trials indicated that cutbacks would penetrate the average cement-treated subgrade layer to depths ranging from $1/4$ to $1/2$ in. and, therefore, even though a superficial layer of asphalt adheres to the concrete, it is expected that there will be a sufficient amount of impregnation in the cement-treated subgrade to resist erosion when water is churned back and forth by the pumping action of the slab ends.

RAPID CEMENT CONTROL TEST FOR CEMENT-TREATED BASES

There has long been a need for a rapid field test to determine the cement distribution in CTB's during construction. The trend in California toward using higher quality aggregates with lower cement contents, as well as the use of road mixing methods, made it imperative that such a test be developed. Such things as the uniformity of materials in the windrow, uniformity of cement spread in advance of mixing, poor mixing due to high speeds of the mixer, size of windrow in relation to the capacity of the mixer, as well as the mechanical condition of the mixer itself can all profoundly influence the uniformity of cement distribution and thereby the uniformity of strength in the CTB. Although central mixing plants are not subject to as many variables as road mixing, serious variations in cement distribution of the final mixture can occur as a result of malfunctioning or poorly equipped plants.

The California Division of Highways laboratory has in the past investigated several test methods, such as chemical analysis, compressive strength and electrical conductivity measurements. For various reasons, these test methods did not prove satisfactory for field control (4). One such method, developed in England, used a chemical titration process. Although not satisfied with the test as a whole, the idea of titration was pursued and a procedure (Appendix D) was developed.

Two different titration procedures are available and the selection of the particular procedure for use on a given project depends on the nature of the aggregates encountered. The first procedure, the acid-base method, is used where the aggregates do not react with hydrochloric acid. In cases where aggregates react with this acid, the second procedure, the constant neutralization method, is used.

Briefly, the acid-base method consists of placing a 300-g sample of treated aggregates (which can contain up to 1½-in. size coarse aggregates) in a plastic container and introducing a measured quantity of 3N hydrochloric acid. This is followed by a standardized stirring procedure, during which the acid neutralizes the cement contained in the sample and causes a proportional reduction in the acidity or Ph of the total solution. The reduction in acidity is determined by withdrawing a measured portion of the liquid from the plastic container, adding a few drops of phenolphthalein indicator solution to the withdrawn portion and titrating it with sodium hydroxide (NaOH). The amount of NaOH required to neutralize the residual acid is inversely proportional to the cement content of the sample.

The acid-base test is comparatively simple and results can be obtained on groups of eight samples in about 45 minutes, excluding sampling and preparation time.

The constant neutralization method is used where the CTB aggregates contain significant amounts of such substances as limestone, calcite or dolomite and can be performed on a maximum of four 300-g test specimens at one time. The specimens are placed in a plastic container to which 250-ml of water and a small quantity of phenolphthalein solution are added. The solution will immediately turn pink due to the release of hydration products from the cement. Then, using a burette containing 3N hydrochloric acid, the operator continuously adds acid and stirs the mixture for one hour to maintain a colorless solution. The amount of hydrochloric acid used in this process is determined from volume measurements with a burette or by weight. Experience has

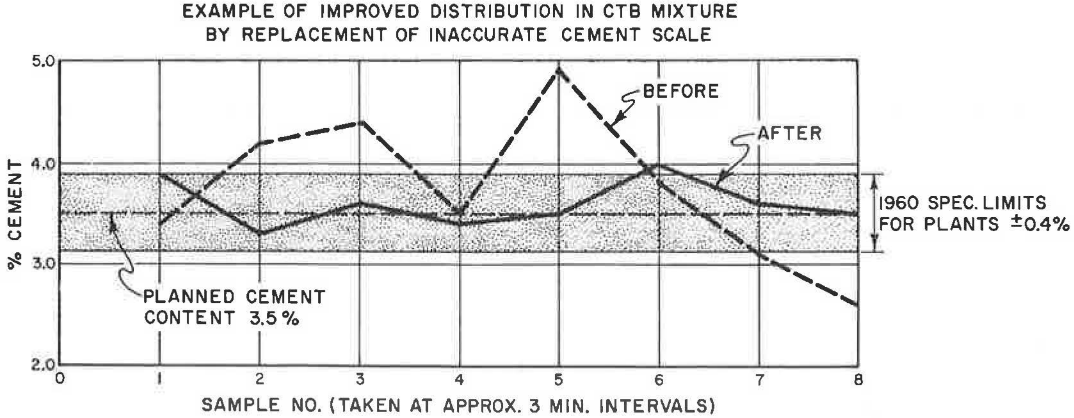


Figure 20. Project A batch plant CTB operation.

shown that the amount of acid used is directly proportional to the cement content of the treated base sample.

This method is based on the principle that the rates of hydrolysis producing the hydroxyl ion (OH-) are greatly different for cement and limestone. The hydrolysis of limestone is very slow compared to that of cement. The particle size of the limestone as compared to the particle size of the cement also retards the observed rate of hydrolysis of limestone.

The application of either of these procedures in the field during construction operations is relatively simple. At the beginning of the testing operations on the project, the operator must first establish a standard curve by testing specimens fabricated to known cement contents with samples of the cement, aggregate, and water to be used on the job. This standard curve then provides the reference for calculating the cement content from test data on field-treated samples. Incidentally, the same test procedures can also be used, without alteration, for determining the percentage of lime in bases that have been treated with commercial hydrated calcium lime.

The titration test has proven to be a very effective construction control tool. California standard specifications now limit the variation from the planned cement content to a maximum of ±0.6 percent cement (by dry weight of aggregate) for road mixing and ±0.4 percent cement for plant-mix operations. This, in combination with the fact that up to 32 samples can be tested on the project in an 8-hr day by one operator, provides the engineer with an enforceable on-the-spot means of finding and correcting deficiencies as they happen. The arrangement of the testing equipment in a convenient and compact kit form (Fig. 19) further enhances the usefulness of the methods for field application.

There are many ways in which the titration may be utilized by the engineer to disclose sources of poor cement distribution during CTB operations. The use of various sampling techniques, together with thorough and detailed knowledge of particular construction processes, often makes it possible to isolate and correct the offending element. The following three actual CTB projects demonstrate the typical role played by the titration test in the control of CTB production.

Figure 20 shows an example involving a batch-type plant-mix operation. When nonuniformity was encountered during sampling of successive batches, it was found that the cement weighing scale was out of calibration. After corrective measures a vast improvement in cement distribution was noted.

Figure 21 shows results from a continuous-mix type plant operation in which samples,

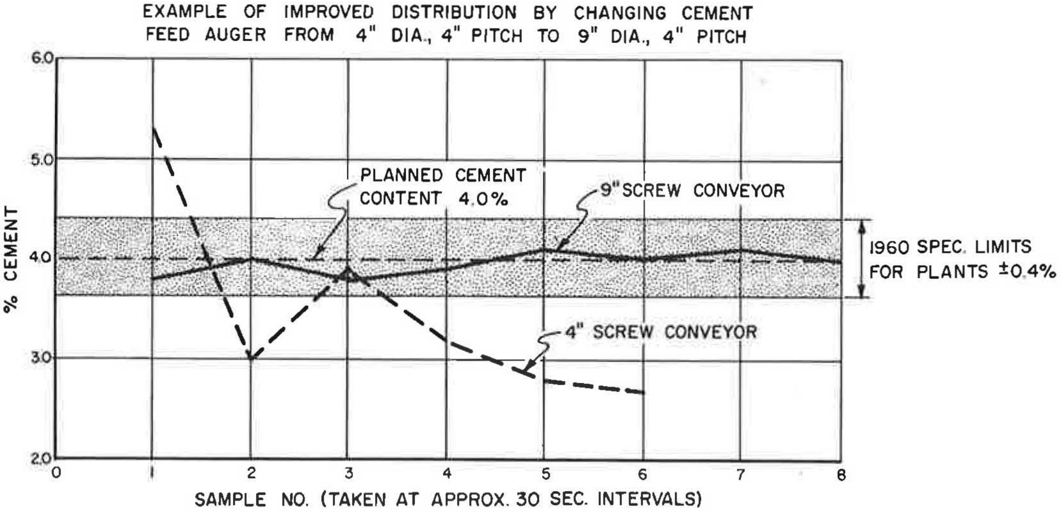


Figure 21. Project B continuous-mix CTB plant.

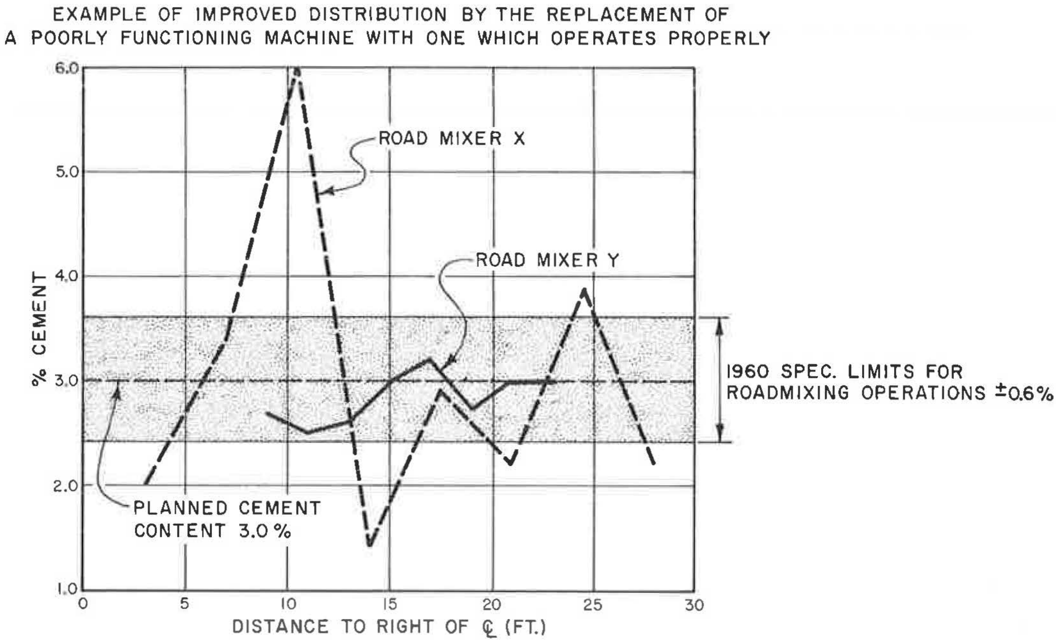


Figure 22. Project C road mixing CTB operation.

taken at approximately 30-sec intervals, indicated fluctuations in cement content from a high of about 5 percent to a low of 2.5 percent cement. The installation of a larger diameter screw in the conveyor solved this problem.

A particular road mixing operation is shown in Figure 22. Although the planned cement content was 3.0 percent, test results on samples taken transversely across the "spread out" material indicated a high of 6 percent and a low of 1.5 percent. Although the contractor was permitted to try several corrective measures, it soon became apparent that the mixing equipment was unsuitable. When another machine was used, the cement distribution became satisfactory, as indicated by Curve Y. The titration tests have been very valuable in securing cement-treated bases of consistently uniform quality.

REDUCING THICKNESS OF UNTREATED BASE BY USING CEMENT-TREATED BASE

In the California Method for determining the design thickness for flexible and composite type pavements, the cohesion or tensile strength of the various layers making up the structural section is evaluated. The design values are established from a large number of cohesiometer tests, correlation with test track data, and correlation with experience on highways.

The following design cohesiometer values are presently used:

Asphaltic concrete	400
CTB Class A	1,500
CTB Class B	750
Road-mixed surfacing	150
Soils, aggregate bases and Class C CTB	100

From original test track studies it was found that the thickness of cover is proportional to $1/\sqrt[5]{coh}$ or $1/coh^{0.2}$. It is often convenient to express the total thickness of cover required in terms of gravel equivalent. The gravel equivalent is the thickness of gravel (sand, crushed stone or other granular material) required to protect the underlying material from a given load, and is based on an assumed cohesion value of 100 for the granular cover material.

Through design relationships and cohesiometer values, the unit thickness of CTB (5) may be determined and expressed in terms of an equivalent thickness of gravel (or other granular material).

Therefore, the equation for unit gravel equivalent may be derived as follows:

Let Tg = thickness of gravel,
Cg = cohesiometer value of gravel,
Tx = thickness of other material, and
Cx = cohesiometer value of other material.

Then

$$\frac{Tg}{Tx} = \frac{\frac{1}{\sqrt[5]{Cg}}}{\frac{1}{\sqrt[5]{Cx}}} = \frac{\sqrt[5]{Cx}}{\sqrt[5]{Cg}} = \sqrt[5]{\frac{Cx}{Cg}}$$

If Tx = 1 in. and Cg = 100 (cohesiometer for untreated soils or gravel), then

$$Tg = \sqrt[5]{\frac{Cx}{100}}$$

Application of this equation to CTB and asphaltic concrete (for comparison), gives the following unit gravel equivalents:

1. Class A CTB
   $Tg = \sqrt[5]{\frac{1500}{100}}$ = 1.72 in. of gravel per in. of CTB
2. Class B CTB = 1.50 in. per in.
3. Asphaltic concrete = 1.32 in. per in.

A reduction in base thickness, from that required for untreated aggregate bases, is made when Class A or B CTB is used.

1. Class A CTB reduces the thickness of untreated base by 42 percent.
2. Class B CTB reduces the thickness of untreated base by 33 percent.

It should be pointed out that when the reduction in thickness from an untreated base layer results in a CTB thickness of less than 6 in., it is advisable, from the construction standpoint and due to variations encountered in the construction of any base, that the CTB layer be built at least 6 in. thick, and preferably not less than 8 in. when used under asphalt concrete surfacing. It was found on the test track that cement-treated bases less than 5 in. thick over a saturated subgrade are subject to early breakup if exposed to even a comparatively small number of truck repetitions.

ACKNOWLEDGMENT

The writers wish to acknowledge the assistance of Daniel Howe and Masayuki Hatano in preparing this report and collecting the data presented.

REFERENCES

1. Stanton, T.E., Hveem, F.N., and Beatty, J.L., "Progress Report on California Experience with Cement Treated Bases." HRB Proc., 23:279-296 (1943).
2. Standard Specifications, State of California (Jan. 1960).
3. Hveem, F.N., "Construction Practices on Cement-Treated Subgrades for Concrete Pavements." HRB Bull. 265, 1-7 (1960).
4. Howe, Daniel R., "A Rapid Cement Control Test for Cement Treated Bases." Third Annual Highway Conf., College of Pacific (March 1-3, 1960).

5. "Soil Stabilization with Portland Cement." HRB Bull. 292, 212 pp. (1961).
6. Test Method No. Calif. 217 (Method of Test For Sand Equivalent).

Appendix A

Test Method No. Calif. 312-B
January 3, 1956
(10 pages)

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

METHOD FOR DETERMINATION OF COMPRESSIVE STRENGTH OF CEMENT TREATED BASES, CLASSES "A" AND "B," AND CEMENT TREATED SUBGRADE

Scope

This method describes the procedure for determining compressive strength as an index of the effectiveness of cement treatment in imparting desirable properties to base and subgrade materials.

PART I. LABORATORY METHOD

Procedure

A. Apparatus

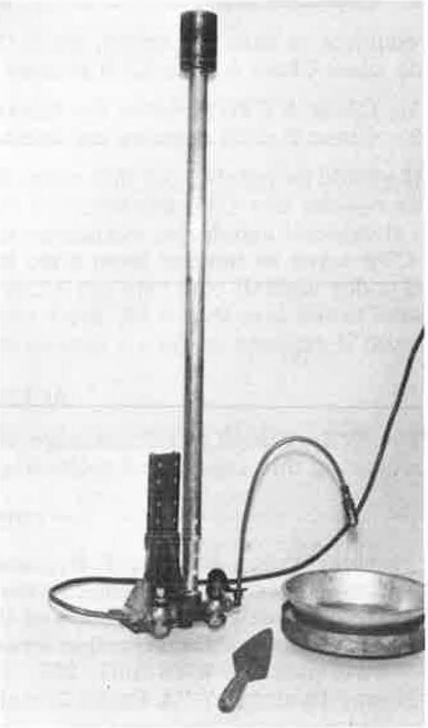
1. Drying and preheating oven thermostatically controlled to 230 ± 10 F.
2. Drying and preheating oven thermostatically controlled to 140 ± 5 F.
3. Water tank, 6 in. deep for use in submerging test specimens.
4. Balance with capacity of 5,000 g., accurate to 1 g.
5. Balance with capacity of 500 g., accurate to 0.1 g.
6. Sample splitter, riffle type, ¾-in. openings.
7. Sieves, U. S. Standard sizes, 1-in., ¾-in., ½-in., ⅜-in., No. 4, square openings.
8. Pans, 10 in. diam. x 2 in. deep.
9. Pans, 6 in. diam. x 1½ in. deep.
10. Metal scoop, No. 3.
11. Funnel weigh scoop and tare weight.
12. Testing machine, 50,000-lb. capacity.
13. Water spray metering device with turntable, Figure I.
14. Mechanical mixing machine.
15. Mixing bowl and concrete cylinder cans.
16. Large mixing spoon.
17. Mechanical compactor, kneading type (optional). See Method No. Calif. 901 for details.
18. Compaction accessories, hand method, Figure II.
19. Compaction accessories, mechanical method, Figure V.
    (The accessories for the hand compaction method can also be used for the mechanical compaction method.)
20. Mechanical device for pushing samples from mold, Figure III.
21. Measuring gauge and stand, Figure IV.

B. Test Record Form

Use work card "Laboratory Record of Cement Treatment," Form T-342, for recording test data.

C. Preparation of Sample

1. Both proper preparation and accurate quartering of test samples, in addition to use of proper testing procedure, are necessary in order to obtain accurate test results and good test reproducibility. See Test Method No. Calif. 201 for description of proper methods to use for initial sample preparation and quartering.
2. Samples submitted for cement treatment tests are divided into the following five categories:
    a. Bin samples or windrow samples.
    b. Stockpile samples.
    c. Pit or quarry samples.



   d. In-place material consisting of existing surfacing and/or base.
   e. Field compacted control samples.

3. The following methods shall be used for preparation of test samples:
   a. *Bin samples or windrow samples* are not subjected to any further processing prior to mixing with cement. Therefore, sample preparation of these samples shall consist only of separation on the coarse aggregate sieves. Since the aggregate is to be later proportioned for mixing, it will be necessary to split each sample into the following fractions:

      Passing 1-in., retained ¾-in.
      Passing ¾-in., retained ⅜-in.
      Passing ⅜-in., retained No. 4.
      Passing No. 4.

      Quartering the samples for separation into smaller fractions shall be accomplished by the methods described under Test Method No. Calif. 201. Do not obtain required amounts by scooping or pouring from containers.
   b. *Stockpile samples* shall be treated in the same manner as bin samples, provided there is to be no further processing in the field prior to addition of cement. However, if further field processing is planned, treat as described below for pit or quarry samples.
   c. *Pit or quarry samples* are processed in various manners. Normally, instructions will be furnished by the sampler as to whether crushing or screening is to be employed. If crushing is to be employed, the material shall be scalped on the sieve designated as the maximum size, and the oversize crushed to pass that maximum size sieve in such a manner that when blended back with its natural component it will conform to the grading requirements for the project. Coatings shall be removed from coated coarse aggregates, and soil lumps shall be reduced to passing No. 4 sieve size. This is necessary in order that all fines be included in test specimens for determination of amount of cement required for desired compressive strength.
   d. Treat the *''in-place'' materials* in the same manner as a pit or quarry sample if the material does not contain bitumen. ''In-place'' materials containing lumps of bituminous mix should have the lumps reduced in size to pass a 1-in. sieve and no sieve analysis is required. Quarter out required amounts for test specimens from this passing 1-in. sieve size portion.
   e. Leave *field compacted control samples* in the tin liner with ends sealed for a 6-day curing period from date of field compaction, then remove from the liner and submerge in water for 1 day to complete the curing period.
4. Weigh representative samples of coarse and fine aggregates to 0.1 g. and heat to dryness at 230 F. to determine initial moisture in aggregate.
5. From the sieve analysis of the sample or samples, design the mix to conform to the specified grading limits by blending or adjusting if necessary. Designing to a smooth grading curve approximating the middle of a specified range is desirable but not always essential. General practice is to produce the best possible grading within the specification limits with the material on hand, but any adjustment should be such that it can be duplicated under actual field conditions. All gradings shall be washed gradings.

   Whenever a larger size than 1-in. maximum is specified, the percentage of material passing the No. 4 sieve is held constant and the percentage passing the 1-in. sieve is equated to 100 percent. The intermediate sizes between the 1-in. and No. 4 are proportioned in the same ratio as the original grading.

   See Method No. Calif. 905 for methods of adjusting gradings when the ''as received'' grading is to be changed.
6. The following example illustrates a method for calculating weights of materials and moisture content for cement treated base and cement treated subgrade test specimens:

   First, make an estimation of the dry weight per cubic foot for one compacted test specimen. Assume: 130 lb. per cu. ft. for trial density, a cement content of 5 percent, 0.8 percent moisture in coarse aggregate, 1.2 percent moisture in fine aggregate and 80 g. of water added for proper consistency. From attached table of weights (Figure VI), select values opposite 130 lb. per cu. ft. and arrange as follows:

Weight per cu. ft.	*Grams of cement and aggregate*	*Grams of cement*
130	1,716	82

Weight of aggregate = 1,716 − 82 = 1,634 g.

Assume grading of sample as follows:

Percent passing	*Percent passing*
1 in. 100	⅜ in. 80
¾ in. 96	No. 4 60

Then from the grading analysis of the sample arrange cumulative percentages of coarse aggregates and percentage of fine aggregates as follows:

Sieve size	*Percent passing*	*Percent retained*
1 in.	100	0
¾ in.	96	4
⅜ in.	80	20
No. 4	60	40

Cumulative weights of aggregates, cement and moisture for one 4 in. × 4 in. compacted specimen are as follows:

	Cumulative dry weights in grams	Cumulative moisture correction	Cumulative corrected weights in grams
Ret. ¾ in.	.04 × 1,634 = 65	.008 × 65 = 1	66
Ret. ⅜ in.	.20 × 1,634 = 327	.008 × 327 = 3	330
Ret. No. 4	.40 × 1,634 = 654	.008 × 654 = 5	659
Pass. No. 4	1.00 × 1,634 = 1,634	5 + .012 (1,634 − 654) = 17	1,651
Cement	1.05 × 1,634 = 1,716	17	1,733
Water		1,716 + 17 + 80	1,813

$$\text{Total moisture in sample} = \frac{17 + 80}{1{,}716} = .057 \text{ or } 5.7 \text{ percent}$$

D. Determining Optimum Moisture

1. When combinations of various sized particles of mineral aggregate and a constant weight proportion of cement are mixed with different quantities of water and then compacted by identical methods, the use of one certain moisture content will usually result in a greater density (as indicated by the compacted dry weight of material for a given volume) than will be the case using any other moisture content for the particular material under consideration. The amount of water thus required for maximum compaction, expressed as percent of the dry weight of the material, is commonly referred to as the "optimum moisture content" for that combination of aggregates and cement.

2. If no previous data on the soil or aggregates in question is available, a trial initial moisture content may be estimated from the appearance of the soil or from its sieve analysis. Usually, tests are started with a moisture content below the expected optimum. As a rule, an initial moisture of 3 percent to 7 percent, depending upon the type of material, will give a good starting point.

3. An estimation is made of the weight of dry material required to fabricate the 4 in. x 4 in. test specimen. This estimate is based upon the type of material to be used for the test. For simplicity, the several different types of materials may be classified according to their densities or compacted dry weights per cubic foot, which normally ranges as follows:

Volcanic tuff	70 to 100 lb. per cu. ft.
Fine sand	110 to 120 lb. per cu. ft.
Silty sand or sandy soil	120 to 128 lb. per cu. ft.
Clayey silt or fine grained soil	125 to 135 lb. per cu. ft.
Decomposed granite	128 to 132 lb. per cu. ft.
Coarse to fine or well graded material	130 to 145 lb. per cu. ft.

This classification is given merely as a guide for the operator in selecting proper weights to begin the tests. However, some materials may produce densities higher or lower than those shown.

4. The following formula may be used for determining the total combined weights of aggregate and cement that are required for the fabrication of 4 in. x 4 in. test specimens of various weights per cubic foot:

$$W_g = \frac{W_f H}{.303}$$

Where:

W_g = Dry weight in grams of 4 in. × 4 in. compacted test specimen.

W_f = Dry weight in lbs. per cu. ft. of compacted test specimen.

H = Height of test specimen in inches.

$.303$ = Constant used to convert weight in grams to weight in lbs. per cu. ft. for a 4-in. diam. specimen having a height H.

Example:

Assume a weight of 107 lb. per cu. ft. for a trial density and a cement content of 2 percent. Substituting in above formula,

$$W_g = \frac{107 \times 4}{.303} = 1412 \text{ g. of cement and aggregate}$$

$$\frac{1412}{100 + 2} \times 100 = 1384 \text{ g. of aggregate}$$

$$1412 - 1384 = 28 \text{ g. of cement}$$

5. In order to simplify the procedure for calculating the amount of aggregate and cement to be used in fabricating one 4 in. x 4 in. test specimen, a table is furnished (Figure VI), for reference. This table gives dry weights of materials in grams required to produce one 4 in. x 4 in. test specimen with cement content varying from 2 percent to 8 percent by weight, and densities varying from 107 to 150 lb. per cu. ft. If quantities of material are needed to make specimens with a density lower than 107 or higher than 150 lb. per cu. ft. the above formula must be used.

6. For the trial or pilot specimen, add water to the mixture in small increments up to a point where visual inspection and hand squeezing of small amounts of the mixture indicate sufficient water to provide good cohesion between the cement and aggregates. For this trial mix, attempt to produce a specimen of 4.000 ± .200 in. in height where 8 to 10 g. of water are squeezed out when the specimen is compressed with the specified static load. After the trial specimen is made, it may be necessary to make adjustments both of the amount of material needed to fabricate one specimen 4 in. high and of the proper amount of water needed to get 8 to 10 g. squeezed out.
7. In determining the optimum moisture, fabricate three test specimens with different moisture contents. For most materials the moisture increment is about 1 percent, but for absorptive materials the moisture increment may be increased to 2 percent. The ideal situation is reached when one of the specimens is at the point of saturation, another is slightly above the point of saturation, (8 to 10 g. of water exuded under the static load), and the third specimen is slightly below the saturation point.
8. The specimens fabricated to determine the optimum moisture are cured and tested for compressive strength.
9. Highest density is usually attained in cement treated base and cement treated subgrade test specimens if a small amount of water is exuded from the specimen when subjected to a static load. From the data obtained in the fabrication of the three specimens, calculate the amount of moisture required to produce maximum density. This amount of moisture expressed as percent of the dry weight of material is known as the optimum moisture.
10. Using the data obtained from the fabrication of the optimum moisture specimens, fabricate three additional specimens in which the cement content is varied (usually 2 percent increments for cement treated bases and 1 percent increments for cement treated subgrades) and the moisture content is held constant at or as near the predetermined optimum moisture as possible.
11. These three additional specimens are for compressive strength determinations and are for the purpose of determining the necessary amount of cement to provide a specified or desired strength under optimum moisture conditions.
12. Fabricate any additional test specimens necessary for special tests such as wetting and drying, or freezing and thawing, in the same manner.
13. Mix the individual test specimens in the following manner:
    a. Mix together the proper proportion of aggregates and cement prior to adding water. After the dry ingredients are thoroughly mixed, add the required amount of water and continue mixing until all of the aggregates are coated.
    b. Any mechanical mixer which will produce a homogeneous mix may be used, or the composite materials may be mixed by spoon and mixing bowl method.
    c. After mixing, place the aggregate-cement-water mixture in a can and cover with a tight fitting lid for a period of 30 min. before compacting the individual test specimens.

E. Compaction of the Test Specimens

The following descriptions cover two methods of compacting cement-treated test specimens. Method "A" covers the hand compaction procedure, and Method "B" covers an alternate procedure of compacting with a mechanical compactor.

It is not intended that the mechanical method of compacting strength test specimens is to replace or do away with the present hand method, it simply provides for an alternate method whereby the kneading compactor may be used whenever it is available for such work.

1. Method "A"—hand compaction.
    a. Assemble the component parts of the compaction mold, Figure II. Insert the long expansion liner, marked No. 2, in steel mold No. 3 so that ends are flush. Insert the tin liner No. 1 from opposite end of mold until butted against expansion liner No. 2; follow the tin liner with the short expansion liner

No. 4, and insert bottom plunger No. 5 and extension sleeve No. 6. Insert the U-shaped spacer No. 7 between bottom of mold and bottom plunger. A set of these spacers should be available with thicknesses of $\frac{1}{16}$ in., $\frac{1}{8}$ in. and $\frac{3}{16}$ in. Use the thinner spacers when compacting granular materials, and use the thickest one when compacting plastic materials. Remove the spacer on completion of the hand compaction, and the space provided by its use results in a double plunger effect under the static load. Place the assembled mold on a solid foundation during compaction.

b. Pour approximately one-half of the prepared sample in the mold. If the material contains rock particles larger than $\frac{1}{4}$-in., rod 20 to 30 times with a $\frac{3}{8}$-in. bullet shaped rod (part No. 9 in Figure II), while pouring in mold to prevent rock pockets forming at the bottom or sides of the specimen. Tamp the first layer of material with 50 blows using the small end of the 6-lb. hand tamper (part No. 10 in Figure II). Physical exertion in tamping should be only sufficient to move the tamper up and down in approximately a 4-in. travel. Guide the tamper over the entire surface of the specimen. The actual compactive effort should be provided only by the combined weights of the tamper and the operator's hand. At the end of the 50 blows the tamper should be penetrating into the specimen about $\frac{1}{2}$ in. Avoid having a smoothly compacted surface at this stage because it will result in a compaction plane in the specimen when the next layer is tamped, and this would prevent the two layers from being bonded together.

c. Place the remaining portion of the sample in the mold (rodded, if sample contains coarse aggregate), and tamp, using 100 blows with the small end of the hand tamper. Level off the top of the compacted specimen by tamping lightly with the large end of the tamper in order to provide a smooth surface on an even plane at right angles to the axis of the mold. Remove the extension sleeve and the spacer, insert the follower (part No. 8 in Figure II), and place the assembly in the compression machine. Gradually apply a total load of 25,000 lb., using 1 min. to attain the first 20,000 lb. and one-half min. for the next 5,000 lb. Hold the total load of 25,000 lb. for 1 min. before releasing.

d. Push the compacted specimen, in its tin jacket, from the mold by means of a suitable device. A mechanical device for this purpose is shown in Figure III. Immediately weigh the specimen and tin jacket.

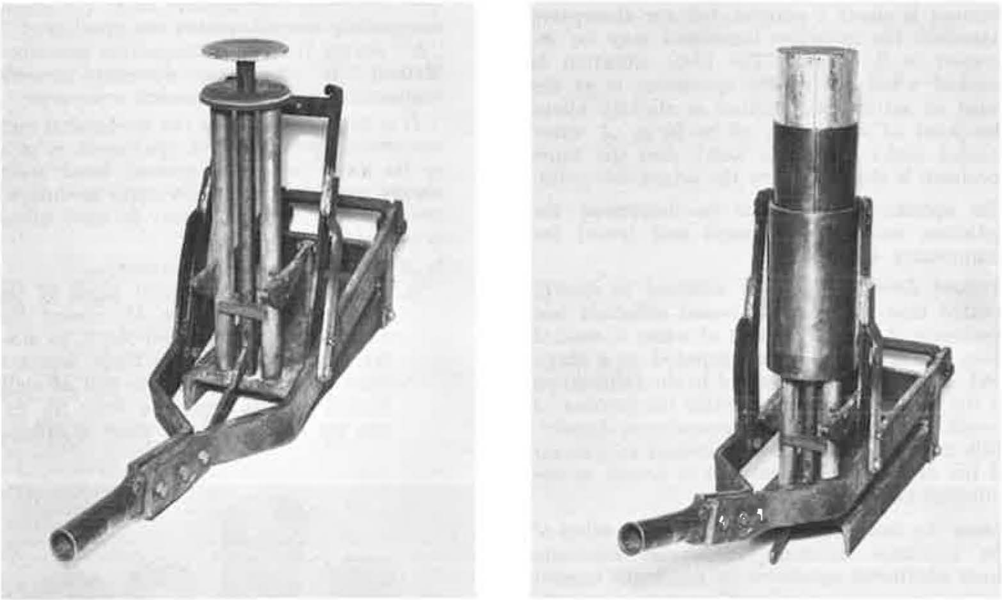


FIGURE III

Measure the height with the measuring gauge, Figure IV, by taking several readings half way between the center and edge of the specimen and computing the average of these measured lengths.

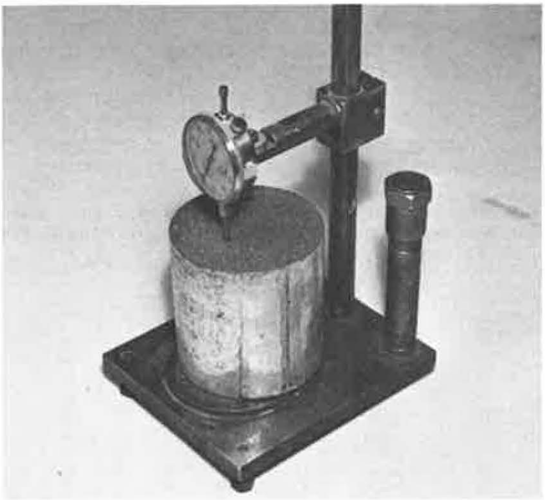


FIGURE IV

e. From the determined amount of moisture in the specimen, calculate the dry weight of the specimen. Knowing the dry weight and the measured height of the specimen, the equivalent compacted dry weight in lb. per cubic foot can be obtained from the table, Figure VI. (If the height is less than 3.988 in. or more than 4.012 in., the formula shown in Section D, Paragraph 4, must be used instead of the table.)

f. Properly mark the test specimen for identification purposes, and the specimen is ready for curing prior to testing.

g. If the specimen is to be tested for compressive strength only, the identification number can be marked on the side of the specimen with a grease pencil. However, if the specimen is to be tested for "wetting and drying" or "freezing and thawing" there is danger of losing such identification due to sloughing; therefore, it is advisable to attach a small numbered brass or copper washer to the top of the specimen by means of a brass screw.

2. Method "B"—mechanical compaction

   a. Refer to Method No. Calif. 901 for method of operation and calibration of the mechanical compactor.

   b. Assemble the component parts of the compaction mold, Figure V. Insert the long expansion liner, marked (B) in steel mold (A) so that ends are flush. Insert the tin liner (C) from opposite end of mold until it butts against expansion liner (B). Follow tin liner (C) with the short expansion liner (D) and insert bottom plunger (E).

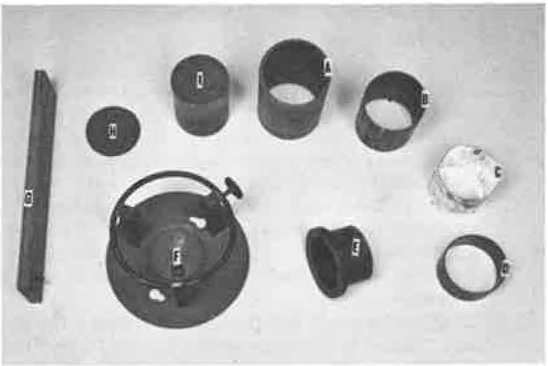


FIGURE V

   c. Place the assembled mold in mold holder (F), and adjust mold vertically to provide approximately $\frac{1}{8}$ in. clearance between lower edge of mold and the base plate of the mold holder. Clamp mold in place, position the assembly on compactor turntable, and lock it on the studs provided.

   d. Place the wood shim (G) under metal feeder trough to provide the additional height necessitated by the longer compaction mold.

   e. Place mixed sample in the feeder trough, and distribute the loose material uniformly along the full length of the trough.

   f. Start compactor and adjust the air pressure to 15 psi gauge reading.

   g. Use a spatula, formed to fit the inside of the feeder trough, and push the lower 3 in. of material from the trough into the mold. With the compactor still in motion, push the remainder of the sample into the mold in 30 equal parts; push one part into the mold with each blow of the compactor foot. After all the material is in the mold, add 10 additional blows to level and seat the material.

   h. Increase air pressure to a previously determined gauge reading that will provide a compactor foot pressure of 350 psi, and place the rubber disk (H) on top of the partially compacted specimen.

   i. Apply 100 tamps to the specimen.

   j. Immediately upon completion of the 100 tamps, remove the rubber disk and insert the follower (I) into the mold.

   k. Place the mold holder containing the mold and test specimen in the testing machine and slowly apply a static load of 1,000 psi (12,500 lbs. total load) to the specimen at a rate requiring $1\frac{1}{2}$ min. to reach the maximum. Hold the load for 1 min. before releasing.

l. The test specimen is now ready to be pushed out of the mold and cured for the compressive strength test.

F. Curing Test Specimens

1. Store or seal all test specimens in such a manner after compaction that no moisture is lost from the specimens during the curing period. This can be accomplished by curing specimens in a moist cabinet, by covering specimens with wet burlap, or by placing lids on each end of the tin sleeve and sealing with adhesive tape.
2. Cure all test specimens for six days. Then remove the tin sleeves and use a wax pencil to write proper identification on the side of the specimens.
3. Next, submerge the specimens in water for one day. This concludes the seven-day curing period, and the specimens are ready to be tested for compressive strength.

G. Testing for Compressive Strength

1. Remove test specimens from the soaking tank and dry the surfaces of the specimen with a cloth.
2. For each specimen, grease two 6 in. x 6 in. glass plates on one side using ordinary lubricating oil. Arrange the glass plates in a double row on a table and place the surface dried test specimens in a row between the two rows of greased plates.
3. Mix enough plaster of Paris with water to form a thick paste sufficient in quantity to cap approximately six specimens, top and bottom.
4. Place an amount of the paste equivalent to a large tablespoonful on top of each of the test specimens and on each of the glass plates in the row nearest the operator.
5. Place the outer row of glass plates that have no paste on them on the top surface of the specimens containing the plaster paste. Force the plates down until the paste covers the entire surface of the specimens. Then, place the specimens with top plates in place on the row of plates that have paste on them, and press the specimens down until the paste covers the entire area of the bottom of the specimens. Adjust the specimens while plaster is still soft so that top and bottom plates are as nearly as possible at right angles to the vertical axis of test specimen.
6. Allow specimens to stand for a period of 30 to 40 minutes to permit hardening of the plaster. Remove the glass plates by tapping the edges lightly with a piece of soft wood. If difficulty is experienced in removing the plates, apply warm water and continue tapping lightly.
7. The specimens are now ready to be placed in the testing machine for compressive strength tests.
   a. If mechanical testing machine is used, the travel of the head shall be at the rate of 0.05 in. per min.
   b. If hydraulic testing machine is used, apply the load at the rate of between 20 and 50 lb. per square inch per second. Ideal rate of loading on a 4-in. diameter specimen for the hydraulic testing machine is 2,200 lb. total load in 5 seconds.
8. Apply the load until ultimate fracture of the test cylinder occurs. An initial fracture will usually occur at approximately 80 percent of the load required for ultimate fracture.

FIGURE VI

TABLE OF WEIGHTS FOR USE IN FABRICATING 4-IN. DIAM. X 4-IN. HIGH TEST SPECIMENS OF VARIOUS WEIGHTS PER CU. FT.

Wt. lbs. cu. ft.	Total grams cement + agg.	Grams cement						
		2 percent	3 percent	4 percent	5 percent	6 percent	7 percent	8 percent
107	1,412	28	41	54	67	80	92	105
108	1,426	28	42	55	68	81	93	106
109	1,439	28	42	55	69	82	94	107
110	1,452	29	42	56	69	82	95	108
111	1,465	29	43	56	70	83	96	109
112	1,478	29	43	57	70	84	97	109
113	1,492	29	44	57	71	85	98	110
114	1,505	30	44	58	72	85	99	111
115	1,518	30	44	58	72	86	99	112
116	1,531	30	45	59	73	87	100	113
117	1,544	30	45	59	74	87	101	114
118	1,558	31	45	60	74	88	102	115
119	1,571	31	46	60	75	89	103	116
120	1,584	31	46	61	75	90	104	117
121	1,597	31	47	61	76	90	105	118
122	1,610	32	47	62	77	91	105	119
123	1,623	32	47	62	77	92	106	120
124	1,637	32	48	63	78	93	107	121
125	1,650	32	48	64	79	93	108	122
126	1,663	33	49	64	79	94	109	123
127	1,676	33	49	65	80	95	110	124
128	1,690	33	49	65	81	96	111	125
129	1,703	33	50	66	81	96	111	126
130	1,716	34	50	66	82	97	112	127
131	1,729	34	50	67	82	98	113	128
132	1,742	34	51	67	83	99	114	129
133	1,756	35	51	68	84	99	115	130
134	1,769	35	52	68	84	100	116	131
135	1,782	35	52	69	85	101	117	132
136	1,795	35	52	69	86	102	118	133
137	1,808	36	53	70	86	102	118	134
138	1,822	36	53	70	87	103	119	135
139	1,835	36	53	71	87	104	120	136
140	1,848	36	54	71	88	105	121	137
141	1,861	37	54	72	89	105	122	138
142	1,874	37	55	72	89	106	123	139
143	1,888	37	55	73	90	107	124	140
144	1,901	37	55	73	91	108	124	141
145	1,914	38	56	74	91	108	125	142
146	1,927	38	56	74	92	109	126	143
147	1,940	38	57	75	92	110	127	144
148	1,954	38	57	75	93	111	128	145
149	1,967	39	57	76	94	111	129	146
150	1,980	39	58	76	94	112	130	147

To obtain weight of aggregate subtract weight of cement from total weight of cement and aggregate.

9. Report the test results as compressive strength in pounds per square inch which equals the total compression load divided by the end area of the test cylinder.

   In the standard 4-in. test cylinder the end area is 12.57 sq. in. An optional method is to multiply the total compression load by .080, in lieu of dividing total load by 12.57.

Test Method No. Calif. 312-B

January 3, 1956

Reporting of Results

Report the test results on Test Report Form T-346. Include grading used, compressive strength, and recommended moisture and cement contents.

PART II. FIELD METHOD

A. Apparatus

1. Balance with capacity of 5,000 g., accurate to 1 g.
2. Split compaction mold, 4-in. diam. x 11.5 in. as shown in Figure VII.

HYDRAULIC COMPACTION APPARATUS

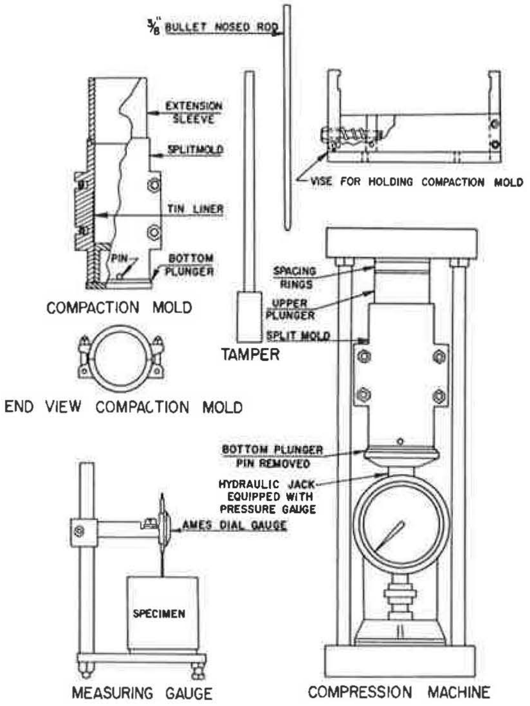


FIGURE VII

3. One compression machine consisting of a 12-20-ton capacity hydraulic jack fitted with a spherically seated head and mounted in a 30-in. frame.
4. Tamper, rod, tin liners and bottom and upper plunger as shown in Figure II (Nos. 1, 5, 8, 9 and 10).
5. Measuring gauge and stand as shown in Figures IV and VII.
6. Special bench vise for holding compaction mold as shown in Figure VII.
7. 6 in. x 12 in. concrete cylinder cans with lids.
8. Two 6 in. x 6 in. glass plates for each specimen.
9. Miscellaneous items such as spoons, spatulas, scoops, scotch tape, etc.

B. Materials

1. A supply of gypsum casting plaster.
2. Cartons for shipping test specimens.
3. Supply of ½-in. adhesive tape.

C. Procedure for the Fabrication of Test Specimens

1. Obtain representative samples of the freshly mixed materials daily. Normally two samples should be taken, one from the mixing plant and one from the street immediately ahead of rolling operations. To protect against the loss of moisture, place all samples in 6 in. x 12 in. concrete cylinder cans and immediately cover.
2. Transport the cans to the point of fabrication and fabricate samples with the least possible delay. Protect against loss of moisture at all times.
3. Immediately upon arrival at the point of fabrication, remix the sample and screen through a 1-in. sieve. Only the minus 1-in. material is used in making test specimens.
4. Quarter out the approximate amounts required for the moisture determination and test specimen. The amount of material for moisture samples should be approximately 1,000 g. The amount of material needed for fabricating a test specimen is shown in the attached Figure VI.

   It is of extreme importance that test specimens be fabricated as soon as possible after the mixing process. The hydration of the cement can cause a serious loss of compressive strength as well as a reduction in the density of the test specimen.
5. Weigh the material for both moisture samples and test specimens to the nearest gram, and weigh as rapidly as possible to avoid loss of moisture. Ordinary baking pans, approximately 9 in. x 5 in. x 4 in., make convenient weighing pans. Protect all material for test specimens against loss of moisture after weighing, and do not delay between weighing and fabricating.
6. Assemble the 4-in. mold with the tin liner in place and the plunger held one space from the bottom by means of the pin. (For some soils it may be necessary to hold the bottom plunger further from the end of the mold in order to prevent the rim of the mold from coming in contact with the shoulder of the plunger before compaction is completed. In such cases, insert the pin through successively higher holes until satisfactory results are secured.)
7. Place the extension sleeve on top of the mold and add approximately half of the weighed sample with a scoop or large spoon. If the material contains rock particles larger than ¼ in., rod 20 to 30 times with a ⅜-in. bullet-nosed rod

during this operation in order to prevent the formation of rock pockets at the bottom or sides of the specimen. Tamp the first layer of material 50 blows with the *small end* of the 6-lb. hand tamper. Physical exertion in tamping should be only sufficient to move the tamper up and down in approximately a 4-in. travel. Guide the tamper over the entire surface of the specimen. The actual compactive effort should be provided only by the combined weights of the tamper and the operator's hand. Avoid a smoothly compacted surface because it will result in a compaction plane in the specimen when the next layer is tamped, and will prevent the two layers from bonding.

8. Place the remaining portion of the sample in the mold and rod if sample contains coarse aggregate. It is not necessary to rod fine material such as sand. Tamp the second layer using 100 blows with the *small end* of the hand tamper. Level off the top of the compacted specimen by tamping lightly with the *large end* of the tamper in order to provide a smooth surface on an even plane at right angles to the axis of the mold. After tamping is completed, remove the extension sleeve.
9. Place the top plunger in position, then place the entire assembly on the hydraulic jack in the compression frame. If necessary, place one or more of the spacing rings between the top plunger and the top of the frame to prevent excessive travel of the jack. *Remove the pin that holds the bottom plunger in place* and gradually apply a total load of 25,000 lb.; use 1 min. to attain the first 20,000 lb., $\frac{1}{2}$ min. for the next 5,000 lb. and hold the 25,000-lb. load for 1 min. Then release the load, place the mold in the vise, take out the plungers, open the mold, remove the specimen with its tin jacket and weigh.
10. Determine the height with the measuring gauge by taking several readings half way between the center and edge of the specimen and computing the average of these measured lengths. The height measurement is illustrated in Figure VII.
11. If water is squeezed out of the specimen during compaction, a notation should be made of the amount as a guide in maintaining optimum moisture control in the field. This is done by taking the difference between the net weight of the specimen and the net weight of the sample used. Headquarters laboratory will normally make optimum moisture determinations on preliminary samples which may serve to compare with field results. However, in applying optimum moisture control in the field, consideration must be given to evaporation losses in mixing and handling and allowances made accordingly.
12. Determine the moisture content of the sample by weighing the material set aside for this purpose (approx. 1,000 g.) to the nearest one (1) gram and drying in a 230-F. oven to constant weight.

    Percent moisture =

    $$\frac{\text{Wet weight} - \text{dry weight}}{\text{Dry weight}} \times 100$$
13. After determining the height and weight of the compacted specimen, print the proper identifying marks on the side of the tin liner with a wax pencil. Place tin caps on each end and seal with masking tape.
14. Cure the specimens for two days in a cool place and then ship the test specimens to the district laboratory for testing.

D. Procedure for the Determination of Compressive Strength in the Field

1. If it is necessary that compressive strengths be determined on the job, the test specimens should be cured for six days. The curing shall be accomplished by placing lids on each end of the tin sleeve, sealing with adhesive tape and then storing in a cool place such as the shady side of a building. However, during cold weather, the test specimens should be protected from freezing. At the end of the six-day curing period, remove the caps and liners and transfer the identifying marks to the side of the specimen with a wax pencil.
2. Immerse the specimens in water for one day in order to complete the seven-day curing period required for the compressive strength test.
3. Remove the specimens from the water bath, wipe the surfaces with a dry rag and cap both ends of each specimen with gypsum casting plaster as follows:
    - a. Select two glass plates, approximately 6 in. x 6 in., for each specimen and lay them out on a table or bench.
    - b. Oil the top of each glass with common motor lubricant.
    - c. Measure out into a suitable container about a cup of gypsum casting plaster for each specimen to be capped.
    - d. Add water and mix to a fairly thick paste.
    - e. Place a tablespoonful of paste on top of each test specimen and immediately force one of the plates down on the paste on top of each specimen to form full caps.
    - f. Place a tablespoonful of paste on each of the other glass plates and press each of the specimens firmly on a glass plate so as to form full caps.
    - g. Allow the caps to harden for a minimum of 30 minutes and then remove the glass plates by tapping the edges lightly with a piece of soft wood. If difficulty is experienced in removing the plates, apply warm water and continue tapping lightly.

4. The specimen may be tested for compressive strength as soon as the glass plates are removed. Center the specimen on the head of the hydraulic jack in the compression machine and apply the load at the rate of between 20 and 50 lb. per square inch per second. A convenient rate of loading when using the compression machine on a 4-in. diameter test specimen is 30,000 lb. per minute. This allows easy conversion in the field to desired load-time units (i.e. 500 lb. per second). Apply the load until ultimate fracture of the test cylinder occurs. An initial fracture will usually occur at approximately 80 percent of the load required for ultimate fracture.
5. Report the test results as compressive strength in pounds per square inch which equals the total compression load divided by the end area of the 4-in. diameter test specimen (12.57 sq. in.). Fig. VIII lists the unit compressive stress in increments of 500 lb. total load.

FIGURE VIII

TABLE OF UNIT COMPRESSIVE STRENGTH

4-in. Diameter Cement Treated Base Specimen

Total load (lbs.)	Compressive strength (psi)	Total load (lbs.)	Compressive strength (psi)	Total load (lbs.)	Compressive strength (psi)
500	40	10,500	840	20,500	1,640
1,000	80	11,000	880	21,000	1,680
1,500	120	11,500	920	21,500	1,720
2,000	160	12,000	960	22,000	1,760
2,500	200	12,500	1,000	22,500	1,800
3,000	240	13,000	1,040	23,000	1,840
3,500	280	13,500	1,080	23,500	1,880
4,000	320	14,000	1,120	24,000	1,920
4,500	360	14,500	1,160	24,500	1,960
5,000	400	15,000	1,200	25,000	2,000
5,500	440	15,500	1,240	25,500	2,040
6,000	480	16,000	1,280	26,000	2,080
6,500	520	16,500	1,320	26,500	2,120
7,000	560	17,000	1,360	27,000	2,160
7,500	600	17,500	1,400	27,500	2,200
8,000	640	18,000	1,440	28,000	2,240
8,500	680	18,500	1,480	28,500	2,280
9,000	720	19,000	1,520	29,000	2,320
9,500	760	19,500	1,560	29,500	2,360
10,000	800	20,000	1,600	30,000	2,400

Procedure for Determining the Density of Test Specimens

The density of the test specimen, based on the dry weight of material, is calculated from the data obtained in the "Procedure for the Fabrication of Test Specimens" using the following formula:

$$D = \frac{30.3W_w}{(100 + M)H}$$

Where:

D = Dry density of the test specimen in lb. per cu. ft.

W_w = Wet weight of the test specimen in grams.

M = Percent moisture of the sample.

H = Height of the test specimen in inches.

30.3 = Constant used to convert weight in grams to lb. per cu. ft. for a 4-in. diameter specimen having the height measured in inches.

REFERENCES

Test Method No. Calif. 201
Method No. Calif. 901
Method No. Calif. 905

End of Text on Calif. 312-B

Test Method No. Calif. 313-B
January 3, 1956
(1 page)

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

METHOD OF FREEZING AND THAWING TEST FOR COMPACTED TEST SPECIMENS OF CEMENT TREATED BASES, CLASSES "A" AND "B," AND CEMENT TREATED SUBGRADE

Scope

This method, which is a modification of A. A. S. H. O. Designation: T 136, is intended for determining visual physical condition, volume change (swell), and compressive strength after the compacted test specimens have been subjected to repeated cycles of alternate freezing and thawing.

Procedure

A. Apparatus

1. Freezer or refrigerator capable of maintaining a temperature of minus 10 F. or lower.
2. All of the apparatus specified for Test Method No. Calif. 312.

B. Test Record Form

Record the test data on work card, Form T-342.

C. Preparation of Test Specimen

Prepare and compact the specimen, using optimum moisture and desired amount of cement, in accordance with Test Method No. Calif. 312.

D. Curing Test Specimen

1. Store or seal test specimen in such a manner that no moisture is lost from the specimen during the curing period. This can be accomplished by curing specimen in a moist cabinet, by covering specimen with wet burlap, or by placing lids on each end of the tin sleeve and sealing with adhesive tape.
2. Cure test specimen for seven days. Remove the tin sleeves and make sure that the numbered identification disk is attached to the top of the specimen.

E. Freezing and Thawing Cycles

Place the specimen in a freezer or refrigerator having a constant temperature not warmer than minus 10 F. for 22 hr., then remove, surface dry, weigh (to detect any sloughing), and measure for height. Place in a moist cabinet and allow to thaw for 22 hr., remove, and again weigh and measure. This constitutes one cycle of freezing and thawing. Continue this freezing and thawing process until 12 cycles have been completed, provided, of course, that serious disintegration of the specimen does not occur sooner.

F. Test Calculations

1. If the specimen has increased in length after completion of the 12 cycles of alternate freezing and thawing, calculate the volume change as percent of original volume. If the specimen has decreased in length, consider the volume change zero.

   The formula for volume change is:

   $$\text{Percent volume change} = \frac{V_2 - V_1}{V_1} \times 100$$

   V_1 = Volume of specimen at beginning of cycle period
   V_2 = Volume of specimen at end of cycle period

   Example:

	Height of specimen	*Diameter of specimen*
Before cycle period	4.092 in.	4.000 in.
After cycle period	4.097 in.	4.005 in.

   $V_1 = (2)^2 \times 3.1416 \times 4.092 = 51.421$ cu. in.
   $V_2 = (2.0025)^2 \times 3.1416 \times 4.097$
   $= 51.614$ cu. in.

   $$\text{Volume change} = \frac{51.614 - 51.421}{51.421} \times 100$$

   $= 0.4$ percent

2. Examine the specimen visually for physical condition, and report as excellent, good, fair or poor.
3. Then submerge the specimen in water for one day and test for compressive strength as specified in Test Method No. Calif. 312.

Reporting of Results

Report the test results on Test Report, Form T-346.

REFERENCES

A. A. S. H. O. Designation: T 136
Test Method No. Calif. 312

End of Text on Calif. 313-B

Test Method No. Calif. 314-B
January 3, 1956
(1 page)

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

METHOD OF WETTING AND DRYING TEST FOR COMPACTED TEST SPECIMENS OF CEMENT TREATED BASES, CLASSES "A" AND "B," AND CEMENT TREATED SUBGRADE

Scope

This test, which is a modification of A. A. S. H. O. Designation: T 135, is intended for determining visual physical condition, volume change (swell), and compressive strength after the compacted test specimens have been subjected to repeated cycles of alternate wetting and drying.

Procedure

A. Apparatus

All of the apparatus specified for Test Method No. Calif. 312.

B. Test Record Form

Record the test data on work card, Form No. T-342.

C. Preparation of Test Specimen

Prepare and compact the specimen, using optimum moisture and desired amount of cement, in accordance with Test Method No. Calif. 312.

D. Curing Test Specimen

1. Store or seal test specimen in such a manner that no moisture is lost from the specimen during the curing period. This can be accomplished by curing specimen in a moist cabinet, by covering specimen with wet burlap, or by placing lids on each end of the tin sleeve and sealing with adhesive tape.
2. Cure test specimen for seven days. Remove the tin sleeves and make sure that the numbered identification disk is attached to the top of the specimen.

E. Wetting and Drying Cycles

1. Submerge the specimen in water for 5 hr., remove, surface dry, weigh (to detect any sloughing), and measure for height. Then place in a drying oven with temperature at 140 F. and leave for 42 hr., at which time remove and again measure and weigh.
   a. The above procedure constitutes one cycle of wetting and drying; and with the time used in transferring, weighing, and measuring adds up to a total time of approximately 48 hr.
   b. Continue this wetting and drying process until 12 cycles have been completed, provided of course that serious disintegration of the specimen does not occur sooner.

F. Test Calculations

1. If the specimen has increased in length after completion of the 12 cycles of alternate wetting and drying, calculate the volume change as percent of original volume. If the specimen has decreased in length consider the volume change zero.

   The formula for volume change is:

$$\text{Percent volume change} = \frac{V_2 - V_1}{V_1} \times 100$$

V_1 = Volume of specimen at beginning of cycle period
V_2 = Volume of specimen at end of cycle period

Example:

	Height of specimen	*Diameter of specimen*
Before cycle period__	4.092 in.	4.000 in.
After cycle period___	4.097 in.	4.005 in.

$V_1 = (2)^2 \times 3.1416 \times 4.092 = 51.421$ cu. in.
$V_2 = (2.0025)^2 \times 3.1416 \times 4.097$
$= 51.614$ cu. in.

$$\text{Volume change} = \frac{51.614 - 51.421}{51.421} \times 100$$

$= 0.4$ percent

2. Examine the specimen visually for physical condition, and report as excellent, good, fair or poor.
3. Then submerge the specimen in water over night and test for compressive strength as specified in Test Method No. Calif. 312.

Reporting of Results

Report the test results on Test Report, Form T-346.

REFERENCES

A. A. S. H. O. Designation: T 135.
Test Method No. Calif. 312.

End of Text on Calif. 314-B

Appendix D

Test Method No. Calif. 338-A
January 1, 1960
(12 pages)

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

DETERMINATION OF CEMENT CONTENT IN CEMENT TREATED AGGREGATE BY THE METHOD OF TITRATION

Scope

This method covers the procedures for determining the percentage of Portland cement in freshly mixed cement treated base on a project under construction. The determinations are based upon chemical titration methods which relate the cement concentration of field treated samples to known solution concentrations. Two different titration procedures are given; first, an acid-base titration method and second, a constant neutralization method. Normally the first method is used when the aggregates *do not* react to hydrochloric acid. This method is fast and experience indicates that it can be used for about 90% of the aggregates in California. However, when aggregates are encountered which react to hydrochloric acid, the second *method* must be used.

This test method is divided into the following parts:

General
I. Method of Test by Acid-Base Titration
II. Method of Test by Constant Neutralization
III. Method of Field Sampling

This test method was designed for Portland cement treated aggregates, but the same procedure can also be used for determining the percentage of lime in aggregates which have been treated with commercial hydrated calcium lime.

GENERAL

The *first* thing that *must* be done when testing for cement content on a cement treated base project is to determine whether significant amounts of such substances as limestone, calcite, dolomite, etc., which are subject to attack by hydrochloric acid, exist in the aggregate. The following procedure accomplishes this and indicates whether Part I or Part II of this Test Method should be used for cement content determinations.

The Acid-Base test, Part I of this Test Method, is performed on duplicate aggregate blank (containing no cement) specimens using the procedures for test specimen preparation, acid digestion and titration given in section E-4 (Part I). This test is then repeated on duplicate test specimens composed of standard or graded Ottawa sand. Glass beads of the type normally used for highway centerline stripes may be substituted if Ottawa sand is not readily available.

The appropriate test procedure for use on a project is based upon the following determination:

> If either of the titration tests using aggregate blanks require an amount of sodium hydroxide which is six (6) mililiters (mls.) or more lower than the average of the Ottawa sand blanks, the Constant Neutralization Method, Part II, shall be used. If the difference is less than 6 ml., the Acid-Base test, Part I, should be used (Part II may also be used in this case if desired).

It is *imperative* that the proper test method be selected *first* before proceeding with a determination of actual cement content.

PART I. METHOD OF TEST BY ACID-BASE TITRATION

Scope

This method describes a procedure for cement content determination based upon the neutralization of the cement with an excess of hydrochloric acid. The residual excess acid, as measured by back titration with an alkali, is inversely proportional to the cement content of the treated base sample. This test procedure cannot be used if acid active aggregates, as indicated at the start of this Test Method under "General", are used in the cement treated material.

Procedure

A. Apparatus

1. 1—100 ml. titrating burette
2. 1—100 ml. pipette
3. 1—Burette stand and burette clamp
4. 4—200 ml. volumetric flasks
5. 8—250 ml. Erlenmeyer flasks
6. 8—plastic beakers approximately 500 mls. min. capacity
7. Glass (or plastic) dropping bottle

8. 8—2 quart wide-mouth polyethylene containers
9. 3—5 gal. plastic "carboys" equipped with siphons, neoprene or tygon tubing, hose clamps etc., for containing working solutions of acid, base and water (do not substitute glass containers for the plastic carboys).
10. 8—Stainless steel stirring rods
11. ⅜ inch sieve, 12" Dia.
12. 1½ inch sieve, 12" Dia.
13. Ohaus balance (or equal), 5 Kg. capacity, graduated to 1 gram
14. Torsion balance, 500 gm. capacity, graduated to 0.1 gram
15. Timer

B. Reagents and Materials

1. *Hydrochloric acid* (approx. 3N)
   Pour the contents of two full standard 6 lb. bottles of concentrated hydrochloric acid C. P. (Service & Supply stock No. 69010.81) into one of the 5 gal. plastic carboys. Add tap water in approximately one gallon increments until 5 gal. of solution is obtained. After each addition of water shake the carboy vigorously for about one minute to obtain a homogeneous mixture.

2. *Sodium Hydroxide* (approx. 1N)
   Use the following procedure for making 5 gallons of solution:

   a. Using a second plastic carboy, dissolve 800 grams of sodium hydroxide pellets (Service and Supply stock No. 69010.83) in about one gallon of hot tap water at a temperature of approximately 110 F. (This can be judged as being about as hot as a person's hand can stand.)

   b. Stopper and shake the carboy vigorously for about one minute.

   c. Continue adding tap water in one gallon amounts and shake after each addition until 5 gallons of solution is attained. After the first gallon of water is added, successively reduce the temperature of each subsequent increment until the last gallon is added at the temperature of the cold tap.

   d. Cool to room temperature, then examine the solution for suspended matter by looking down through the neck opening of the carboy while using a flashlight around the outside of the bottom for illumination. The solution *must* be clear for use. Experience indicates that when suspended matter in the form of a precipitate is present it is usually the result of either insufficient heat in the mixing water or insufficient agitation. Dissolving the objectionable precipitate requires reheating the 5 gallons of solution. Since this is normally impractical, it is better to discard it and mix a new solution.

   Note: The operator(s) mixing the acid and base solutions are required, for safety, to wear chemical protective gloves, goggles, and aprons.
   Do not use compressed air to agitate the solutions in the carboys.

3. *Phenolphthalein indicator solution* (1% solution)
   Dissolve 5 grams of phenolphthalein powder U.S.P. in 250 mls. of ethanol. Dilute with 250 mls. of distilled water (Service & Supply stock No. 69010.85 for prepared 1% solution).

4. *Ottawa sand* (Standard or Graded).
   Glass beads of the type normally used for highway centerline stripes may be substituted if Ottawa sand is not readily available.

C. Test Record Form

Use work card "Field-Laboratory Record of Titration Tests", Form T-3040, for recording project, calibration, and field test data (See Figure II). (Service & Supply Stock No. 12953.40)

D. Titration of Field Samples

1. Determine whether the aggregate reacts with acid, as described at the beginning of this Test Method under "General". If the aggregate does not react with acid, proceed with this method.

2. Secure from one to eight field samples of cement treated aggregate by means of the procedure specified in Part III. Each field sample should weigh at least three kilograms.

3. Prepare from one to eight 300 gram specimens as follows:

   a. Determine total weight of each field sample to nearest 5 grams.

   b. Remove and waste any aggregate retained on the 1½ inch sieve.

   c. Separate sample on the ⅜ inch sieve and determine the proportion passing the ⅜ inch sieve on the basis of total sample weight including retained 1½ inch material.

   d. Recombine the retained and passing ⅜ inch portions for the 300 gram test specimens in their "As received" proportions, place each specimen in a separate two quart plastic container, and line the containers up along the front of the work bench.

   e. In field testing operations there is a lapse of time between the mixing of the cement treated base and the final preparation of the 300 gram test specimens. This is the time required to obtain a sample, transport it to the testing location and prepare the 300 gram test specimens. Since the cement is hydrating during this period some changes will occur in the test determinations depending on the length of the time lapse. Therefore, in order to correct for these changes, establish by trial the time lapse required for the particular project and use this as the "standing period" specified in section E-3-g of this Part I. The standing period used for subsequent samples should be within ± 15 minutes of this established period.

4. Measure accurately for each specimen 200 ml. 3N HCl, using the volumetric flask; pour into an Erlenmeyer flask and set one flask behind each specimen container.
5. Measure accurately for each specimen 400 ml. tap water; pour into a plastic beaker and set one beaker behind each Erlenmeyer flask of acid.
6. Start the timer, add the 200 mls. of acid to the first specimen and stir for 45 seconds. Add 200 mls. of acid to the second specimen and stir 45 seconds. Continue this procedure until 200 mls. of acid has been added to all specimens and they have each been stirred 45 seconds.
7. When timer reaches 6 minutes, start stirring of each specimen for 45 seconds, in sequence.
8. When timer reaches 12 minutes, again stir each specimen for 45 seconds, in sequence.
9. When timer reaches 18 minutes, add the 400 mls. of water from the plastic beaker to the first specimen and stir for 45 seconds. Add 400 mls. of water to the second specimen, stir 45 seconds, and continue procedure until 400 mls. of water has been added to all specimens and they have each been stirred for 45 seconds.
10. Rinse out the Erlenmeyer flasks.
11. Allow specimens to settle until timer reaches 30 minutes, then pipette a 100 ml. portion of the residual acid solution from the first specimen and deposit in one of the clean Erlenmeyer flasks. At 45 second intervals, pipette 100 mls. of solution from each of the other specimens in sequence and deposit in clean Erlenmeyer flasks.
12. Add two droppers full (approximately 40 drops) of phenolphthalein solution to the solution in each of the Erlenmeyer flasks.
13. Titrate the solution in each flask by adding 1 normal sodium hydroxide (NaOH) from the burette until a deep red color is formed which does not fade when the flask is shaken for one minute. If allowed to settle for a few minutes, the clear liquid should remain red. Record the burette readings to the nearest 0.2 ml. for the solution in each flask. (See the reverse side of work card, Form T-3040, as shown in Fig. II).
14. Using the resulting titration values, determine the percentages of cement in the specimens from the standard curve (see section E and Fig. II).

E. Determination of a Standard Curve

1. The standard curve establishes the relationship between the amount of sodium hydroxide used in titration and the percent cement in a test specimen for the particular aggregates, cement, and water used on the project.
2. Two points are needed to establish the standard curve; one point will be determined from duplicate tests on specimens containing 5% cement and the other from duplicate tests on aggregate blank specimens.
3. Use the following procedure to fabricate and test the calibration test specimens containing 5% cement.
   a. Determine a representative field moisture (%) for the cement treated material from road moisture samples obtained after mixing.
   b. Refer to Table No. 1 and select the weight of aggregate corresponding to the appropriate moisture content and 5% cement. This figure is the dry weight of untreated aggregate required to make a 5% calibration test specimen of 300 grams. If the samples of untreated aggregate initially contain moisture, then adjust the weight of aggregate plus water to conform to the tabular values.
   c. From the project records determine the average percentage of untreated aggregate passing the ⅜ inch sieve. This percentage passing the ⅜ inch sieve along with the portion retained on the ⅜ inch sieve will be the proportion used for the calibration specimens.
   d. Remove and waste any aggregate in the sample of untreated material retained on the 1½ inch sieve and then separate the sample on the ⅜ inch sieve. Recombine for duplicate calibration test specimens with the aggregate weight found in (b) above, and with the ⅜ inch sieve proportions found in (c) above.

TABLE NO. 1

QUANTITIES OF MATERIALS FOR CALIBRATION SPECIMENS CONTAINING 5% CEMENT

% Moisture	*Dry Wt. of Agg. in grams*	*Mls. of Water*	*Wt. of Cement in grams*
5	271	15	13.6
6	270	17	13.5
7	267	20	13.3
8	265	22	13.2
9	263	24	13.1
10	260	27	13.0
11	257	30	12.9
12	255	32	12.8
13	252	35	12.6
14	250	37	12.5
15	249	39	12.4
16	247	41	12.3
17	244	44	12.2
18	242	46	12.1
19	240	48	12.0
20	238	50	11.9

e. Pour the weighed aggregates and cement (cement weighed to 0.1 gram) into the two quart plastic container and dry mix together thoroughly with a stainless steel stirring rod.

f. Add the mls. of water given in Table No. 1 and again mix thoroughly.

g. In order to correlate with the field testing operations, allow the mixture to stand in the covered plastic container for a period of time which corresponds to the time required to obtain a field treated sample, transport it to the testing location and prepare the 300 gram test specimens (see Section D-3-e). Normally the standing period should not be less than 30 minutes nor more than 90 minutes.

h. At the conclusion of the standing period, start the acid digestion and titration test procedure given in the preceding Section D.

4. Use the following procedure to fabricate and test the aggregate blank specimens:

a. Using the representative field moisture (%) as determined in Section E-3-a, refer to Table No. 2 for the proper quantities of dry aggregate and water to be used.

TABLE NO. 2
QUANTITIES OF MATERIAL FOR AGGREGATE BLANK SPECIMENS

% Moisture	*Dry Wt. of Agg. in grams*	*Mls. of Water*
5	286	14
6	283	17
7	280	20
8	277	23
9	275	25
10	273	27
11	270	30
12	268	32
13	266	34
14	263	37
15	261	39
16	259	41
17	256	44
18	254	46
19	252	48
20	250	50

b. Use these weights of dry aggregate to fabricate duplicate aggregate blank specimens in the manner described in Section E-3-d.

c. Pour the aggregates and water into the two quart plastic container, mix thoroughly, allow to stand for the standing period as specified in Section E-3-g, then test in accordance with preceding Section D.

5. Plot the burette milliliter readings (abscissa) against the percent cement (ordinate) for both the duplicate 5% cement specimens and the duplicate aggregate blank specimens (0% cement) on the graph provided on Form T-3040 as illustrated in Figure II. Draw a straight line between the averages of the paired tests. This is the standard curve for determining the cement content of field mixed cement treated specimens.

F. Precautions

1. This test should not be performed by persons subject to color "blindness".
2. Keep all reagent bottles closed when not in use to prevent evaporation and the consequent changes in concentration.
3. Rinse and drain all glassware and plasticware when the day's work is done.

Notes

A standard curve should be determined *at least once per day.* In event of any change in source of aggregate or cement or new working solutions of acid or hydroxide, another determination should be made.

Hazards

Extreme care should be exercised in handling the concentrated solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH). If either of these chemicals are spilled in the eyes, on the skin, or on the clothing, the affected areas should be flushed immediately with liberal quantities of water.

Care should also be exercised in the use of the dilute solutions of HCl and NaOH as these dilute solutions can also be harmful to the eyes, cuts, skin, or clothing.

The following safety recommendations regarding the use of this test method are by the State Division of Highways Safety Engineer:

1. Since the test is a chemical analysis requiring the handling of acids and bases, it is recommended that its use be limited to Materials & Research trained and controlled personnel.
2. Information on the test should include a requirement that when handling either acids or bases, personnel will be required to wear chemical protective gloves, goggles, and aprons.
3. Test instructions should contain a requirement that glass carboys are not to be substituted for plastic carboys described in the test apparatus and that all containers having either acids or bases, or solutions made from them, are to be clearly labeled.
4. It is suggested that there be some notation that while there is limited danger associated with getting a part of the final solution in the mouth after it has acted on the cement, extreme care should be taken not to permit any of the pure acid to get into the mouth.

5. Personnel conducting experiments should know that Phenolpthalein has been known to cause allergic symptoms in humans and has a powerful laxative effect. It can be toxic. Exposed personnel who are sensitive to it should wear protective clothing, if necessary, to avoid bodily contact. Do not ingest.

PART II. METHOD OF TEST BY CONSTANT NEUTRALIZATION

Scope

This method is an alternate to the Acid-Base test procedure, Part I, and is used for determining cement content when the aggregate is found to react with acid. The determinations are based upon the continuous neutralization of an aqueous solution from the cement treated aggregate specimen for a specified time period. This is accomplished by adding sufficient acid to just neutralize the OH ion which is continuously being liberated during the hydration of the cement. The amount of acid used is directly proportional to the cement content of the treated base sample.

Procedure

A. Apparatus

1. 1—100 ml. titrating burette or other device permitting slow, easily controlled addition of acid.
2. 1—burette stand and burette clamp.
3. 4—2 quart wide mouth polyethylene containers.
4. 2—5 gal. plastic "carboys" equipped with siphons, neoprene or tygon tubing, hose clamps, etc., for containing acid working solution and water (do not substitute glass containers for the plastic carboys).
5. Glass dropping bottle.
6. 4—stainless steel stirring rods.
7. 3/8 inch sieve, 12 inch Dia.
8. 1½ inch sieve, 12 inch Dia.
9. Ohaus balance (or equal), 5 kg. capacity, graduated to 1 gram.
10. A balance having a minimum capacity of 1 kg. and graduated to 0.1 gram.

B. Reagents

1. *Hydrochloric acid* (approx. 3N)

   Pour the contents of two full standard 6 lb. bottles of concentrated hydrochloric acid C.P. (Service & Supply stock No. 69010.81) into one of the 5 gal. plastic carboys and dilute with tap water in the manner indicated in Part I, Section B-1, to make 5 gal. of solution.

   NOTE: The operator(s) mixing the acid solution are required, for safety, to wear chemical protective gloves, goggles, and aprons. *Do not* use compressed air to agitate the solution in the carboy.

2. *Phenolphthalein indicator solution* (1% solution)

   Dissolve 5 grams of phenolphthalein powder U.S.P. in 250 mls. of ethanol. Dilute with 250 mls. of distilled water. (Service & Supply stock No. 69010.85 for prepared 1% solution.)

C. Test Record Form

Use work card "Field-Laboratory Record of Titration Tests", Form T-3040 for recording project, calibration, and field test data (See Figure III). (Service & Supply stock No. 12953.40.)

D. Test Procedure for Field Samples

1. Secure one to four 3 kilogram field samples of the cement treated aggregate using the procedure specified in Part III. Prepare 300 gram test specimens as desscribed in Part I, Section D-3.
2. Place each specimen in a separate 2 quart plastic container.
3. Add 250 ml. of tap water to each specimen and start timer.
4. Add two droppers full (approximately 40 drops) of phenolphthalein solution to each container. The water solution will normally turn red due to the presence of cement.
5. Weigh each plastic container, including its contents, to the nearest 0.5 gram.
6. At 10 minutes after the addition of water, start adding the 3N hydrochloric acid with a titrating burette while *stirring continuously*.
   a. The initial amount of acid to be added is based upon the planned cement content of the mix and is determined from Figure I.

      Example: Suppose the planned cement content for a given project is 3½%. Enter the abscissa of the chart in Figure I at 3½% and find the intersection with the upper diagonal line. Reading from the ordinate to the nearest milliliter, it is noted that 15 mls. of HCL is required. Use the titrating burette to measure and add the 15 mls. of acid to the specimen in the plastic container. When the proper amount of acid is used, the red color in the solution will disappear.

   b. When the red color starts to reappear, after the initial introduction of acid, use the burette to make a second addition of acid in the amount (mls.) indicated by the lower diagonal line (at the planned cement content) of the chart in Figure I.

7. After the first two additions of acid, make subsequent additions at random as the red color reappears using only the minimum amounts necessary to cause the color to just disappear and maintain a neutral solution. This requires *constant* attention by the operator.
8. Besides the stirring accomplished during the actual introduction of the acid, additional light stirring (for about 3 seconds) should be performed every minute with every fifth minute being devoted to deep stirring (for about 6 seconds).
9. Continue this procedure of adding acid and stirring until one hour after the initial addition of water to the specimen (see 3 above). Then reweigh the plastic container and contents to the nearest 0.5 gram.
10. From the difference between the initial and final gross test specimen weights (see Fig. III), calculate and record the total weight of hydrochloric acid used to neutralize the solution.
11. Using the resulting weight of hydrochloric acid, determine the percentage of cement in the specimens from the standard curve (see Section E and Fig. III).

E. Determination of a Standard Curve

1. The standard curve establishes the relationship between the amount of hydrochloric acid used to neutralize the OH ion in the cement and the percent cement in the test specimen for the particular aggregates, cement, and water used on the project.
2. Normally only one point is needed to establish the standard curve and this point is determined from duplicate tests on specimens containing 5% cement. However, if a small sample of untreated aggregate in water shows a red color on adding a few drops of phenolphthalein solution, indicating the presence of water soluble alkalis, then duplicate aggregate blank specimens should also be tested.
3. Fabricate calibration test specimens using the applicable portions of Part I, Section E-3 & E-4.
4. Perform the Constant Neutralization test procedure in accordance with Section D of Part II with the exception that the first and second addition of acid shall be based upon 5% cement content using Figure I (which gives 21 and 8 mls. respectively).
5. Using the graph provided on Form T-3040, plot the grams of acid used against percent cement for both duplicate 5% cement specimens (also aggregate blank specimens if tested; see 2 above) as illustrated in Figure III. Draw a straight line from the origin of the chart (or the average of the duplicate blanks) to the average of the tests on the 5% specimens. This is the standard curve for determining the cement content of field mixed cement treated specimens.

F. Precautions

1. This test should not be performed by persons subject to "color blindness".
2. It is very important that the operator *vigilantly* maintain a neutral solution by repeatedly adding acid as soon as the pink color reappears. Neglect of this item will cause inaccuracies in the cement determinations.
3. After first two additions of acid, use only the amount necessary to just eliminate the red color. Excess acid may attack the aggregates, particularly in the later phases when the amount of hydration products from the cement remaining in the mixture may be low, resulting in erroneous cement determinations. It is also possible that the use of excessive acid in the final stages could cause the total quantity of acid to exceed that which would have normally been attained at the end of the one hour time period.
4. Keep all reagent bottles closed when not in use to prevent evaporation and the consequent changes in concentration.
5. Rinse and drain all glassware and plasticware when the day's work is done.

Notes

While the total amount of acid used for neutralization is determined by weight in this procedure, it may also be measured in volumetric units using titrating burettes. However, since it is customary to test more than one specimen at a time (four simultaneous tests are the maximum number for this procedure) and volume measurements would require a burette for each sample, it is more convenient to use the weight basis. A titrating burette is normally used in this procedure only as a convenient implement for introducing the acid and not as a measuring device (except when metering the 1st & 2nd additions of acid in accordance with Section D-6 of Part II).

The purpose of the chart in Figure I is to provide a sufficient time lapse, before the red color reappears in the solution, for the operator to adequately process four specimens in the early stages of the test. The indicated amounts are calculated to allow a slight excess of acid which under normal circumstances will usually retard the color return for 2 or more minutes. Studies indicate that the amounts of acid prescribed

Test Method No. Calif. 338-A
January 1, 1960

CONSTANT NEUTRALIZATION PROCEDURE
Hydrochloric Acid Starting Quantities

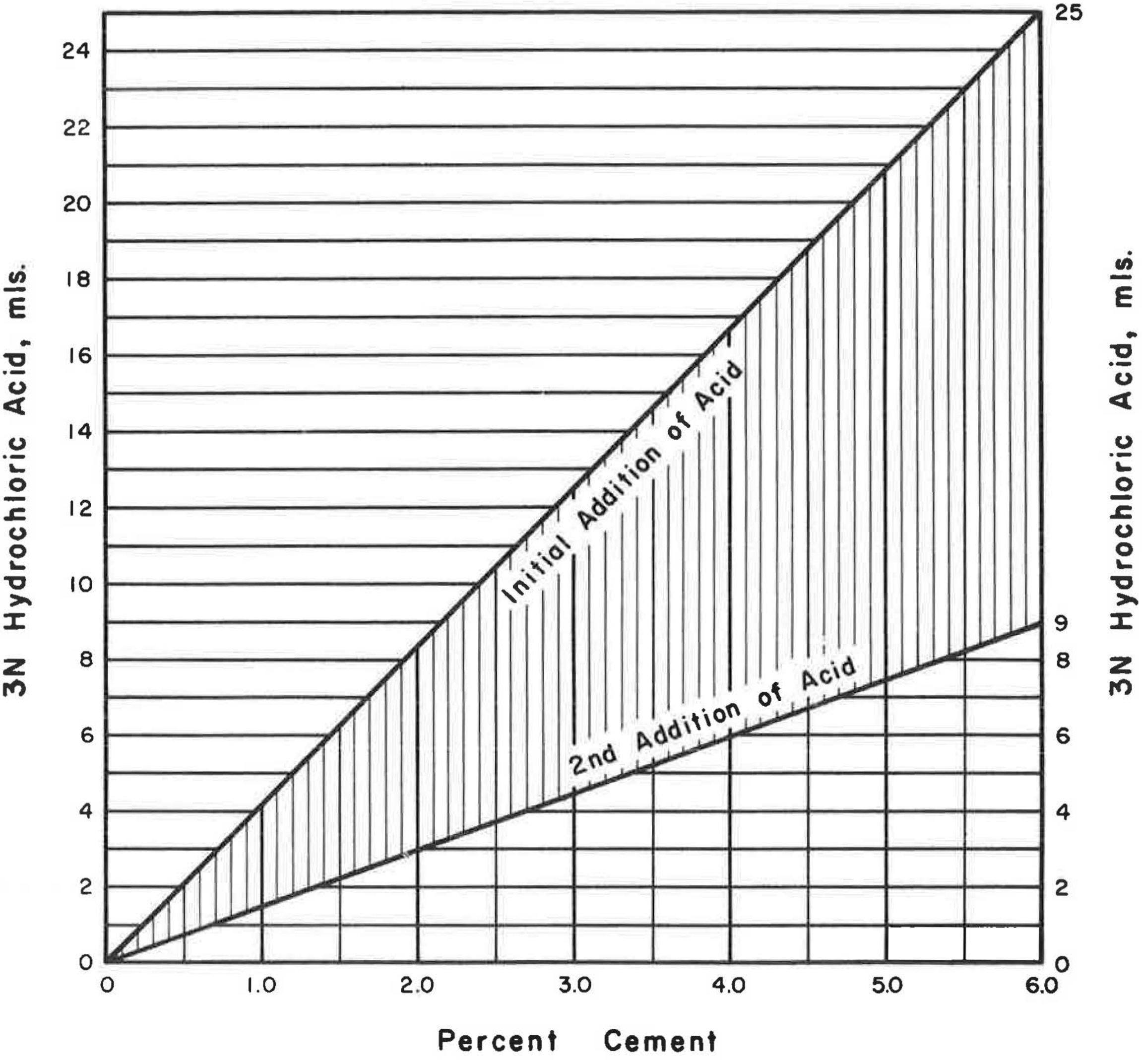


FIGURE I

in Figure I for the first two additions, will not affect the accuracy of the test, however care must be exercised, in performing the balance of the procedure, not to permit any further excess of acid.

A standard curve should be determined *at least once per day*. In the event of any change in source of aggregate or cement or a new working solution of acid, another determination should be made.

PART III. METHOD OF FIELD SAMPLING

Scope

This method describes the procedure for sampling cement treated bases to obtain representative portions for cement determinations.

All sampling should be performed for a definite purpose such as to determine the efficiency of the mixer, the relationship between the cement and aggregate feeds, or the general overall variations in cement content during the day.

Procedure

A. Equipment

1. Pick
2. Shovel
3. Hand Scoop
4. Covered containers of 3 Kg. min. capacity

B. Test Record Form

Keep all pertinent data regarding the project and individual samples on Form T-3040 "Field-Laboratory Record of Titration Tests". (See Figures II & III)

C. Size of Sample

Each sample should weigh approximately 3,000 grams.

D. Sampling for Determination of Efficiency of Mixers

1. *Central Batch Plant*

   The most desirable method of sampling a central batch plant is after the material has passed through the paving machine or spreader box. This sampling will check the combined efficiency of the mixer and paving machine. If too large a variation is found in the different samples, further sampling directly from the plant must be done in order to isolate the trouble.

2. *Continuous Mix Plant*

   A continuous mix plant generally feeds the mix onto a conveyor belt. Samples should be taken directly from the belt. Samples taken at five or ten minute intervals from the belt will give a good check on the efficiency of the plant, including the cement and aggregate feeds.

3. *Road Mixers*

   Sampling should be from the material just as it leaves the mixer. To check the efficiency of the mixer, several samples (generally four) should be taken transversely to the direction of spread. When sampling from a windrow, remove about four inches of the surface material, then take one sample near each toe and one near each edge of the top for a total of four samples. When sampling from spread out material, take the four samples equidistant from each other across the spread, starting about one foot inside each edge. Each sample should represent the full depth of spread.

E. Sampling to Check Overall Fluctuation in Cement Content

After the mixers and spreaders have been checked and it is determined that the equipment is in satisfactory adjustment, it is important to take occasional samples to make sure that the cement feed is remaining constant. Therefore, several samples should be taken in the direction of spread. Each sample should represent the average transverse section.

REFERENCES

A California Method.

End of Text on Calif. 338-A

STATE OF CALIFORNIA—DIVISION OF HIGHWAYS
MATERIALS AND RESEARCH DEPARTMENT
Field-Laboratory Record of

TITRATION TESTS

For Cement Content Determination of CTB
FORM T-3040 (REV. 11-59)

Sheet 1 of 1 Sheets
Date NOVEMBER 6, 1957
Operator TROTTER

PROJECT DATA

Dist. III	Co. Pla.	Rte. 37	Sec. B	Contract 57-3TC21-F

Limits BETWEEN HEATHER GLEN & COLFAX

Source of Aggregate BEAR RIVER

Cement Brand IDEAL

Cement Mixer Make: MADSEN

Road Mix X Plant { Batch ☐ Cont. ☐

Cement Spreader HOPPER TRUCK

PLANNED	Thickness 8"	Cement* 4.5 %	Water 6.0 %

DETERMINATION OF METHOD OF TEST

Item	Specimen Type	Jar No.	NaOH Ml.	Control Values
A	Ottawa Sand	7	102.6	Av. Mls.
		8	102.9	102.7
B	Aggregate Blank	9	100.0	Lowest Value
		10	101.5	100.0
C	Av. of A—Lowest Value of B			2.7

If C<6 mls use Acid-Base Proc. (Part I) . . . . ☒ *
If C≥6 mls use Const. Neut. Proc. (Part II) . . . . ☐

CALIBRATION RECORD FOR STANDARD CURVE

Station of Agg. Sample	Curve No.	% Cement	Jar No.	NaOH Ml. ** ~~HCl-Gr.~~
360+00	2	0	9	100.0
" "	2	0	10	101.5
360+00	2	5	9	53.4
" "	2	5	10	52.6

FIELD NOTES

MIXER SPEED 22'/min.

U.B. AGGS. 1½" max.

Use Reverse side for Tests of Field Samples

* Check appropriate item.
** Cancel words not applying.

STANDARD CURVE

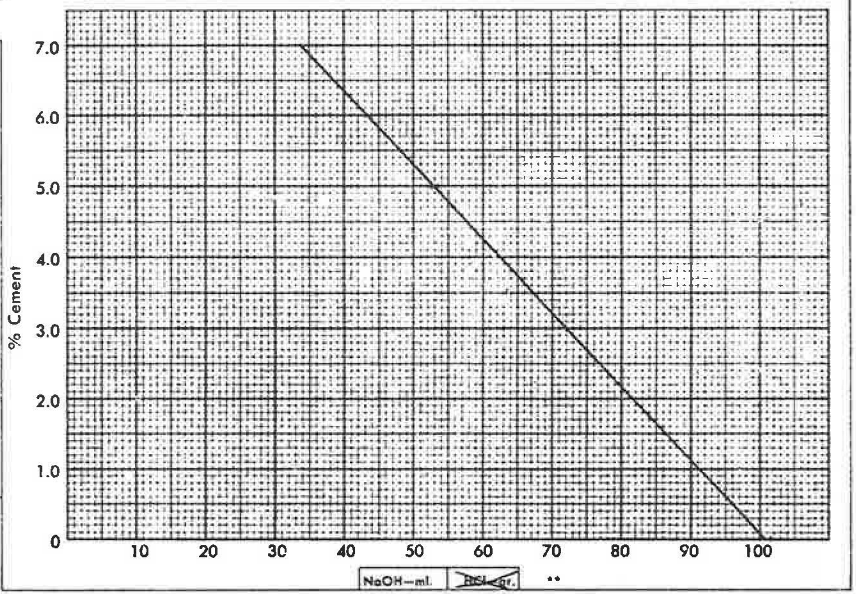


FIGURE II

TEST RECORD OF FIELD SAMPLES

Station	Position	Depth	Time Mixed	Sample Proportions			Jar No.	Gross Jar Wt. Grams †		NaOH Ml. ** ~~HCl Cc.~~	% Cement	Remarks
				Sample Wt. Grams	Pass ⅜ Grams	% Pass ⅜		Start	Final			
349+50	12' Lt. ℄	0-8"	0830	4070	1470	36	1			67.4	3.5	AVERAGE = 3.9%
	14' Lt. ℄	"	"	4740	1700	36	2			64.0	3.9	
	16' Lt. ℄	"	"	4350	1840	42	3			61.4	4.1	
	18' Lt. ℄	"	"	4880	1840	38	4			61.6	4.1	
365+90	12' Lt. ℄	0-8"	0950	3890	1650	42	5			53.6	5.0	AVERAGE = 4.4%
	14' Lt. ℄	"	"	4995	2175	44	6			61.4	4.1	
	16' Lt. ℄	"	"	3435	1775	52	7			59.6	4.3	
	18' Lt. ℄	"	"	3960	1895	48	8			60.6	4.2	
374+00	20' Lt. ℄	0-8"	1030	3550	1100	31	1			58.6	4.4	AVERAGE = 5.0%
	22' Lt. ℄	"	"	3965	1825	46	2			49.0	5.5	
	24' Lt. ℄	"	"	3600	1690	47	3			51.8	5.2	
	26' Lt. ℄	"	"	3420	1410	41	4			55.0	4.8	
343+50	27' Lt. ℄	0-8"	1340	4075	1550	38	5			55.0	4.8	AVERAGE = 4.6%
	29' Lt. ℄	"		5280	2240	42	6			65.6	3.7	
	31' Lt. ℄	"		5600	2260	40	7			53.6	5.0	
	33' Lt. ℄	"		3235	1105	34	8			54.8	4.8	
342+00	24' Lt. ℄	0-8"	1340	4560	1970	43	1			58.8	4.4	
340+00	24' Lt. ℄	"	1350	4430	1810	41	2			55.6	4.7	
336+00	24' Lt. ℄	"	1415	6030	2810	47	3.			51.0	5.3	

** Cancel words not applying

† Required only for the Constant Neutralization Procedure (Part II, Test Method No. Calif. 338)

EST. 2023 6116 11-59 8M △ SPO

FIGURE II—Continued

STATE OF CALIFORNIA—DIVISION OF HIGHWAYS
MATERIALS AND RESEARCH DEPARTMENT
Field-Laboratory Record of
TITRATION TESTS
For Cement Content Determination of CTB
FORM T-3040 (REV. 11-59)

Sheet 1 of 1 Sheets
Date SEPT. 26, 1958
Operator TROTTER

PROJECT DATA

Dist.	Co.	Rte.	Sec.	Contract
IV	CC	75-107	A, Wlc.	58-4TC17-FI

Limits

Source of Aggregate SELECTED PIT - DIV. OF HIGHWAYS
Cement Brand S.CLARA - TYPE II
Cement Mixer Make MADSEN
* Road Mix ☐ Plant { Batch ☐ Cont. ☒
Cement Spreader BELT

PLANNED	Thickness	Cement	Water
	6"	6.0 %	17.0 %

DETERMINATION OF METHOD OF TEST

Item	Specimen Type	Jar No.	NaOH Ml.	Control Values
A	Ottawa Sand	3	94.4	Av. Mls. 94.6
		4	94.8	
B	Aggregate Blank	1	58.2	Lowest Value 58.2
		2	61.6	
C	Av. of A—Lowest Value of B			36.4

If C<6 mls use Acid-Base Proc. (Part I) . . . . . ☐ *
If C≧6 mls use Const. Neut. Proc. (Part II) . . . . . ☒

CALIBRATION RECORD FOR STANDARD CURVE

Station of Agg. Sample	Curve No.	% Cement	Jar No.	~~NaOH Ml.~~ ** HCl—gr.
PIT SAMPLE	1	5	5	65.1
		5	6	64.4

FIELD NOTES

See Reverse Side for Tests on Field Samples

* Check appropriate item
** Cancel words not applying

STANDARD CURVE

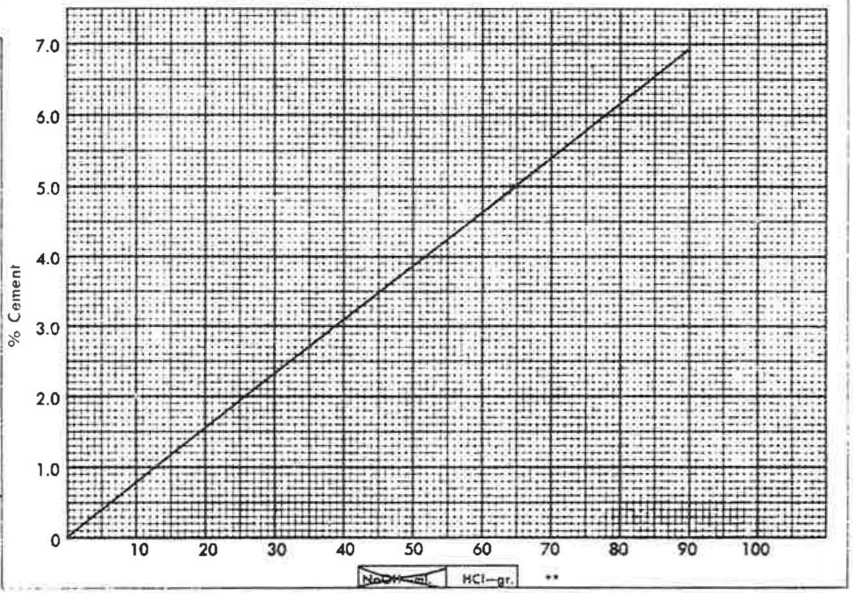


FIGURE III

TEST RECORD OF FIELD SAMPLES												
Station	Position	Depth	Time Mixed	Sample Proportions			Jar No.	Gross Jar Wt. Grams †		~~NaOH Ml.~~ **	% Cement	Remarks
				Sample Wt. Grams	Pass ⅜ Grams	% Pass ⅜		Start	Final	HCl Gr.		
SAMPLED FROM BELT @			0835	4280	3170	74	10	808.5	883.0	74.5	5.7	AVERAGE = 6.0%
30 SEC. INTERVALS				4010	3050	76	11	813.3	892.5	79.0	6.1	
				3815	2990	78	12	806.5	891.5	85.0	6.5	
				4435	3295	74	13	816.0	890.5	74.5	5.7	
SAMPLED FROM BELT @			1100	3600	2880	80	10	830.5	901.0	70.5	5.4	AVERAGE = 5.7%
30 SEC. INTERVALS				3775	3050	81	12	808.5	878.5	70.0	5.4	
				4140	3470	84	13	809.0	880.0	71.0	5.5	
				3700	3005	81	14	811.5	897.5	86.0	6.6	
SAMPLED FROM BELT @			1350	5110	3910	77	10	810.5	876.0	65.5	5.0	AVERAGE = 5.1%
30 SEC. INTERVALS				4770	3690	77	12	812.5	884.0	71.5	5.5	
				4060	2920	72	13	808.0	871.0	63.0	4.9	
				4270	3380	79	14	808.0	873.5	65.5	5.1	

** Cancel words not applying

† Required only for the Constant Neutralization Procedure (Part II, Test Method No. Calif. 338)

EST. 2923 9116 11-59 8M △ GPO

FIGURE III—Continued

British Practice in the Design and Specification of Cement-Stabilized Bases and Subbases for Roads

D. J. MACLEAN and W. A. LEWIS, Road Research Laboratory, Department of Scientific and Industrial Research, United Kingdom

Current British practice in the design and specification of soil-cement for use in the base or subbase for roads is described. The composition of the material, the structural design of roads incorporating soil-cement, and the methods used to control the quality of the material in practice are covered. A current specification for soil-cement is appended.

The suitability of a soil for stabilization is based on requirements similar to those in the United States: good grading, low plasticity of the fines, and freedom from deleterious chemical constituents. The pedological classification of a soil profile is used to estimate the depth of soil unsuitable for stabilization because of organic content; measurements of the pH of a soil-cement paste 1 hour after mixing are used as a check on the presence of deleterious organic matter.

Soil-cement is usually required to have a strength and state of compaction higher than certain specified values. The contractor selects the necessary cement content. Procedure involves carrying out tests to determine unconfined 7-day compressive strength of cylindrical or cubical specimens of soil-cement mixtures containing different cement contents. The specimens are usually prepared at a moisture content and dry density as close as possible to what is obtained in practice. If the specimens have a significantly different dry density, a correction is applied to the strength.

Tests to assess the effects of water and frost on a cement-stabilized soil are made only when the soil has certain characteristics: expansive clay minerals in cohesive soils and porous particles in granular soils.

Soil-cement has been widely used since 1945 for the construction of housing estate roads and low-traffic rural roads. These roads usually comprised a 6-in. thickness of stabilized soil with a minimum 7-day compressive strength of 250 psi, with a double surface dressing or thin pre-mixed bituminous carpet.

Cement-stabilized materials have also been used for the construction of main roads, in particular as the subbase of concrete and bituminous-surfaced roads. The most recent specification requires that the stabilized soil should have a minimum unconfined 7-day compressive strength of either 400 psi for cylindrical specimens or 500 psi for cubical specimens.

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

The quality control of soil-cement during construction is based largely on tests to check the strength and state of compaction of the laid material. If the compaction state of the laid material differs significantly from that of the test specimens, corrections are made to obtain a more reliable strength indication.

•THE SYSTEMATIC use of soil-cement as a road base material in Great Britain dates from 1939, when the Road Research Laboratory followed up a study of American experience by carrying out a program of laboratory tests and small-scale field trials on soil-cement mixtures.

From the outset, it was decided to depart from American practice by using the unconfined compressive strength instead of the results of two American durability tests for evaluating the composition of soil-cement mixtures. This probably arose partly from doubts of the relevance of the results of the American tests to the performance of soil-cement as a road base material in the less severe climate of Great Britain and partly from a desire to adapt the equipment and test procedure used for the testing of concrete.

Strength as a criterion for the quality of soil-cement has provided a valuable basis for obtaining a better understanding of the properties and factors that can affect soil-cement performance in practice, and has helped to insure production at more uniform standards with consequent improvement in performance.

With increasing knowledge of factors affecting soil-cement strength, it became apparent that small differences in moisture content and state of compaction from the specified requirements could have as great an effect on the properties of the soil-cement as a significant error in cement content. This led to the view that specifications for soil-cement should require that strength and state of compaction exceed specified values and that the contractor should select a cement content which would consistently meet the strength requirement. This may represent a significant difference from the usual American practice.

Soil stabilization work in Great Britain is usually carried out either by direct labor under the control of the road authority or by a contractor working to the road authority's specification. In both approaches, however, decisions have to be made as to the suitability of a particular soil for stabilization, the moisture content and the state of compaction, and the required cement content for minimum strength.

COMPOSITION OF CEMENT-STABILIZED SOILS

The basic procedure followed in design of the composition of cement-stabilized soils broadly comprises four stages:

1. Laboratory tests of the physical and chemical properties of the soils are made to select soils suitable for stabilization with portland cement.
2. Laboratory tests, and sometimes full-scale trials, are made to determine the moisture content and state of compaction that can be consistently achieved with the cement-stabilized soil under average practical conditions.
3. Laboratory tests are made to determine the cement content which, for the selected moisture content and state of compaction, will enable the cement-stabilized soil to achieve a specified compressive strength.
4. The durability of the compacted cement-stabilized soil is tested by determining its resistance to the action of frost and of soaking in water.

It is not always essential to carry out all four stages and, with the necessary experience, stages 1 and 4 are often omitted.

SELECTION OF SOILS SUITABLE FOR STABILIZATION

The selection of soils suitable for stabilization is based on the following:

1. The particle-size distribution and plasticity properties must be such that the soil can be stabilized with an economical amount of cement and successfully processed with available mixing and compaction plant.
2. The soils have to be sufficiently free from undesirable chemical constituents that can either prevent the hardening of the cement-stabilized soil or cause a loss of durability through the subsequent disruption of the cement bonds.

Soil Type

Laboratory research and practical experience show that the amount of cement required for stabilization may become excessive with two groups of soil: (a) with certain granular soils, the material may be so single-sized that a considerable amount of cement is required to fill the relatively large volume of voids before any significant gain in strength is obtained (Fig. 1), and (b) with certain cohesive soils the inherent strength of the soil crumbs may be so low as to require a high cement content to provide a sufficiently strong matrix to achieve adequate stabilization (Fig. 2).

To eliminate single-sized granular soils it has been suggested that for British conditions the material should have a uniformity coefficient of not less than 10 (the coefficient of uniformity is the ratio of the particle size for which 60 percent of the material is finer to the particle size for which 10 percent is finer). This limitation will of course depend on the availability of other road-making materials, and in areas where single-sized sands predominate it may be possible economically to justify the use of high proportions of portland cement for stabilization.

Cohesive soils are unsuitable for stabilization if their liquid limit exceeds 45 percent and their plastic limit exceeds 20 percent. The difficulty of successfully stabilizing cohesive soils arises from the inability of many types of mixing plant to break down the soil into sufficiently small aggregations so that a satisfactory micro-distribution of cement can be achieved. Experiments have been made at the Road Research Laboratory (1) to determine the effect of the degree of pulverization on the unconfined compressive strength of a cement-stabilized clay soil. Some results of these experiments (Fig. 3) show that to obtain an adequate strength with a reasonable cement content the degree of pulverization has to be such that the majority of the aggregations are finer than $^3/_{16}$ in. There is a British Standard test (2) for determining the degree of pulverization, defined as the ratio of the weight of aggregations finer than $^3/_{16}$ in. to the total weight of the soil, expressed as a percentage. A degree of pulverization of at least 80 percent is often required for successful cohesive soil stabilization. Investigations of mixer performance shows that this degree of pulverization can be obtained with cohesive soils only by using single-pass mix-in-place plant. Stationary

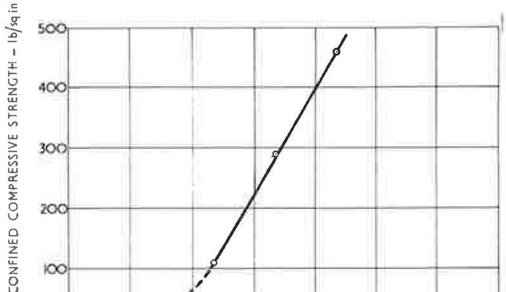


Figure 1. Relation between unconfined compressive strength and cement content for a stabilized uniformly-graded sand.

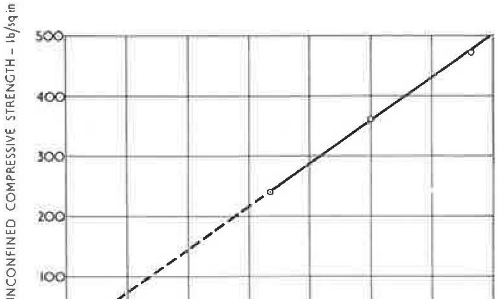


Figure 2. Relation between unconfined compressive strength and cement content for a stabilized heavy clay.

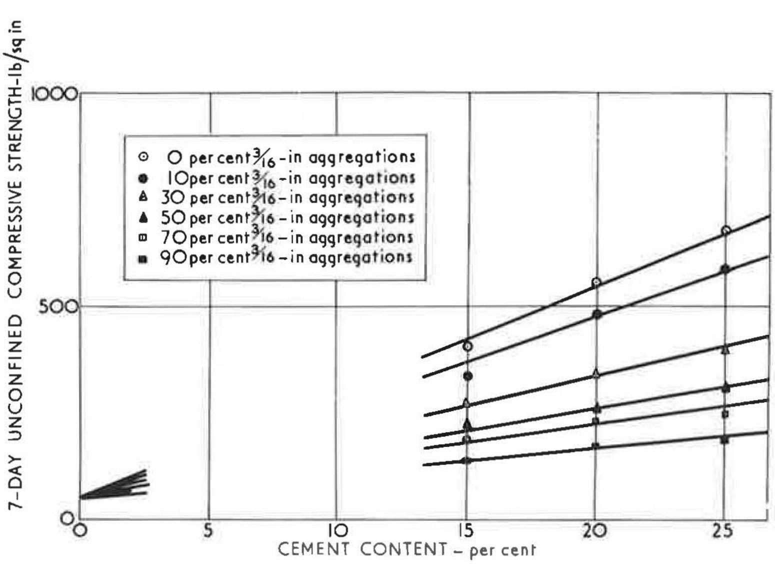


Figure 3. Relation between unconfined compressive strength of stabilized clay and cement content for various percentages of $^3/_{16}$-in. aggregations.

plant is suitable for mixing only granular soils with low cohesivity (3). Figure 4 shows results obtained with a pan-type mixer. With a sandy gravel and sand, both of low cohesivity, it was possible using a 1- to 2-min mixing time to produce a soil-cement mixture having strengths approaching those produced by an efficient laboratory-type mixer. With the cohesive clayey gravel and silty clay soils, only about 50 percent of the strength of the laboratory-mixed material was obtained in 1 to 2 min. When stationary plant is to be used for mixing, therefore, a further limitation has to be imposed on the permitted types of soil to be stabilized; that is, this type plant can only be used satisfactorily with non-plastic materials or granular materials containing less than about 10 percent plastic fines. The suggested limits of grading for single-pass mix-in-place and stationary plant work are shown in Figure 5.

Organic Matter

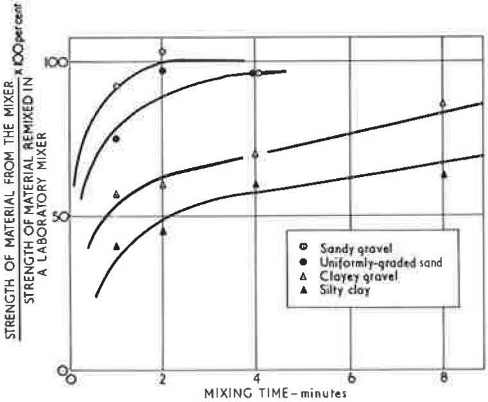


Figure 4. Results of mixing tests on stabilized soils produced by a pan-type concrete mixer.

The presence of organic matter in the surface layers of soil often renders them unsuitable for stabilization with cement. In Great Britain organic matter can extend down to a depth of as much as 5 ft. The presence of a deep surface layer of organic soil may make it impracticable to use the mix-in-place method or to use the site as a borrow pit for materials, and a simple means of recognizing this situation is required. It has been found that the pedological classification of soil profiles used in agriculture meets this particular requirement (4). Over most of the country only five types of profile have to be considered: (a) the calcareous and (b) the high base status brown earth, for which no difficulty arises from the presence of organic matter right up to the

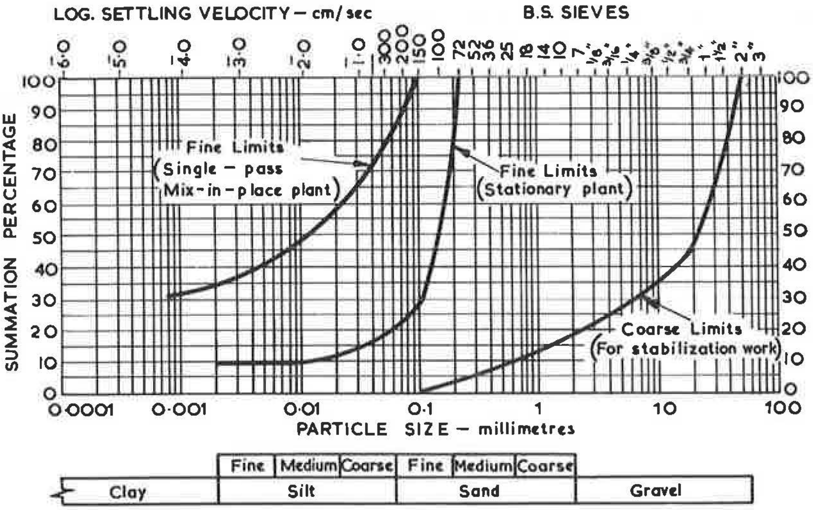


Figure 5. Limits of recommended grading for single-pass mix-in-place and stationary plant soil stabilization.

surface of the ground; (c) gley profiles, for which soils are suitable for soil stabilization usually below a depth of 15 in.; and (d) the low base status brown earth and (e) the podzol profiles, for which the soils are suitable for stabilization only at depths exceeding 2 to 5 ft. The whole of Great Britain is in the process of being mapped on a pedological basis, so that it is sometimes possible to identify the type of pedological profile directly from maps. Where this is not possible it is relatively simple to recognize the type of profile by visual examination in trial pits, and the two types of brown earth profile can be distinguished by a pH test on the soil.

Furthermore, a simple diagnostic test can be used to detect the presence in soils of organic matter that can prevent the normal hardening of portland cement (4). This test consists of making up a soil-cement paste using 10 percent portland cement and determining the pH value of the paste 1 hour after the addition of the water. A pH value below 12.1 indicates the presence in the soil of organic matter capable of preventing or hindering the proper hardening of the cement (Fig. 6). A higher value than 12.1 does not necessarily insure satisfactory hardening of stabilized soil; therefore, the test is used as a rejection test for unsuitable soils.

The determination of the calcium absorption capacity of soils has also been investigated as a diagnostic test for the presence of organic matter. This test has been of value in detecting organic matter in clean sands, a minimum calcium absorption value of 75 mg per 100 g of dry soil being the appropriate criterion. However, the test was found to be unsuitable for use with clay soils because of the ability of the clay fraction to absorb calcium in addition to the organic matter.

Sulfates

It is not uncommon for calcium sulfate, and more occasionally magnesium sulfate, to occur naturally in British soils. Research carried out at the Road Research Laboratory has shown that when such soils are stabilized with portland cement the resulting material may lack durability, particularly if subjected to an increase in moisture content after the material has hardened (5). It appears that disintegration of the stabilized soil may be brought about by a reaction between clay and sulfate ions in the presence of lime and excess water. There is some evidence to suggest that this reaction can result in the formation of ettringite (calcium sulfo-aluminate); this mineral occupies a greater volume than the reactants from which it is formed; an expansion which destroys

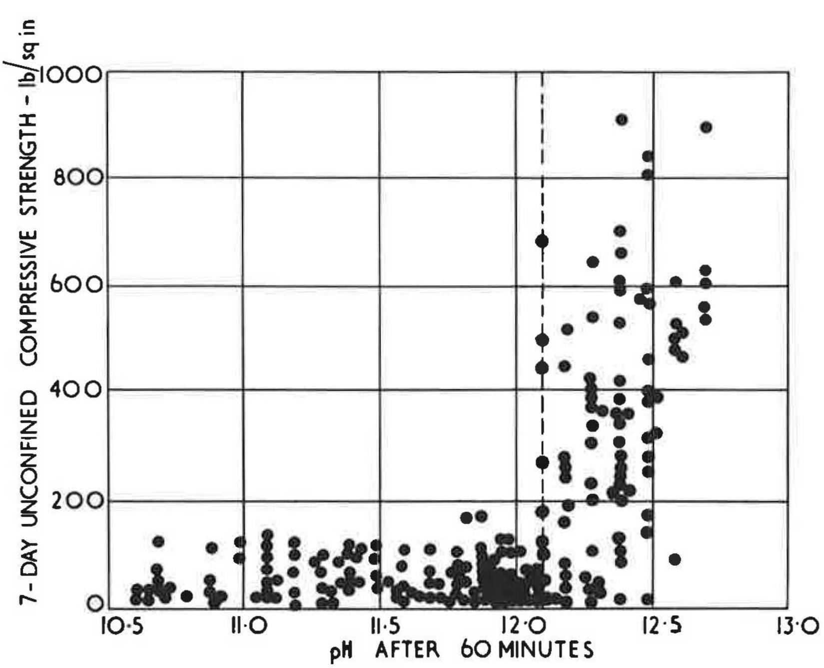


Figure 6. Relation between unconfined compressive strength and pH value for 10:1 soil-cement mixtures.

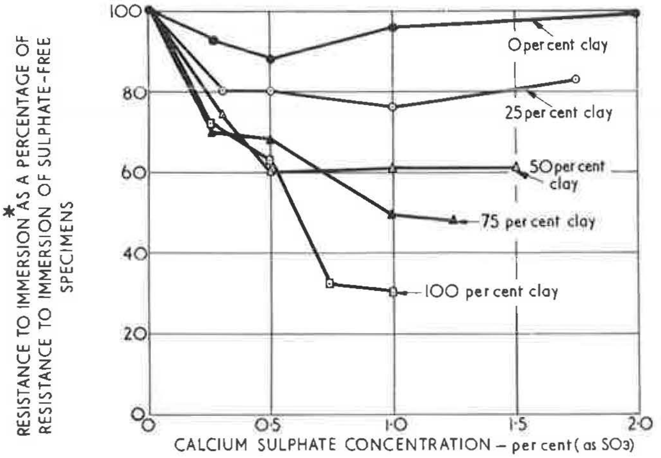
the bonds in the cement-stabilized soil can result. The extent of the reaction depends on the amount of clay in the soil, and it has been shown that the rate of disintegration increases with increase in proportion of clay in the stabilized soil (Fig. 7). It appears that a soil is unsuitable for stabilization with cement if it contains a very small proportion of sulfate ions, although it is not possible to put a value on this proportion from available knowledge. Determination of the sulfate content of soil is carried out according to test No. 8 of British Standard 1377:1961 (6). The sulfate is extracted from the soil with hydrochloric acid, and the sulfate is then precipitated from the solute as barium sulfate by the addition of barium chloride solution.

MOISTURE CONTENT AND COMPACTION STATE OF STABILIZED SOIL

After establishing the soil as suitable for stabilization, investigations are then made to determine the proportion of cement that will achieve a given compressive strength in practice. This involves making up specimens of soil-cement using different proportions of cement and determining their strength usually at 7 days but sometimes at 28 days. The test specimens have to be prepared at a moisture content and state of compaction as close as possible to those obtained in average practice. The next stage is to decide on the particular values of moisture content and state of compaction to use in these tests.

Moisture Content

The primary consideration affecting moisture content selection is that it should permit high states of compaction to be achieved in practice. Extensive investigations have been made at the Road Research Laboratory into the performance of plant for compacting soils (7, 8, 9, 10). These investigations have shown that with many types of granular soil a reliable guide to the moisture content to be used in practice is the optimum moisture content obtained with the British Standard compaction test using



*(Resistance to immersion = $\frac{\text{Strength of specimens cured for 7 days, soaked for 7 days} \times 100 \text{ percent}}{\text{Strength of specimens cured for 14 days}}$)

Figure 7. Effect of calcium sulfate on strength of cement-stabilized clay-sand mixtures.

heavy compaction (equivalent to the modified AASHO test). There are, however, some types of sandy soil for which laboratory compaction tests do not accurately reflect the compaction characteristics in practice; in such cases full-scale trials are required to determine the appropriate moisture content for the stabilized soil.

In the case of cohesive soils the test specimens are often made up at a moisture content 2 percent below the plastic limit of the freshly-mixed soil-cement; this moisture content not only permits high states of compaction to be obtained in practice, but also insures a low water absorption capacity in the hardened soil-cement. This latter point is regarded as being important, since failures in cemented bases have occurred in cases where the material was compacted at too low a moisture content.

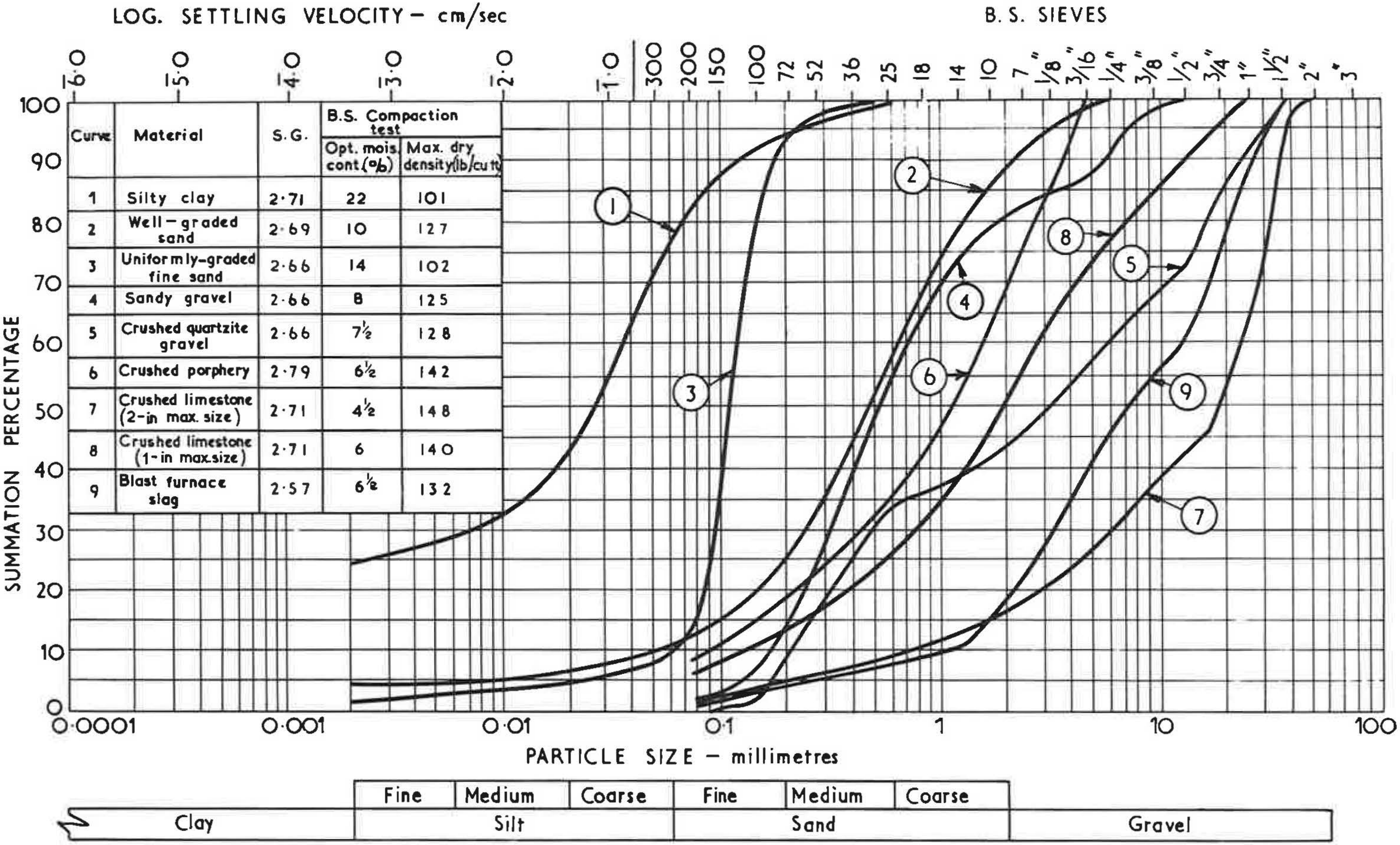
State of Compaction

Specimens are prepared at a dry density as close as possible to that likely to be obtained in practice. It is assumed that a well-compacted stabilized soil will have a dry density equivalent to an air content of 5 percent. The appropriate dry density can then be determined from the specific gravity of the soil particles and the cement and the moisture content of the soil-cement. The required weight of the soil-cement mixture is compacted in a constant volume mold.

An alternative method of preparing test specimens of stabilized soil is to compact the material to refusal using an electric vibrating hammer in a manner similar to that used for making concrete test specimens. Dry density is usually slightly higher than can be consistently produced by the compaction plant normally used for soil stabilization work.

If the specimens have a dry density significantly different from the design value, a correction can be applied to the result of the strength test to allow for the effect of dry density on the strength of the stabilized soil. The importance of making this correction cannot be overstressed as failure to achieve the design state of compaction in practice can have a profound effect on the strength and durability of the material.

Evidence (3) of the importance of obtaining a high state of compaction in cement-stabilized material was obtained in an investigation carried out with a range of soils and crushed stone (Fig. 8). A linear relation was obtained between the unconfined compressive strength of the cement-stabilized material and its dry density when the test results were plotted on logarithmic scales (Fig. 9). The relation between



Curve	Material	S.G.	B.S. Compaction test Opt. mois. cont (%)	 Max. dry density (lb/cu ft)
1	Silty clay	2·71	22	101
2	Well-graded sand	2·69	10	127
3	Uniformly-graded fine sand	2·66	14	102
4	Sandy gravel	2·66	8	125
5	Crushed quartzite gravel	2·66	7½	128
6	Crushed porphery	2·79	6½	142
7	Crushed limestone (2-in max. size)	2·71	4½	148
8	Crushed limestone (1-in max.size)	2·71	6	140
9	Blast furnace slag	2·57	6½	132

Figure 8. Materials used in study of effect of dry density on unconfined compressive strength.

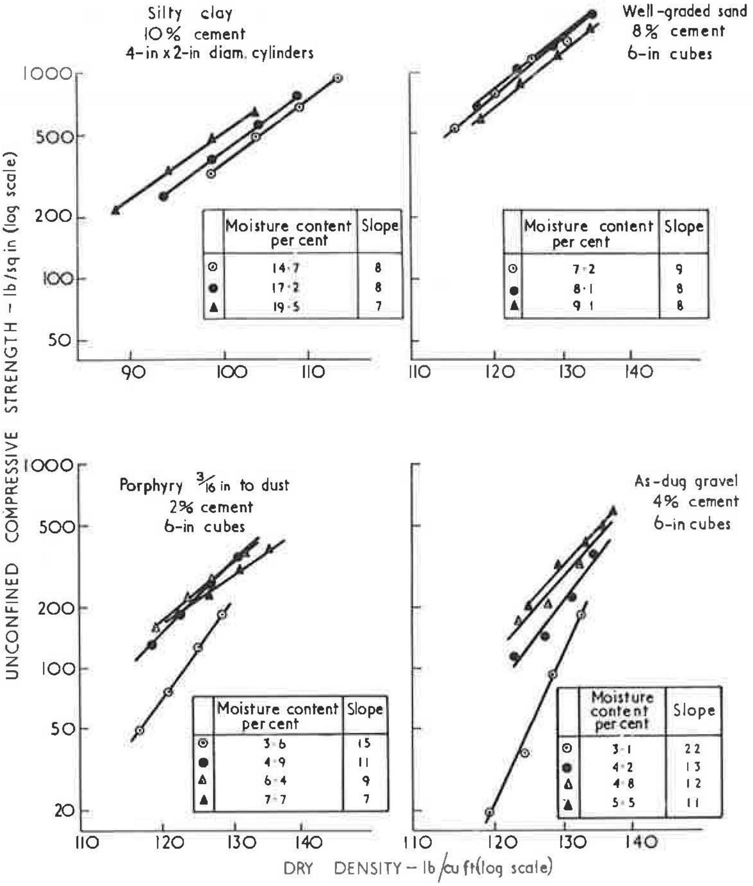


Figure 9. Relations on logarithmic scales between 7-day unconfined compressive strength and dry density for stabilized materials at various moisture contents.

strength and density was

$$S = KD^n$$

in which

S = compressive strength;
D = dry density; and
n and K = constants

The slope of the relation n was found to depend on the type of material and its moisture content, the effect of the cement content being very small. Table 1 shows that the value of n ranged between 5 and 22 with an average of about 10. Considering the average value of n = 10, this means in effect that a change in dry density of only 1 percent will produce a change in the strength of the stabilized materials of 10 percent. This effect is now widely recognized in Great Britain and it has had a considerable influence on both the design and control of cement-stabilized bases.

DETERMINATION OF CEMENT CONTENT TO MEET A SPECIFIED STRENGTH

The tests for determining the strength of stabilized soils are those described in British Standard 1924:1957 (2). These tests require the use of cylindrical specimens having a height-diameter ratio of 2:1. Soils are divided into three groups: fine-grained, medium-grained and coarse-grained. The lengths of specimens for these three soil groups are 4, 8 and 12 in., respectively. Experience has shown that the 12-in. specimens are too cumbersome and heavy to handle; it seems probable that the next revision of the standard specification will require 6-in. cubical specimens instead of 12-in. long cylindrical specimens for coarse-grained soils.

The specimens are cured at constant moisture content; this is usually achieved by coating the surface with paraffin.

Strength determination is usually made at 7 days; the relation is then determined between the 7-day strength of the stabilized soil and the cement content, from which the design cement content is determined. Allowance is made, in the specification for the stabilized soil, for the higher strength achieved with cubical specimens as compared with cylindrical specimens with a height/diameter ratio of 2:1.

Check tests are made in accordance with British Standard specification No. 12 (11), and particular attention is paid to the results of mortar cube tests. In this way allowance can be made for any difference in the properties of the cement used in the laboratory tests and the actual stabilization work.

DURABILITY OF CEMENT-STABILIZED SOILS

Experience suggests that cement-stabilized soil made strictly in accordance with specifications is a durable road base material with considerable water and frost resistance. In cases where the material has lacked durability, there has usually been evidence either that the material was inadequately compacted during construction or that it was mixed at too low a moisture content. Laboratory tests made to examine the effects of water and frost on cement-stabilized soils confirm that materials made to specification are highly durable; the addition of even small amounts of cement to a frost-susceptible material reduces the heave to negligible proportions (Fig. 10). Therefore, durability tests are made only when it is suspected that the stabilized soil may be affected by either water or frost as a result of some special soil characteristics. There are two characteristics which have to be considered with British soils: the presence of expansive clay minerals in cohesive soils and weak porous particles in granular soils. With stabilized cohesive soils, the swelling of small aggregations of clay on an increase in

TABLE 1

VALUES OF CONSTANT n IN THE RELATION $S = KD^n$ FOR MATERIALS STABILIZED WITH PORTLAND CEMENT

Material	Cement Content (%)	Moisture Content (%)	n
Silty clay	10	14.7	8
		17.2	8
		19.5	7
Well-graded sand	8	7.2	9
		8.1	8
		9.1	8
Uniformly-graded fine sand	4	14	8
	7	14	8
	10	14	8
Sandy gravel	4	3.1	22
		4.2	13
		4.8	12
		5.5	11
Crushed quartzite gravel (1½-in. max.)	4	3.7	13
		5.4	12
		7.2	11
		8.7	10
Crushed porphyry (3/16-in. max.)	2	3.6	15
		4.9	11
		6.4	9
		7.7	7
Crushed limestone (2-in. max.)	2	3.3	12
		4.0	9
		4.9	6
		5.5	5
Crushed limestone (1-in. max.)	2	3.0	17
		4.3	11
		5.8	7
		7.0	5
Blastfurnace slag (1½-in. max.)	2	4.1	16
		5.3	12
		5.7	11
		6.4	8

moisture content may disrupt the bonds holding the aggregations together and result in considerable softening of the material (Fig. 11). With stabilized granular soils having porous particles, disruption of the material can result by the action of frost forming ice lenses from water drawn through the relatively permeable porous material (Fig. 12). In such cases, tests are useful for determining whether a stabilized material having adequate durability can possibly be produced by increasing cement content.

In B.S. 1924:1957 there is a test for determining the effect of soaking stabilized soils in water. The unconfined compressive strength of a number of cylindrical specimens of stabilized soil is determined at 14 days. Half the specimens are cured at a constant moisture content for this period. The remaining specimens are cured at a constant moisture content for 7 days and then immersed in water for 7 days. The average strength of the immersed specimens is expressed as a percentage of that of the specimens maintained at a constant moisture content. Stabilized soils which are known to be durable rarely suffer a loss in strength of more than 10 percent when subjected to this immersion test.

In the frost test (B.S. 1924:1957), the reduction in the unconfined compressive strength of cylindrical specimens subjected to repeated cycles of freezing and thawing is expressed as a percentage of the strength of specimens of the same age but which have been maintained to constant temperature and moisture content. All specimens are cured at constant temperature for 7 days. Then half the specimens are subjected to 14 cycles of freezing for 16 hours at -5 C and of thawing for 8 hours at 25 C. Freezing conditions are applied only to the top face of the specimen and the bottom face is immersed to a depth of ¼ in. in water at a temperature of 8 C. The test has been criticized on the grounds that there is insufficient control of the temperature conditions during the freezing period and the test needs further development.

Another frost test makes use of compacted samples 6 in. high and 4 in. in diameter. Nine such specimens can be

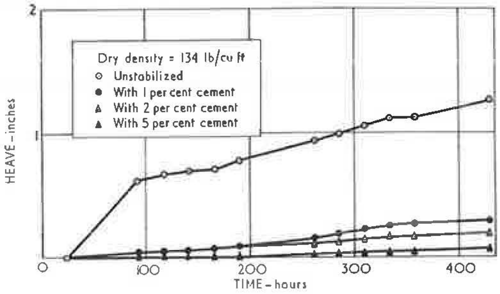


Figure 10. Effect of cement content on frost heave for natural gravel samples stabilized with ordinary portland cement.

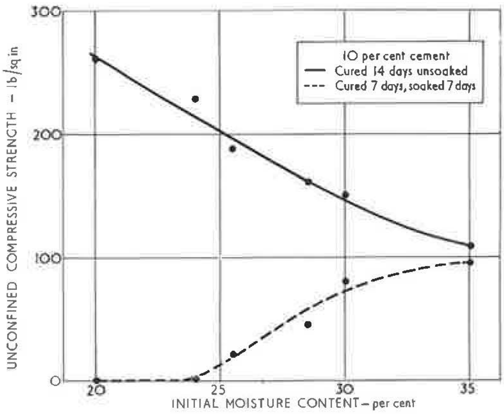


Figure 11. Effect of initial moisture content on loss in strength of cement-stabilized cohesive soil due to immersion in water.

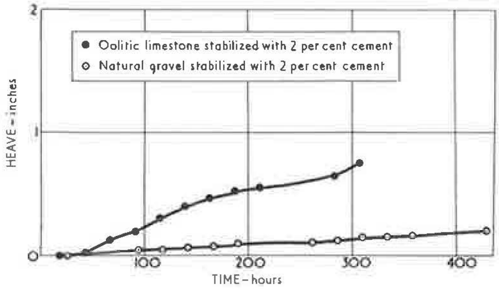
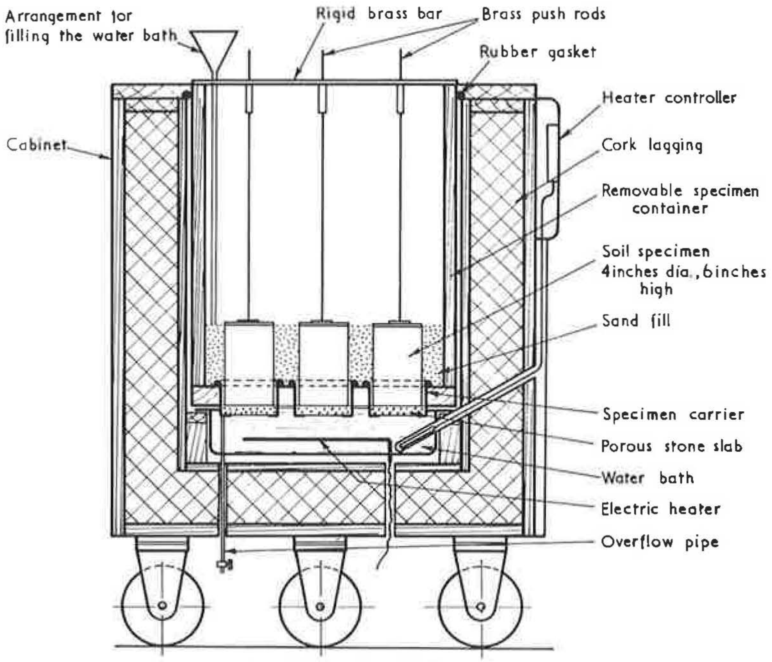


Figure 12. Comparison of frost heave of a porous limestone and a non-porous gravel, both stabilized with ordinary portland cement.

accommodated in the apparatus shown in Figure 13. The specimens stand together on porous ceramic trays in contact with water maintained at a temperature of 4 C. The space between the samples is packed with a coarse dry sand leaving only the upper surfaces exposed to a refrigeration temperature of -17 C. The top of each specimen reaches an equilibrium temperature of -6 C to -10 C, and the zero isotherm remains at a point approximately halfway down the sample. The temperature conditions specified remain constant for about 14 days. Any heave that occurs in the materials is measured daily by push rods in contact with waxed-hardboard caps which cover the top faces of the samples. The frost susceptibility of the stabilized materials is assessed in terms of the amount of heave that occurs after 250 hours of freezing. A heave of more than $\frac{1}{2}$ in. is regarded as an indication of inadequate resistance to severe frost conditions. This criterion is only tentative, and laboratory research is being carried out to investigate its validity.



SECTIONAL ELEVATION

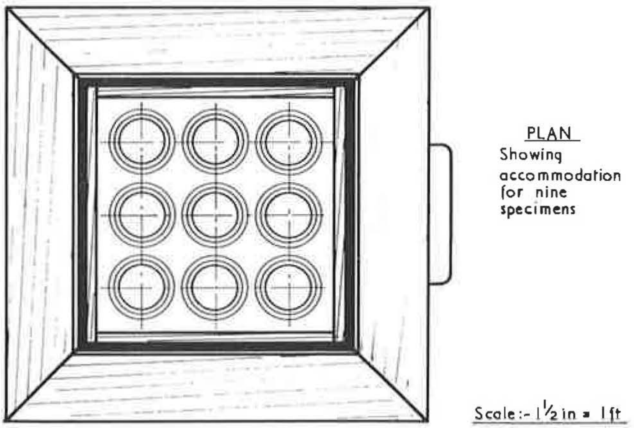


Figure 13. Apparatus for investigating frost action in road materials.

PAVEMENT DESIGN

Although cement-stabilized soil was used during World War II for the construction of military airfields, it has only been used on a considerable scale in Great Britain as a constructional material for roads since 1945. The following briefly summarizes the use of stabilized soil in road pavements.

Minor Roads

Minor roads are principally housing estate roads, lightly-trafficked rural roads, and car parks carrying less than 150 commercial vehicles per day. Soil-cement bases approximately 6 in. thick and usually surfaced with a double surface dressing or thin pre-mixed bituminous surfacing have been used in a considerable mileage of such roads.

The bases were constructed mostly by the mix-in-place method on subgrades having a California bearing ratio (CBR) value generally more than 6 percent. Preliminary design work on the composition of the stabilized soil was usually not as rigorous as that described previously in this paper. After determining the soil suitable for stabilization, laboratory tests determined the cement content necessary for an unconfined compressive strength of 250 psi with cylindrical specimens at 7 days. The origin of this criterion was research carried out at the British Road Research Laboratory in 1939, which showed that a minimum compressive strength of 250 psi at 7 days was required to insure that cement-stabilized soils satisfied the requirements of the American wet-dry and freeze-thaw tests.

Main Roads

The structural design of main roads is governed by the requirements of Road Research Note No. 29 (12). This document, prepared by the British Ministry of Transport and the Road Research Laboratory, contains recommendations for the design of bituminous-surfaced and concrete pavements for various categories of roads defined by the amount of commercial traffic they will carry 20 years after construction. Cement-stabilized soil is permitted as the base material for flexible roads carrying light and medium traffic (i.e., roads estimated to carry less than 1,500 commercial vehicles per day in 20 years). In addition cement-stabilized soil is permitted as the subbase material for both bituminous-surfaced and concrete roads. The material is not permitted as the base of bituminous-surfaced roads carrying heavy traffic, since excessive deformation has occurred on some stretches of heavily-trafficked bituminous roads having cemented bases.

For any given category of road, the pavement design of bituminous-surfaced roads consists of a constant thickness of surfacing and base with a variable thickness of subbase depending on the CBR value of the subgrade. A 6-in. minimum thickness of stabilized soil base is recommended for roads estimated to carry from 150 to 450 commercial vehicles per day; for roads estimated to carry between 450 and 1,500 commercial vehicles per day an 8-in. base is required. With the heaviest category of traffic (more than 4,500 commercial vehicles per day) the thickness of the recommended subbase can be as much as 20 in. (for CBR of 2 percent), but in such cases it is usual to employ a suitable granular subbase material and to stabilize if necessary the top 6 in. of the subbase, generally by mix-in-place method. This stabilized layer, in addition to complying with the strength requirements of the subbase (a CBR value of at least 20 percent for the heaviest category of traffic), also provides an excellent working platform for the construction of the base. By constructing a stabilized soil subbase during good weather, it has often been possible to carry on with the construction of the base during the wet winter months, whereas work has sometimes had to be suspended when unstabilized gravel subbases have been used in similar circumstances.

Two large full-scale road experiments will be carried out by the Road Research Laboratory to determine the conditions under which cemented bases can be successfully used in heavily-trafficked bituminous roads (13). Various compositions of the cemented base will be obtained by using different gradings of aggregate and different strengths of the cemented material. Bases will be constructed to a constant thickness beneath a constant thickness of bituminous surfacing at one site, and certain compositions will be laid to form bases having a range of thicknesses under different types and thicknesses of surfacing at a second site.

SPECIFICATIONS FOR CEMENT-STABILIZED SOIL BASES AND SUBBASES

In the early stages of the development of soil stabilization in Great Britain, the specifications were usually of the "methods" type; that is, the mix proportions (cement content and moisture content) were specified together with the details of the plant to be employed and the method of carrying out the work. The control of the process was thus limited very largely to supervision of the constructional procedure.

With the increased use of stabilized soil and the greater knowledge of the properties of the material which have become available in recent years, the form of the specification has changed. The properties of the final product are specified, and the contractor is given considerable freedom in the way the process is carried out. Although in this "end product" form of specification a considerable amount of control testing is required, it results in a stabilized layer having more consistent properties which is likely to perform more satisfactorily under heavy traffic conditions. It also results in some saving in the amount of cement required for a given strength for the layer and encourages the development and use of improved processing plant.

The main factors now specified are the minimum state of compaction of the layer and the minimum unconfined compressive strength of specimens of the stabilized material made to the same state of compaction. The most suitable moisture content for the stabilized material is determined from preliminary full-scale trials carried out under the supervision of the engineer responsible for the work.

Compaction Requirement

It is usual to define the minimum state of compaction required in stabilized soil in terms of a maximum permissible air content for the material. The maximum air voids usually permitted is 5 percent; that is, the stabilized material has to be compacted to a dry density of at least 95 percent of the saturation dry density. This corresponds approximately to a relative compaction of at least 100 percent of the maximum dry density of the British Standard compaction test which is almost identical to AASHO T99-57.

Specification of the state of compaction in terms of air voids is considered to have the advantage of simplifying the control work where variations in the type of material occur. Thus, in the relative compaction procedure such variations would require frequent laboratory compaction tests to be made before the results of the dry density measurements could be interpreted, whereas the value of the air void requirement can be applied without change to a wide range of materials provided they are reasonably well graded. It must be stressed that in the air void method the actual control of the state of compaction is carried out by dry density measurements and it is only in the interpretation of such results that the air void criterion is employed.

Strength Requirements

It is now usual in Great Britain to specify the strength of stabilized materials in terms of a minimum unconfined compressive strength of the completed layer. No consistent value of the minimum strength has been employed in the past but generally it has been of the order of 250 to 500 psi for specimens cured at constant moisture content for 7 days. The most recent Ministry of Transport specifications for stabilized soil require a minimum value of 400 psi for cylindrical specimens having a height/diameter ratio of 2:1 and 500 psi for cubical specimens. These values refer to the mean of five test specimens made on samples of the stabilized material taken at random over each 1,000 sq yd of completed layer. The specimens have to be compacted to a dry density within 2 pcf of the average value being achieved in the stabilized layer.

Although a lower strength has often been required for subbases in comparison with that for bases, the present tendency is to require the same strength for both as it is now recognized that subbases must withstand high stresses from construction traffic.

A recently revised version of the Ministry of Transport Specification (14) is given in the Appendix.

CONTROL TESTS FOR CEMENT-STABILIZED SOILS

Tests for Compaction and Moisture Content

The sand-replacement test is usually employed for determining the dry density of compacted stabilized layers. Generally, five test determinations are made at random over each 1,000 sq yd of completed layer to provide a mean value for control purposes. Where the results are required very quickly, rapid methods of moisture content determination (15) may be employed but these are not considered to be as reliable as the oven-drying procedure.

Comparisons have been recently made of the results of dry density measurements of cement-stabilized bases determined by the sand-replacement test and on cores cut from the hardened material. This work has indicated that the sand-replacement test can sometimes be seriously in error, the results being up to 10 pcf higher than those for the cores. The probable cause of this error is the slight inwards collapse of the sides of the hole made for the sand-replacement test during excavation. The largest errors have occurred when wet unstable granular material has been employed. As a result of these findings, dry density measurements by the sand-replacement test are now delayed for at least 4 hours after the completion of the compaction work to allow the material to acquire an initial set. This procedure has been shown to result in reasonable agreement between the core measurements and the sand-replacement test.

Studies have been made at the British Road Research Laboratory of the use of gamma and neutron radiation methods of determining the density and moisture content of compacted layers, but this work has indicated that serious errors in the measurement of dry density can result from existing methods. Both the type of material and the density gradients in the compacted soil layer have been found to have a serious effect on the calibration of the apparatus.

Strength Tests

The control of the strength of stabilized soil in practice is carried out by determining the unconfined compressive strength of the material using the previously described test procedures.

For control purposes the strength of stabilized material is normally determined at 7 days. However, research (16) carried out by the Road Research Laboratory has indicated that it is possible to test specimens at an age of only 1 or 2 days and to use the results to predict the strength likely to develop at 7 or 28 days. The accuracy of such predictions (about 10 percent) is thought to be sufficiently close for field control work and any disadvantage in the error of prediction would be offset by the advantage of obtaining the strength at an early stage. However, this early testing procedure has not yet been applied on any large-scale work.

Cement Content Tests

Current British specifications for soil stabilization do not normally specify the cement content of the material. The contractor is responsible for complying with the minimum strength requirements. However, instances may sometimes arise where it is desirable to determine the cement content (such as in connection with trials to determine the performance of the processing plant). Either the E. D. T. A. method or the flame photometer method can be employed. Full details of these two methods are given in Road Research Road Note No. 28 (17). Both methods are based on a comparison of the calcium content of the soil-cement mixtures with that of the soil and cement. For this reason neither method is applicable to soils with high or variable calcium contents and in such cases a less accurate method based on determination of the sulfate content has to be employed.

ACKNOWLEDGMENT

This work was carried out as part of the program of the Road Research Board of the Department of Scientific and Industrial Research, United Kingdom. The paper is published by permission of the Director of Road Research.

REFERENCES

1. Grimer, F. J., and Ross, N. F., "The Effect of Pulverization on the Quality of Clay-Cement." Proc. 4th Internat. Soil Mech. Conf., 2: 109-13 (1957).
2. British Standard No. 1924:1957, "Methods of Test for Stabilized Soils." British Standards Institution, London (1957).
3. Grimer, F. J., "Studies of Factors Affecting the Strength of Cement-Stabilized Materials." Dept. of Scientific and Industrial Research, Road Research Tech. Paper No. 65 (in press).
4. Sherwood, P. T., "Effect of Soil Organic Matter on the Setting of Soil-Cement Mixtures." Dept. of Scientific and Industrial Research, Road Research Tech. Paper No. 61 (1962).
5. Sherwood, P. T., "Effect of Sulfates on Cement- and Lime-Stabilized Soils." HRB Bull. 353, 98-107 (1962).
6. British Standard No. 1377:1961, "Methods of Test for Soil Classification and Compaction." British Standards Institution, London (1961).
7. Williams, F. H. P., and Maclean, D. J., "The Compaction of Soil: A Study of the Performance of Plant." Dept. of Scientific and Industrial Research, Road Research Tech. Paper No. 17 (1950).
8. Lewis, W. A., "Further Studies in the Compaction of Soil and the Performance of Compaction Plant." Dept. of Scientific and Industrial Research, Road Research Tech. Paper No. 33 (1954).
9. Lewis, W. A., "Investigation of the Performance of Pneumatic-Tyred Rollers in the Compaction of Soil." Dept. of Scientific and Industrial Research, Road Research Tech. Paper No. 45 (1959).
10. Lewis, W. A., "Recent Research into the Compaction of Soil by Vibratory Compaction Equipment." Proc. 5th. Internat. Soil Mech. Conf., 2: 261-8 (1961).
11. British Standard No. 12: 1958, "Portland Cement (Ordinary and Rapid Hardening)." British Standards Institution, London (1958).
12. "A Guide to the Structural Design of Flexible and Rigid Pavements for New Roads." Dept. of Scientific and Industrial Research, Road Note No. 29 (1960).
13. Lee, A. R., and Croney, D., "British Full-Scale Pavement Design Experiments." International Conf. on Structural Design of Asphalt Pavements, Univ. of Michigan (1962).
14. "Specification of Road and Bridge Works." Ministry of Transport and Civil Aviation (in press).
15. Croney, D., and Jacobs, J. C., "The Rapid Measurement of Soil Moisture Content in the Field." Roads and Road Construction, 29: 343, 191-4 (1951).
16. Grimer, F. J., and Krawczyk, J., "Strength/Age Relations for Soil-Cement with Particular Reference to the Prediction of Later Strengths from Earlier Ones." Magazine of Concrete Research (1963, in press).
17. "The Determination of the Cement or Lime Content of Cement- or Lime-Stabilized Soils." Dept. of Scientific and Industrial Research, Road Note No. 28 (1960).

Appendix

CURRENT SPECIFICATION IN USE IN GREAT BRITAIN FOR CEMENT STABILIZED MATERIALS

This specification, which forms part of the revised specifications for Road and Bridge Works (14), was prepared by the British Ministry of Transport with the assistance of the British Road Research Laboratory.

(804) Cement stabilized materials for bases and sub-bases

Materials to be stabilized

The material used for stabilization may be a naturally occurring soil, a washed or processed granular material, crushed rock or slag, an industrial waste product (such as pulverised fuel ash, burnt colliery shale) or any combination of these providing the material is free from organic contamination which would affect the setting of the cement and does not contain such a proportion of sulphates or other chemical that the long-term durability of the stabilized material will be affected (See notes for guidance of engineers).

The material shall be well-graded with a coefficient of uniformity of not less than 10 and have a grading finer than the following limits:-

B.S. sieve size	Percentage passing
2 in	100
$1\frac{1}{2}$ in	95
$\frac{3}{4}$ in	45
$\frac{3}{8}$ in	35
$\frac{3}{16}$ in	30
No. 7	25
No. 25	12
No. 52	5
No. 200	0

If the material is plastic it shall have a liquid limit not greater than 45 per cent and a plastic limit not greater than 20 per cent as determined in accordance with B.S. 1377.

In the event of the contractor offering a material having a grading curve falling slightly outside the limits stated in the Table, or plasticity properties slightly outside the limits given above, the Engineer may approve its use subject to his being satisfied, as a result of such tests as he may require that it meets all other requirements specified in the Clause. The material may, however, prior to its use in the works be subject to disapproval by the Engineer if, although having acceptable grading or plasticity properties, it is shown during the preliminary trials to be incapable of producing a well closed final surface to the compacted layer, or if the compacted stabilized material is considered to be frost susceptible.

Cement and cement content

Ordinary Portland or other approved cement to B.S. 12: Portland cement or Portland blastfurnace cement to B.S. 146: Portland blastfurnace cement shall be used. The cement content shall be such as to provide a crushing strength to the requirements stated in this Clause.

Moisture content

The moisture content of the mixed cement stabilized material shall be determined from preliminary field compaction trials, using the mixed material, employing the type of plant and method of operation which has been approved by the Engineer for the main work and using the optimum moisture content as determined in the laboratory compaction test using heavy compaction (B. S. 1377 Test 11) as a basis.

If water has to be added during mixing it shall be free from organic contamination and the source of supply shall be approved by the Engineer.

Method of stabilization to be used

If the layer to be stabilized does not exceed 6 inches in compacted thickness it may be constructed in one layer within the range 3-6 in after compaction using either mix-in-place or stationary plant for the mixing process provided the plant meets the requirements specified in the Clause. If the course to be stabilized exceeds 6 inches in compacted thickness it shall be constructed in two or more layers each within the range 3 in to 6 in in thickness when compacted. When two or more layers are employed the mix-in-place process will only be permitted for the construction of the bottom layer.

Mixing

The plant to be used for pulverising and mixing the stabilized material shall be approved by the Engineer on the basis of preliminary trials to establish that the plant is capable of producing the degree of mixing and uniformity of the stabilized material specified in this Clause.

If stationary plant is used it shall be of the power driven paddle or pan type and may be of the batch or continuous type. When mix-in-place construction is employed with plastic soils, the mixer shall be of the single-pass type and the degree of pulverisation as determined in accordance with B. S. 1924 achieved in one pass shall not be less than 80 per cent. With non-plastic materials both single and multi-pass equipment will be permitted.

The proportioning of the cement in the stabilized mixture shall be by weight or, if approved by the Engineer, by volume.

If batch mixers are used the appropriate measured amounts of material and cement shall be delivered into the mixer. Water may be added during mixing to bring the moisture content of the resulting mixture to the optimum moisture content for compaction as determined by the preliminary trials. Special care shall be taken with batch type paddle mixers to ensure that the cement is spread uniformly in the loading skip so that it is fed uniformly along the mixing trough and that with both paddle and pan mixers the cement is proportioned accurately by a separate weighing or proportioning device from that used for the material being stabilized. Mixing shall be continued until the mixture has the uniformity required by this Clause and for not less than one minute unless a shorter minimum period is permitted by the Engineer, after satisfactory preliminary trials.

If continuous mixing is used the paddles, baffles and rate of feed of material shall be adjusted to give a uniformly mixed material. The spray bar distributing water into the mixer, if it is required, shall be adjusted to give uniformity in moisture content throughout the mix.

If the mix-in-place process is used the mixers shall be equipped with a device for controlling the depth of processing and the mixing blades shall be maintained or reset periodically so that the correct depth of mixing required is obtained. The cement shall be spread ahead of the mixer by means of a cement spreader of a type to be approved by the Engineer fitted with control gates or other device to ensure a uniform and controllable rate of spread of cement both transversely and longitudinally.

If multi-pass equipment is being employed, the soil shall first be pulverised to the required depth and degree with successive passes and the moisture content adjusted if it is more than 3 per cent below the value required for compaction. The cement shall then be spread and mixing continued with successive passes until the required depth and uniformity of processing has been obtained.

With single-pass equipment the forward speed of the machine shall be selected, in relation to the rotor speed, such that the required degree of mixing, pulverisation and depth of processing is obtained.

The machine shall also be set so that it cuts slightly into the edge of the lane processed previously so as to ensure that all the material forming the layer has been properly processed. If it is necessary to adjust the moisture content of the material to the optimum for compaction, water shall be added during the mixing operation using a water sprayer of such a design that the water is added in a uniform and controlled manner both transversely and longitudinally.

Where hard non-plastic soil is encountered in situ, the Engineer may approve the use of a scarifier or preypariser ahead of the mixer but with plastic soils no prior scarification will be permitted unless the soil is subsequently recompacted before processing with the single-pass mixer.

The output of the mixing plant shall be such that a minimum rate of 20 linear yards per hour measured longitudinally of completed stabilized layer can be maintained in order to permit satisfactory compaction of the material.

Transporting and spreading plant-mixed material

The plant used for transporting the mixed material shall have a capacity suited to the output of the mixing plant and the site conditions and shall be approved by the Engineer. All transporting plant shall be capable of discharging cleanly.

The mixed material shall be removed directly from the mixer after mixing and transported directly to the point where it is to be laid. The mixed material shall be covered during transit and while awaiting spreading to prevent it from drying off from wetting by rain. Where the stabilized material is being used to form a base layer it shall be spread and tamped evenly without delay by an approved paver to the levels and shape to give, after compaction, the specified thickness of layer and surface regularity. Where the conditions or location preclude the operation of a paver or where the material is being used for a sub-base layer, the material may be spread by any other approved method which is shown to avoid segregation of the material and which will produce after compaction the specified levels, shape, thickness of layer and surface regularity.

Compaction

Compaction shall commence as soon as possible after the mixed material has been spread and shall be completed within a period of two hours of mixing or such shorter period as may be necessary in drying weather.

Compaction shall be carried out initially with a 2-3 ton smooth-wheeled roller followed by an 8-10 ton smooth-wheeled roller, and finished, if necessary with a 2-3 ton smooth-wheeled roller or the compaction can be carried out by such other means such as pneumatic-tyred or vibrating rollers, dropping weight or vibrating plate compactors as are approved as a result of compaction trials. The work of compacting shall be continued in such a manner as to produce throughout the full depth of layer an average dry density corresponding to not more than 5 per cent air content at the moisture content at which the stabilized material is compacted or such other air content as the Engineer shall permit as a result of the preliminary compaction trials.

Where it is necessary to employ more than one layer of stabilized material, the material for each successive layer shall, subject to the following proviso, be placed and compacted within two hours of the completion of the compaction of the layer beneath. Where it is not possible to achieve this and the two hour limit has to be exceeded, the surface of any layer remaining so exposed shall be subjected to the curing process required by this Clause.

Special care shall be taken to obtain full compaction in the vicinity of both transverse and longitudinal construction joints and the Contractor shall, if required, provide special small compactors to assist in this work. Any loose uncompacted material left in the vicinity of construction joints shall be removed prior to the placing of fresh stabilized material.

The approval of the Engineer shall be subject to each layer on completion of compaction being well closed, free from movement under the roller, from compaction planes, ridges, cracks or loose material and, within the tolerance for surface finish allowed in this Clause, true to the lines and levels shown on the Drawings. All loose, segregated or otherwise defective areas shall be broken out to the full depth of the layer and recompacted. If this cannot be carried out within the two hour limit specified, the material broken out shall be removed and replaced with freshly processed and properly compacted material without extra charge.

Protection and curing

The surface of any layer of stabilized material, unless it is to be covered within two hours by another layer of the material or other pavement course, and any exposed edges shall be cured as soon as compaction is completed for a period of at least 7 days. Curing shall be achieved by any of the following methods:

(1) Covering the surface of the stabilized layer with an approved impermeable plastic sheeting laid so that joints in the sheeting are overlapped by at least 3 feet and held down at intervals by suitable means so that the sheets will not be blown off the layer by wind.

(2) Spraying the surface of the stabilized layer with an approved quick breaking 55 per cent bitumen emulsion at a rate not lighter than 6 sq yd per gallon of emulsion. Where it is necessary to limit heat absorption of the base, the bitumen emulsion shall, where directed by the Engineer, be lightly blinded with coarse sand or fine gravel at a rate of 200 sq yd per ton.

(3) On very small schemes where the Engineer does not require methods (1) or (2) to be used, covering the stabilized layer with at least a 3-in thick layer of suitable soil which is kept in a damp condition by periodic spraying with water during the curing period and completely removed on completion of this period.

During frosty weather the stabilized layer shall be protected by means of a layer of straw or soil at least 3 inches thick covered by impermeable sheeting and maintained in position during the period of the frost. Work involving the use of cement shall not be continued when the descending air temperature in the shade falls below 38° F nor shall it be resumed until the ascending air temperature reaches 38° F. Frozen material shall not be used in the stabilized layer and all work shall be discontinued if, in the opinion of the Engineer the weather is in any way likely to be harmful to the process of construction.

Where side forms are used in the construction of the stabilized layer they shall be firmly secured in place and not removed until at least 6 hours after the completion of the compaction work. The edges exposed by such removal shall be protected from drying and be cured for a period of at least 7 days after construction by use of any of the methods listed above.

Construction traffic or other vehicles shall not use any stabilized layer until it has been cured for a period of at least 7 days. Thereafter, the use of the stabilized layer by traffic shall be subject to the requirements of Clause 27/20.

If shown on the Drawings or stated in the Bill of Quantities, the stabilized layer shall, after curing and approval be then sealed by surface dressing in accordance with Clause 914 but using instead variations (b), (e) and (f) in Clause 710.

Joints

The Contractor shall so organise his work that longitudinal joints against hardened stabilized material are avoided as far as possible. Wherever possible, in any day's work the area constructed shall extend the full width of the carriageway. At the end of each day's work on completion of compaction, the transverse edge of the layer shall if stabilized be feathered out and shall be cut back vertically to the full depth of construction of the layer before work starts again. Alternatively the work may be terminated against an approved stop end. When the joint has been cut back or formed it shall be adequately protected from drying out. On resumption of work the vertical face of the joint shall be brushed to remove loose material and freshly mixed stabilized material shall be butted tightly against the previous work. Joints in the layers, where more than one layer is required, shall be staggered a distance of 5-10 ft.

Accuracy of surface and the thickness of stabilized base or sub-base

The surface of the completed stabilized base or sub-base shall be within the limits of tolerance of surface irregularity permitted in the specification for surface levels of flexible and concrete bases (Clause 27/18).*

Preliminary trials

At least ten days before the main work of stabilization is started, the Contractor shall construct an area of stabilized material of 500-1,000 sq yd extent as stated in the Bill of Quantities as a preliminary trial at a site to be approved by the Engineer. For this trial the Contractor shall use the materials, mix proportions, mixing, laying, compaction plant and construction procedure that is proposed for the main work. The preliminary trial is to test the efficiency of mixing, spreading and compaction plant and the suitability of the methods and organisation proposed by the Contractor. The results of the dry density measurements made in the stabilized trial area will be used to confirm the moisture content and minimum state of compaction to be attained in the main stabilization work.

Crushing strength and uniformity of the mixed material

The average crushing strength at an age of 7 days for each batch of 5 test specimens made and tested in accordance with Clause 30/2 shall not be less than 400 lb/sq in for cylindrical samples having a height/diameter ratio of 2:1 or 500 lb/sq in for cubical specimens. If an area of stabilized layer represented by the 5 test specimens has an average strength less than the specified minimum values the area shall be replaced with acceptable stabilized material.

In addition to complying with the minimum specified strength, the stabilized material shall have such a uniformity that the root mean square value of the coefficient of variation of crushing strength of five successive batches of 5 test specimens shall not exceed 40 per cent.

30/2 Provision for testing cement-stabilized material

Samples of the mixed cement-stabilized material shall be taken from the site immediately prior to the compaction of the material. Five samples shall be taken at random times and spacings over each 1,000 sq yd of completed layer or such other area as the Engineer decides. From each sample a cylindrical or cubical test specimen as appropriate to the type of material shall be prepared, cured and tested using the procedure given in B. S. 1924. The specimen shall be compacted to a state of compaction within 2 lb/cu ft of the average density being achieved in the compacted stabilized layer. After preparation the specimens shall be cured at constant moisture content within the range of curing temperatures given in B. S. 1924 for a period of 7 days. The specimens shall then be weighed, the dimensions checked and tested in unconfined compression. A representative portion of the crushed material from each specimen shall be retained for a moisture content determination in accordance with B. S. 1924 and the dry density of each test specimen shall then be determined using the weight, dimensions and moisture content of the material.

The results of the 5 test specimens shall be averaged to give a representative figure for the area from which the samples were originally taken.

To obtain a comparable average value for the state of compaction of the stabilized layer, 5 determinations of dry density shall be made at random over each 1,000 sq yd of stabilized layer or such other area as the Engineer decides and the results averaged. The method to be used for determining the dry density shall be the sand-replacement method according to British Standard 1377; Methods of Testing Soils for Civil Engineering purposes. The measurements shall be made at least 4 hours after the completion of the compaction work and preferably within a period of 24 hours.

---

*This clause requires an accuracy of surface level of ± $3/4$ in for sub-bases and ± $1/2$ in for bases of flexible pavements.

Development of a Freeze-Thaw Test for Design of Soil-Cement

KALANKAMARY P. GEORGE and DONALD T. DAVIDSON, respectively,
Research Associate, and Late Professor of Civil Engineering, Iowa State University

A laboratory test for evaluating the durability of stabilized fine-grained soils subjected to repeated freeze-thaw cycles has been developed. Test specimens (2-in. by 2-in. diameter) molded in Iowa State compaction apparatus are used. The specimens are kept frozen from top with free water available at bottom. Winter climatic conditions in Iowa roads are simulated taking into consideration field conditions, such as a freezing temperature, availability of free water, and a proper temperature gradient. Climatic data and freeze-thaw calculations showed that 10 cycles of freezing and thawing are sufficiently severe for testing base courses. The criteria were the unconfined compressive strength and the index of resistance to the effect of freezing, defined as the ratio of the unconfined compressive strength of the freeze-thaw specimen to that of the immersed control specimen.

Laboratory test results are correlated with those of field trial sections of soil-cement base courses. Based on the performance of the test sections in which the cement content was the variable, an adequate soil and cement combination was chosen. The laboratory test results of the soil-cement are proposed as tentative criteria for the design of soil-cement for base courses. From the results of one year's study of the experimental pavement sections, 7 percent cement seems to be adequate for making durable soil-cement, though standard tests warranted 11 percent cement admixture.

•ALL components of pavements for roads and airfields must resist the destructive effects of alternate cycles of freezing and thawing in climates having such seasonal changes. Some laboratory techniques have been developed to evaluate the durability of stabilized soils subjected to repeated freeze-thaw cycles. The two most widely used methods are AASHO Method T 136-57—ASTM Method D 560-57 and British Standard Test 1924:1957.

The AASHO-ASTM method covers procedures for determining the soil-cement brushing weight losses, moisture changes, and volume changes produced by repeated freezing and thawing of hardened soil-cement specimens (1). Soil-cement weight loss as a result of brushing after freeze-thaw cycles is usually the critical criterion affecting mix design. A frequent criticism of this method of test is that it is overly severe and does not simulate field conditions.

The British Standard test determines the change in unconfined compressive strength of specimens of stabilized fine-grained soil when subjected to cycles of freezing and thawing under specified conditions. The British test is not restricted to soil-cement and perhaps simulates field freeze-thaw conditions better than the brushing test.

The British freeze-thaw test has been modified in the soil research laboratory of the Iowa Engineering Experiment Station (4). The principal modification was the use

---

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

of 2- by 2-in. instead of 2- by 4-in. specimens. The field conditions simulated in the modified test were the same as stipulated by the British test, though climatic changes in Iowa were more severe than those in England. The British test conditions should therefore be modified to suit Iowa climate.

DEVELOPMENT OF THE FREEZE-THAW TEST PROCEDURE

The field conditions for deleterious frost action are a freezing temperature, a readily available source of water, a thermal gradient and cycles of freezing and thawing.

In the AASHO-ASTM test the specimen is frozen but, unlike in the field, the freezing temperature is all around the specimen. Hence the thermal gradient is between the interior and all exterior faces of the specimen, not between the top and bottom as in pavements. Furthermore, the freezing temperature and number of freeze-thaw cycles are not varied to simulate climatic changes. The specimen absorbs water during thawing, but during freezing the need of water for ice segregation is satisfied mainly by the redistribution of moisture inside the specimen. Another severe test condition which does not simulate a field condition is the brushing weight loss of the specimen.

The British freeze-thaw test appears to have been designed to approximate the worst conditions for freeze-thaw damage to stabilized pavement components in England; freezing is from the top down. A realistic freezing temperature, a temperature gradient, and a constant source of unfrozen water during freezing and thawing are used. The emphasis is placed on the strength loss of the freeze-thaw specimen as compared with that of the control (continually immersed) specimen. However, no provision is made in the test procedure for adapting the test to different climatic conditions.

The purpose of the test procedure developed in this report is, therefore, to simulate the field freeze-thaw conditions in Iowa.

Establishment of Freezer Temperature

It was decided to have the temperature inside the freezing cabinet equivalent to the average minimum air temperature in Iowa during the winter months, November to March. An analysis of available climatic data of the U. S. Weather Bureau (15) for the 130 observation stations in Iowa over the 8-yr period 1952 to 1960 showed the temperature to be 20 F.

Establishment of Temperature Inside Vacuum Flask

The thermal gradient between the top and bottom surfaces of a specimen in contact with free water governs the movement of water to the freezing zone. Inasmuch as the top of the specimen is subjected to freezing temperature corresponding to the minimum average air temperature in winter, the bottom of the specimen should be kept at a temperature corresponding to that at a specified depth in the pavement or in the underlying soil. An analysis was made of available soil temperature data for Iowa (15) from the five observation stations over the 10-yr period 1950 to 1960. The maximum temperatures at depths of 1, 4, and 20 in. were averaged; the value obtained was 35 F. The overall effect of averaging the temperatures at depths up to 20 in. was to exaggerate the temperature gradient simulated in the 2-in. high test specimen.

Evaluation of Number of Cycles

Strength properties of a test specimen in the laboratory or the load-carrying capacity of a pavement component may be expected to vary appreciably with repeated freezing and thawing. In the thawed state the soil may lose as much as 80 percent of the strength it had prior to freezing (14). Hence thawing represents the critical period for a road base or subbase. Also, a single freezing may cause sufficient ice segregation to reduce the density and increase the porosity so that subsequent frost action effects become more intense. Therefore the load-bearing capacity is greatly influenced by the number and extent of freezing and thawing cycles.

The determination of the number of freeze-thaw cycles was based on a study of the daily maximum and minimum air temperatures obtained from Iowa weather records.

Daily winter temperatures at the 130 observation stations were averaged for a 2-yr period (1958-1960). From the daily maximum and minimum air temperatures, two graphs were plotted (Fig. 1). The median of the two graphs determined the number of cycles of freezing and thawing.

The frost penetration depths (Figs. 2 and 3) are based on the modified Berggren formula (7). The surface of a pavement would experience about 10 cycles of freezing and thawing and the top of the base might experience about 9 cycles in a single winter season.

The depth of freezing and the number of cycles of freezing and thawing have been verified in the field by installing thermocouples under a soil-cement base in Iowa primary highway 37. The results verify the theoretical calculations, in respect to the depth of freezing (Fig. 4). However, the surface experienced relatively few cycles of freezing and thawing. Therefore, the use of 10 cycles of freezing and thawing in the Iowa freeze-thaw test appears sufficiently severe.

Establishment of Thawing Temperature

Rapid thawing of a pavement component to shallow depths produces the most unfavorable condition of supersaturation above a residual layer. Thus, the rate and depth of thawing should be a major factor governing the strength of the material subjected to thawing. Therefore, the evaluation of a thawing temperature suitable to Iowa climate was significant.

Approximate calculations showed that a thawing temperature of 40 F was probably a satisfactory value to use in the test. However, recognizing that complete melting during spring thaw takes place at rather elevated temperatures, a value of 77 F was proposed for the test procedure.

Criteria for Evaluation of Test Results

Unconfined Compressive Strength.—The Iowa freeze-thaw test criterion is that the soil-cement test specimens subjected to 10 cycles of freezing and thawing must have an unconfined compressive strength of minimum specified value.

Index of Resistance to Effect of Freezing.—An important criterion of durability is the change in the unconfined compressive strength of test specimens subjected to freezing and thawing. The index of resistance to the effect of freezing is defined by the ratio of the unconfined compressive strength of the freeze-thaw specimen to that of the control specimen, expressed as a percentage. This criterion is a measure of the relative strength gaining capability of soil-cement during cycles of freezing and thawing.

Heave upon Freezing.—The question has often been asked as to how accurately the results of a laboratory freezing test indicate the frost behavior of a soil in the field, especially since the major problem is loss of strength on melting. According to Lambe (12), the rate of heave does give an indication of strength of thawed soil. The more the test specimen heaves, the greater the amount of water imbibed during freezing. The greater the quantity of water present on thawing, the lower the strength of the thawed soil. A subjective measure of heave may be the maximum heave expressed as a percentage of the initial height.

Moisture Content and Distribution After Tests.—Another criterion to evaluate the results of the Iowa freeze-thaw test may be the change in moisture content of the test specimens. The moisture content of soil at the beginning of freezing largely determines the amount of segregated ice and amount of heaving of the soil on freezing. The moisture content of the test specimens before and after testing and its distribution through the height of the specimen after cycles of freezing and thawing are shown in Figure 5. The soil is a friable loess. On the basis of these data, it may be concluded that the increase in the amount of soil moisture and its distribution through the soil seems to be a promising criterion warranting further investigation.

DEVELOPMENT OF STRENGTH CRITERIA

This part of the research was intended to relate pavement performance to the cement content of the soil-cement base in the field and thus develop criteria for soil-cement design.

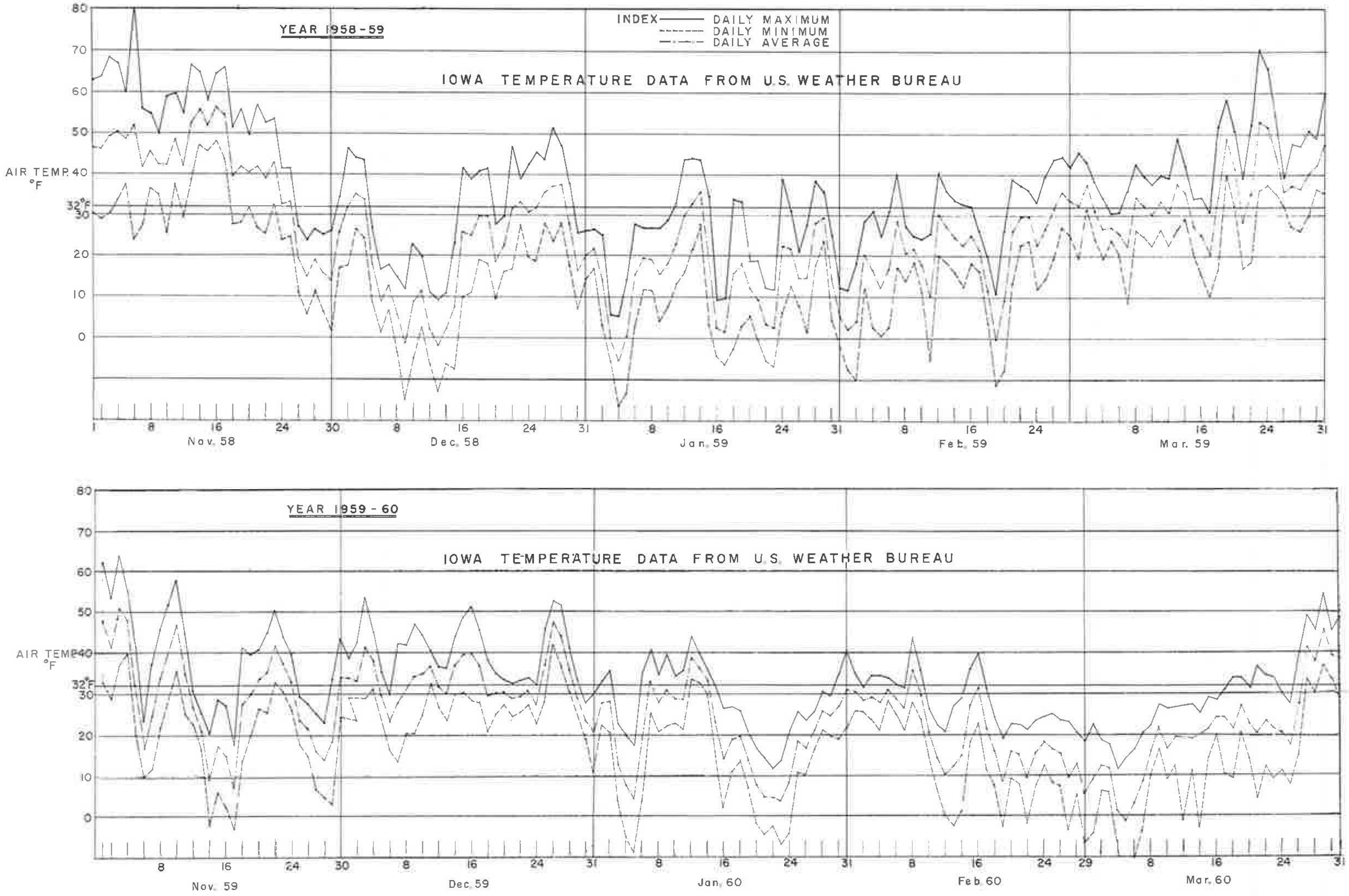
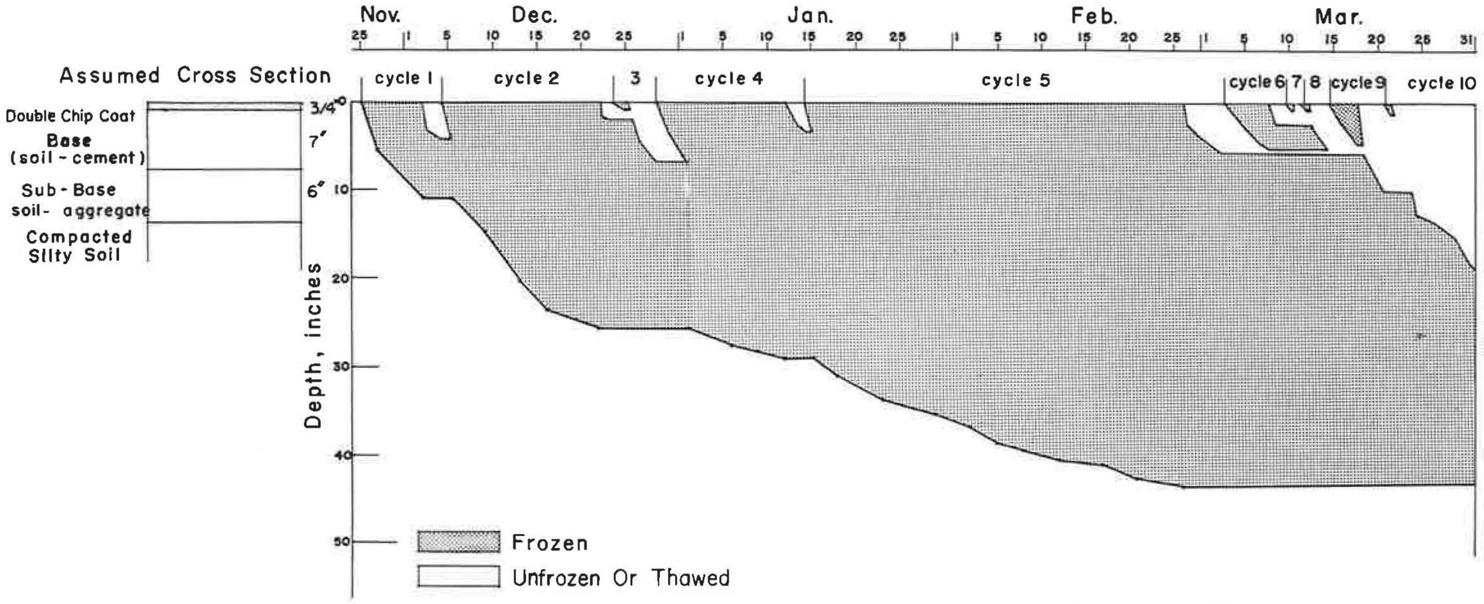
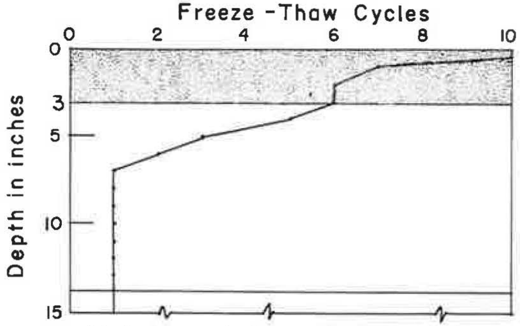


Figure 1. Average U. S. Weather Bureau 1958-1960 temperature data from 130 stations in Iowa.

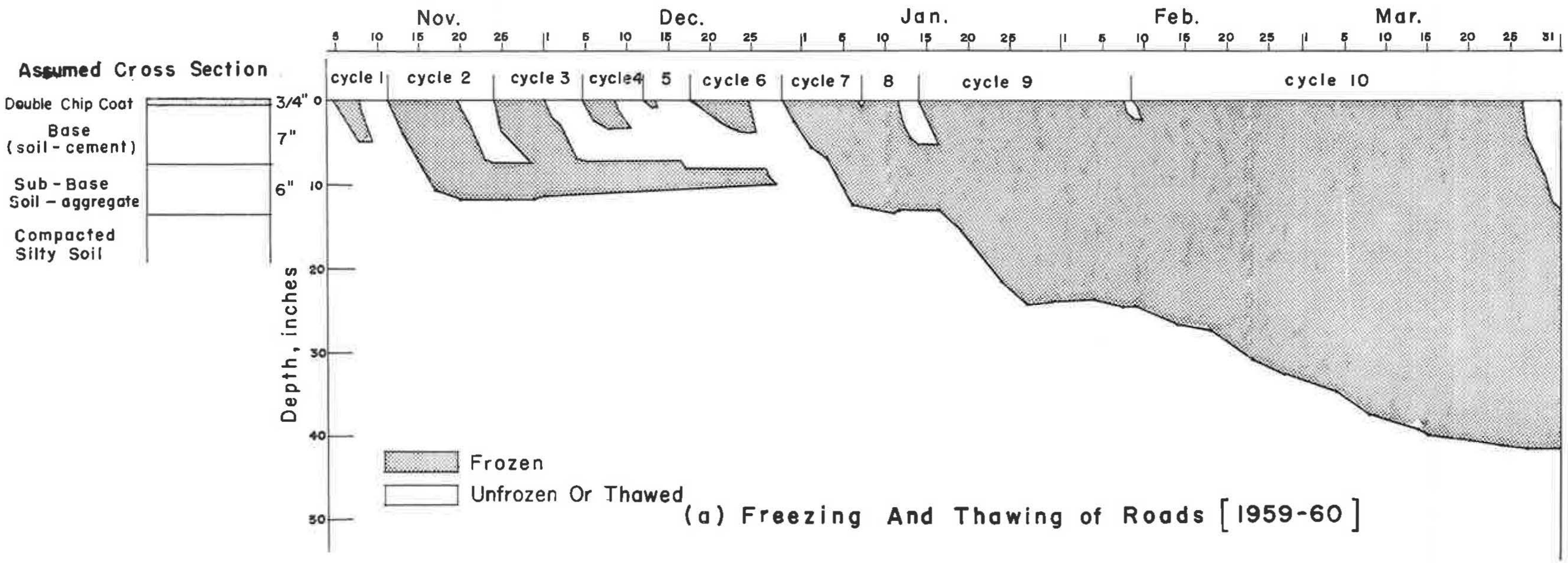


(a) Freezing And Thawing of Roads [1958-59]

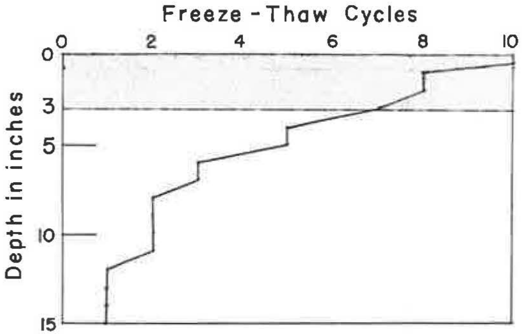


(b) Cumulative Freeze-Thaw Cycles at Different Depths

Figure 2. Freezing and thawing depths calculated from air temperature.

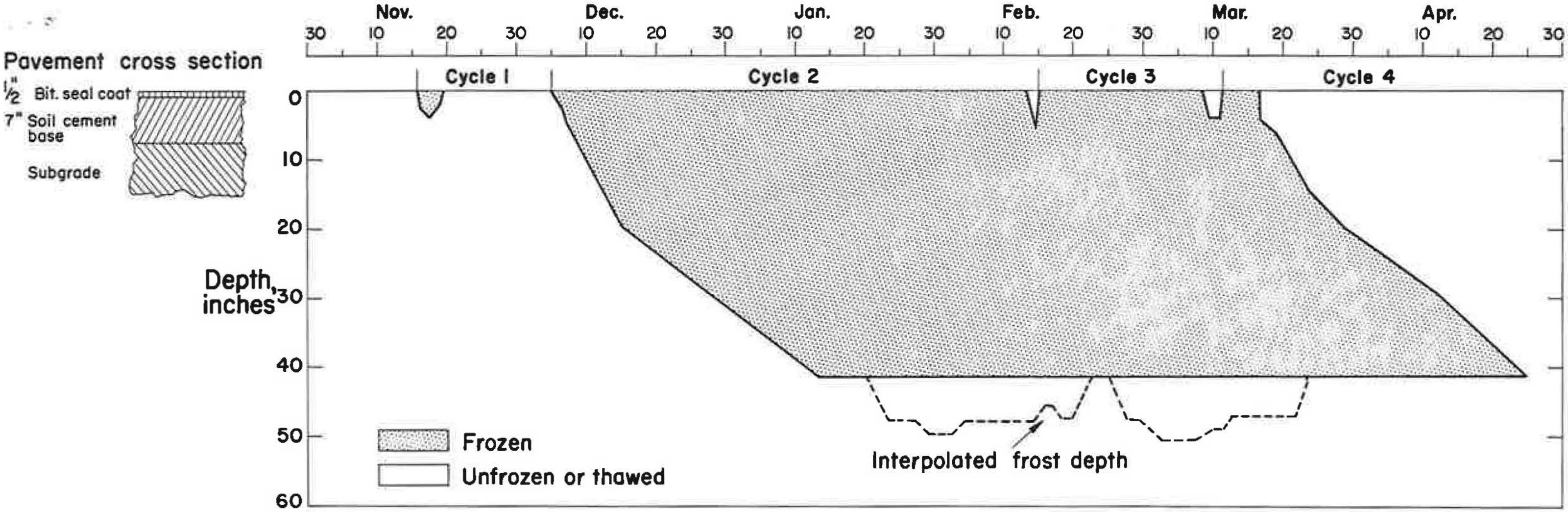


(a) Freezing And Thawing of Roads [1959-60]

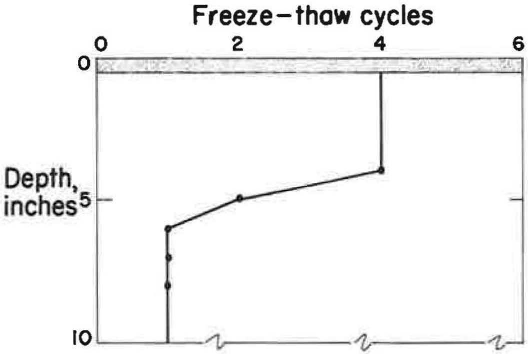


(b) Cumulative Freeze - Thaw Cycles at Different Depths

Figure 3. Freezing and thawing depths calculated from air temperature.



(a) Freezing and thawing of experimental pavement (1961-1962)



(b) Cumlative freeze-thaw cycles at different depths

Figure 4. Freezing and thawing depths observed in experimental soil-cement pavement.

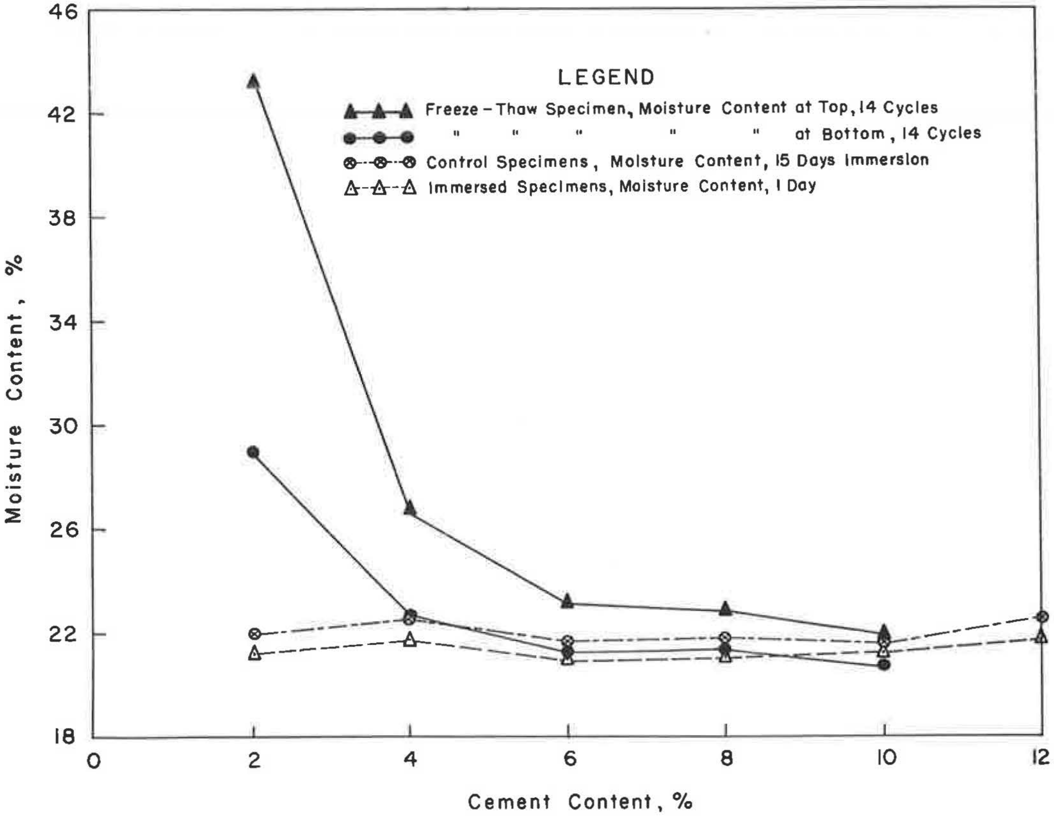


Figure 5. Effect of cement content on moisture content, and distribution of freeze-thaw test specimens.

Experimental Soil-Cement Construction

The experimental project included the placing of a soil-cement base in a 12.83-mi stretch (from Dunlap to Soldier) of Iowa primary highway 37, during August 1961. To insure uniformity in the soil-cement bases the soil was taken from a borrow area near the center of the project. The location of this area permitted central plant operation and control. The cement content required by the AASHO-ASTM standards for the available fine-grained soil was 11.0 percent (based on the dry weight of the soil). But for research purposes the cement content was varied from 7.0 to 13.0 percent in 12 experimental sections. These experimental sections are from 1,000 to 1,500 ft in length, with cement contents assigned to random test sites scattered along the entire 12.83-mi span. Cement contents of 7, 9, 11 and 13 percent were selected for the experimental soil-cement base. Some portions of the subgrade which were badly damaged were rebuilt to obtain more uniform subgrade support before the soil-cement base was laid.

Pertinent soil characteristics are given in Table 1. The borrow area was located in the Monona County thick loess area. A typical particle size distribution of the borrow soil was silt-size particles 77 percent, and 5-μ clay 21 percent with a border line classification between A-4 and A-6. The predominant clay mineral was montmorillonite. A Type 1 cement complying with section 4101 of the 1960 Iowa standard specifications was used throughout the construction. A uniform 7-in. base thickness was selected for the project.

As an aid to curing, RC-0 was applied to the compacted base within 24 hours after construction. MC-4, complying with section 4138 of the 1960 Iowa standard specifications, was used for the single bituminous coat. The ½-in. cover aggregate was crushed limestone.

TABLE 1

SOIL CHARACTERISTICS (SAMPLED DURING CONSTRUCTION) OF EXPERIMENTAL SOIL-CEMENT BASE SECTIONS

Test Section No.	Design Cement Content (%)	Properties of Natural Soil					
		Plastic Limit (%)	Plasticity Index (%)	Passing No. 200 Sieve (%)	Clay[a] $<5\mu$ (%)	Classification	
						AASHO	Textural
3	13	22	13	99	24	A-6(9)	Silty clay loam
4	7	23	11	99	18	A-6(8)	Silt
5	9	23	10	100	20	A-4(8)	Silt
6	13	23	9	99	20	A-4(8)	Silty loam
7	11	23	10	100	18	A-4(8)	Silty loam
8	7	23	12	99	24	A-6(9)	Silty clay loam
9	11	21	13	99	24	A-6(9)	Silty clay loam
10	9	22	13	100	22	A-6(9)	Silty clay loam
11	11	21	12	99	22	A-6(9)	Silty clay loam
12	7	21	10	100	22	A-4(8)	Silty clay loam
13	9	21	12	100	22	A-6(9)	Silty clay loam
14	13	20	15	99	20	A-6(10)	Silty clay loam

[a] Predominant clay mineral—montmorillonite.

Evaluation of Performance

Preliminary studies of the performance of the test sections cover a period of slightly over one year from time of construction. Pertinent field tests were conducted after the frost was out during April 1962, and in the fall of 1962 at one year. The loss of strength in the spring has been previously observed to be of the order of 60 percent (14).

Deflection as Related to Pavement Performance.—Deflections of the surface of the pavement were measured with the Benkelman beam under a moving vehicle whose axle load was 18,000 lb, the maximum allowable single-axle load in Iowa. The spring period was selected for measurement because in frost-susceptible areas flexible-type pavements are most likely to undergo distress at this time. Deflections during the spring correlate more closely with pavement performance than do those during other periods (9). In the data reported are the maximum and rebound deflections in outer and inner wheelpaths. A brief description of the procedure to obtain the deflections is presented in Hoover (10).

Results.—The relation between the maximum outer wheelpath deflection and cement content was investigated by an analysis of variance. No deflection difference was found at the 5 percent significance level between soils treated with 7 percent cement and those treated with 13 percent. Further efforts were directed towards detecting and stripping of the effects of other variables, such as California bearing ratio (CBR) of the subgrade, unconfined compressive strength of the soil-cement base, and location and lane of the Benkelman deflection data.

The CBR of the subgrade appears to influence deflections at the surface, indicated in the AASHO Test Road, where the subbase was somewhat more effective than the base in restricting deflections during both the fall and spring periods (9). The differences in the CBR of the subgrade (at the time of obtaining the deflection data) could explain part of the variation in the deflection data. In-place CBR's were measured at each location where Benkelman deflections were obtained, six for each test section. The mean value of CBR for each test section ranged from 35 to 59 percent for the weakest to the strongest subgrade.

The compressive strength of the soil-cement probably influences the flexural deflection, because the flexural strength of soil-cement is about one-fifth of the compressive strength (2). Preliminary regression analysis of the individual test sections to some extent supported this hypothesis, since two-thirds of the regression coefficients were negative, indicating that low deflection tended to be associated with high compressive strength.

TABLE 2

Factor Influencing Deflection	Regression Coefficient	t-Values, Calculated	t-Values, Tabulated[a]	Remarks
Cement, %	-2.007	3.74	1.99	Significant
Subgrade CBR	-0.011	1.91	1.99	Barely significant
Location	+0.035	4.41	1.99	Highly significant
Unconfined compressive strength	-0.001	0.12	1.99	Insignificant

[a] At 5 percent level.

The consistent increase in deflection readings from the north end of the road towards the south warranted an investigation. The finding seemed reasonable in that the clay content of the subgrade increased from north to south. Preliminary investigations showed that the increase was of significance in influencing deflection.

The effect of lane on deflection, considered a variable, later proved not very significant. Whether the sections occurred in cut or fill was not considered because the sections were randomly distributed along the road.

A multiple regression analysis was conducted to gain an insight into the factors which influence deflection under wheel loads. The deflection is determined by cement content, subgrade CBR value at 0.1 in. deflection, unconfined compressive strength of the soil-cement base, and location of stations along the test road. The outer and inner wheelpath deflections were regressed against the four variables. The same regression was repeated excluding the unconfined compressive strength, the purpose of which was to investigate the interaction between the former and the cement content to influence deflection.

The prediction equation is

$$D_{owp} = 65.7 - 2.007\,(C_m) - 0.011\,(C_{br}) + 0.035\,(L_O) \tag{1}$$

in which

D_{owp} = outer wheelpath Benkelman beam deflection, in 0.001 in.;
C_m = cement content, percentage of dry weight of soil;
C_{br} = California bearing ratio of subgrade, lb at 0.1 in. deflection; and
L_O = location of station, in 100 ft from north to south.

The term corresponding to the unconfined compressive strength is purposely omitted because it plays little part in predicting the deflection. The regression coefficients and their test of significance are summarized in Table 2.

The regression computation for the inner wheelpath deflections led to the same general deduction in that the unconfined compressive strength was not a pertinent factor in predicting deflection.

The analysis led to some useful conclusions concerning the significance of the various parameters influencing flexural deflection:

1. The higher the cement content the lower the deflection, the rate of decrease being 2 units (0.001 in.) per percent cement content. The interval 2 ± 1 units includes the true value unless a 1-in-20 chance has occurred. (These limits are hereafter designated as 95 percent confidence limits.)
2. The CBR of the subgrade is barely significant, the higher the CBR the lower the deflection.
3. Location along the road was significant. The higher deflections were towards the south end of road, the rate of increase being 0.035 units (0.001 in.) per hundred ft of road (95 percent confidence limits 0.035 ± 0.015 units).

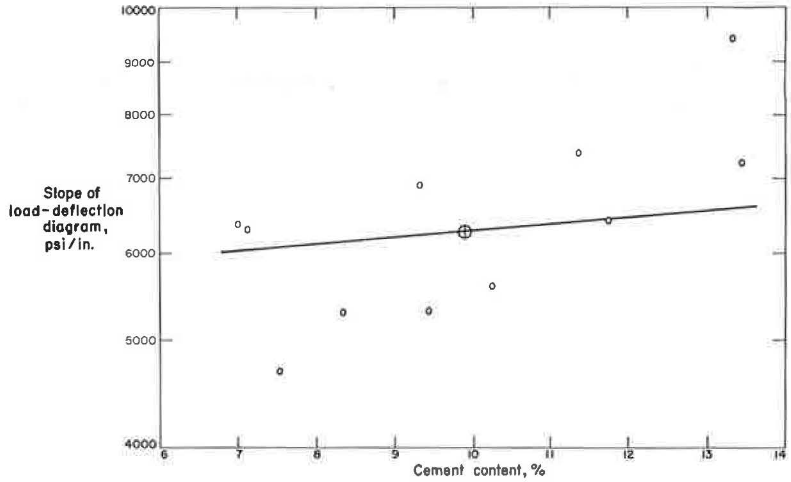


Figure 6. Effect of cement on bearing strength of soil-cement base (each point is average of six tests).

4. The unconfined compressive strength is not significant.

5. A comparison of the outer wheelpath and inner wheelpath prediction equations shows that inner wheelpath deflections are not highly influenced by the given factors, as evidenced by low t-values and small mean residual. This conclusion is in keeping with the previous finding that pavement deflection in the inner wheelpath is more uniform than in the outer wheelpath and changes only slightly with the season (6).

In the AASHO Road Test (9), the expected life of different test sections as indicated by the decline in the serviceability index to a specified value was related to spring deflections. Serviceability index, which may apply either to the present or the future, is a mathematical combination of values obtained from certain physical measurements of a large number of pavements so formulated as to predict either the present or the future ability of a pavement to serve the traffic (9). For example, a spring deflection of 0.025 in. is safe for a pavement that is expected to carry 1,000,000 18-kip axle loads without dropping the serviceability index below 2.5. Calculations on similar lines showed that the test sections built of 7 percent cement-treated base could safely carry 500,000 18-kip axle loads without dropping the serviceability index below 1.5. These predictions, however, do not take into account the gain in strength with age of soil-cement.

Bearing Capacity by Plate Bearing Tests.—Plate bearing tests were performed during April. It has been hypothesized by the Research Department of the Iowa Highway Commission that the depth of influence in the plate test is approximately 1¾ times the diameter of the plate. Thus a 4-in. diameter plate may be used to evaluate the characteristics of soil-cement base 7 in. thick with minimum influence from lower layers. The slope of the load-deflection diagram expressed in pounds per square inch per inch is reported for each location.

It was suspected that the unconfined compressive strength of the base and/or the CBR of the subgrade might influence the bearing strength as determined by plate bearing tests. Preliminary analysis showed that these factors did not have any significant influence on the slope of the load deflection diagram. However, the bearing strength shows a slight increase with increase in cement content (Fig. 6). The presence of closely spaced cracks in soil-cement of 7 percent admixture might be one of the reasons contributing to its larger deflection.

Surface Crack Studies.—Cracking is an element of structural deterioration that detracts from serviceability and performance of flexible pavement. Cracks do not in themselves have much effect on the ability of the pavement to serve traffic, provided

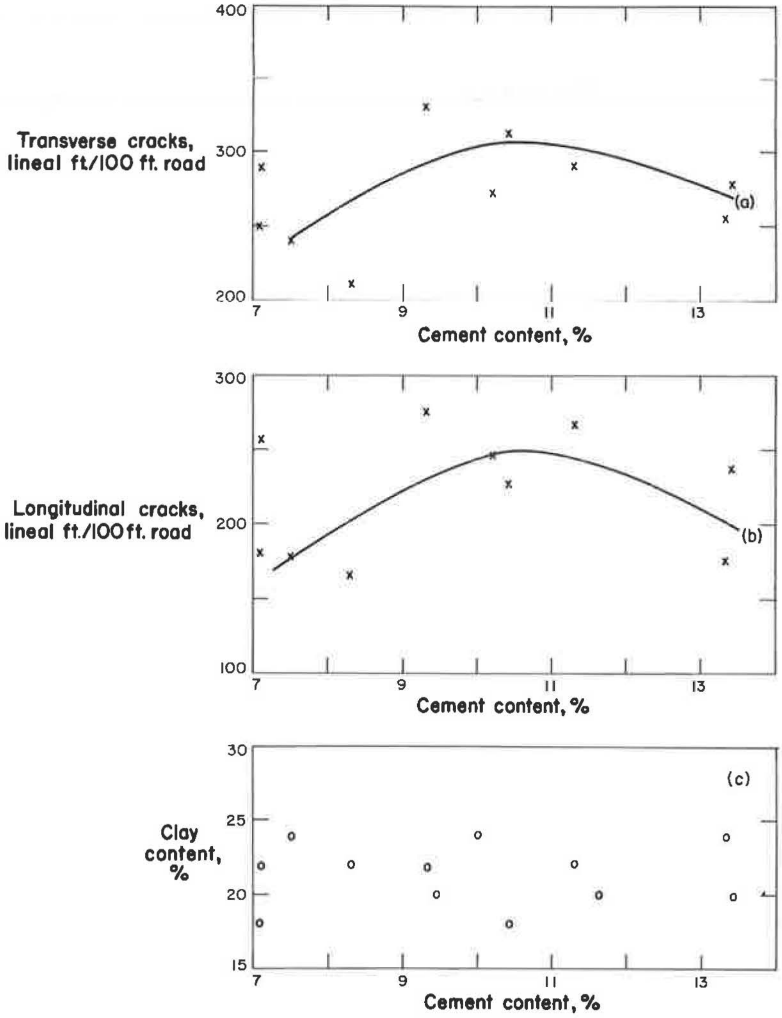


Figure 7. Relation between extent of (a) transverse cracks and cement content, (b) longitudinal cracks and cement content, and (c) clay content of twelve different test sections and corresponding cement contents.

they are intact. However, weakening of the pavement structure is likely because of infiltration of surface water.

More cracks appeared during periods of cold weather, at which time a crack survey was conducted. The results obtained, expressed in lineal feet per 100 feet of pavement, are plotted against cement content (Figure 7 a and b). The extent of cracking seems to be optimum for a soil-cement whose cement content is around 10.5 percent.

Before an explanation for this maximum cracking is offered, the various factors causing shrinkage and cracking should be analyzed. Shrinkage cracks are inherent in soil-cement. Recent research in shrinkage of soil-cement by H. Nakayama and R. L. Handy indicates that this phenomenon is primarily influenced by the amount of $5\text{-}\mu$ clay in the soil. Cracks in soil-cement also could be flexural failures. The reasoning is essentially as follows: the flexural strength corresponding to an unconfined compressive strength of 300 psi is of the order of 60 psi (2). Using the Westergaard method of rigid pavement design, flexural stresses of the order of about 100 psi should be devel-

Figure 8. Crack pattern of 4-in. diameter road cores with cement contents of 13 and 7 percent after one year.

oped under a wheelload of 18,000 lb, which is far above the cracking stresses. Had shrinkage cracks not occurred, flexural cracks would have, causing the soil-cement to act as a flexible material.

The clay content of the soil-aggregate that went into the soil-cement remained almost the same for all sections (Fig. 7c).

As the cement content is increased, the flexural strength is also increased, the beam action is more pronounced as a result, and flexural cracks occur at longer spacings. The adverse effect of high cement content on shrinkage is overrun by the higher flexural strength.

At lower cement contents the cracks were closely spaced. Being very fine, they are likely to be overlooked and often they were not visible on the pavement surface. Numerous concealed cracks revealed during coring operations supported this hypothesis.

Several cores drilled after one year were examined for defects. These were mainly vertical cracks whose width and depth were noted and the crack plane of each was checked for direction and interlocking characteristics. The results indicated that cracking is inevitable and to some extent is required of soil-cement construction. Closely spaced shrinkage cracks were observed in all test sections. Of the 66 cores examined, 39 exhibited essentially vertical cracks (Fig. 8). These data support the finding of Mitchell (13).

Unconfined Compressive Strength of Road Cores.—Summarizing the analysis of the pavement performance, it may be concluded that test sections built of 7 percent cement-treated soil were as structurally sound as the sections built of high quality soil-cement. However, it may be pointed out that future structural deterioration of the test pavement is unlikely because soil-cement gains strength with age, and the first spring thawing period is the most critical time. This can be verified by comparing the core strengths at the age of eight days with those at 245 days during spring thaw, and at the age of one year, in August 1962. The core strengths are graphed against the log of the age of soil-cement (Fig. 9), because strength of soil-cement correlates best with time of curing in a semilogarithmic manner (3). The relatively flat slope of strength-age relationship during the winter is an indication of the retarding effects of freezing temperatures.

CRITERIA FOR DESIGN OF SOIL-CEMENT

Most highway agencies base the mix design of soil-cement on the standard durability tests, which have been successfully used for many years. A soil-cement base must remain stable to resist effects of weathering and traffic. The durability tests primarily measure deterioration or weight loss of specimens by physical weathering, and it is implied that the soil-cement which meets the durability requirements satisfies strength requirements as well. However, it would be desirable if both requirements could be met in one test procedure. This requirement may be simplified in that failure of a soil-cement base by deformation is most likely a shear failure; therefore, a safe minimum allowable unconfined compressive strength after adequately severe simulated weathering treatments may be considered as a valid criterion of strength and durability (4). Simplicity and reliability of test procedures are other reasons for favoring unconfined compressive strength criteria.

The performance of the 7 percent soil-cement mixture in the field test sections is satisfactory. However, the question arises as to the successful performance of a soil-cement mixture of lower cement content. Though a 5 percent soil-cement mixture was

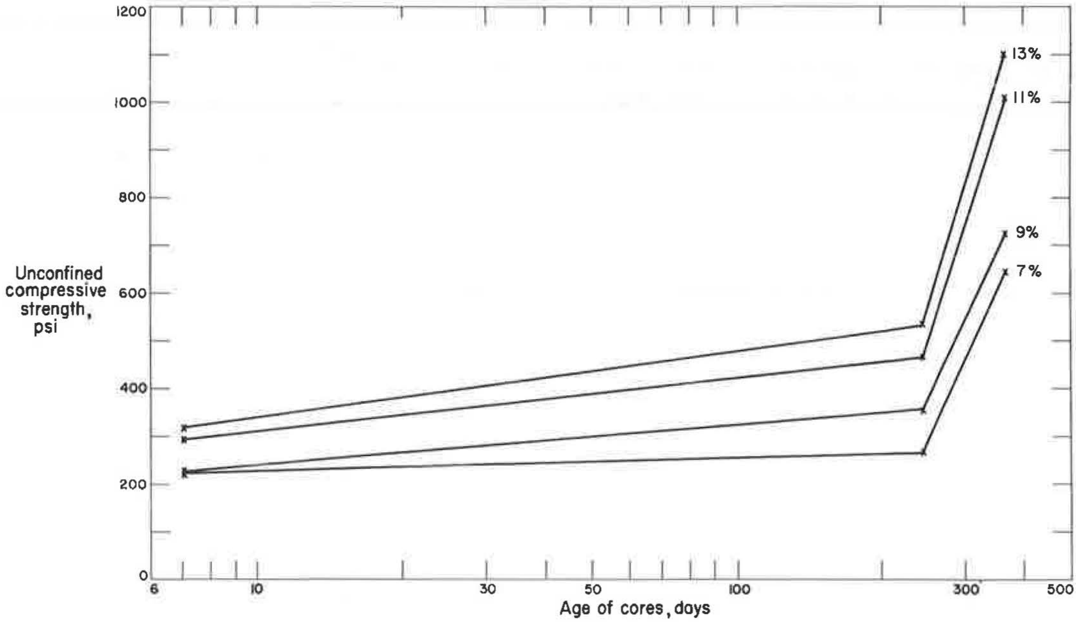


Figure 9. Effect of age on unconfined compressive strength of Proctor-size road cores after immersion in distilled water for 24 hours before test.

not a part of the experiments in the field the indications are that it may not perform satisfactorily under field freezing and thawing conditions. Figure 10 shows two test specimens which were subjected to Iowa freeze-thaw tests. That the 5 percent soil-cement mixture did not survive the freeze-thaw test in the laboratory indicated its inadequate strength gaining capability. Therefore, the adequate and economical cement content to use would be the lowest percent admixture that survived in the field and laboratory. The properties of a soil-cement mixture with 7 percent of cement will be the tentative criteria for mix design with fine-grained soils. The results given are for soil samples obtained from the borrow area during the time of construction of the experimental base. Details regarding molding, curing and testing of the 2-in. diameter by 2-in. high specimens have been given (4).

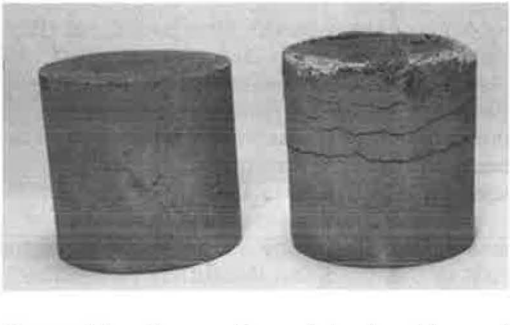


Figure 10. Freeze-thaw deterioration of 2-in. diameter by 2-in. high test specimens with cement contents of 7 and 5 percent after 10 cycles of freezing and thawing.

Results.—Based on the analysis and acceptable performance of the 7 percent test section, the following criteria may be used as the basis for selecting minimum cement contents for durable soil-cement. Mixes are prepared in the laboratory and molded into 2-in. diameter by 2-in. high test specimens.

1. The minimum unconfined compressive strength shall be 453 psi when tested after 7 days' curing in a humid room followed by 24 hours' immersion in distilled water, 95 percent confidence limits from 431 to 475 psi.
2. The minimum unconfined compressive strength after being subjected to the Iowa freeze-thaw test (Appendix A) is 500 psi, 95 percent confidence limits from 472 to 528 psi.

Seven-day-cured and one-day-immersed strengths may be reliably used for preliminary studies since they correlate with the corresponding freeze-thaw results; the correlation coefficient is 0.869, based on analysis of 40 pairs of strength results with a wide range of cement contents. Strength after freeze-thaw may be predicted by multiplying the 7-day strengths by 1.221 ± 0.342 (95 percent confidence limits on the prediction coefficient). A statistical t-test was conducted on the regression coefficient to test the approach to unity; the ratio may be considered 1.0, subjected to a chance variation of 1-in-20.

Another criterion is a measure of the relative strength gaining capability of soil-cement during adverse conditions of freezing and thawing. This measure is obtained by dividing the strength at the end of the test by the continually immersed specimen at room temperature. The indices of resistance of soil-cement mixtures are plotted against the corresponding cement contents (Fig. 11). Also shown is the ratio of the unconfined compressive strengths of specimens buried adjacent to the test road and similar specimens stored in the humid room for a 1-yr period. These specimens were molded from the soil-cement mixture mixed in the pugmill during construction of the test sections.

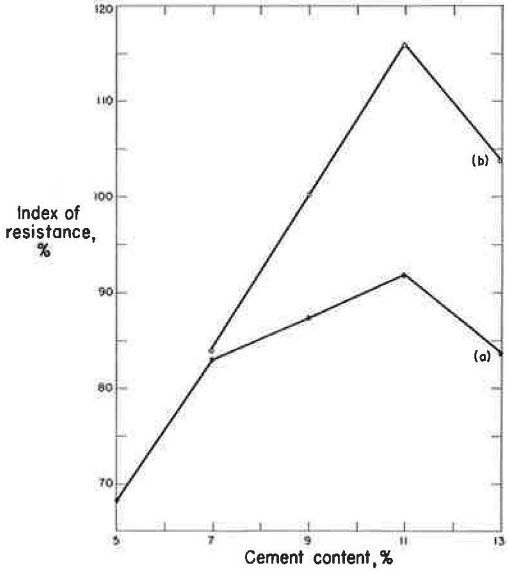
Comparison of a and b (Fig. 11) shows either that the laboratory freeze-thaw test is more severe than what actually occurred in the field, or that transient field temperature and moisture conditions provide better curing than the carefully controlled conditions of the laboratory moist-cure room. The latter explanation appears more acceptable, since field-cured strengths of hand-molded specimens with 11 and 13 percent cement actually exceeded those obtained in the laboratory.

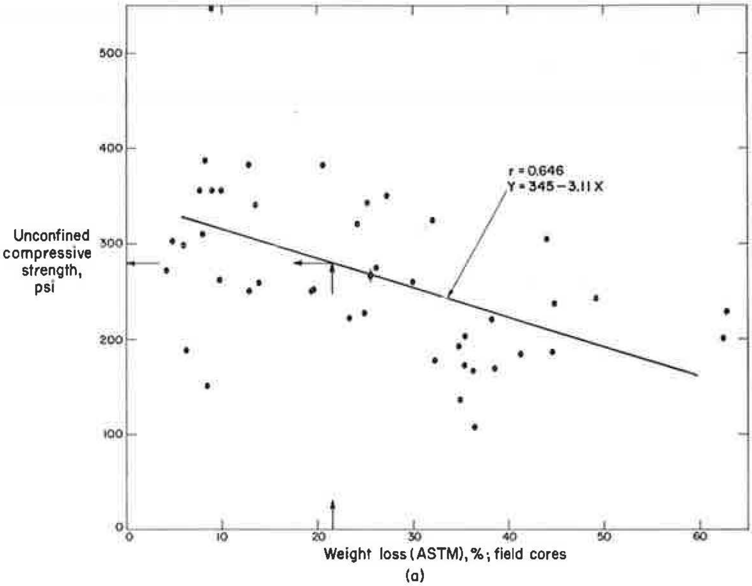
The index of resistance drops below 80 percent for mixtures of less than 7 percent cement. Therefore, the third criterion tentatively reads as follows:

3. Laboratory test specimens shall give a minimum index of resistance to freezing of 80 percent after being subjected to Iowa freeze-thaw test.

Strength Criterion by Correlation.—A correlation for the 8-day unconfined compressive strength with the ASTM weight loss has been established (correlation coefficient 0.646), the test specimens being road cores from the experimental soil-cement base (Fig. 12a). Because these results cover a wide range of cements feasible for soil-cement, and because the properties obey a linear relationship in the range of cement

Figure 11. Relationship between (a) index of resistance to freezing and cement content and (b) index of resistance to freezing of field weathered test specimens and cement content (2-in. diameter by 2-in. high specimens).





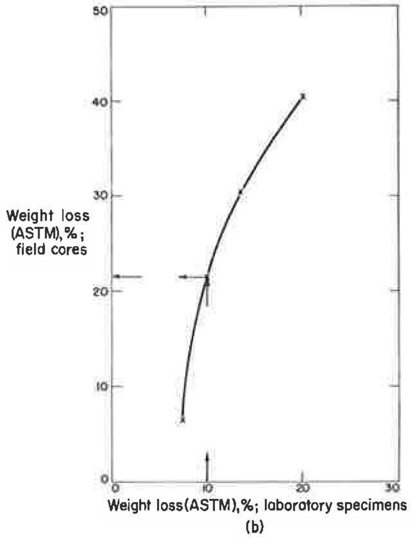


Figure 12. (a) Relation between unconfined compressive strengths of specimens after aging 7 days and immersion of one day and brushing weight loss; and (b) relation between brushing weight loss (ASTM) on field cores and brushing weight loss (ASTM) on laboratory molded Proctor-size specimens.

contents, the prediction of unconfined compressive strength from weight loss is permissible. The expected unconfined compressive strength is

$$Y = 345.3 - 3.108 X \tag{2}$$

in which

Y = unconfined compressive strength (psi) of road cores at 8 days following 24-hr immersion, and
X = ASTM brushing weight loss.

The permissible weight loss of this soil is 10 percent (ASTM Designation D560-57) to produce durable soil-cement. Corresponding weight loss for road cores of similar soil-cement as that of laboratory specimens would be predicted from a plot of weight loss on laboratory specimens against the laboratory results on field cores. The predicted weight loss is 21.5 percent (Fig. 12b). Knowing the weight loss, the expression can be used to predict the expected compressive strength.

When X = 21.5, the population mean of the unconfined compressive strength is estimated at 278 psi with 95 percent confidence limits from 251 to 305 psi. In other words if a field strength of 250 to 300 psi is guaranteed, the soil-cement would necessarily meet the requirements of durability. In the United Kingdom, a minimum compressive strength of 250 psi at 7 days has been used successfully to design soil-cement for bases of lightly trafficked roads, but for heavily trafficked roads, a minimum strength of the order of 400 psi at 7 days is probably required (4). An immersed strength of about 275 psi after 7 days' curing has been suggested by the soil-cement bureau of the Portland Cement Association as tentative strength value for friable loess.

It has been found that the construction methods used in the soil-cement base construction result in a field strength of about 55 to 65 percent of the laboratory results. Experience in Great Britain showed a value of 60 percent was probably satisfactory (2, 13).

The cement content is therefore that necessary to give a laboratory compressive strength equal to 278/0.6 = 463 psi. Thus, the 7-day immersed compressive strength would be 463 ± 45 psi. The statement is correct except for the diverse kind of sample that occurs about once in 20 trials.

CONCLUSIONS

An Iowa freeze-thaw test, modeled after the modified British freeze-thaw test but designed to simulate more closely winter field conditions in Iowa, has been developed (Appendix A). Essentially the Iowa freeze-thaw test method covers the determination of the change in the unconfined compressive strength of specimens of stabilized fine-grained soil when subjected to cycles of freezing and thawing under conditions summarized as follows:

1. A freezer temperature (20 F) equivalent to the daily average minimum air temperature in Iowa during winter months.
2. A meaningful temperature (35 F) inside the vacuum flask at the bottom of the specimen simulating the soil temperature during winter months.
3. The number of freeze-thaw cycles (10) is obtained from a plot of the average air temperature, in that freezing of pavements is considered to occur when the air temperature falls below 32 F, and thawing when the temperature increases above 32 F.
4. For simplicity of procedure, the specimens are to be thawed at room temperature, 77 F.
5. The important criteria for the evaluation of the Iowa freeze-thaw test are the unconfined compressive strength and index of resistance to freezing. Heave upon freezing and the moisture content and distribution after freeze-thaw tests offer good promise as criteria.

To establish design criteria, several sections of Iowa primary highway 37 were built with soil-cement mixtures with varying cement contents. The central plant construction insured uniform soil characteristics. Cement contents ranged from 7 to 13 percent whereas the ASTM Method D560-57, warranted the use of 11 percent cement.

The experimental test sections were evaluated on the basis of the Benkelman beam deflection, plate bearing tests, crack survey, and the unconfined compressive strengths of road cores. Based on the acceptable performance of the 7 percent test section, the laboratory test results of 2-in. diameter by 2-in. high test specimens of the soil-cement are proposed as tentative criteria for the design of soil-cement:

1. The minimum unconfined compressive strength shall be 433 ± 23 psi when tested after 7 days' curing followed by 24 hours' immersion in distilled water.
2. The minimum unconfined compressive strength after being subjected to the Iowa freeze-thaw test shall be 459 ± 41 psi.
3. Laboratory test specimens shall give a minimum index of resistance to freezing of 80 percent after being subjected to the test.

In view of the correlation (r = 0.646) between the unconfined compressive strength and the ASTM brushing weight loss on road cores, the validity of the 7-day strength criterion is verified.

ACKNOWLEDGMENTS

This report was part of the research under Project 451-S of the Engineering Experiment Station, Iowa State University, under the sponsorship of the Iowa Highway Research Board, Iowa State Highway Commission.

The authors extend appreciation to R. L. Handy, H. T. David, J. M. Hoover, and T. Demirel.

REFERENCES

1. ASTM Standards. Part 4 (1958).
2. Blake, L. S., and Lilly, A. A., "Laboratory Test Procedures for Soil-Cement Pavement Construction." Tech. Report TRB-177, Cement and Concrete Association, Great Britain (Feb. 1955).
3. Circeo, L. J., Davidson, D. T., and David, H. T., "Strength Maturity Relations of Soil-Cement Mixtures." HRB Bull. 353, 84-97 (1962).
4. Davidson, D. T., and Bruns, B. W., "Comparison of Type I and Type III Portland Cement for Soil Stabilization." HRB Bull. 353, 28-45 (1960).
5. Davidson, D. T., Pitre, G. L., Mateos, M., and George, K. P., "Moisture-Density, Moisture-Strength and Compaction Characteristics of Cement-Treated Soil-Mixtures." HRB Bull. 353, 42-63 (1962).
6. Miller, C. F., Jr., and Bisset, J. R., "Flexible Pavement Performance Studies in Arkansas." HRB Bull. 321, 1-15 (1962).
7. George, K. P., "Development of a Freeze-Thaw Test for Evaluating Stabilized Soil." M. S. thesis, Iowa State Univ. (1961).
8. George, W. S., "Statistical Methods." Iowa State Press (1956).
9. "The AASHO Road Test: Report 5—Pavement Research." HRB Special Report 61 E, 352 pp. (1962)
10. Hoover, J. M., Huffman, R. T., and Davidson, D. T., "Soil Stabilization Field Trials, Primary Highway 117, Jasper County, Iowa." HRB Bull. 357, 41-68 (1962).
11. "Soil-Cement Stabilization." (Mimeo.) Iowa Highway Research Board, Iowa State Highway Commission (June 1962).
12. Lambe, T. W., "Modification of Frost-Heaving of Soils with Additives." HRB Bull. 135, 1-23 (1956).
13. Mitchell, J. K., and Freitag, D. R., "A Review and Evaluation of Soil-Cement Pavements." Soil Mech. and Found. Eng. Division, Proc., ASCE, 85:49-73 (Dec. 1959).
14. Motl, C. L., "Report of Committee on Load Carrying Capacity of Roads as Affected by Frost Action." HRB Bull. 96, pp. 1-23 (1954).
15. "Climatological Data." U. S. Department of Commerce, Weather Bureau, Vols. 61-71 (1950-1960).

Appendix A

IOWA FREEZE-THAW TEST

Scope

1. This method covers the determination of the change in the unconfined compressive strength of 2-in. high by 2-in. diameter specimens of stabilized fine-grained soil when subjected to cycles of freezing and thawing under specified conditions which simulate winter climate in Iowa. The Iowa freeze-thaw test is a modification of the British (B. S. 1924: 1957) freeze-thaw test.

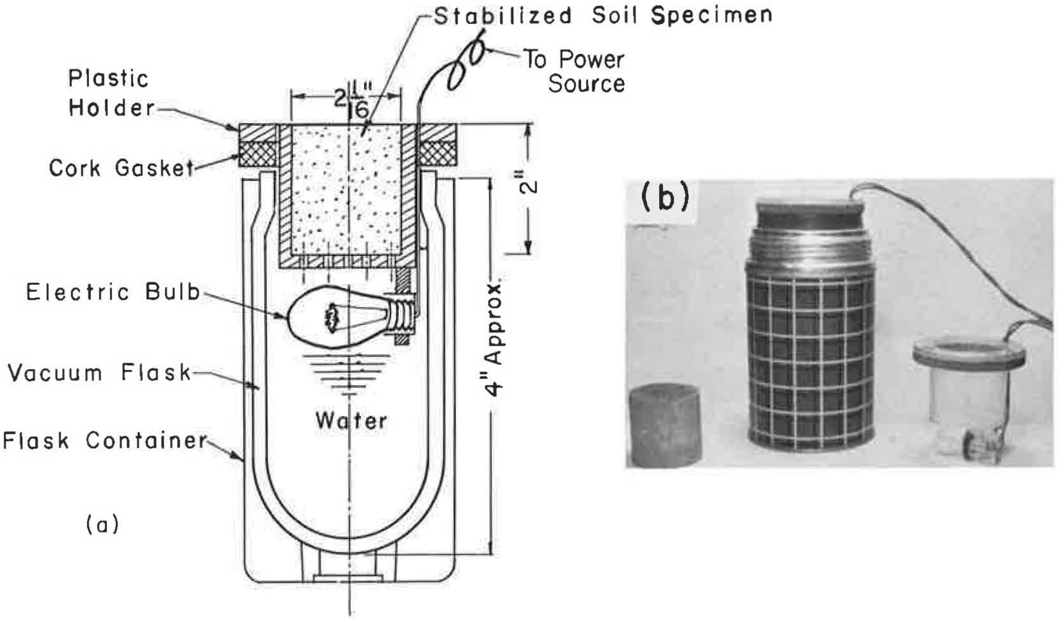


Figure 13. (a) Freeze-thaw test apparatus; (b) vacuum flask, stabilized soil specimen and specimen holder.

Apparatus

2. The apparatus required (Fig. 13) is as follows:

   a. A commercial vacuum flask having a neck with an internal diameter of approximately 2.5 in. and an internal depth of at least 4 in.
   b. A specimen holder of low thermal conductivity and resistant to deformation under the test conditions, and capable of supporting a stabilized specimen 2 in. high and 2 in. in diameter within the vacuum flask, so that the upper flat surface of the specimen is flush with the top of the flask (Fig. 13). The base of the carrier is perforated to permit free access of water to the underside of the specimen. A heating element with an output of about 0.5 watt is attached to the bottom of the specimen holder (a 6-watt bulb connected through a variable transformer using a 110 ac supply is found to be very convenient).
   c. A refrigerated space maintained at a temperature of 20 ± 2 F which is large enough to contain the vacuum flask with its enclosed specimen. A thermometer is installed inside the refrigerated space.
   d. A supply of asphalt or resin-base paint.
   e. A supply of self-adhering membrane (the commercial product Saran Wrap is satisfactory).
   f. About 100 ml of distilled water, cooled to 35 ± 2 F.
   g. A height gage equipped with an Ames dial having an accuracy of 0.001 in.

Preparation of Specimens for Test

3. For each determination two identical specimens 2 ± 0.05 in. high and 2 in. in diameter are prepared as has been described (4). (If greater accuracy is desired, 6 or 8 identical specimens may be prepared for each determination).

Test Procedure

4. a. After the desired curing period of 7 days' moist curing, any covering material on the specimens is removed and both specimens are weighed. If moist

curing either specimen has lost more than two grams in weight during storage at a temperature of 70 ± 1 F and a relative humidity of at least 95 percent, both specimens are discarded.

b. After weighing, a coating of asphalt or resin-base paint about 1 mm thick, is applied to the top surfaces of both specimens and allowed to dry. The specimens shall then be immersed in distilled water at 77 ± 4 F.

c. After immersion for 24 hours, the specimens are removed from the water and dried with blotting paper. The height and weight of the specimens are measured. One specimen is subjected to freezing and thawing and the other to immersion. A collar, 1.5 in. deep, of a self-adhering membrane is placed around the top of the freeze-thaw specimen.

d. Sufficient water at a temperature of 35 deg shall be poured into the vacuum flask so that when the specimen dealt with in c above is inserted in the holder and the latter placed in the flask, the bottom $\frac{1}{4}$ in. of the specimen is immersed in water. The height of the specimen while inside the holder shall be measured using the Ames dial. The vacuum flask and its contents is placed in the refrigerated space maintained at 20 ± 2 F. As the water inside the vacuum flask cools down (after about one hour) it is heated up and brought to the required temperature of 35 ± 2 F. The vacuum flask is kept in the refrigerated space for 16 hours.

e. The flask and contents are removed and the height of the specimen while inside the holder measured. The specimen is thawed for 8 hours at a temperature of 77 ± 4 F. If, after thawing, the level of the water inside the vacuum flask has dropped so that it is no longer in contact with the base of the specimen, water at 35 F is added to restore the level.

f. The procedure described in d and e above constitutes one cycle of freezing and thawing. Testing shall continue until the specimen has been subjected to 10 such cycles.

g. At the conclusion of the freezing and thawing cycles, the thawed specimen is removed from the holder and, together with the second specimen control which has been stored in water during the entire period, allowed to drain for 15 minutes. The heights and weights of both specimens are determined.

h. The unconfined compressive strengths of the two specimens are then determined. Each specimen is placed centrally on the lower plate of the compression testing machine, and the load is applied so that the rate of deformation is uniform and approximately 0.10 in./min. The maximum load in pounds exerted by the testing machine is noted and recorded (p_f for the freeze-thaw specimen and p_c for the control specimen).

i. The moisture contents of representative samples of fragments taken from the specimens are determined.

Calculations

5\. a. The unconfined compressive strengths (p_f and p_c) of the two specimens are calculated from the formula:

$$p = 0.318\ P \text{ (psi)}$$

where P = the maximum load recorded in pounds.

b. The index of resistance to the effect of freezing (R_f) is calculated from the formula:

$$R_f = \frac{100\, p_f}{p_c} \ (\%).$$

Reporting of Results

6\. a. The important criteria used to evaluate the stabilized soils are p_c, p_f, and R_f, which are reported.

b. The report may include the "heave" expressed as a percentage of the initial height and the final moisture content and its distribution.

c. Also the details of composition of the stabilized soil mixture and the dry density at the time of molding are reported.

Developments in Durability Testing of Soil-Cement Mixtures

R. G. PACKARD and G. A. CHAPMAN, respectively, Chief and Laboratory Engineer, Soil-Cement Laboratory, Paving Bureau, Portland Cement Association

The conditions of soil-cement specimens during freeze-thaw and wet-dry tests were measured by several methods (weight loss, moisture gain, length change, compressive strength, pulse velocity and visual rating).

Results are analyzed to show: (1) a comparison of the efficiencies of the measuring techniques; (2) information, essential to the interpretation of data, on the effects of freezing-thawing and wetting-drying on soil-cement specimens; (3) an accumulation of sufficient data to indicate that the length change technique represents a superior, alternate method to the weight loss technique used in the standard freeze-thaw test (suggested length change criteria are well correlated with established weight loss criteria); and (4) a promising, but preliminary, correlation of an accelerated (5-day) freeze-thaw test to the standard freeze-thaw procedure.

Application of these results is indicated for improving the standard freeze-thaw and wet-dry procedures and in the analysis or development of other soil-cement testing procedures.

•ONE of the most important properties of soil-cement is that the stability of the material is retained over years of exposure to the destructive forces of weather. In this property lies the definition of the minimum quality of the material, the reason for its acceptance and widespread use and, pertinent to this discussion, the objectives of the mix design of the material.

The desire to obtain this property (whether it is termed retention of stability, long-term performance or, simply, durability) permeates the history and development (1) of soil-cement mix design procedures, specifically the standard (2) freeze-thaw and wet-dry tests and related criteria (3).

Recognizing that the investigation of durability is a major objective in soil-cement testing the Soil-Cement Laboratory of the Portland Cement Association has continued to study means of making the reliable standard procedures faster, more exact, and easier to use.

In this progress report, data from previous studies (4) on 4 soils are verified and amplified by similar investigations of 104 soils of various types, and the results of new studies are presented.

METHODS FOR MEASURING DETERIORATION IN FREEZE-THAW AND WET-DRY TESTS

In the development of environmental change testing procedures, a measure of the quality of the soil-cement specimens is required. The following is a discussion of some of the methods that have been used for soil-cement procedures. It is felt that the comments would apply not only for the standard freeze-thaw and

---

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

wet-dry tests but for other types of environmental testing involving changes in moisture and temperature.

Three methods for measuring the resistance of cement-soil mixtures to wet-dry and freeze-thaw are specified in the standard procedures: weight loss, volume change and moisture gain. Others that have been studied are: length change, various measures of strength, pulse velocity and visual inspection. Each of these measures some property that reflects the quality of the cement-soil mixture. For some, the measurement may be more directly and sensitively related to deterioration. Therefore, it would be expected that, although all the methods may correlate for major differences in durability, minor differences might not be rated in the same order by all of the measures.

Before discussing the merits of each method, it is well to consider the objectives of the measurements. Retention of stability under the exposure conditions is the primary criterion. Therefore, the changes in values are of more concern than the absolute values. This is also true in concrete durability testing where criteria are based on changes in strength, changes in elastic properties or changes in shrinkage and expansion characteristics.

For soil-cement, Table 1 shows how each measure is related to various aspects of the condition of soil-cement specimens. Deterioration or loss in hardness is the condition to be measured. The other conditions, to the degree that they can be independent of the first condition, are extraneous and only mask the detection of deterioration.

TABLE 1

DEPENDENCE OF MEASURES ON THE CONDITIONS OF SPECIMENS

	CONDITIONS(1) OF SPECIMENS			
MEASURE	DETERIORATION (LOSS IN HARDNESS)	ABSOLUTE HARDNESS	SURFACE CONDITION (If different from overall condition)	SOIL TYPE
Final Weight Loss	x	x	x	x
Rate of Weight Loss	x x	x	x	x
Length Change (Volume Change)	x x	Independent	Almost Independent	Independent
Moisture Gain	x	Almost Independent	Almost Independent	x
Strength After Test (Absolute Value)	Independent	x x	Almost Independent	Independent
Strength Change (Loss during test)	x x	Almost Independent	Almost Independent	Independent
Pulse Velocity After Test (Absolute Value)	Independent	x x	Almost Independent	Independent
Pulse Velocity Change (Loss during test)	x x	Almost Independent	Almost Independent	Independent
Manual Inspection	Independent	x	x	Independent

Note: x - dependent, x x - greatly dependent

(1) Some of the conditions are obviously related. They are considered here separately only to the degree of independence with the first condition.

Some of the measures are expressed both as the absolute value and as the change in the value occurring during the test.

Weight Loss

This method (the manual brushing technique and the PCA weight loss criteria) has been the most extensively used procedure. It has been amply demonstrated that reliable results are obtained for all soil types. The absolute value of the final weight loss is strongly related to the degree of deterioration, but it is also dependent on the extraneous factors of absolute hardness, surface condition and soil type (Table 1). If a specimen were of unchanging hardness during the test and no deterioration occurred, a uniform rate of weight loss would be obtained. This rate would depend on the hardness of the specimen and the abrasive characteristics (soil type). It is well known that specimens passing the weight loss criteria may have large differences in hardness as measured by compressive strength. The abrasive variable is recognized in the three separate weight loss criteria for different soil types.

For some silty soils and fine sands, a surface scaling occurs in the freeze-thaw test which may complicate analysis of weight loss data. The degree of significance of this scaling to field performance is not fully established. The large surface-to-volume ratio of laboratory specimens would emphasize surface effects to a degree that may not be representative of the performance of pavements in service. Surface effects in concrete durability tests are not considered as necessarily reflecting the overall condition of the specimen.

The rate of weight loss is considered to be more dependent on deterioration and less dependent on the extraneous conditions (Table 1). Although the rate of weight loss is not specifically studied in this paper, it has been the general experience that increasing rates reflect deterioration.

Moisture Gain

The moisture gain technique specified in the standard procedures was not found to be a sensitive measure of deterioration for all soil types. The criterion of a moisture gain more than void accommodation at the time of molding reflects deterioration only in very advanced stages. Many specimens will fail weight loss criteria with moisture gains much less than that required for saturation. The moisture changes on freezing-thawing and wetting-drying are primarily a function of the capillary and permeability properties of the mixture.

In the freeze-thaw test, the capillary moisture gain is greatly influenced by soil type and to this extent moisture gains below the saturation limit are not necessarily indicative of deterioration.

In the wet-dry test, there is a progressive drying of most fine-textured mixtures that is independent of deterioration except in advanced stages.

However, deviations from moisture change patterns established by nondeteriorated specimens on a particular soil may indicate failure for specimens at lower cement contents even though a saturation limit is not reached.

Length Change (Volume Change)

The volume change technique specified in the standard procedures was found not to be a sensitive measure of deterioration for all soil types. The criterion of a 2 percent volume change reflected deterioration only in very advanced stages and many specimens fail weight loss criteria with much lower volume changes.

Recent studies measuring lengths with greater precision indicate that expansions of about 0.1 percent indicate failure for soil-cement specimens. This is the same value as the criteria (5) used in concrete durability testing.

Precise length change measurements are considered to be a very sensitive and direct measure of deterioration (Table 1). Studies with these measurements indicate that they are completely independent of absolute hardness, an extraneous factor. The amount of shrinkage on freezing or drying depends mostly on soil type and slightly on cement content. Variations in moisture content during the tests will also affect the degree of shrinkage or expansion. However, the expansions that accompany deterioration are of sufficient magnitude that they are not masked by effects of soil type, cement content or moisture content.

The direct relation of length change to deterioration is well established in concrete freeze-thaw tests. Other deteriorating effects such as sulfate attack, alkali reactivity, and distress due to differential thermal coefficients are also readily detected by length change measurements.

Strength Measurements

Because of the variation in soils' reactions with cement, it is not thought possible to set a single-valued strength criterion that will insure durability for all soil types. The change in strength, rather than the absolute value at start or end of a durability test, is the measure of deterioration.

For compressive strengths after periods of continuous moist-curing, about a 10 percent variation in accuracy is expected on single determinations. Variations are even greater on specimens after freeze-thaw or wet-dry tests. These variations indicate that replication of specimens may be required to develop or accurately apply compressive strength change criteria.

For concrete specimens, effects of freezing on compressive strengths are strongly influenced by the aggregates and the strengths are less sensitive to changes in the paste (5). There is some evidence that this may be true for soil-cement. Compressive strengths may not be detecting minute cracks or localized weaknesses. It is therefore felt, but not examined, that tensile or flexural tests might more sensitively detect deterioration in durability testing.

All things considered strength change testing, with adequate replication, is considered to be a direct measure of deterioration not significantly dependent on the extraneous factors.

Sonic Methods

Most of the previous comments for strength testing also apply to measures of the pulse velocity by the soniscope. Pulse velocities are basically related to the density of a material. However, for any given soil the density of cement-soil mixtures varies only slightly and therefore, the pulse velocity is related to the strength of the mixture. Examples of the relations are shown for one soil (see Fig. 7); additional data are presented in a previous study (4).

Pulse velocity is not thought to be significantly affected by the extraneous variables (Table 1). Although gross deterioration is reflected in major decreases in velocity, there are qualifications which may preclude the use for sensitive and accurate detection of deterioration. The sound pulse selects the best path through the specimen and may not indicate the overall condition. Furthermore, there are large experimental variations in velocities between replicate specimens and between repeated measurements on single specimens.

It seems likely that measurements of resonant frequency by sonic methods, although not examined in this study, might be a sensitive detector of deterioration for soil-cement durability tests. For concrete freeze-thaw tests, there is a close correlation of resonant frequency and expansion in measuring deterioration. The standard size soil-cement specimen would have to be modified to obtain a greater length to diameter (or width) ratio suitable for the sonic test.

EFFECTS OF FREEZING AND THAWING ON SOIL-CEMENT SPECIMENS

The following effects of freezing and thawing were measured under the standard freeze-thaw test conditions unless otherwise noted. Details of the measuring techniques are given in the Appendix.

Length and Moisture Changes

The normal pattern of length changes for undeteriorated soil-cement specimens is shown in Figure 1. The degree of shrinkage on freezing depends primarily on soil type and especially on the silt and clay content. On thawing, specimens expand back towards the original length but never significantly exceed the original length (unless considerable moisture is absorbed in the thawing process). This constant pattern of length changes continues throughout successive freeze-thaw cycles a gradual rise occurs in the pattern, however the relative amount of shrinkage and expansion (the amplitude) remains about the same.

The effect of soil type on length change is indicated in Figure 2. Shrinkages on freezing vary from 0.05 percent for soils of AASHO Classifications A-1, A-2 and A-3 up to 0.60 percent for soils of the A-7 classification. For each soil group there is considerable variation from the average shrinkage.

These observations are made for undeteriorated specimens only. When deterioration starts there is a rise in the volume change pattern (Fig. 3). The envelope of frozen lengths usually starts to rise first, showing successive reductions in shrinkages on freezing. The thaw envelope also rises but to a lesser degree. As deterioration progresses both the thawed lengths and the frozen lengths exceed the original length. At severe deterioration the freeze envelope rises above the thaw envelope, i.e., expansion on freezing, shrinkage on thawing. Several characteristics of the pattern are suggested as detectors of deterioration: (a) expansion on thawing above original length or above first thaw length, (b) expansion on freezing above original length or above first freeze length, (c) progressive reduction in the magnitude of shrinkage on freezing, and (d) expansion on freezing. It is indicated that only two measurements, one at the start of a freeze-thaw test and one at the end, may be required to detect deterioration.

The shrinkage of soil-cement specimens containing appreciable fines is much greater than that which can be explained by thermal effects. Thermal coefficients for soil-cement range from 4 to 7 μ-in./in./° F. It is presumed that this extra shrinkage is caused by a redistribution of the water in the specimen similar to mechanisms that occur in concrete as reported by Powers (6). That is, the unfrozen water in the fine fraction of the mixture is migrating toward the ice being formed in larger void spaces, in effect drying the fine fraction. An example of the amount of shrinkage attributed to drying of the fines can be ascertained by comparing the shrinkage of moist specimens to those of dry specimens (Fig. 4). Inasmuch as drying shrinkage depends on the amount of fines, this mechanism is accepted as explaining the direct relation of increasing freezing shrinkages with increasing silt-clay contents.

This introduces the possibilities of failure mechanisms other than the growth of ice crystals. For soil-cement, as in concrete, hydraulic pressures may contribute to deterioration. The differential

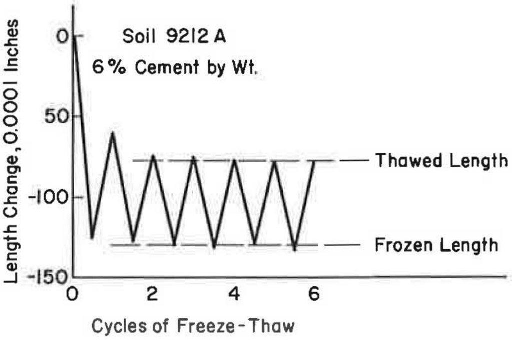


Figure 1. Length changes during freeze-thaw test.

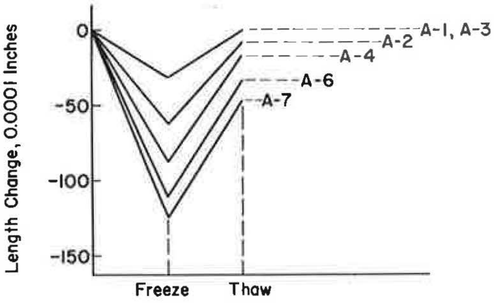


Figure 2. Effect of soil type on length changes during freeze-thaw test.

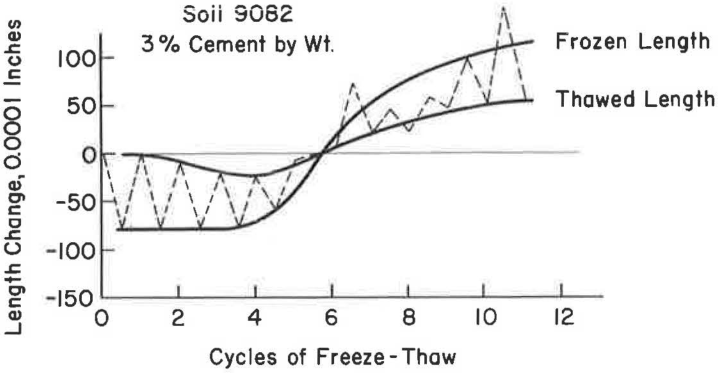


Figure 3. Length changes for deteriorating specimen in freeze-thaw test.

volume change mechanism is presumed to have a greater effect on soil-cement than it does on concrete. This seems logical in view of (a) the greater freezing shrinkages obtained for soil-cement, which depending on soil type, may be several times that of concrete, and (b) the likelihood of having wider differences in the volume change properties of the different soil constituents than for concrete aggregates.

A study of the pattern of length changes on several cycles of freezing and thawing indicates that the initial weakening is not necessarily caused by the ice growth mechanism. In many cases, specimens will maintain a constant pattern of shrinkage and expansion through several cycles of freezing and thawing and then start expanding abruptly (Fig. 3). If the specimens had absorbed additional water the abrupt failure could possibly be explained by the critical saturation concept (6). However, additional moisture was not absorbed. If ice growth damaged the specimen in the first cycle then progressive damage would follow in subsequent cycles and result in a rising pattern of length changes from the start. It is presumed that during the cycles of the constant length change pattern, other mechanisms such as volume changes or hydraulic pres-

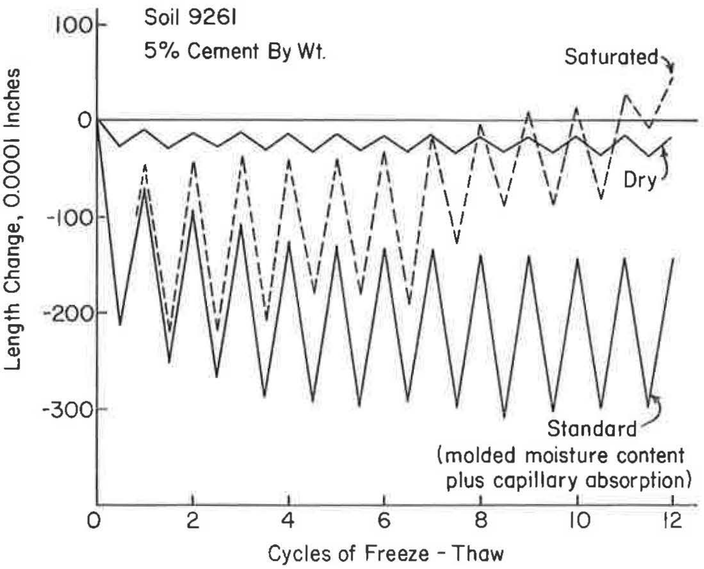


Figure 4. Effect of moisture condition in freeze-thaw test.

sures may gradually break down sufficient bonds of cementation. Between the condition of the steady length change pattern and the severe condition of expansion on freeze it is thought that ice growth becomes the predominant failure mechanism only after the material is weakened by other mechanisms.

Thus, for all soils except those completely devoid of fines, the standard freeze-thaw test measures the deteriorating effects of drying shrinkage.

A mechanism of failure that is discounted for most materials is the failure of the aggregate particles. Even though the plus No. 4 sieve material is saturated at the time of molding, observation of failed specimens indicates that the aggregate particles are not broken. Failure occurs in the matrix between the aggregate particles. An exception to this is made for soft or unstable aggregates such as shales.

Records of the weights of specimens during freezing and thawing indicate that the 25 to 35 percent voids in soil-cement specimens are not completely filled with water; there is from 5 to 10 percent air in the specimens and this air-water-solids proportion does not change during the freeze-thaw test for most soils. Failure of specimens in the standard freeze-thaw test does not depend on the absorption of additional water. On the other hand, specimens of A-4 soils absorb considerable water in the test regardless of their hardness or state of quality. Of course, the absorption of water in itself produces a greater susceptibility to frost damage in the case of silts leading sometimes to a higher cement requirement than that for clay soils.

Undeteriorated specimens of A-1 and A-2 soils on the average gain little moisture, if any, during the test; extremes up to 2 percent by dry weight have been absorbed by some specimens. A-3 soils also usually gain very little but extremes can run up to 4.0 percent for very fine sands. Specimens of A-4 soils usually pick up considerable moisture, from 3 to 5 percent, with extremes of poorly-graded silts gaining 7 percent moisture, just short of saturation. A-6 soils absorb only a small amount of moisture, extremes up to 3 percent.

Scaling of some silt and fine sand specimens is associated with large moisture gains. In this study, all specimens that scaled had previously absorbed at least 4 percent additional moisture. Specimens of the same soils at higher cement contents also absorbed large amounts of moisture but did not scale.

Experiments with soil-cement specimens subjected to concrete freeze-thaw tests (freeze and thaw in water) show that the saturated condition is much more severe than the unsaturated condition used in the standard soil-cement freeze thaw test. Figure 4 shows length changes for freeze-thaw cycles conducted under three moisture conditions: saturation (immersion in water before test and during thaw cycles), the standard method (water absorbed through a felt pad), and completely dry. It is obvious that a much greater cement content would be required to resist the freeze-thaw test at saturation.

Therefore, the severity of a particular freeze-thaw test depends largely on the moisture conditions prevailing in the procedure. Since the moisture content in road bases is usually less than saturation a test condition of saturation is unrepresentatively severe. The capillary absorption condition used in the standard test seems more rational since it permits an absorption that will be determined by the natural capillarity and permeability properties of the soil-cement.

Strength Changes

During the 12 thaw periods of the standard freeze-thaw test there is an opportunity for a strength gain equivalent to about 10 days of additional moist-curing. Figure 5 shows the strength gain during the standard test. Curve 1 represents the strength gain during continuous moist-curing conditions. The maximum values of strength possible for undamaged specimens are represented by curve 2 which terminates at a strength equivalent to 17 days of continuous moist cure. Curve 3 represents the strengths of a specimen slightly damaged by freezing and thawing within the limits permitted by weight loss criteria. Thus, for specimens passing the weight loss criteria it is expected that strengths-after-test will exceed the 7-day strengths by an amount that is in proportion to the increase in strength due to an additional 10 days of moist-curing. Failing specimens may have strengths below curve 3 and yet exceeding the 7-day value while badly deteriorated specimens would have strengths-after-test below the 7-day value.

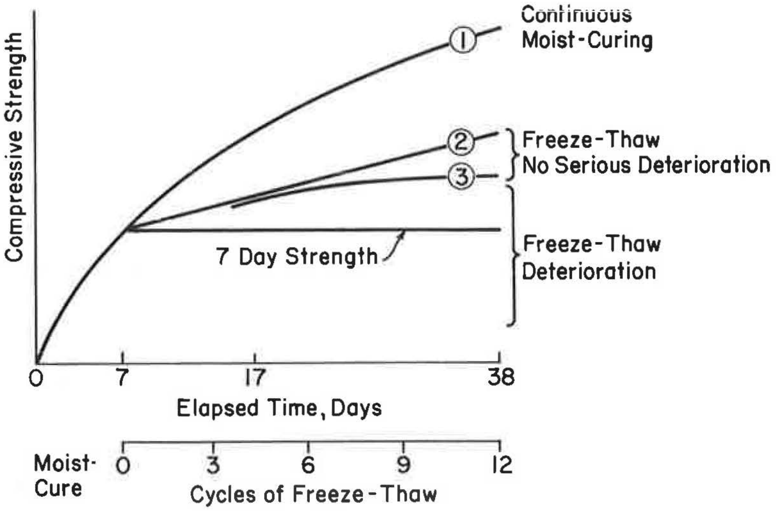


Figure 5. Strength gain during freeze-thaw test.

EFFECTS OF WETTING AND DRYING ON SOIL-CEMENT MIXTURES

An earlier study (4) reported results of weight losses, moisture changes, compressive strength changes and pulse velocity changes during the standard wet-dry tests on four soils. Further examination of the effects of wetting and drying may be of value in two applications: (a) to determine if a more sensitive and direct measure of deterioration can be developed for the standard wet-dry test, and (b) to evaluate the conditions of the test when it is used alone (with no freeze-thaw test) as a durability test for mild climates. The critical evaluation of the standard wet-dry test and suggestions for modification refer to the second application only.

Length changes in the standard wet-dry test depend on soil type. Shrinkages on the first drying vary from 0.05 percent for granular soil-cements to 1.00 percent for clayey soil-cements. For any given soil the first drying shrinkage is roughly twice the freezing shrinkage occurring in the freeze-thaw test. Subsequent length changes on wetting and drying are usually less than length changes in the freeze-thaw test.

The pattern of length changes for undeteriorated specimens is fairly constant (Fig. 6). For deteriorated specimens there is a definite rise in the length change pattern.

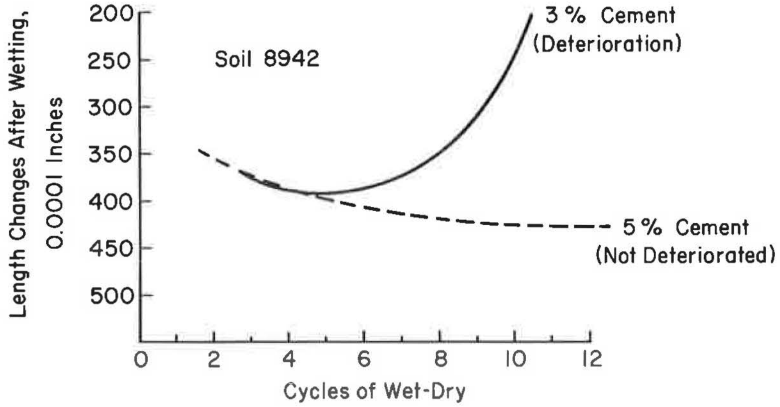


Figure 6. Length changes during wet-dry test (wet lengths only).

Additional studies are required to determine if a length change criteria can be developed to correlate with weight loss criteria in the standard wet-dry test.

The moisture contents of specimens in the wet-dry test also depend on soil type. On drying, moisture contents vary from about 2 to 6 percent moisture, the dryer for soils containing less clay. On wetting, the moisture contents vary from the saturated moisture content to several percentage points below the molded moisture content, the wetter for soils containing less clay. In additional experiments with soil-cement specimens of heavy clay soils, weights on wetting and drying indicate that the specimens are progressively dried in the standard test. In the latter cycles the specimens do not absorb sufficient water to test their great volume change properties. On examination of some of these specimens after wetting it was observed that water had penetrated only into the outside $^{3}/_{8}$ in. of surface.

Results of the earlier study show that the 160 F oven-drying condition used in the standard test causes an accelerated strength gain. This may unduly benefit specimens that might not otherwise pass the wet-dry test. The strength data for specimens of 14 soils at a range of cement contents are summarized in Table 2. Additional data (Table 3, Appendix) show the magnitude of the strength gains in the wet-dry test.

In a separate experiment the 160 F ovendrying condition was replaced by 73 F air-drying. Weight losses, length changes, compressive strengths and pulse velocities were measured on specimens of a gravelly loamy sand. Results of all the measures showed that the 73 F drying condition was more severe than the standard 160 F drying condition.

In the development (1) of the standard testing procedures the possibility of an accelerated strength gain due to the high temperature in the wet-dry test was recognized. This is one of the reasons that the two procedures, the freeze-thaw test and the wet-dry test, were selected to be used together to measure the properties of soil-cement mixtures.

Another effect of the 160 F drying condition is shown in Figure 7. The relation between compressive strengths and pulse velocities is considerably different after the wet-dry test (160 F) from the relation after other environmental conditions (73 F) of continuous moist-curing of freeze-thaw cycles. After the wet-dry test, the pulse velocities decrease appreciably (compared to the pulse velocity at start of test) even though the compressive strengths have increased. Apparently, the high temperature has modified the structure or composition of the soil-cement. A possible, but unsupported, explanation is that the high temperature has caused minute cracking that does not affect compressive strength but greatly reduces pulse velocity.

The volume changes of specimens are also affected by the high temperature. On drying, the drying shrinkage is counteracted by the thermal expansion of heating to 160 F; and on wetting, the expansion due to wetting is counteracted by the cooling to room temperature. This counteraction is shown in Figure 8 where the thermal effect is greater than the moisture effect for specimens of a gravelly loamy sand. For different soil types it is estimated that the thermal effect cancels from 16 percent (for clayey soil-cement) to 100 percent (for soils containing 10 percent clay or less) of the length changes due to wetting and drying.

TABLE 2

SUMMARY OF STRENGTH GAINS IN WET-DRY TEST

Strength after wet-dry test				Weight Loss in wet-dry test, per cent	
per cent of 7 day strength					
average	extremes	average	extremes	average	extremes
240	160-420	160	101-290	11	1-57

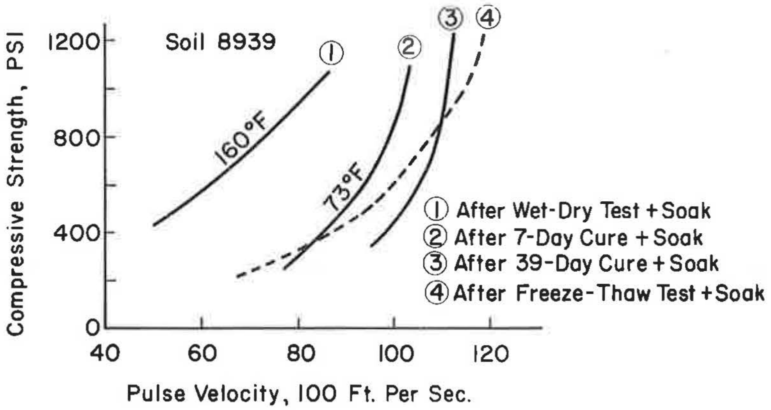


Figure 7. Relation of compressive strength to pulse velocity, different environmental conditions.

The significance of these effects of the temperature and moisture conditions must be considered if only the standard wet-dry test is to be used in the development of procedures for mild climates. For such use, not the intention of the original development of the standard procedure, several modifications of the conditions used in the standard wet-dry test are suggested. First, and most important, it is suggested that the 160 F ovendrying condition be replaced by air-drying at room temperature or at a temperature not exceeding the extremes of natural conditions. This would reduce the previously discussed accelerated strength gains, changes in structure of material and counteraction of volume changes by thermal effects. Second, it is suggested that a greater degree of wetting and a lesser degree of drying would be more representative of natural conditions. This would eliminate the progressive drying and small moisture changes obtained with heavy clay soil-cement. It is felt that this could be accomplished in the same or less time than that required for the standard cycle by use of a deep soaking tank to accelerate wetting, and by use of a shorter drying period.

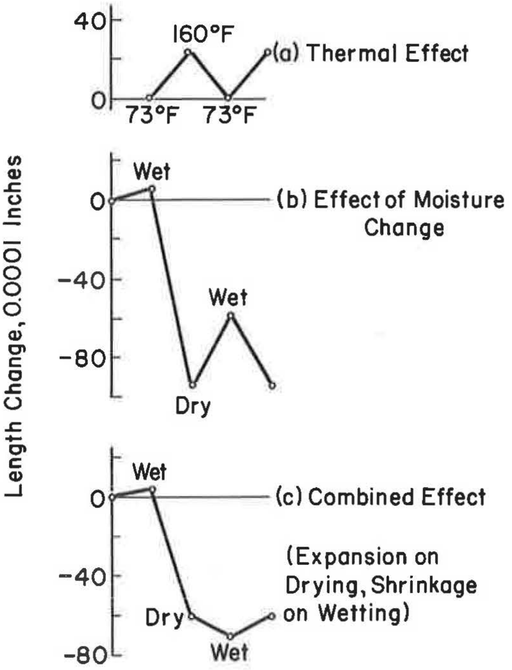


Figure 8. Effects of temperature and moisture changes on lengths of specimens in wet-dry test (soil 9328 - gravelly loamy coarse sand).

CORRELATION OF LENGTH CHANGE, WEIGHT LOSS AND COMPRESSIVE STRENGTH DATA IN THE STANDARD FREEZE-THAW TEST

Weight losses, moisture gains, length changes, compressive strengths and condition by manual inspection were recorded during standard freeze-thaw tests on 235 specimens molded of 86 soils. Measuring techniques are described in the Appendix and results are given in Table 4.

An attempt was made to correlate results from the various measures to indicate if the specimens had passed or failed the test as primarily defined by weight loss criteria. Best agreement was found by using the following limits of test values of the various measures:

1. Weight Loss.—PCA criteria of maximum weight loss of 14 percent, 10 percent or 7 percent depending on soil classification.
2. Length Change.—Twelfth cycle frozen length not exceeding first cycle frozen length by 0.0050 in. (The other changes in the length change pattern discussed previously may be equally effective in detecting deterioration.)
3. Compressive Strength.—When strengths-after-test are 145 percent or more of the 7-day values the specimens always pass the test; when strengths-after-test are less than 90 percent of the 7-day value the specimens always fail the test; strengths between 90 and 145 percent of the 7-day value may occur for failed or passed specimens with increased probability of passing as the upper limit is approached.
4. Condition of Specimens by Inspection After Test.—A minimum condition of "fair to O.K." as defined by arbitrary ratings is usually required for passing the test.

The results are discussed primarily as comparisons of length change data and weight loss data because the former is considered to be the most accurate and sensitive detector of deterioration, and the latter is considered to give the cement requirement best correlated to field performance.

Expansions and weight losses are in exact agreement as to whether the specimens passed or failed the test in 205 out of 235 cases (87 percent). They are in slight disagreement in 21 cases (9 percent) and in serious disagreement in 9 cases (4 percent). The cases of exact agreement are shown in Figure 9 and cases of slight and serious disagreement in Figure 10. Strength-after-test data are also indicated as additional evidence of failure or passing.

Data in Figure 9 establish the significance of the strength-after-test data. For all the points in the lower left quadrant, expansions and losses agree that the specimens passed the test. For all the points in the upper right quadrant, expansions and losses agree that the specimens failed the test. Major strength increases indicated by the "x" and "o" points, fall into the proper quadrant with only five exceptions. Thus, expansions and losses are in 100 percent agreement. For these data, strength data corroborate in 93 percent of the cases.

In Figure 10, expansions and losses are in slight or serious disagreement. In all cases strength data agree with the expansion data, disagreeing with weight loss data, as to whether the specimen has failed or passed.

For most of the cases where excessive losses occurred without serious expansion, the losses were affected by scaling of the specimens. These are indicated in the upper left quadrant by squares.

Figure 11 illustrates a consideration for points in the lower right quadrant of Fig. 10. In some cases where expansion did not start until the latter cycles the test was continued past 12 cycles. Weight losses then increased beyond that allowed by criteria showing the deterioration measured by expansion a few cycles earlier.

The particular expansion value shown in these plots is considered to be in the correct range but not as a final criterion to be adopted without more development. Thus, close agreement between expansion and weight loss data was obtained in 96 percent of the cases, strengths corroborating. For the few cases of disagreement between expansion and weight loss data, consideration of strength data, delayed weight losses and instances of scaling validate the expansion data. For all cases, there is strong evidence that length changes are more sensitive, accurate and immediate in detecting deterioration.

Due to the demonstrated efficiency of length change measurements in the standard freeze-thaw test the technique is recommended for: the development of modifications or accelerations of the standard procedures, the development of durability tests for specific climate areas, or the comparison of the severity of different durability tests.

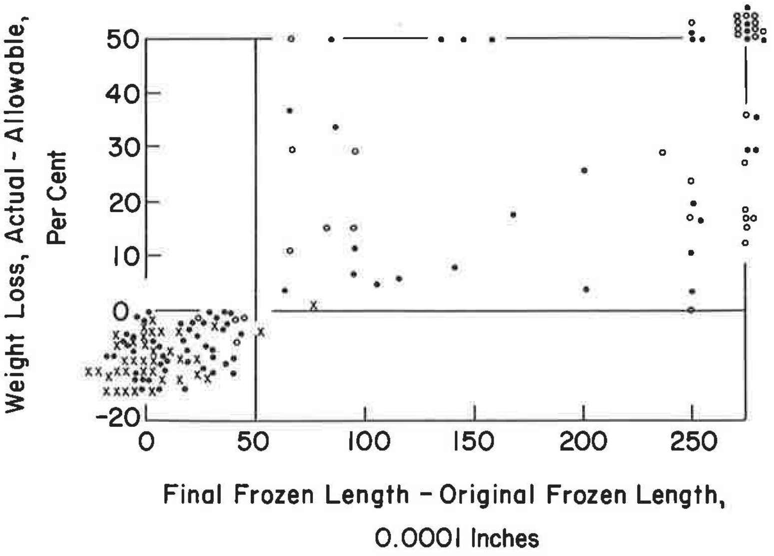


Figure 9. Correlation of measures in freeze-thaw test, agreeing cases—x = strength-after-test more than 145 percent of 7-day strength; o = strength-after-test less than 90 percent of 7-day strength; ● = strength between above limits or incomplete data.

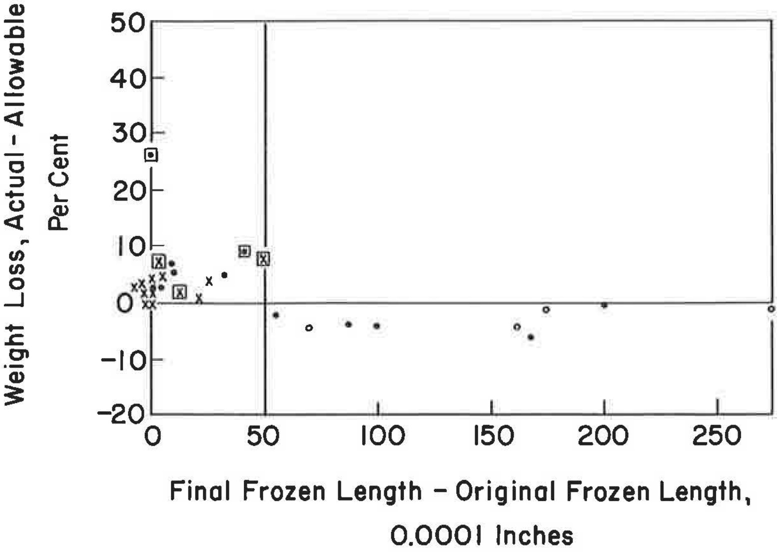


Figure 10. Correlation of measures in freeze-thaw test, disagreeing cases— x = strength-after-test more than 145 percent of 7-day strength; o = strength-after-test less than 90 percent of 7-day strength; ● = strength between above limits or incomplete data.

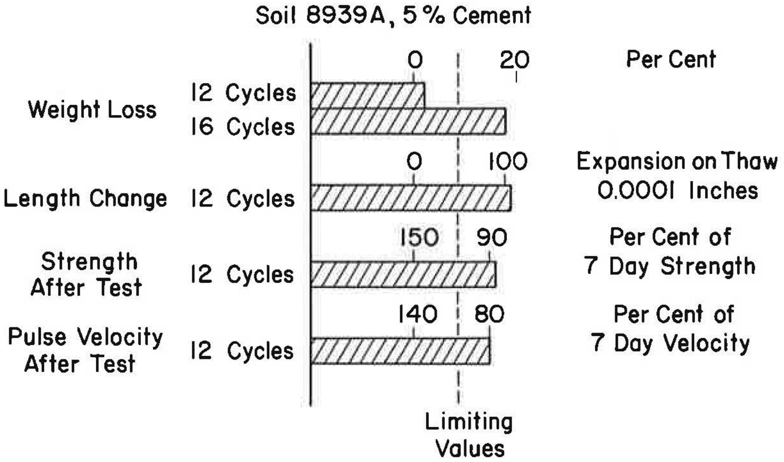


Figure 11. Comparison of freeze-thaw results by four measures.

ACCELERATED PROCEDURE FOR FREEZE-THAW TESTING

It is well established that the standard ASTM freeze-thaw test procedure and the related PCA criteria determine a cement content that will produce a durable construction material. However, the fact that the standard test requires from 6 to 7 weeks to complete restricts its use in some instances.

It is not intended to establish a new and independent method of determining the cement requirement for soil-cement mixtures. It is intended, rather, that the accelerated procedure would result in a cement requirement equal to that obtained from the standard procedure.

In the 24-hr thaw periods of the standard test only 8 hours are required for thawing and for a rise of specimen temperature (interior) to room temperature; the other 16 hours represent additional moist-curing.

In the 24-hr freeze periods, 8 hours at the most are required to bring the specimen temperature (interior) to -10 F. During the other 16 hours it is presumed that no significant damage is effected in this closed system of freezing (additional water not available).

Thus, of the 24 working days required for 12 cycles of the test, there is a period equivalent to 16 days that might be eliminated without defeating the objectives of the test.

Reduction in the length of thaw periods would result in changes in the interpretation of results from various measures of the quality of specimens. Strengths would be lower in an accelerated test because there is less moist-curing time. Weight losses in the accelerated test would be greater, with an equal amount of brushing, than those in the standard test since the specimens cannot gain as much strength. Moisture gains would be lower because there is less time available for absorption; however, this would be significant only for those few soils that absorb appreciable moisture in the standard test. On the other hand, it is thought that interpretation of length change data would be almost the same as in the standard test.

The effect of rate of cooling, in addition to the effect of shorter thaw periods, was considered important. It was decided not to exceed the rate of cooling (10° F. per hr) of the freezer used for the standard test, especially since an increased rate might alter the failure mechanism as it does in concrete freeze-thaw testing (6).

It was desired to select an accelerated freeze-thaw cycle whose severity did not greatly exceed the standard cycle. The cycle selected, after preliminary studies with several variations of freeze-thaw temperatures and durations, is shown in Figure 12 and compared to the time-temperature used with standard tests.

The equipment for automatic freezing and thawing is shown in Figure 13. Its essential components are: a copper-lined cabinet insulated with styrofoam, a $\frac{1}{3}$-hp condenser with reverse cycle controlled by solenoid actuated valves, 60 ft of $\frac{3}{8}$-in. copper tubing coiled in the sides and bottom of the cabinet, an air-circulating fan and an adjustable,

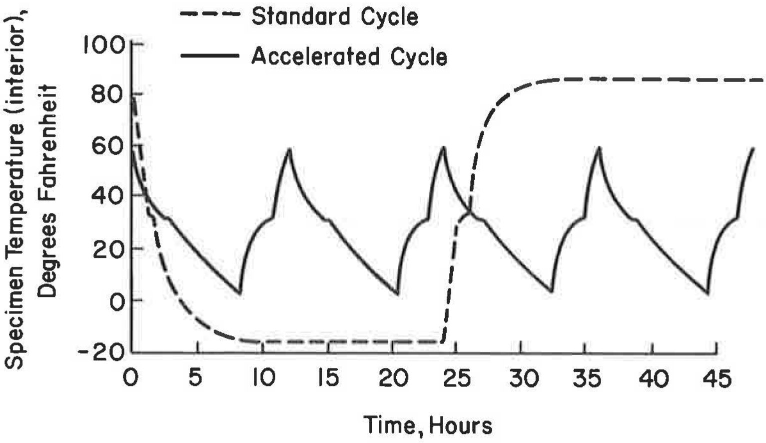


Figure 12. Time-temperature cycles for freeze-thaw test, standard and accelerated.

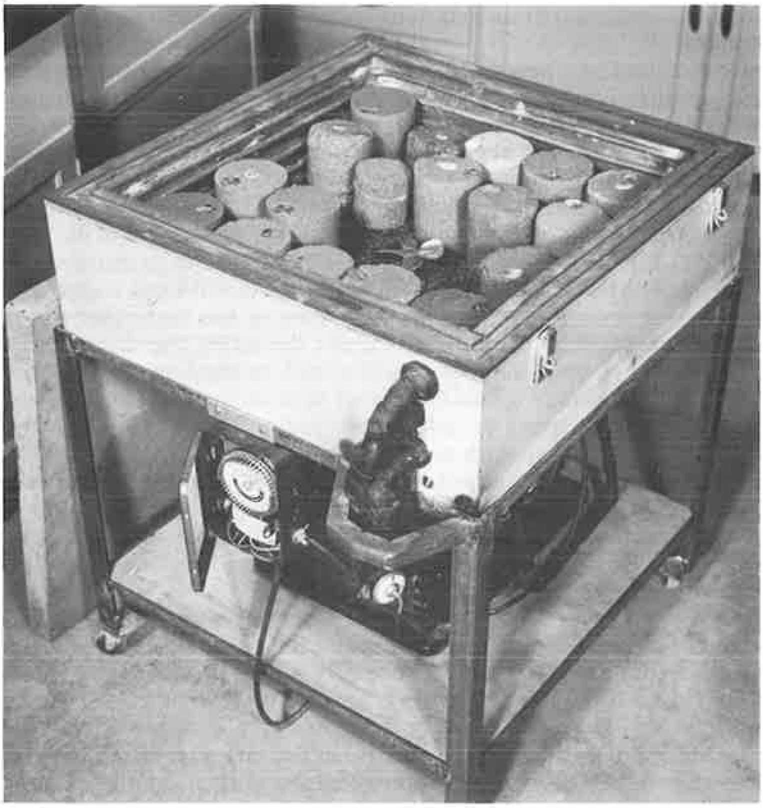


Figure 13. Automatic freeze-thaw equipment used in accelerated tests.

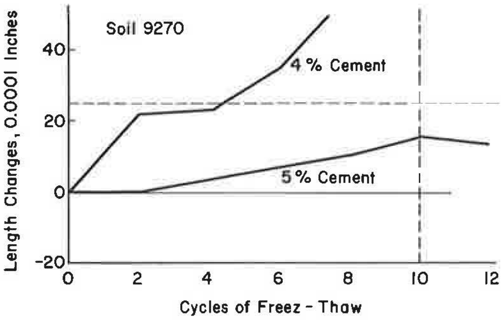


Figure 14. Length changes in accelerated freeze-thaw cycles (thawed lengths only).

electric time switch. The cabinet has a capacity for 24 specimens; dummy specimens were used when required to keep the freezing load constant.

Standard size specimens molded from 20 soils were subjected to the accelerated test after a 7-day moist-curing period. Sixteen cycles were conducted at a rate of 2 cycles per day, four times faster than the standard rate. During the test the specimens were kept on a water-saturated felt pad.

Data on length changes, compressive strength, and weight losses were recorded on replicate specimens during the test (Table 5, Appendix). These data were studied to define a limiting value at a particular cycle that would consistently give the same cement requirement already determined by standard procedures.

Figure 14 shows typical length change data for the accelerated cycles. Specimens at the cement content (5 percent by weight) required to pass the standard test exhibited only slight expansions. Specimens with less cement expanded considerably. (Expansion values in this section are changes in the thawed lengths computed from either the original length or the first measure thaw length, whichever gives the greater value.)

The accuracies of six limiting expansion values in giving the standard cement requirements are compared in Figure 15. An expansion value of 0.0025 in. after 10 cycles of testing was selected as the most suitable. Consideration was given to the selection of a minimum number of unsafe cases, i.e., cement requirements from the accelerated test less than that for the standard test.

Compressive strength tests were performed on specimens at three stages of testing; before starting the freeze-thaw (at 7 days' moist cure), during the test after either 6 or 8 cycles, and at the completion of the test which was usually at 16 cycles but sometimes at 12 cycles. An exact trend of strength changes differentiating between failed and passed specimens was not established. However, the 6-, 8-, 12- or 16-cycle strengths

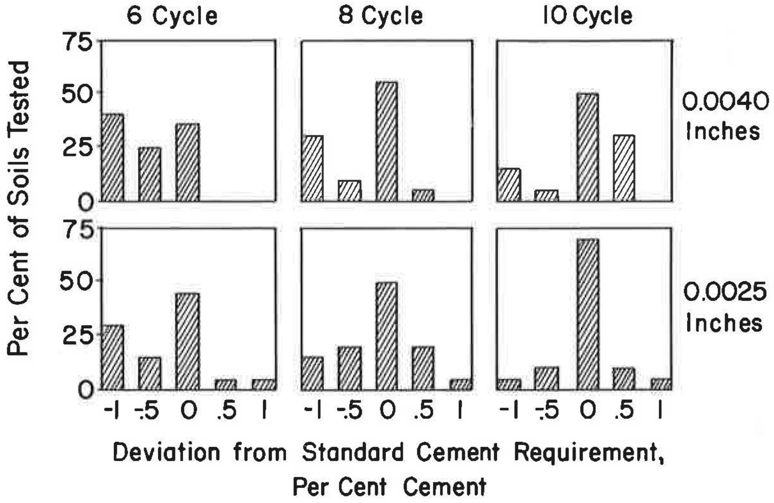


Figure 15. Accuracies of 6 limiting expansion values in accelerated freeze-thaw test, expressed as deviation in cement requirements (accelerated— standard).

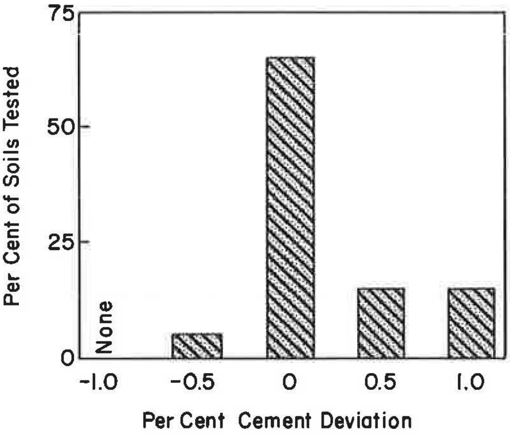


Figure 16. Accuracy of dual limiting values in accelerated freeze-thaw test, expressed as deviation in cement requirements (accelerated — standard).

of specimens with adequate cement contents exceeded 75 percent of the 7-day strength value with few exceptions.

Weight loss data are also given in Table 5. Due to the automatic cycling, brushing of specimens could only be performed on alternate cycles on weekdays and not at all on weekends. In view of the variations inherent in this procedure, no analysis of weight loss data was made.

The best correlation to the cement requirements of the standard procedure was found by applying dual limiting values: a limiting expansion value of 0.0025 in. at 10 cycles and a 10-cycle compressive strength greater than 75 percent of the 7-day value. With these limiting values the cement requirement by the accelerated procedure was the same or up to 1 percent cement greater than the standard requirement for 19 of the 20 soils tested. For one soil the accelerated requirement was 0.5 percent cement less than the standard requirement. The distribution of these deviations is shown in Figure 16.

Within the limited scope of this study there is a promising indication that a greatly accelerated freeze-thaw procedure can be developed to determine the same cement contents as required by the standard freeze-thaw test. However, these results are definitely exploratory and the testing of a great quantity and variety of soils is needed to develop fully a completely reliable accelerated method. Further studies are contemplated and will include more soils, different time-temperature cycles and attempts to develop criteria based on a single measuring technique.

SUMMARY

Methods for Measuring Deterioration in Freeze-Thaw and Wet-Dry Tests

Several methods (weight loss, volume change, moisture gain, length change, strength, sonic methods and visual inspection) were discussed relative to the degree of their direct relation to deterioration. Of these methods, four are judged as primarily measuring deterioration and relatively free of extraneous influences: length change, strength change, rate of weight loss and pulse velocity change. The length change method is superior in immediate sensitivity and accuracy in detecting deterioration.

It is emphasized that in the development of criteria for any of these measures, correlation to weight loss results and weight loss criteria will insure a tie-in to long-term field performance.

Effects of Freezing and Thawing on Soil-Cement Specimens

Data were presented to show that the initial volume (length) and moisture change characteristics of specimens on freezing and thawing depend primarily on soil type and, only to a limited extent, on cement content.

When deterioration occurs volume changes become abnormal (expansion), whereas the moisture change characteristics may not be immediately affected.

In the standard freeze-thaw test there is evidence that ice growth becomes a failure mechanism only after the material is weakened by other forces such as hydraulic pressures and absolute or differential volume changes. It is indicated that failure occurs when sufficient bonds of cementation are broken in the matrix between particles and, for most materials, that failure does not occur by disruption of the saturated aggregate particles.

For all but the very clean soils results show a degree of volume change, increasing with the amount of fine particles in the soil, that cannot be assigned to thermal effects alone. Similar to mechanisms in concrete, and more pronounced, these volume changes are attributed to the dessication of the fines on freezing.

Comparison of the results of two experimental freeze-thaw tests (one with dry specimens and one with saturated specimens) with the results of the standard test (moisture content depending on capillary absorption) indicates the great influence of the moisture conditions used in a freeze-thaw test.

The effect of freezing and thawing on the compressive strengths of specimens is considered essential to the interpretation of strength-after-test data in the standard test.

Effects of Wetting and Drying on Soil-Cement Specimens

The initial volume (length) and moisture change characteristics of specimens on wetting and drying are shown to depend primarily on soil type and, only to a limited extent, on cement content. When deterioration occurs volume changes become abnormal (expansion), whereas the moisture change characteristics may not be immediately affected.

Effects of the high temperature used in the standard wet-dry test are apparent in three manifestations: an accelerated strength gain, a counteraction of the volume changes due to moisture changes by thermal effects and a possible alteration of the structure of the material. Experimental wet-dry tests (drying at 73 F) were more severe than the standard test (drying at 160 F).

Knowledge of these effects is essential to the interpretation of volume change, moisture change and compressive strength data in the standard wet-dry test.

(Because of these effects the standard wet-dry test is not considered suitable for use alone as a procedure for non-frost areas. For this purpose modifications in the moisture and temperature conditions are suggested that are more representative of natural conditions. These modifications may serve better as a basic procedure on which to develop criteria related to long-term field performance in non-frost areas.)

Correlation of Length Change, Weight Loss and Compressive Strength Data in the Standard Freeze-Thaw Test

A close correlation was obtained between weight loss and length change data for 235 specimens molded of 86 soils. In 96 percent of the cases, expansions of 0.1 percent served to correlate with PCA weight loss criteria to indicate failure of the test specimens. In the few cases of disagreement between length change and weight loss data there is substantial evidence that the length change data are more sensitive.

A general, but not exact, correlation was established for compressive strength data.

It is suggested that sufficient data have been accumulated on a wide range of soil types to show that the length change method represents an improved, alternate technique to the brushing procedure used in the standard freeze-thaw test.

Accelerated Procedure for Freeze-Thaw Testing

Parallel tests, with standard freeze-thaw cycles and accelerated freeze-thaw cycles, were conducted on specimens molded from 20 soils. An automatic freeze-thaw cabinet was used for the accelerated cycles. Length changes, compressive strengths, weights and moisture contents were recorded to measure the quality of the specimens. These data were analyzed in an attempt to correlate results of the accelerated procedure to give the same cement requirement as determined by the standard procedure. A fair correlation was obtained with length change and strength data.

The promising indications that an accelerated test, correlated to the standard test, can be performed in 5 days are considered exploratory and in need of verification with a large number of soils of varied types.

REFERENCES

1. Catton, M. D., "Early Soil-Cement Research and Development." Proc., ASCE Paper 1899, Vol. 85, No. HWI (Jan. 1959).
2. "Standard Method for Wetting-and-Drying Test of Compacted Soil-Cement Mixtures." ASTM Designation: D 559-57 and "Standard Method for Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures." ASTM Designation: D 560-57, ASTM Standard, Part 3.
3. "Soil-Cement Laboratory Handbook." 5th Ed., Portland Cement Association (1959).
4. Packard, R. G., "Alternate Methods for Measuring Freeze-Thaw and Wet-Dry Resistance of Soil-Cement Mixtures." HRB Bull. 353, 8-41 (1962).
5. Verbeck, G. J., "Nondestructive Tests for Deterioration of Concrete Specimens Subjected to Frost Action." Research Dept. Bull. 79, Portland Cement Association (1956).
6. Powers, T. C., "Basic Considerations Pertaining to Freezing-and-Thawing Tests." Proc., ASTM Vol. 55 (1955).

Appendix

TESTING PROCEDURES, MEASURING TECHNIQUES AND MATERIALS

Standard size specimens, 4.0-in. diameter by 4.6-in. height were molded at optimum moisture and maximum density as determined by the standard procedure, ASTM Desigation: D 558-57. Specimens were molded at a range of cement contents above and below the cement requirement determined by the standard freeze-thaw test and PCA weight loss criteria. These were moist-cured for 7 days before freeze-thaw and wet-dry tests.

The freeze-thaw and wet-dry tests were conducted on specimens that were brushed using the standard brushing procedures and on companion specimens that were not brushed but used for length and compressive strength measurements.

Weights of all specimens were periodically recorded during these tests so that weight losses and moisture changes could be computed.

Length measurements at each environmental change were made on unbrushed specimens. During the moist-curing period copper washers (No. 9 rivet burrs) were cemented as reference points to the top and bottom of each specimen. The mixture used for attaching reference points was prepared as follows: Polyester Selectron 5119 (Pittsburgh Plate Glass) and silica flour were mixed to the consistency of a heavy syrup; to this was added an accelerator, MEK Peroxide (American Resin Corp.) at the rate of 1 drop per gram. Lengths were measured in a length comparator equipped with a dial gage graduated to 0.0001 in. The first (reference) length was measured at the end of the 7-day curing period.

Compressive strengths were determined after certain periods of moist-curing, at certain cycles of the freeze-thaw and wet-dry test and at the end of these tests. In all cases the specimens were soaked for 4 hours before being capped and broken.

Pulse velocities by soniscope were determined on some of the specimens during and after the tests. Velocities reported here were determined on specimens that had been soaked for 4 hours.

In the current studies of length changes, strength changes and pulse velocities, specimens of 104 soils were investigated. These soils represent a wide range of soil types from most of the states. General comments on strengths and durabilities are based on standard soil-cement tests on hundreds of soils.

The Type I portland cement was a blend of four commercial brands purchased in the Chicago area.

Tabulation of Results

Tables 3, 4 and 5 list the essential portion of raw data on weight losses, length changes, and compressive strengths obtained in the standard freeze-thaw test, the accelerated freeze-thaw test and the standard wet-dry test.

TABLE 3

RESULTS OF WET-DRY TESTS

Weight Losses and Compressive Strengths

			Compressive Strength, psi		
Soil No.	Cement content, per cent by weight	Weight loss, per cent	7 day	28 day	After Wet-Dry test
S-1	3	17	248	334	450
	5	4	385	637	950
	7	2	595	809	1400
SD-20	3	7	302	376	585
	5	4	458	650	1000
	7	1	602	820	1180
SD-35	3	12	248	310	600
	5	5	357	510	940
	7	3	372	650	1550
SD-40	3	12	283	375	540
	5	6	398	540	950
	7	3	372	590	1300
SD-45	3	20	274	340	585
	5	6	293	445	1050
	7	2	414	585	1420
MC-1	3	10	137	155	280
	5	4	210	250	610
	7	3	255	300	875
MC-2	3	20	220	280	440
	5	9	360	530	900
	7	4	400	740	1270
MC-3	3	15	250	360	560
	5	6	360	560	860
	7	3	515	790	1265
FS	7	32	137	261	280
	9	11	220	366	480
	11	7	254	537	540
FS-8	4	57	86	145	230
	6	20	172	251	388
	8	9	269	408	435
FS-16	3	34	94	209	394
	5	13	172	296	465
	7	6	255	393	540
FS-24	3	25	150	228	380
	5	12	239	357	450
	7	5	342	538	545
FS-32	3	27	205	350	460
	5	12	318	468	590
	7	6	481	729	770
FS-48	4	11	320	417	670
	6	5	403	519	795
	8	3	407	618	1060

TABLE 4

RESULTS OF FREEZE-THAW TESTS

Weight Losses, Length Changes and Compressive Strengths

Soil No. & AASHO Class.	Cement Content, per cent by wt.	Weight Loss, per cent	Length Changes 0.0001 Inches				Compressive Strength, psi	
			1st Freeze	1st Thaw	12th Freeze	12th Thaw	7 Days	After Test
8295	3	14	-	0	238+	100+	200	235
A-4(5)	3	14	-92	43	120+	85+	216	250
	5	6	-97	2	-37	-2	224	284
	5	8	-102	-30	-25	-12	243	407
	7	3	-76	-17	-	-	341	481
	7	3	-86	-28	-94	-36	403	525
	10	2	-67	-3	-62	-4	577	740
	10	2	-66	-3	-36	-4	586	796
	13	1	-58	-19	-59	-2	867	987
	13	1	-61	-5	-46	3	851	1002
8939	2	29	-	5	66	100	190	377
A-1-b(0)	2	26	-12	5	78	87	197	332
	3	3	-	0	16	30	312	605
	3	3	-	0	39	40	326	581
	5	2	-	0	-	-	702	1131
	5	2	-	0	-29	-10	703	1154
	7	1	1	0	-33	3	1123	1831
	7	2	-35	2	-20	12	1120	1791
8939A	3	54	-	15	-	300	180	85
A-1-b(0)	5	2	-	5	-	240	538	463
(No. +4)	7	1	-	3	-	-5	907	1140
8942	3	-	-189	60	774+	451+	92	78
A-6(10)	3	-	-184	45	535+	291+	84	92
	5	19	-198	-40	435	357	215	273
	5	23	-212	-45	285	189	240	271
	7	10	-	-	36	94	325	407
	7	9	-107	-2	116	134	363	461
	10	2	-147	-28	-175	-78	477	608
	10	2	-136	-17	-174	-63	504	-
	13	1	-118	-22	-154	-60	573	652
	13	1	-122	-26	-149	-62	506	678
9064	4	28	-130	-35	35	25	356	390
A-4(3)	6	1	-105	-40	-80	-40	465	609
9069	8	34	-28	3	124+	66+	530	822
A-4(8)	8	38	-31	3	318+	144+	525	771
	10	15	-31	3	111	95	693	1082
	10	18	-30	3	80	85	640	1051
	12	8	-29	2	-16	17	780	1280
	12	7	-28	3	4	39	772	1345

TABLE 4

RESULTS OF FREEZE-THAW TESTS (Continued)

Soil No. & AASHO Class.	Cement Content, per cent by wt.	Weight Loss, per cent	Length Changes 0.0001 Inches				Compressive Strength, psi	
			1st Freeze	1st Thaw	12th Freeze	12th Thaw	7 Days	After Test
9069 (Cont')	14	2	-34	2	-29	2	1060	1392
A-4(8)	14	2	-36	3	-25	6	948	1562
9073	2	100	-50	-15	35	30	-	151
A-1-b(0)	4	4	-50	5	-5	15	-	490
9076	7	39	-90	2	145	105	445	174
A-4(5)	9	21	-95	-15	-30	0	670	478
9079	9	6	-75	10	-50	10	-	544
A-6(9)	11	5	-70	10	-40	20	379	560
9081	4	3	-35	2	-32	-10	468	572
A-1-b(0)								
9082	3	100	-75	0	200	75	-	-
A-2-4(0)	5	19	-70	-5	-65	5	346	685
9085	5	17	-30	-5	-30	-5	111	119
A-2-4(0)	7	7	-30	-5	-25	-5	-	310
9089	4	100	-30	-15	105	80	-	88
A-3(0)	6	51	-20	0	45	60	-	221
9090	2	13	-30	15	13	30	-	236
A-1-b(0)	4	3	-20	5	4	14	-	490
9091	11	10	-100	0	-65	25	512	-
A-4(8)	13	3	-90	-5	-60	5	-	-
9092	9	16	-190	-40	-76	-12	-	358
A-4(8)	11	4	-170	-35	0	0	509	473
9110	8	7	-25	0	-39	-3	254	495
A-3(0)	10	5	-25	0	-34	15	421	748
9112	10	2	-140	-40	-129	-37	286	414
A-4(4)	12	1	-115	-30	-109	-38	360	572
9113	8	100	-25	0	282	216	191	231
A-2-4(0)	10	2	-25	0	-38	4	354	701
9115	12	6	-20	0	-32	5	296	597
A-3(0)	15	0	-30	-5	-26	2	-	876
9116	11	4	-30	0	-37	-7	142	454
A-3(0)								
9117	3	4	-25	0	-34	-9	409	486
A-1-b(0)	5	2	-20	0	-28	-4	748	955
9118	3	8	-30	5	137	97	359	355
A-1-b(0)	5	0	-30	5	-13	9	748	796
9119	4	10	-25	0	45	25	195	154
A-1-b(0)	6	2	-25	0	-25	0	451	470
9130	2	100	-30	2	113	118	-	-
A-1-a(0)	3	6	-25	0	-23	0	438	-
9131	9	18	-105	10	-55	15	267	490
A-4(8)	11	7	-90	-10	-40	20	439	675
9132	8	13	-290	-70	-119	3	631	591
A-2-4(0)	10	7	-265	-105	-224	-98	715	716

TABLE 4

RESULTS OF FREEZE-THAW TESTS (Continued)

Soil No. & AASHO Class.	Cement Content, per cent by wt.	Weight Loss, per cent	Length Changes 0.0001 Inches				Compressive Strength, psi	
			1st Freeze	1st Thaw	12th Freeze	12th Thaw	7 Days	After Test
9137	6	100	-30	10	200+	200+	-	-
A-3(0)	6 + $CaCl_2$	100	-30	10	200+	200+	-	-
9138	7	11	-140	-40	-105	-20	407	385
A-2-4(0)	9	5	-110	-5	-74	6	503	565
	11	3	-100	-5	-70	7	-	748
9139	6	21	-100	65	200+	200+	-	289
A-4(4)	8	8	-165	-40	-125	-26	620	489
	10	4	-140	-20	-99	-2	740	533
	12	2	-140	-30	-103	-18	-	748
9140	6	14	-75	-5	-47	15	-	369
A-2-4(0)	8	7	-45	5	-34	12	314	568
	10	3	-45	5	-36	16	-	653
9141	5	23	-28	5	15	31	-	150
A-3(0)	7	12	-25	5	-10	24	-	231
	9	8	-31	-11	-15	22	-	382
	11	7	-14	6	-15	21	513	537
	13	5	-27	7	-16	23	-	716
9143	4	48	-20	5	66	77	-	120
A-1-b(0)	6	8	-20	5	6	40	313	303
9144	3	3	-25	0	-19	7	329	844
A-1-b(0)	5	1	-25	0	-32	-8	469	1099
9145	3	7	-28	-2	-21	10	180	688
A-1-b(0)	5	3	-28	-2	-18	17	428	705
9146	3	9	-21	-15	-48	-27	304	-
A-1-b(0)	5	2	-26	-3	-20	-13	650	-
9147	4	20	-17	-2	-8	18	-	254
A-2-4(0)	6	11	-33	-8	-44	-15	318	258
9148	4	100	-25	5	490	223	-	21
A-1-b(0)	6	13	-25	5	18	38	216	178
	8	5	-25	5	-7	23	428	404
9151	8	10	-35	35	135	138	418	387
A-4(3)	10	6	-35	30	-35	38	505	637
9152	4	5	-25	5	-8	25	227	629
A-1-b(0)	6	2	-25	5	0	24	461	1083
9153	6	6	-70	-5	-60	-8	-	-
A-2-4(0)	8	4	-85	-10	-60	-20	-	-
9155	7	100	-60	15	353	270	302	88
A-4(2)	9	36	-50	-5	-27	10	501	522
9156	4	14	-60	-20	-3	41	250	263
A-4(1)	6	8	-80	20	-40	30	450	511
9157	3	29	-92	-15	-15	-9	310	205
A-2-4(0)	5	6	-75	-10	-36	4	455	422

TABLE 4

RESULTS OF FREEZE-THAW TESTS (Continued)

Soil No. & AASHO Class.	Cement Content, per cent by wt.	Weight Loss, per cent	Length Changes 0.0001 Inches				Compressive Strength, psi	
			1st Freeze	1st Thaw	12th Freeze	12th Thaw	7 Days	After Test
9160	10	15	-35	0	40	60	91	156
A-3(0)	12	6	-35	0	-20	15	127	250
9161	6	100	-25	0	188	158	-	65
A-3(0)	8	13	-25	8	-30	15	-	110
9162	5	0	-25	0	-37	-11	-	697
A-1-b(0)								
9164	3	100	-160	-40	600+	500+	-	-
A-4(5)	5	29	-170	-45	110	68	250	95
9165	2	100	-30	30	127	92	-	174
A-1-b(0)	3	10	-30	15	40	38	263	290
9169	2	3	-91	-66	-45	-20	-	318
A-1-b(0)	3	1	-145	-107	-25	15	420	438
9174	5	15	-130	10	(1)	(1)	391	430
A-2-4(0)	7	9	-105	5	(1)	(1)	423	578
9175	4	100	-95	5	700+	500+	-	33
A-6(5)	5	100	-115	10	700+	500+	284	16
	6	100	-102	8	700+	500+	-	16
	7	100	-98	2	700+	500+	439	17
9181	4	100	-39	-4	761	390	147	25
A-2-4(0)	6	100	-26	7	376	215	206	216
	8	33	-50	-10	60	45	-	650
9182	3	100	-25	5	400+	100+	46	0
A-1-b(0)	7	16	-25	5	-19	3	207	293
9183	12	100	43	83	200+	100+	114	0
A-2-4(0)	8 $CaCl_2$	17	-25	0	-25	0	107	499
	10 $CaCl_2$	4	-25	0	-25	0	173	689
9203	6	6	-25	0	-25	0	325	611
A-4(0)	8	4	-25	0	-25	0	-	979
9204	3	16	-20	0	-30	-2	205	439
A-1-b(0)	4	11	-20	0	-27	4	350	629
	5	5	-20	0	-33	-2	494	812
9207	4	9	-30	10	-27	15	-	400
A-4(2)	6	4	-30	5	-50	-10	210	412
9209	3	31	20	30	183	144	221	174
A-2-4(0)	5	14	-5	10	240	202	345	304
9210	4	21	-70	-25	25	45	-	371
A-1-b(0)	5	6	-60	-10	-40	-5	281	524
9211	8	15	-25	0	-28	-9	129	-
A-3(0)	10	10	-25	0	-26	-3	325	408
9212A	6	5	-123	-60	-222	-78	-	231
A-4(2)	8	6	-116	-62	-190	-80	302	446

(1) -- Loose reference point, no expansion at 9 cycles.

TABLE 4

RESULTS OF FREEZE-THAW TESTS (Continued)

Soil No. & AASHO Class.	Cement Content, per cent by wt.	Weight Loss, per cent	Length Changes 0.0001 Inches				Compressive Strength, psi	
			1st Freeze	1st Thaw	12th Freeze	12th Thaw	7 Days	After Test
9217	11	10	-	-	-	-	-	-
A-7-6(14)	13	7	-100	12	-75	-1	420	451
	15	4	-100	0	-81	-9	436	422
9218	4	27	-36	19	200+	200+	-	178
A-4(1)	6	9	-69	-3	-47	-1	325	287
9221	10	14	-35	3	-44	4	362	362
A-4(8)	12	2	-35	3	-48	-4	-	372
9222	6	15	-73	-9	-85	-5	-	178
A-4(6)	8	9	-78	-8	-80	-21	-	275
9246	5	100	-48	-3	200+	200+	170	100
A-3(0)	7	9	-20	15	-29	-2	220	229
	7	7	-32	5	-29	-10	-	169
9247	10	10	-123	-12	-87	-19	326	375
A-4(8)	12	3	-	-	-	-	587	-
9248	4	66	-40	0	200+	200+	225	-
A-1-b(0)	4	40	-51	-9	-53	3	-	-
	6	4	-15	-5	-46	-14	334	494
	6	7	-5	5	-7	-1	310	491
9261	3	46	-200	-35	190	127	466	358
A-4(2)	3		-200	-20	107	132	-	-
	5	11	-197	-56	-252	-104	592	-
9262	7	16	-35	-15	-51	-21	116	338
A-1-b(0)	9	4	-30	15	-35	-14	188	463
9263	2	31	-70	-18	211	47	242	80
A-2-4(0)	2	30	-65	5	207	29	-	64
	3	10	-70	-12	-94	-55	393	459
	3	6	-78	-12	-111	-63	-	527
9268	4	20	-45	5	-34	-5	340	365
A-2-4(0)	4	17	-30	5	-44	1	-	384
	5	9	-40	3	-35	3	455	531
	5	10	-45	6	-22	-2	-	427
9269	3	-	-31	5	1	14	-	212
A-2-4(0)	5	13	-18	-2	-47	-11	288	450
	6	8	-20	7	-14	19	370	443
	6	-	-35	7	-30	5	-	421
9270	3	18	-46	18	101	67	268	197
A-2-4(0)	4	15	-25	40	-5	35	412	437
	4	-	-15	15	-6	30	412	425
9271	2	-	-66	-29	129	33	260	140
A-2-4(0)	3.5	17	-35	3	-48	-7	-	338
	3.5	16	-45	-5	-33	9	-	-
	5	10	-32	15	-35	6	410	466
	5	-	-41	0	-36	-1	410	427

TABLE 4

RESULTS OF FREEZE-THAW TESTS (Continued)

Soil No. & AASHO Class.	Cement Content, per cent by wt.	Weight Loss, per cent	Length Changes 0.0001 Inches 1st Freeze	1st Thaw	12th Freeze	12th Thaw	Compressive Strength, psi 7 Days	After Test
9282	6	17	-114	-31	-106	-46	202	312
A-4(8)	8	14	-81	-23	-78	-39	260	411
	8	-	-99	-29	-78	-47	-	420
	10	12	-70	-15	-74	-33	330	460
	12	6	-	-	-	-	395	-
9283	9	100	-155	-40	200+	200+	353	258
A-4(8)	9	100	-115	-18	200+	200+	-	268
	11	100	-139	-42	189+	47+	425	-
	13	40	-131	-28	157	146	484	-
9287	3	12	-37	-5	-27	-4	393	525
A-1-a(0)	5	3	-18	-8	-28	-3	538	995
	5	3	-10	2	-34	-7	-	935
9294	4	15	-52	-4	-20	1	355	350
A-2-6(1)	6	10	-55	-10	-14	20	393	429
	8	5	-47	-8	-26	13	528	481
9295	6	76	-40	-10	200+	200+	194	-
A-2-4(0)	6	34	-40	-10	200+	200+	-	-
	8	11	-48	-1	-60	-24	223	468
	10	6	-48	-1	-60	-24	-	495
9296	4	44	-30	-3	35	31	246	77
A-1-b(0)	4	100	-38	-4	27	28	-	315
	6	3	-34	-2	-64	-30	394	780
9307	7	12	-23	2	-21	-14	194	318
A-3(0)	9	10	-25	-2	-22	-1	-	-
	9	10	-25	-2	-12	-6	250	275
9312	8	6	-58	25	-90	24	364	510
A-4(7)	8	5	-108	-17	-97	-27	-	540
9319	3	43	-57	-5	35	15	251	209
A-2-4(0)	5	4	-45	-5	-30	-12	406	595
	5	4	-45	-5	-26	7	-	604

TABLE 5

RESULTS OF ACCELERATED FREEZE-THAW TESTS

Weight losses, Length changes and Compressive strengths

Reference No.	Soil No.	Cement content used in actual test procedure	2 cycle	6 cycle	10 cycle	12 cycle	Compressive strength % of 7 - day strength				Loss in weight after 10 cycle % of original wt.
							6 or	8 cycle	12 or	16 cycle	
1	9246	7.0	- 2	8	43	80	150			127	10
		8.0	5	2	11	14			163		
2	9247	10.0	- 9	19	63	80	50			35	7
		11.0	5	6	106	400		38	45		27
3	9248	6.0	- 5	23	48	58		98		90	10
		8.0	- 9	-10	-10	- 6		145	150		1
4	9261	5.0	-14	40	160	250		47		35	22
		6.0	-30	-32	0	-10		62		62	1
5	9262	7.0	-10	-11	-12	- 2		124		168	14
		9.0	-14	- 8	- 8	- 8	164			144	1
6	9263	2.0	-13	1	17	28		68		64	-
		3.0	- 4	10	15	30				81	-
7	9268	4.0	3	4	10	17		59	72		7
		5.0	- 2	7	10	0		65	68		3
8	9269	4.5	31	80	122	360		56	70		21
		5.0	- 5	1	5	5					6
9	9270	4.0	22	23	34	70	50			72	8
		5.0	0	5	16	14			65		2
10	9271	3.5	- 7	- 5	- 3	5		80		78	5
		5.0	6	5	18	28	70			88	5
11	9282	8.0	- 9	-14	Failed	-		117		100	4
		10.0	- 4	-16	-13	- 5		126		129	1
12	9283	9.0	79	150	Failed	-		57	5		100
		11.0	-24	100	Failed	-		-	25		100
13	9287	3.0	- 6	- 2	3	33			100		2
		5.0	- 7	- 5	3	8		126		108	3
14	9295	6.0	- 4	- 4	33	44		82		28	6
		7.0	-13	-13	4	21		105		115	0
15	9294	6.0	1	13	50	95		66	37		6
		7.0	0	5	18	40		137	130		0
16	9296	4.0	- 4	5	50	100		84	80		16
		5.0	-15	-13	5	21		100	125		2
17	9297	5.0	-	6	41	35	65		60		13
		7.0	1	3	4	5	-		140		-
18	9307	7.0	- 3	- 4	0	0		-	125		-
		8.0	- 3	- 2	3	10		100	130		5
19	9319	3.0	- 9	-13	27	30	73			59	42
		5.0	- 4	-10	- 7	- 5	84			116	4
20	9309	3.0	-	-	-	-	58		62		2
		5.0	- 2	5	9	17	-		125		-

Determination of Cement Content of Soil-Cement Mixtures

W. E. CURTIS, Bernard, Curtis, Hoggan, Engineering and Testing, Ltd., and
A. J. FORBES, Associated Engineering Services, Ltd., Edmonton, Alberta

Rapid and accurate determination of the cement content of soil-cement mixtures and cores becomes increasingly important with advanced construction techniques and improved designs. This paper covers an investigation and evaluation of six methods of test currently in use by various agencies. These methods are: (a) ASTM Designation D806 - 57; (b) Versene method; (c) flame photometer method; (d) California method; (e) conductivity method; and (f) chemical analysis method.

The results indicate that all the test methods investigated could be utilized but that certain methods are more desirable than others for particular conditions. This investigation indicates that either the California or the conductivity method of test would be the most practical for field use and that the ASTM, Versene or the California method would be suitable for most laboratory requirements. (Cement contents throughout this paper are expressed as percent weight of dry soil.)

•WITH increasing use of soil-cement in Canada, many agencies have realized the need for a fast and accurate method of determining the cement content of the mix. In 1960, the Alberta Department of Highways began a laboratory investigation to determine the most suitable method of cement content determination for their particular needs. To correlate field data with laboratory design, it was necessary to determine the percentage of cement present in field cores.

The investigation was carried out on six different methods currently being used to determine the cement content of soil-cement mixtures.

ASTM PROCEDURE

In the ASTM procedure (Designation D806 - 57), chemical analysis is employed to determine the cement content of samples of soil-cement mixtures either during or after construction. Samples of the raw soil, cement, and soil-cement mixture must be obtained and analyzed. Hydrochloric acid is added to each sample and the resulting precipitate discarded. The remaining filtrate is made slightly ammonaical and the hydroxides are allowed to settle out. The precipitate is washed with ammonium nitrate and the hydroxides are dissolved with hydrochloric acid. The solution is again made slightly ammonaical, the precipitate washed with ammonium nitrate and the hydroxide precipitate discarded. Ammonium hydroxide is added to the filtrate, the solution is heated and ammonium oxylate is added. The calcium oxylate is filtered off, washed into a beaker, diluted, and sulfuric acid then added. This is then titrated with a standard potassium permanganate solution.

The above procedure is carried out for the three samples (raw soil, cement and soil-cement mixture) and the cement content then computed by means of the following formula:

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

$$\text{Cement content } (\%) = \frac{J - I}{5H - I} \times 100$$

in which

H = potassium permanganate ($KMnO_4$) solution required for titration of cement sample, ml;
I = $KMnO_4$ solution required for titration of the raw soil, ml; and
J = $KMnO_4$ solution required for titration of the soil-cement, ml.

VERSENE METHOD

The Versene method is recommended for all soils except those containing a large or variable amount of calcium. It is recommended by the British Road Research Laboratory for most purposes, including field tests, and results can be obtained in appreciably less time than with the ASTM method. This method can also be used for determination of the lime content of soil-lime mixtures.

The cement content is estimated by measuring the combined calcium and magnesium contents of the natural soil, the stabilized soil, and the cement. Titration is performed with the disodium salt of ethylene-diamine tetracetic acid (EDTA). Filtrations are eliminated but since the soil, cement and soil-cement mixture are analyzed separately, the errors introduced are largely canceled out.

Hydrochloric acid is added to the samples to dissolve compounds of calcium, magnesium, iron, aluminum, etc., and then ammonium hydroxide is added to an aliquot portion until red litmus turns blue. The hydroxides of iron, aluminum, and titanium are precipitated out, if present, while the calcium and magnesium stay in solution. An aliquot portion containing the calcium and magnesium in solution is then titrated with EDTA using eriochrome blue-black as an indicator until all traces of pink have disappeared.

FLAME PHOTOMETER METHOD

The flame photometer method is preferable to the two previous ones where a large number of determinations are required on the same soil sample and time is an important factor. The equipment necessary for the test is, however, fairly expensive and a supply of gas, electricity, and compressed air is required.

The method involves initial calibration of the photometer followed by spraying a portion of the processed soil-cement solution through the photometer.

CALIFORNIA METHOD

The California method for finding the cement content of freshly mixed soil-cement is based on chemical titration methods which relate the cement concentration of samples to known solution concentrations. Two types of titrations are performed: an acid-base titration and a constant neutralization method. The acid-base titration is used when the aggregates do not react with hydrochloric acid and the constant neutralization method is used when there is a reaction.

The acid-base test is based on the neutralization of the cement with an excess of hydrochloric acid. The residual excess acid, as measured by back titration with an alkali, is inversely proportional to the cement content of the soil-cement mixture.

The constant neutralization procedure is based on the continuous neutralization of an aqueous solution from the cement-treated aggregate specimen for a specified period of time. This is accomplished by adding just sufficient acid to neutralize the OH ion that is continuously being liberated during hydration of the cement. The amount of acid used is directly proportional to the cement content of the soil-cement mixture.

CONDUCTIVITY METHOD

The conductivity method (Washington State Highway Commission) is based on the change of conductivity of water with the addition of cement. Suitable calibration curves are first obtained by measuring the conductivity of diluted mixtures containing a known percentage of cement. The cement content is then determined by measuring the conductivity of the diluted sample and referring to the standard calibration curves.

The method of test is quite inexpensive and may be used in the field as well as in the laboratory, but there are several aspects of procedure that must be carefully controlled to insure accurate results. Some of the factors which may or may not have an appreciable effect on results are as follows:

1. Use of field water supply instead of distilled water;
2. Type of cement or aggregate;
3. Sample size and grading;
4. Length of tempering time (4);
5. Length of dilution time;
6. Amount of dilution;
7. Effect of agitation; and
8. Temperature.

CHEMICAL ANALYSIS METHOD

The chemical analysis method is essentially the same as the ASTM procedures except for the following points. The iron and aluminum are not precipitated but just the calcium in the form of the oxylate. The hydrochloric acid is neutralized with ammonium hydroxide and acetic acid added to decrease the pH from 7 and thus leave the iron and aluminum in solution. An excess of ammonium oxylate is then added to the calcium to give a precipitate of calcium oxylate. The procedure then continues as for the ASTM procedure.

TEST PROGRAM

Work on the test program was carried out from 1958 to 1961 and comprised the following phases:

1. Comparison of the ASTM and Versene methods of test on laboratory samples of known cement content (summer and fall 1958);
2. Comparison of results obtained by ASTM and Versene methods on field specimens consisting of both formed specimens and cores of soil-cement;
3. Comparison of results obtained by experienced and inexperienced technicians using the Versene test method (1959);
4. Comparison of results using ASTM, Versene, chemical analysis and conductivity methods of test (conductivity tests performed by Alberta Research Council);
5. Comparison of results using California method and chemical analysis method on a prepared sample of known cement content; and
6. Comparison of the six methods of cement content determination to evaluate suitability for the particular requirements.

RESULTS AND CONCLUSIONS

Table 1 gives a comparison between cement contents from the field and those obtained by the Versene method in laboratory test. The cement content from the field was an average value for the day, and the cement content of samples obtained for the laboratory tests could have varied appreciably from this average. The laboratory result was considerably lower than the field result in most cases except for the Updike Lake material where the field control was difficult to obtain and results may not be completely reliable. The soil-cement samples tested consisted of a fine sand of the dune sand type for the Reaume, Elhardt and Ravenshaw pits, a coarse well-graded sand for the Sheppert pit and a crushed sandstone for the Updike Lake pit.

The results of a comparison of the ASTM and the Versene methods (Table 2) show that both methods of test give sufficiently accurate results for either field or laboratory usage. The Versene method appears to give slightly higher values than the ASTM

TABLE 1

COMPARISON OF FIELD ESTIMATED CEMENT CONTENT AND LABORATORY CEMENT CONTENT (Versene Method), 1959

Pit Name	Sample No.	Lab. Cement (%)	Field Cement (%)
Reaume[a]	44102	9.3	10.7
	44106	11.1	10.8
	44108	9.7	13.3
	44104	11.1	—
	44254	10.8	11.0
	44259	10.4	11.2
Updike Lake[b]	44134	12.8	10.9
	44138	14.6	13.4
	44142	21.1	13.2
	44144	19.2	13.2
	44146	12.5	17.0
	44152	13.8	12.0
	44116	9.6	6.0
	44118	15.8	10.5
	44126	15.8	11.0
	44128	10.4	12.1
	44130	12.8	8.4
Elhardt[c]	43465	4.9	8.0
	43467	7.1	8.0
	43832	5.3	8.0
Ravenshaw[d]	43732	7.3	9.0
	43734	6.8	9.0
	43736	8.1	9.0
	43738	6.4	9.0
	43740	8.6	9.0
	43742	6.2	9.0
	43744	6.6	9.0
	43746	6.2	9.0
	43748	10.5	9.0
	43750	12.4	9.0
	43839	10.0	9.0
	43843	10.4	9.0
	43845	6.3	9.0
Sheppert[e]	44572	7.9	10.5
	44578	4.0	9.0
	44582	3.8	6.8
	44584	6.6	6.7
	44586	5.8	7.0
	44590	5.3	7.0
	44594	7.1	7.0

[a] Project 2-K-2
[b] Project 2-K-2
[c] Project 12-B-1
[d] Project 12-B-1
[e] Project 28-B-2

TABLE 2

RESULTS OBTAINED FROM THE ASTM AND VERSENE METHODS, 1959

Sample	Cement Content (%)		
	Standard	ASTM Method	Versene Method
1	6[a]	6.0	5.8
2	8[a]	7.9	8.0
3	10[a]	9.9	10.0
41166	9.0[b]	9.2	9.7
41186	9.0[b]	10.4	10.7
41200	9.0[b]	9.4	10.6
41196	9.0[b]	10.0	11.3
41575	9.0[b]	5.5	6.3

[a] Known.
[b] Unknown (approximate).

method which may be due in part to small quantities of aluminum or iron being present which are taken as calcium in the Versene determination. It may also be due to a poor end point obtained in the titration process.

The comparison of results obtained with experienced and inexperienced personnel using the Versene method of test (Table 3) definitely indicated the need for experienced personnel. A good knowledge of quantitative chemistry plus knowledge of the physical and chemical properties of cement must be combined with good laboratory technique to insure accurate results.

Table 4 compares results using the ASTM, Versene, chemical analysis and conductivity methods. On the basis of the limited data available, the Versene and conductivity methods appear to give the best average value of test. The ASTM method appears to give a slightly low value of test and the chemical analysis method a slightly high value. However, it is felt that considerably more investigation and test data would be required to substantiate or disprove these findings.

Table 5 indicates that the California method gives excellent results when checked against a known admixture content, whereas the chemical analysis method again indicates results slightly on the high end of the scale. However, test data to verify this indication are very limited.

TABLE 3

RESULTS OBTAINED WITH EXPERIENCED AND INEXPERIENCED TECHNICIANS (Versene Method), 1959

Sample No.	Cement Content (%)	
	Experienced	Inexperienced
1	8.7	8.5
2	9.5	9.6
53797	7.6	14.8 13.0 11.0 10.8
53801	5.6	12.2 10.4 9.0 8.8
53802		10.0 8.3
53804	8.2	8.1 8.3 7.1 6.9
53805	7.5	15.1 13.9 11.9 11.7
54521	6.3	13.3 11.0 10.0 9.5
54522	8.7	11.5 12.3 10.7 10.3
54526	6.8	13.8 10.1 6.2 6.1
54528	6.4	13.7 13.2 11.9 10.8

TABLE 4

RESULTS USING VARIOUS METHODS FOR DETERMINING CEMENT CONTENT, 1960

Sample No.	Cement Content (%)			
	ASTM Method	Versene Method	Chemical Analysis	Conductivity Method
53797	6.6	7.6	8.3	
53801	4.4	5.6	6.7	
53804		8.2	8.6	
53805	6.4	7.5	8.1	
54521	7.1	6.3	7.5	6.5
54522	7.9	8.7	8.1	7.8
54526	6.2	6.8	8.4	7.4
54528	5.8	6.4	8.4	7.0
54523		11.0 (?)		7.4
54524		10.9 (?)		7.5
54525		11.5 (?)		8.3
54527		11.1 (?)		7.5
54529		9.2 (?)		7.3

TABLE 5

COMPARISON OF RESULTS FROM CHEMICAL ANALYSIS AND CALIFORNIA METHOD

Sample No.	Cement Content (%)		
	California Method	Chemical Analysis	Known Admixture Content (%)
1	5.0	6.9	5[a]
2	9.7	11.5	10[a]
3	11.0	12.7	10[b]

[a] Cement.
[b] Lime.

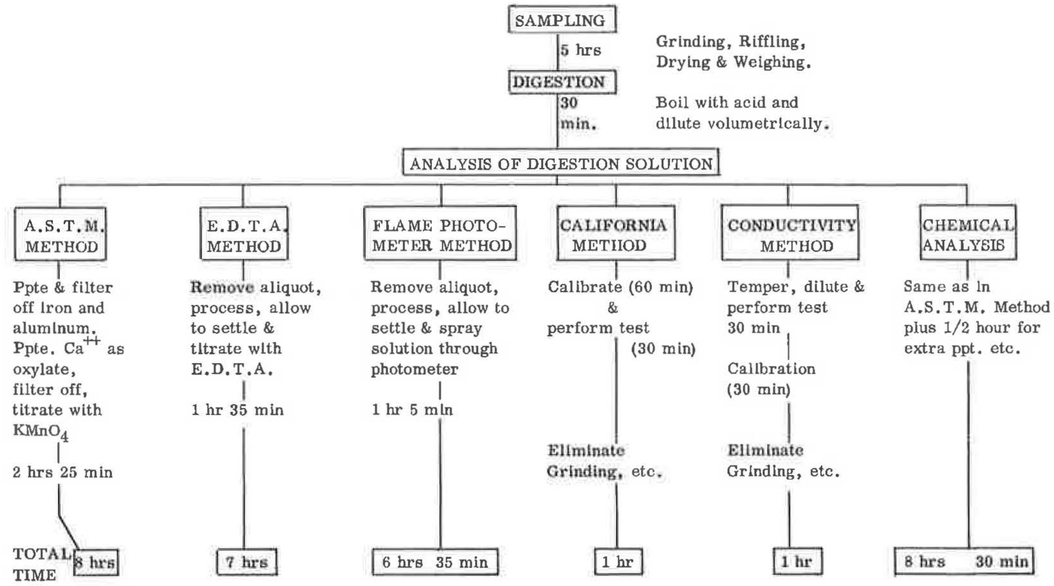


Figure 1. Time required for determination of cement content of soil-cement mixtures by six methods of analysis.

TABLE 6

COMPARISON OF VARIOUS METHODS OF CEMENT CONTENT DETERMINATION OF SOIL-CEMENT MIXTURES

Property	ASTM	Versene	Chemical Analysis	Conductivity	Flame Photometer	California
Speed	5	4	6	1	3	2
Accuracy	2	1	4	1	3	2
Initial cost of equipment	2	1	3	5	6	4
Training of technicians	5	4	6	1	3	2
Space required	3	3	3	1	2	2
Portability	4	3	5	1	6	2
Maintenance cost	5	4	6	1	3	2
Supervision required	4	3	5	1	5	2
Computations	5	4	6	1	3	2
Reproducibility of results	5	4	6	3	2	1
Technician's safety	5	3	6	1	2	4
Availability of utilities	3	4	5	1	6	2

Figure 1 compares the time required in the various methods of test to determine cement content of a soil-cement mixture. Information obtained during the local investigation is substantiated with that from reference material. The California and the conductivity methods require much less time to run inasmuch as the time of grinding, riffling and drying has been eliminated. The time required for the calibration of apparatus has not been included in these two methods but in any event they would take less than one-quarter the time of the other test methods. This time factor plus the indicated accuracy of these two test methods certainly makes them worthy of further investigation.

Table 6 was prepared to show the relative merits of each type of test and to serve as a guide in choosing a test method for a particular purpose. Preparation was to a large extent from noted reference material as personal experience has not been sufficient to permit accurate appraisal of all list methods. The following points are indicated:

1. After calibration of the equipment, the conductivity, flame photometer and California methods are appreciably faster than other methods of test;
2. The ASTM and Versene methods appear to be most accurate, while the chemical analysis method appears to give slightly high results; however, all methods appear to be sufficiently accurate for job control purposes; and
3. In practically all other ways noted, the conductivity and California methods of test appear to be most suitable for use.

The ASTM and Versene methods are rated low in this comparison on the basis of reproducibility of results because trained and experienced technicians are required. Comparisons in general are on the basis of tests carried out by relatively unskilled technicians, who might be found in the average testing laboratory.

In Table 6, number 1 in each case represents the most desirable method; number 6 represents what is considered to be the poorest. For example, the fastest method is rated as number 1; the slowest method as number 6.

Items which may have an important bearing on the general test results are listed below.

1. Relatively small samples are used in the ASTM, Versene and Chemical Analysis methods of test and segregation or poor sampling of materials could therefore have an appreciable effect on the test results.
2. The ASTM method required a double precipitation to be performed and it is considered that there is a possibility of losing calcium or having a portion of the ferrous iron and aluminum stay in the precipitate, which would give erroneous results. In cases where the soil contains an appreciable amount of these materials, the error could perhaps become significant. The Versene method of test reacts the same way and here the error could possibly be even greater.
3. In the Chemical Analysis method, the iron and aluminum is not precipitated but only the calcium in the form of the oxylate. The hydrochloric acid is carefully neutralized with ammonium hydroxide and the pH decreased from 7 by the addition of acetic acid to leave the iron and aluminum in solution. An excess of ammonium oxylate is then added to the calcium to give a precipitate of calcium oxylate. The procedure from this point is identical to the ASTM method.
4. The ASTM, Versene and Chemical Analysis methods of test require test equipment such as an analytical balance, electric oven, dessicator and glassware and are therefore relatively expensive tests to set up in the field. In contrast, the California method uses plastic ware which is an advantage.
5. The Flame Photometer method requires experienced, well trained technicians and the equipment has a high initial cost.
6. The end point in titration is much more distinct for the Chemical Analysis than for the ASTM or Versene methods. This could possibly be improved in the California method by titrating 25 ml of solution rather than 100 ml.
7. The Conductivity method could possibly require frequent re-calibration of the equipment as slight vibration or jarring of the equipment may give sufficient change to the conductivity cell to alter results. In addition, the technician must know the electric circuits involved and have a knowledge of chemistry.

On the basis of the above, it would appear that, of the methods investigated, the California or the Conductivity method would be the most practical for field use. In the main laboratory, the ASTM method, the Versene method or the California method would probably be most suitable. These test methods also permit determination of the lime content of soil-lime mixtures and can be used for hydrated or unhydrated soil-cement.

ACKNOWLEDGMENTS

The authors would like to express their appreciation to Messrs. Shields and Ricks of the Alberta Research Council for their kind cooperation and advice on this program. Also to Mr. Cronkhite, Chief Construction Engineer, and other members of the Alberta Department of Highways for making information in this paper available, and finally to staff members of the Highways Testing Laboratory for their careful work and interest in the program.

Appreciation is also expressed to authors of reference material from which sources information has been freely drawn.

REFERENCES

1. "Soil-Cement Laboratory Handbook." Portland Cement Association.
2. State of California: Materials Testing Manuel Test Method No. 338A.
3. "California Highways and Public Works." (Nov. - Dec. 1959).
4. LeClerc, R. V., and Sandahl, H. E., "A Rapid Method for Determining Cement Content of Plastic Cement-Treated Base." HRB Bull. 267, 1-13 (1960).
5. Sherwood, P. T., "Laboratory Method for the Determination of the Cement or Lime Content of Cement of Lime Stabilized Soil."
6. Sherwood, P. T., "The Determination of the Cement Content of Soil-Cement."
7. Gilliland and Hunter, "Rapid Method for Estimating Cement Content of Soil-Cement and Blended Cement."
8. "The Laboratory." Fisher Scientific Co., Ltd.
9. American Society for Testing Materials: Method D806 - 57.

Appendix

PROCEDURE[1] FOR HIGHWAYS TESTING LABORATORY, EDMONTON, CANADA

Test Method

1. Weight out, on the analytical balance the following amounts of the samples:

Raw soil, 5, 000 g
Soil-cement mixture, 5, 000 g
Cement, 1, 000 grams[2]

Place the materials in a 250-ml beaker.

---

[1] Adapted from ASTM Procedure D806 - 57.

[2] Exact weights of 5,000 g for the samples of soil and soil-cement and 1,000 g for the sample of cement do not have to be used. This will save time in weighing. If exact weights are not used then the following corrections must be made:

$$1^1 = \frac{I(5)}{\text{Wt. Used}} \qquad J^1 = \frac{J(5)}{\text{Wt. Used}} \qquad H^1 = \frac{H(1)}{\text{Wt. Used}} \qquad \%\ \text{Cement} = \frac{(J^1 - I^1)}{5H^1 - I^1} \times 100$$

2. (a) Add 50-ml HCL (1:1) to the soil and soil-cement.
   (b) To the cement add 40-ml water. Stir into a paste. Then add 10-ml concentrated HCL (Sp. Gr. = 1.18).
3. Cover and boil gently for 5 minutes.[3]
4. Add 25-ml hot water. Stir, allow to settle through a Whatman No. 1 filter paper, preferably 15 cm in diameter. If the filtration period exceeds one hour, substitute a No. 41 paper in subsequent similar cases.
5. Receive the filtrate in a 250-ml volumetric flask.
6. Wash the residue once by decantation using hot water, then transfer it to the filter paper.
7. The beaker should be rapidly policed, the loosened material being transferred to the filter paper.
8. Wash the material on the filter 4 times with hot water (use about 10 to 15 ml each time).
9. Discard the filter.
10. Dilute the filtrate in the volumetric flask to 250 ml with cold water and mix the contents by agitating.
11. Using a 50-ml pipette remove a 50-ml aliquot into the original 250-ml beaker.
12. Dilute to about 100 ml.
13. Make the solution slightly ammoniacal by adding NH_4OH. (Put a small piece of red litmus paper into the beaker; stop adding NH_4OH after the litmus paper has turned blue.)
14. Boil 1 to 2 minutes and then allow the hydroxide to settle.
15. Filter through an 11-cm Whatman No. 1 filter paper.
16. Receive the filtrate in a 600-ml beaker.
17. Wash the original 250-ml beaker into the filter with hot NH_4NO_3 (20 g per l) once. Follow by washing the hydroxide precipitate once or twice with hot NH_4NO_3(20 g per l).
18. Set the filtrate aside (in the 600-ml beaker).
19. Transfer the paper and precipitate to the original beaker. Add 20-ml hot HCL (1:3) and then add 50-ml water.
20. Make the solution slightly ammoniacal (as in 13) and boil 1 to 2 minutes.
21. Allow the precipitate to settle.
22. Decant through a Whatman No. 1 filter paper, receiving the filtrate in the 600-ml beaker containing the first filtrate (previously set aside).
23. Wash and police the 250-ml beaker in which the precipitation took place.
24. Wash the precipitate on the filter paper 3 or 4 times with NH_4NO_3 (20 g per l).
25. Discard the hydroxide precipitate (on the filter paper).
26. Add 2-ml concentrated NH_4OH (Sp. Gr. 0.90) to the filtrate (which will now have a volume of 250 to 350 ml).
27. Heat the solution to boiling.
28. Add 10-ml hot saturated $(NH_4)_2C_2O_4$ (ammonium oxalate) solution.
29. Keep the mixture near boiling until the precipitate becomes granular.
30. Set aside the beaker on a warm hot plate for 30 minutes or more.
31. Before filtering off the CaC_2O_4 (calcium oxalate) precipitate, verify completeness of precipitation by adding a little more $(NH_4)_2C_2O_4$. If the solution remains clear then precipitation is complete. Make sure that a slight excess of NH_4OH is present. (Dip a stirring rod into the solution and put a drop of it on some red litmus paper, which will turn blue if there is an excess of NH_4OH.)
32. Filter through an 11- or 15-cm Whatman No. 2 filter paper, making sure that all the precipitate is being retained.
33. With a rubber policeman clean the 600-ml beaker in which precipitation took place and transfer the contents to the filter with a stream of hot water.
34. Wash the precipitate on the filter 6 to 10 times with a stream of hot water (not over 75 ml).

---

[3] Vigorous or extended boiling of samples is seldom necessary and often results in much slower filtration.

35. Carefully open the filter paper and wash the precipitate with water into the 600-ml beaker (where precipitation was effected). Save the filter paper.
36 Dilute to 200 ml.
37. Add 10-ml H_2SO_4 (1:1).
38. Heat the solution short of boiling and titrate it with $KMnO_4$ solution to a persistent pink color. (The $KMnO_4$ solution need not be a standard solution, but the same solution shall be used in titrating all the components.)
39. Add the filter paper and macerate it.
40. Continue the titration slowly until the pink color persists for 10 seconds.
41. Record the volume of $KMnO_4$ used.

Calculation:

$$\% \text{ Cement} = \frac{(J - I)}{5H - I} \times 100$$

in which

H = $KMnO_4$ solution required for titration of the sample of cement, ml;
I = $KMnO_4$ solution required for titration of the sample of raw soil, ml; and
J = $KMnO_4$ solution required for titration of the sample of soil-cement mixture ml.

Relationship Between Cement Content And Freeze-Thaw Loss of Soil-Cement Mixtures

L. J. CIRCEO, D. T. DAVIDSON, and H. T. DAVID, respectively, Captain, Corps of Engineers, U. S. Army; Late Professor of Civil Engineering, and Associate Professor of Statistics, Iowa State University

Although the freeze-thaw test was originally devised to measure the hardening effect of portland cement on soils, it has become useful as a reliable criterion for the determination of the durability of a soil-cement mixture. One great disadvantage of this test is the large amount of labor necessary to complete the freeze-thaw test. This paper attempts, by correlation analysis, to reduce the amount of testing required to conduct a reliable freeze-thaw evaluation of a soil-cement mixture.

A strong logarithmic relationship was found to exist between the cement content and the freeze-thaw loss of a soil-cement mixture. Two freeze-thaw tests will establish the logarithmic relationship for any soil type. From this relationship the cement content that will produce a satisfactory freeze-thaw loss could be accurately determined.

An analysis of the logarithmic relationships for all soils used in this investigation indicated a linear relationship between the slopes and intercepts of the relationships. Theoretically this would indicate a common point through which all relationships would pass. This was found applicable for A-1 through A-4 soils. For these soils one freeze-thaw test is sufficient to establish the logarithmic relationship and thereby determine the cement content required to produce a durable soil-cement mixture.

The slope of the logarithmic relationship was found to be independent of the number of cycles used to conduct the freeze-thaw test. A logarithmic relationship at a reduced number of cycles could be indicative of the possibility of reducing the number of cycles needed to conduct the freeze-thaw test with no loss in the significance of the test.

By the use of these relationships the labor involved in the conduct of the freeze-thaw test can be reduced. The accurate determination of the cement content required to produce a specified freeze-thaw loss will result in more economical mix design of soil-cement mixtures.

• THE cement content required to stabilize a soil adequately will usually determine the economic feasibility of cement stabilization. A soil-cement mixture can be considered to be adequately stabilized if it meets specified design criteria of strength and durability. There are several shortcut methods used to evaluate the required cement content; however, for major projects a complete series of detailed tests is

---

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

usually necessary. These tests include compressive strength evaluation, wet-dry and freeze-thaw tests.

Compressive strength tests are usually supplementary to the wet-dry and freeze-thaw tests. An adequately hardened soil-cement mixture will increase in compressive strength with time of curing. Generally an increasing unconfined compressive strength of 300 psi or more at 7 days will pass the wet-dry and freeze-thaw tests satisfactorily (3).

The wet-dry and freeze-thaw tests are usually considered indicative of the durability of soil-cement mixtures. The wet-dry test produces high shrinkage stresses; the freeze-thaw test produces high expansive stresses. These tests were developed to introduce destructive forces which a soil alone could not withstand, but which a structural material would resist. Thus, they are more valuable in analyzing a soil-cement mixture as a structural material rather than as a criterion of durability. The adequacy of a soil-cement mixture as a structural material would also confirm its ability to withstand weathering (1).

The freeze-thaw test is generally the critical test in determining the required cement content except for mixtures containing relatively large amounts of silt and clay. For other mixtures than these, it is standard practice to mold only one wet-dry specimen at the median cement content, while a freeze-thaw specimen for each cement content investigated is usually molded (3). Thus the freeze-thaw test is the major test in evaluation of a soil-cement mixture, requiring considerable time and labor to conduct.

The freeze-thaw test is, by its nature, very subjective. Individual brushing techniques can cause a wide variance in the results of the test. A relationship between the cement content and the freeze-thaw loss would serve to reduce the error which is inherent in the test.

CEMENT FREEZE-THAW LOSS RELATIONSHIP

An investigation to develop a relationship between the cement content and the freeze-thaw loss of a soil-cement mixture was first considered. An excellent logarithmic relationship was observed using 172 sets of data taken from data sheets of the Portland Cement Association. Typical relationships for the soil types studied are shown in Figure 1. The r^2 correlation coefficients of the logarithmic relationships were evaluated by the IBM 650 digital computer. All r^2 correlation coefficients are extremely high (Appendix). Thus it appears that the cement content by weight can be related, to within good approximation, to the freeze-thaw loss of a soil-cement mixture by a logarithmic relationship.

The cement content by volume was also investigated. Good logarithmic relationships were also obtained; however, the cement content on a weight basis resulted in the best relationships. Although A-1 and A-5 soils were not investigated, it is believed that these would also follow a logarithmic relationship.

The logarithmic relationship is

$$\log C = A + B \log L \tag{1}$$

in which

C = cement content,
L = freeze-thaw loss,
A = intercept, and
B = slope.

Using this relationship, the cement content which will give the maximum allowable freeze-thaw loss can be chosen. Normally, soil-cement mixtures with at least three cement contents are tested. A logarithmic plot may be constructed with these data. The relationship will reduce the error caused by any possible outliers in the data, reducing the possibility of arriving at erroneous conclusions.

Another advantage of this relationship is the reduction of the necessary number of cement contents to be tested. An experienced tester could conduct the freeze-thaw test

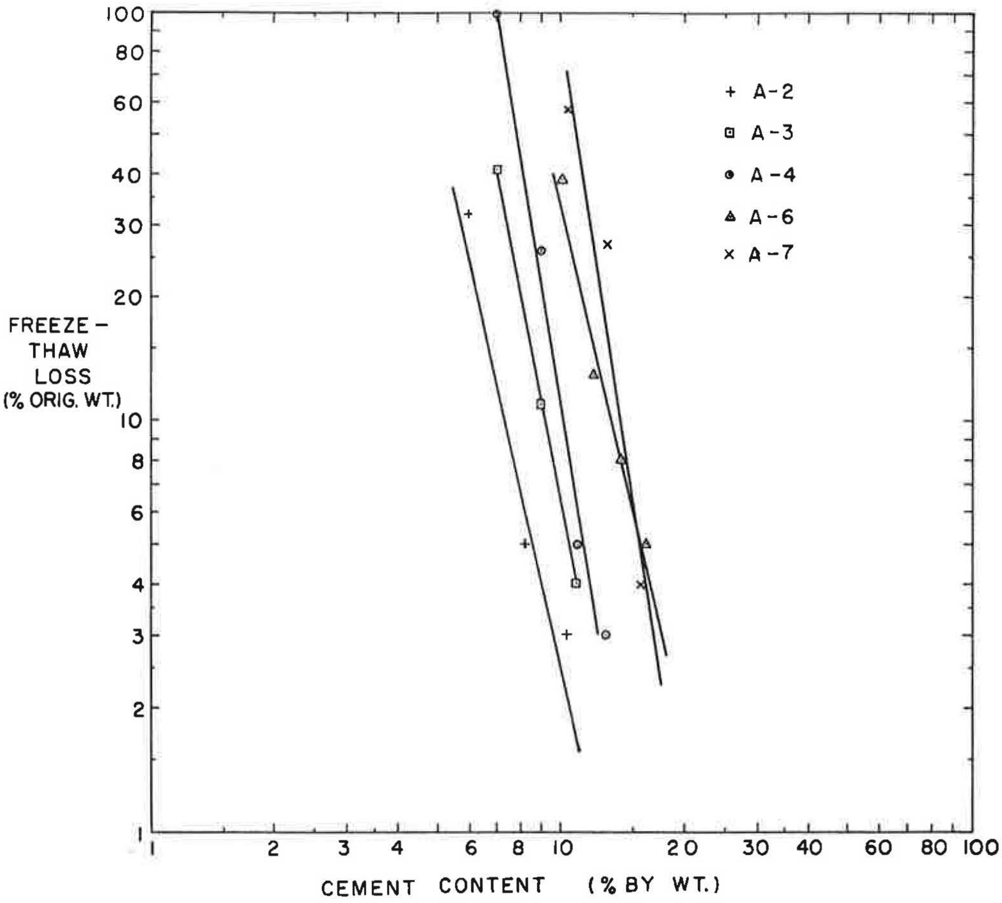


Figure 1. Relationship between the cement content and the freeze-thaw loss of soil-cement mixtures.

with 2 cement contents. A logarithmic plot connecting these points would establish the relationship. The cement content which will give the required freeze-thaw loss may then be determined. It is best to obtain data that will fall on either side of the specified freeze-thaw loss. An experienced tester should be able to choose cement contents which produce these results most of the time.

The ability to select the exact cement content to produce a specified freeze-thaw loss will result in more economical mix designs. Many soils have recommended cement contents which produce 2 percent freeze-thaw loss because the next lower cement content tested had a freeze-thaw loss greater than the allowable. The logarithmic relationship would indicate a cement content between these two. This intermediate cement content might be considered adequate for the particular soil-cement mixture.

All cement contents below the one that will produce 100 percent freeze-thaw loss will also indicate 100 percent loss. In the same manner, all cement contents greater than the one which will give little or no freeze-thaw loss will also produce no freeze-thaw loss. A relationship passing through 100 percent freeze-thaw loss and/or 0 percent loss might lead to erroneous conclusions. Therefore a relationship connecting points between these extremes is recommended.

SLOPE-INTERCEPT RELATIONSHIP

An analysis of the equations obtained for the cement freeze-thaw relationship was undertaken. A linear relationship was found to exist, approximately, between the slope (B) and the intercept (A) of the equations (Fig. 2). The granular soils tend to lie along the lower edge of the scatter diagram (i.e., parallel to the axis of scatter), with a transition to the fine-grained soils at the upper edge.

The line which best represents this scatter diagram can be represented by

$$A + 1.118\,B = 0.62 \tag{2}$$

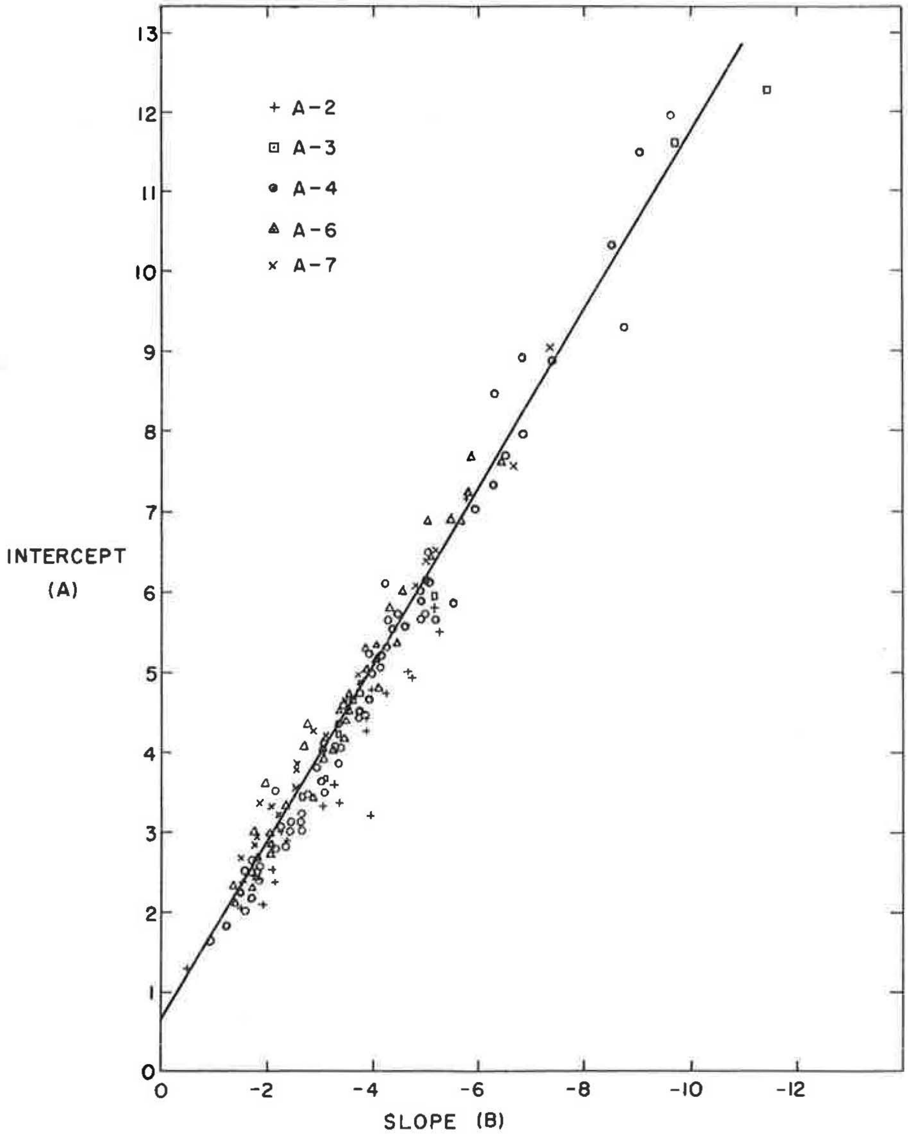


Figure 2. Relationship between slope and intercept of logarithmic cement-freeze-thaw relationships.

TABLE 1

THEORETICAL COMMON INTERSECTION OF LOGARITHMIC FREEZE-THAW RELATIONSHIPS BY SOIL TYPE

Soil Type	Common Intersection	
	Cement (%)	Freeze-Thaw Loss (%)
A-2	12.2	2.0
A-3	13.0	2.8
A-4	13.0	2.8
A-6	13.0	5.0
A-7	12.0	10.2

From this equation it can be seen that any point on the line (an individual cement freeze-thaw relationship) is equal to a constant; thus,

$$A_1 + 1.118\,B_1 = A_2 + 1.118\,B_2 = \dots = A_n + 1.118\,B_n \qquad (3)$$

Theoretically this would indicate that all logarithmic cement freeze-thaw relationships would pass through a common point. This point is at C = 1.118 and L = 0.62. On the logarithmic scale: C = 12.5 percent cement and L = 4.2 percent freeze-thaw loss.

In Table 1, the common intersection increases in the percent freeze-thaw loss as the clay content increases. A soil-cement mixture requiring a cement content greater than the cement coordinate of the common intersection would have to assume a positive slope, making the relationship invalid. Therefore, the common intersection concept cannot be used with these soils. A-6 and A-7 soils have a usual cement requirement in the range of 9 to 16 percent (3). For the most part this common intersection would not be applicable for these soils. Therefore A-6 and A-7 soils were eliminated from this investigation.

A-2, A-3, and A-4 soils were then investigated to determine if this common intersection was applicable to the data. Inasmuch as the common intersections for these soil types are similar, an average intersection of C = 12.6 and L = 2.4 was assumed. Graphical analysis of the data indicated that the relationships passed through, or in the vicinity of, the intersection (Fig. 3).

Thus, with a common point through which all relationships of A-2, A-3, and A-4 soils pass established, only one freeze-thaw test is necessary to establish the relationship and select the required cement content to produce a durable soil-cement mixture. It is believed that A-1 soils will follow a similar relationship.

Analysis indicated that certain limitations should be placed on the data to obtain best results. The cement content of the freeze-thaw specimen should be below 9 percent cement. The freeze-thaw loss should fall below 50 percent soil loss and above the maximum allowable soil loss.

A comparison was made between the actual and predicted cement contents required to produce a durable soil-cement mixture (Appendix). For this investigation 10 percent freeze-thaw loss was considered the maximum allowable loss for all soil types. The actual cement content was determined by the logarithmic relationship using all data points. The predicted cement content was determined by the line from the data point in each set that had the lowest cement content, subject to the limitations, passed through the common intersection. The predicted values compare well with the actual cement content. Most predicted values are greater than the actual values, placing the prediction on the safe side. The actual and predicted cement contents compare favorably with those recommended by the PCA (Appendix).

A freeze-thaw test conducted with one cement content, subject to the prescribed limitations, would be sufficient to evaluate the required cement content properly. For any random soil it might be difficult to choose a cement content which would fall within the limitations; however, in the case of a soil series or soils where the cement content is approximately known, a cement content can be selected and the method used advantageously. For example, this method could be used in conjunction with the "shortcut test procedures for sandy soils" for major projects in order to determine better the required cement content. Although A-6 and A-7 soils were not investigated, this method might also be applicable for these soils which require low cement contents.

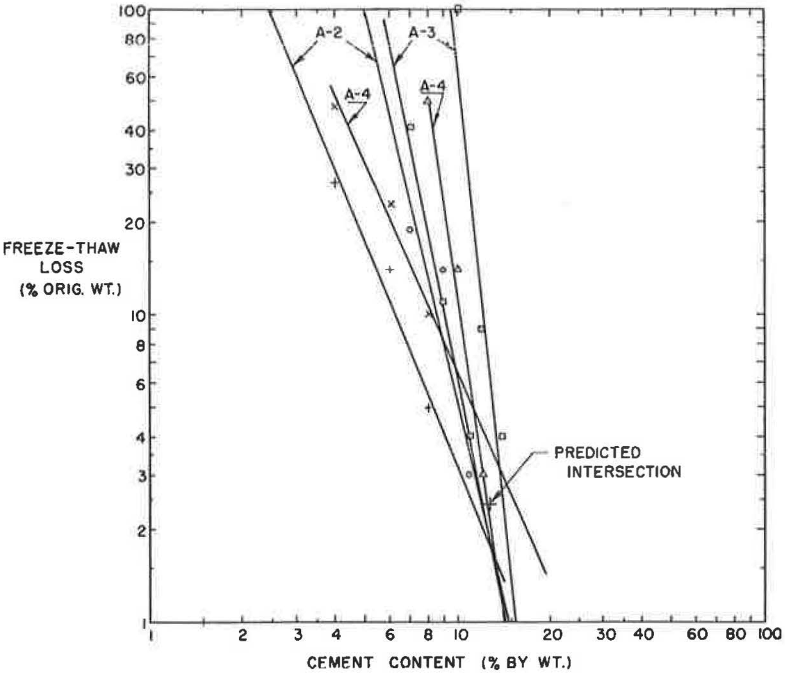


Figure 3. Ability of logarithmic cement freeze-thaw relationships of granular soil-cement mixtures to pass through the common intersection.

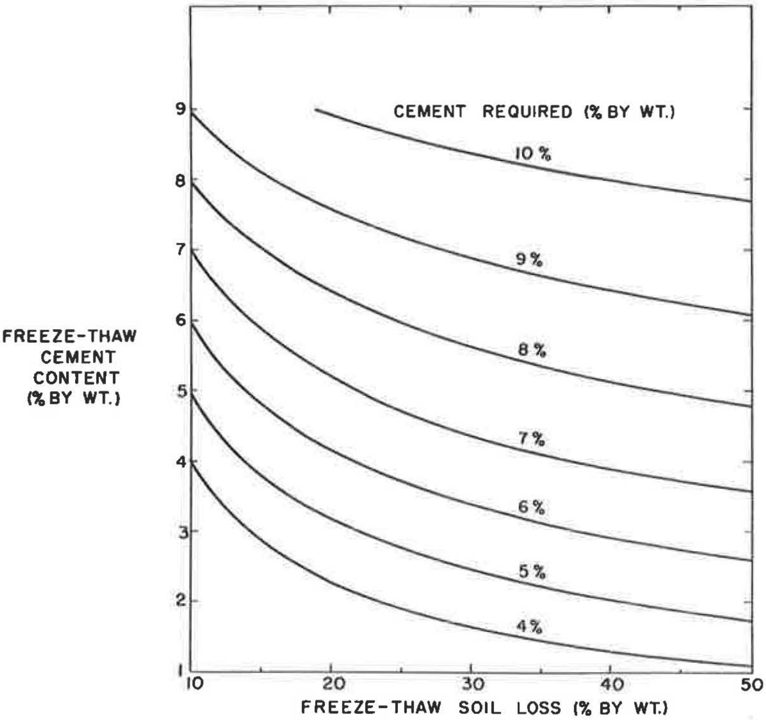


Figure 4. Recommended cement content of granular soil-cement mixtures by common intersection method.

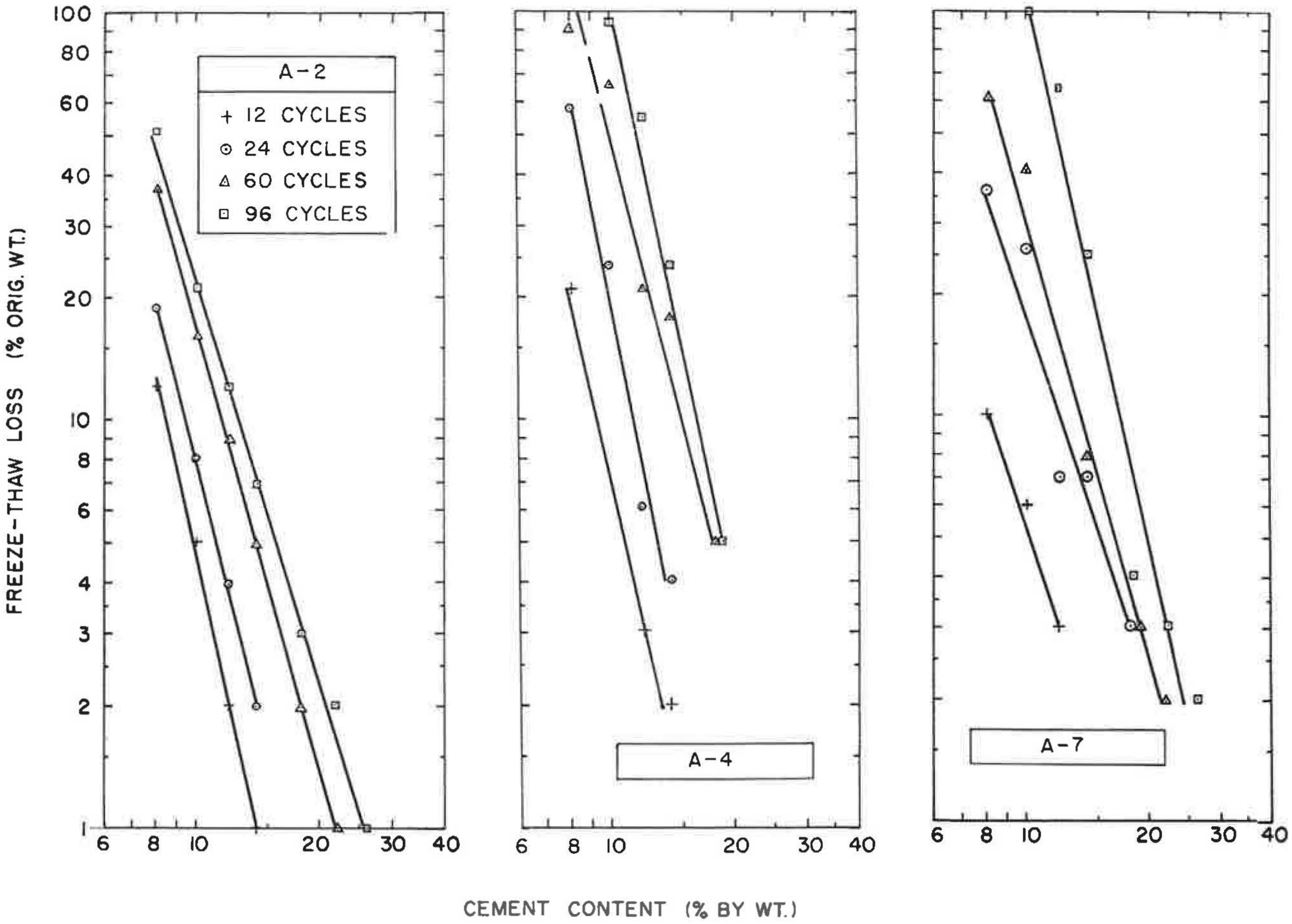


Figure 5. Effect of number of freeze-thaw cycles on logarithmic cement freeze-thaw relationship.

Figure 4 shows the selection of the required cement content with one freeze-thaw specimen at 10 percent freeze-thaw loss. Similar graphs can be constructed for other allowable losses. Safety factors are easily incorporated.

INFLUENCE OF NUMBER OF FREEZE-THAW CYCLES ON LOGARITHMIC RELATIONSHIP

An extensive investigation was undertaken by Felt (2) to observe the influence of cement content on freeze-thaw loss. Freeze-thaw tests from 12 to 96 cycles were conducted. These data were investigated to observe the influence of the number of freeze-thaw cycles on the logarithmic relationship.

The freeze-thaw test conducted at greater than 12 cycles will also produce an excellent cement freeze-thaw logarithmic relationship (Fig. 5). It seems that, as a first approximation, the relationships of each soil can be considered parallel, indicating that the slope of the logarithmic relationship is independent of the number of cycles used to conduct the freeze-thaw test.

The specifications for maximum allowable loss are 14 percent for A-2 soils, 10 percent for A-4 soils, and 7 percent for A-7 soils after 12 freeze-thaw cycles (3). It

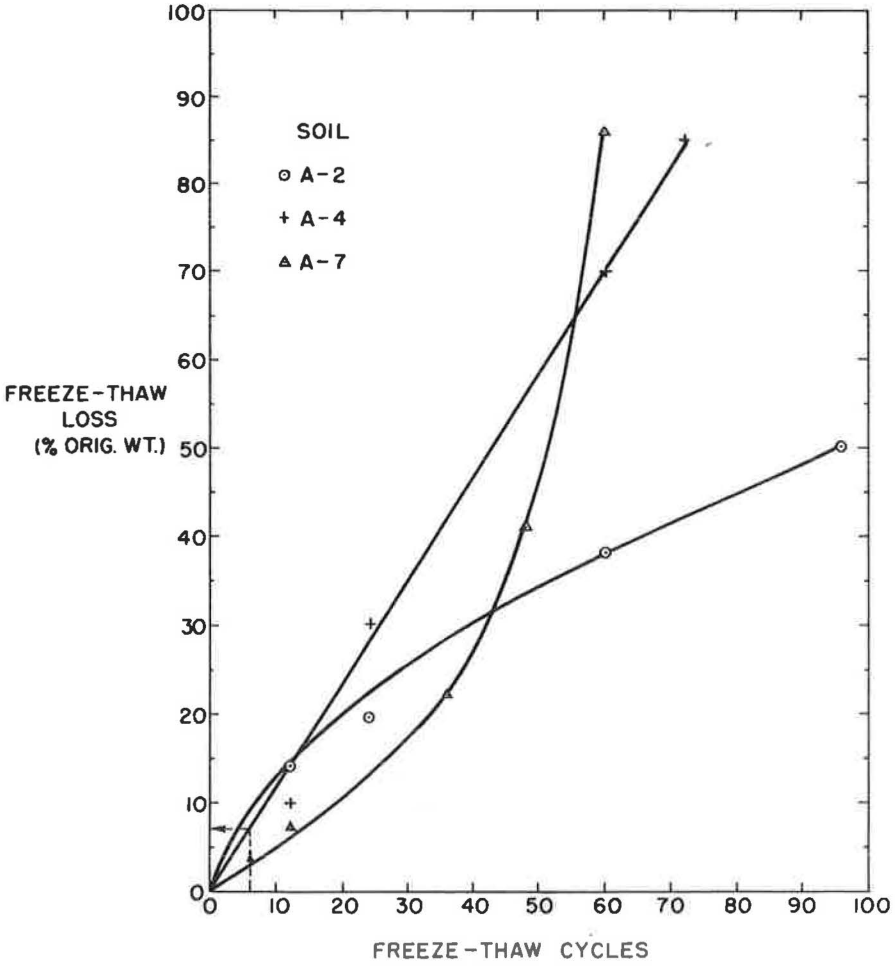
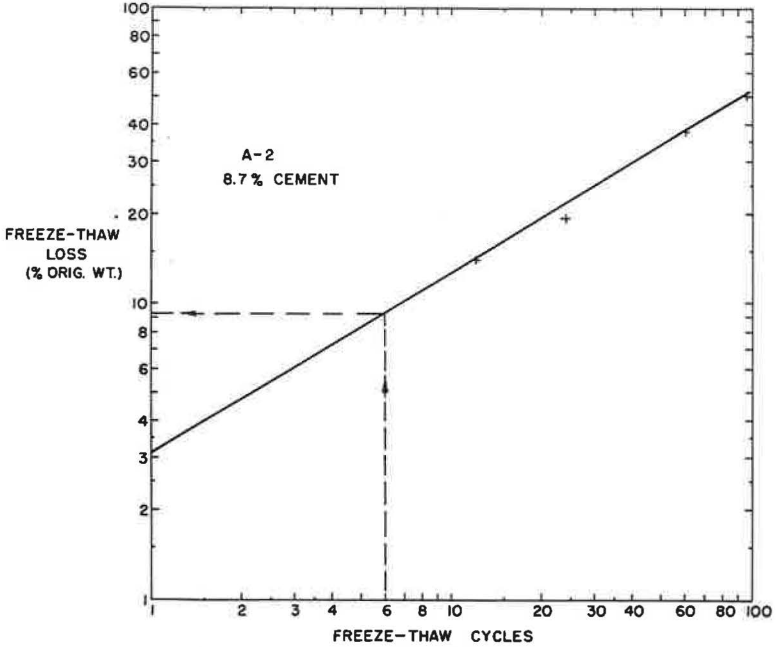


Figure 6. Effect of number of freeze-thaw cycles on maximum allowable freeze-thaw loss.



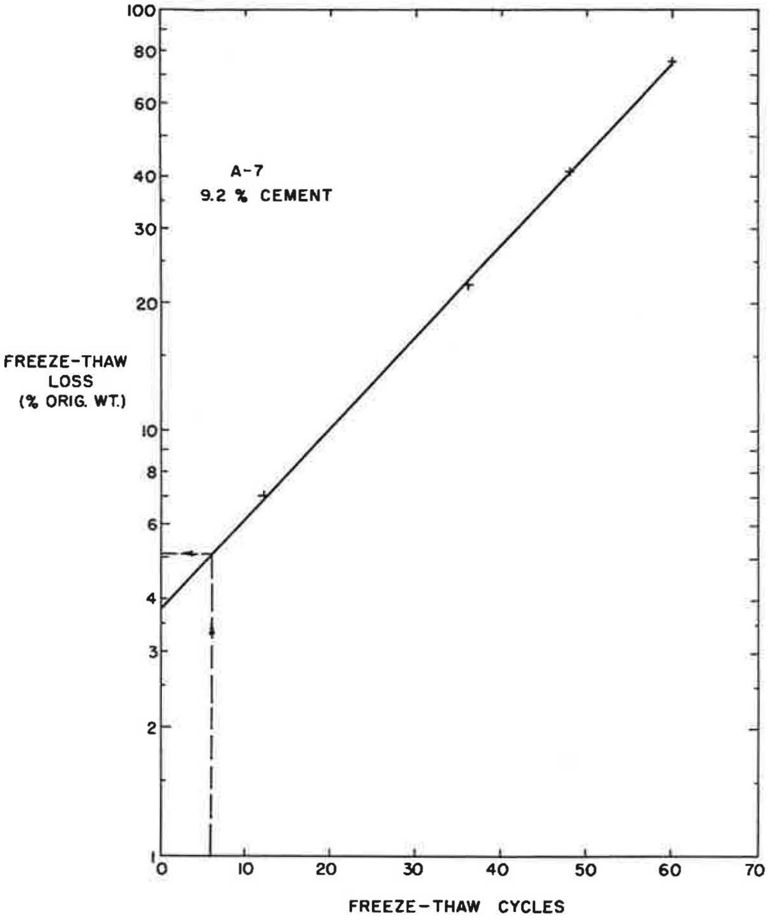


Figure 7. Linear relationships of A-2 and A-7 soils required to evaluate maximum allowable freeze-thaw loss at a reduced number of freeze-thaw cycles.

is apparent that the specifications for maximum freeze-thaw loss can be altered as the number of cycles are varied. For example, in the case of the A-2 soil, 14 percent freeze-thaw loss indicates a cement content of 7.9 percent cement. This cement content corresponds to 50 percent loss at 96 cycles. Thus if 96 cycles were used as the criterion in the freeze-thaw test, 50 percent loss would be considered the maximum allowable freeze-thaw loss.

Figure 6 plots the maximum freeze-thaw loss permitted as the number of cycles of the 3 soils are varied. There is transition in the shape of the curves from the sandy to clayey soils. The A-4 soil assumes a linear relationship; the A-2 soil becomes linear on a logarithmic scale; the A-7 soil becomes linear on a semilogarithmic scale (Fig. 7).

By the use of these linear relationships it should be possible to choose a maximum freeze-thaw loss at any number of cycles. This assumes that the relationship is valid at less than 12 cycles. This would be advantageous in developing a method to conduct the freeze-thaw test at a reduced number of cycles. For example, at 6 cycles of freeze-thaw the cement content for the A-2 soil could be evaluated at 9 percent freeze-thaw loss. In the same way the A-4 and A-7 cement contents could be evaluated at 7 percent and 5 percent freeze-thaw loss, respectively.

This reduction in the number of cycles would reduce by 50 percent the time required to conduct the test. A minimum number of cycles required to produce significant results could be determined. Further investigation is necessary to evaluate this theory properly for all soils.

CONCLUSIONS

This investigation indicates that the freeze-thaw loss of soil-cement mixtures follows certain approximately predictable paths. An approximate logarithmic relationship was found to exist between the cement content and the freeze-thaw loss of a soil-cement mixture. This relationship is useful for determining, approximately, the cement content which will produce the allowable freeze-thaw loss. Freeze-thaw tests with two cement contents will establish the relationship. When more than two cement contents are used, the relationship will obviate any outliers that might exist in the data, reducing the error inherent in this type of subjective testing.

The logarithmic relationships for A-2, A-3, and A-4 soil-cement mixtures were found, approximately, to intersect at a common point. It is conjectured that all granular soil-cement mixtures follow this rule. This can be of great value when properly applied. A granular soil for which the cement content (below 10 percent) is approximately known would require a freeze-thaw test with one cement content to establish the relationship and determine, to within reasonable approximation, the required cement content.

The slope of the logarithmic relationship was found independent of the number of freeze-thaw cycles. This introduces the possibility of conducting the freeze-thaw test at a reduced number of cycles.

By the use of the above methods the time and labor involved in the conduct of the freeze-thaw test can be greatly reduced, though a detailed analysis of the precision to be expected from these methods has not been conducted. The accurate determination of the cement content required to produce a specified freeze-thaw loss will result in more economical mix design of soil-cement mixtures.

REFERENCES

1. Catton, M. D., "Research on the Physical Relations of Soil and Soil-Cement Mixtures." HRB Proc., 23:821-855 (1940).
2. Felt, E. J., "Factors Influencing Physical Properties of Soil-Cement Mixtures." HRB Bull. 108, 138-162 (1955).
3. "Soil-Cement Laboratory Handbook." Portland Cement Association. (1959).

ACKNOWLEDGMENTS

The research for this report is under the sponsorship of the Iowa Highway Research Board, Iowa State Highway Commission.

The authors wish to express appreciation to R. L. Handy, T. Demirel and other members of the Soil Research Laboratory, Engineering Experiment Station, for information and advice.

Appreciation is also expressed to L. T. Norling, G. K. Ray, and R. G. Packard of the Portland Cement Association.

Appendix

Soil Type	r^2 Correlation Coefficient[a]	Cement Content (% by weight)		
		Actual[b]	Predicted[b]	PCA[c]
A-2	0.990	5.0	5.0	6.0
	0.999			
	0.981	6.8	8.0	7.1
	0.999	8.7	8.9	8.2
	0.950	7.6	8.3	7.0
	0.811	12.0	10.7	12.0
	0.999	6.2	6.3	6.1
	0.944	5.9	6.6	6.1
	0.793			
	0.953	7.8	7.8	7.8
	0.798			
	0.823	9.0	8.5	9.3
	0.916			
	0.998	7.8	7.5	7.4
	0.975			
	0.896			
	0.994	4.5	4.8	5.0
	0.986	3.8	5.0	4.6
	0.950	6.5	6.4	6.3
	0.996	5.1	5.9	5.4
A-3	0.992	7.3	7.5	7.9
	0.996	8.0	8.4	9.8
	0.987	9.7	10.5	10.6
	0.951			
	0.999	9.2	9.4	9.3
	0.947			
	0.999			
A-4	0.999	8.7	8.8	9.0
	0.949			
	0.846	6.8	7.5	8.7
	0.976	9.0	8.5	9.2
	0.975			
	0.907	9.9	10.0	10.3
	0.971			
	0.908			
	0.987	10.4	10.2	10.8
	0.982			
	0.945	7.2	7.2	7.6
	0.950			
	0.987			
	0.938	9.7	9.9	10.5
	0.937	7.5	8.3	8.1
	0.972			
	0.910			
	0.829	9.7	8.4	8.8
	0.948			
	0.878	7.4	8.6	8.3
	0.924			
	0.978			
	0.923	6.9	7.3	8.2
	0.905	9.6	9.8	10.5
	0.905	8.2	8.1	9.3
	0.882			
	0.978			
	0.913			
	0.806	11.2	10.0	11.9
	0.965			
A-4	0.866			
	0.900			
	0.979			
	0.997			
	0.874			
	0.868			
	0.908	10.0	9.5	11.0
	0.934			
	0.875			
	0.965			
	0.999	9.3	9.5	10.7
	0.932			
	0.950	5.9	5.6	6.1
	0.959			
	0.983	7.6	7.4	8.1
	0.806	10.6	10.8	12.1
	0.994			
	0.894	9.1	9.4	9.8
	0.963			
	0.938	8.7	8.5	9.0
	0.946	8.8	10.1	10.1
	0.934			
	0.994	12.2	10.5	12.6
	0.990	10.0	9.6	10.2
	0.980	10.3	10.3	10.8
	0.962	8.2	8.2	9.0
	0.998	10.2	9.4	10.3
	0.974			
	0.893	8.3	8.6	9.0
	0.993	8.0	7.8	9.3
	0.963	7.7	7.5	7.6
	0.978	7.2	6.6	7.6
	0.891			
	0.873	7.0	6.8	7.6
	0.992			
	0.928			
	0.878	6.5	7.2	6.8
	0.982	8.2	7.4	8.1
	0.998			
	0.806			
	0.897			
	0.986			
	0.977	6.8	6.2	7.2
	0.968	7.2	7.2	7.2
A-6	0.997			
	0.991			
	0.953			
	0.999			
	0.940			
	0.998			
	0.991			
	0.993			
	0.965			
	0.907			
	0.969			
	0.943			
	0.928			
A-6	0.998			
	0.974			
	0.990			
	0.991			
	0.973			
	0.997			
	0.992			
	0.985			
	0.948			
	0.977			
	0.999			
	0.995			
	0.831			
	0.896			
	0.994			
	0.997			
	0.986			
	0.999			
	0.914			
	0.880			
	0.997			
	0.948			
	0.998			
	0.999			
	0.910			
	0.888			
	0.992			
	0.988			
	0.958			
	0.820			
	0.931			
	0.915			
	0.889			
A-7	0.997			
	0.860			
	0.955			
	0.909			
	0.886			
	0.969			
	0.979			
	0.961			
	0.998			
	0.999			
	0.879			
	0.937			
	0.922			
	0.932			
	0.937			
	0.972			
	0.927			
	0.983			
	0.979			
	0.959			
	0.999			
	0.909			
	0.993			

Supplement

A further investigation (4) into the possibility of reducing the number of cycles required to conduct the freeze-thaw test has recently been concluded. The results are generally in agreement with those observed in the paper.

To investigate the applicability of these relationships below 12 cycles, data on 4 soils from a recent PCA study (5) were used. These soils were classified as A-1-b(0), A-4(5), A-4(8) and A-6(10). Freeze-thaw loss at 2, 4, 6, 8, 10 and 12 cycles of testing was evaluated. Either 4 or 5 cement contents were used for each soil.

A graphical analysis indicated that excellent logarithmic cement freeze-thaw relationships exist between 6 and 12 cycles. Below 6 cycles the freeze-thaw loss was almost negligible and relationships were poor. The A-1-b and A-4 soils produced parallel relationships, whereas the slope of the A-6 soil increased directly with the number of cycles. A statistical analysis of the data of Felt (2) indicated similar results. The existence of the logarithmic relationship between 6 and 12 cycles of freeze-thaw testing would indicate that interpretable results are possible at a reduced number of cycles.

The reduction in the number of cycles was further investigated with data supplied by R. G. Packard, Portland Cement Association. Seventy-three sets of data representing most soil types were investigated. Logarithmic relationships at 6 and 12 cycles were determined; the cement content at the specified 12-cycle freeze-thaw loss was chosen, and this cement content was used to evaluate the freeze-thaw loss which would result from 6 cycles of testing. The freeze-thaw loss at 6 cycles corresponding to the required cement content at the specified 12-cycle freeze-thaw was evaluated, and is shown with the standard deviation at 6 cycles in Table 2.

The standard deviations observed are due to both the 6 cycle and 12 cycle logarithmic relationships. Thus, the standard deviation caused by imprecision of the 6-cycle interpolation is less than that in Table 2. The table is an evaluation of the required 6-cycle loss predicted from a knowledge of the 12-cycle loss. However, due to the parallelism observed in the relationships of the granular soils, the predicted standard deviation of the loss at 12 cycles from a knowledge of the 6-cycle loss would be the same on the logarithmic scale. Therefore, the coefficient of variation (the relative standard deviation) of 12 cycles of freeze-thaw testing predicted from 6 cycles would be the same as the coefficient of variation of 6 cycles of freeze-thaw testing predicted from 12 cycles.

These soils have been grouped according to their allowable freeze-thaw loss at 12 cycles (i.e., at 14 percent, 10 percent, and 7 percent). The results (Table 3) indicate that at 6 cycles a reduced freeze-thaw loss is obtained which is fairly consistent for each soil group. Thus, it is evident that 6 cycles of testing might produce interpretable and reproducible results from which a valid criterion can be established.

TABLE 2
AVERAGE FREEZE-THAW LOSS AFTER 6 CYCLES OF TESTING NECESSARY TO PRODUCE THE REQUIRED CEMENT CONTENT OF INDIVIDUAL SOIL TYPES

Soil Type	No. of Soils Tested	Freeze-Thaw Loss (% orig. wt.)	
		Avg.	Std. Dev.
A-1	7	6.4	2.2
A-2	14	6.5	1.8
A-3	5	5.0	1.1
A-4	22	3.3	1.2
A-6	13	3.2	0.9
A-7	12	2.3	1.2

TABLE 3
AVERAGE FREEZE-THAW LOSS AFTER 6 CYCLES OF TESTING NECESSARY TO PRODUCE THE REQUIRED CEMENT CONTENT OF FREEZE-THAW SOIL GROUPS

Soil Type	No. of Soils Tested	Freeze-Thaw Loss (% orig. wt.)	
		Avg.	Std. Dev.
A-1, A-2, A-3	26	6.0	1.7
A-4	22	3.3	1.2
A-6, A-7	25	2.8	1.0

REFERENCES

4. Circeo, L. J., "Abbreviated Freeze-Thaw Test Procedures for Soil-Cement Mixtures." Ph.D. thesis, Iowa State University (1963).
5. Packard, R. G., "Alternate Methods for Measuring Freeze-Thaw and Wet-Dry Resistance of Soil-Cement Mixtures." HRB Bull. 353, 8-41 (1962).

Reactions Accompanying Stabilization Of Clay with Cement

A. HERZOG, Senior Lecturer in Civil Engineering, University of New South Wales, Newcastle, Australia, and
J. K. MITCHELL, Assistant Professor of Civil Engineering, and Assistant Research Engineer, Institute of Transportation and Traffic Engineering, University of California, Berkeley

This paper reports the results of an investigation aimed at delineating the nature of the reactions accompanying the stabilization of clay with portland cement. Consideration of the nature of cement hydration, the physico-chemical characteristics of clays and lime-clay interaction leads to the hypothesis that during the hydration of a clay-cement mixture, hydrolysis and hydration of cement could be regarded as primary reactions which form usual cement hydration products, increase the pH, and liberate lime. The high pH and $Ca(OH)_2$ concentration could initiate attack of the clay particles and also cause breakdown of amorphous silica and alumina which then could combine with calcium to form secondary cementitious material. A clay-cement skeleton and a clay matrix are likely.

Mechanical, X-ray diffraction, and chemical tests on kaolinite and montmorillonite stabilized with portland cement and with pure tricalcium silicate (C_3S), the major strength-producing compound in portland cement, gave results in agreement with this hypothesis. Compacted clay-cement mixtures were cured at 100 percent relative humidity and at 60 C (to accelerate hardening) and studied at different times after molding. X-ray analyses showed that calcium hydroxide was formed in hydrating clay-cement, but was rapidly used up in reaction with the clay. Minor alteration of the kaolinite-cement X-ray pattern and marked alteration of the montmorillonite-cement X-ray pattern were indicated after curing periods of 12 weeks, suggesting clay mineral structure breakdown and/or interaction with cement at particle surfaces. The pH of hydrating neat portland cement or neat C_3S did not change with time but decreased during the curing of clay-cement, suggesting the presence of $(OH)^-$ consuming processes. The results of conductivity measurements indicate that soluble salts formed by cement hydration are converted to less soluble compounds in the presence of clays. Water soluble calcium and free calcium hydroxide analyses showed that all clay-cement specimens contained only a fraction of the $Ca(OH)_2$ that would be present if normal hydration of the cement took place without cement-clay interaction. The "missing" $Ca(OH)_2$ may have participated in a pozzolanic-type reaction with the clay mineral. Clay-cement cannot, therefore, be regarded as a simple mixture of hydrated cement particles bonding together unaltered clay particles, but must be considered as a system in which both the clay and hydrating cement combine through secondary reactions.

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

•SOIL-CEMENT stabilization has been used on an ever-increasing scale in recent years because of the significant improvement in soil properties that may be achieved as a result of cement treatment. The improvement in engineering properties of cement-treated soils is believed to be due mainly to the hardening of portland cement, but the processes taking place during the hydration of cement in the presence of clays are not fully understood.

If the hardening of soil-cement were due solely to the hydration of cement, the soil could be regarded as a chemically inert component. The cement particles would bind adjacent soil grains together during hardening and form a more or less continuous skeleton of a hard, strong material enclosing a matrix of unaltered soil. This skeleton could also be expected to "plug" some of the voids of the soil, reducing permeability and swelling, and increasing the resistance of soil-cement to the deleterious effects of changes in ambient moisture conditions.

If, in addition to hardening, reactions take place between hydrating cement and clay components, additional cementitious material could be generated leading to the strengthening of the bonds between the soil grains themselves and between soil and cement particles. At the same time, the clay participating in such reactions could suffer alteration to the extent that it would become less plastic and less expansive when exposed to water.

Improved techniques of soil-cement stabilization may be expected to follow the development of a better understanding of cement-clay interaction. Several investigators have recently postulated that the cementitious material formed during the hardening of soil-cement differs in composition from hardened cement paste. Lambe, Michaels and Moh (16) suggest that strong bases formed during the hydration of cement dissolve silica and alumina from clay, and that calcium ions liberated during the hydrolysis of cement react with dissolved silica and alumina forming cementitious material. Interaction between soil and cement has also been suggested by others (2, 3, 10, 18).

This paper reports the results of an investigation aimed at further delineating the nature of the reactions accompanying the stabilization of clay with portland cement. In the formulation of a hypothesis for cement-clay interaction, a general understanding of the nature of cement hydration, the physico-chemical characteristics of clays, and lime-clay interaction is useful. These factors are summarized briefly.

HARDENING OF NEAT PORTLAND CEMENT PASTE

The dry portland cement particle is a heterogeneous material containing small crystals of C_3S, C_2S, C_3A, and a solid solution commonly described as C_4AF (21). (According to nomenclature used in cement chemistry $C = CaO$, $S = SiO_2$, $A = Al_2O_3$, $F = Fe_2O_3$, $H = H_2O$.) A simplified summary of reactions during the hydration of cement is given by Lea and Desch (17). During cement hydration the components react with water. Hydrated silicates, hydrated aluminates and calcium hydroxide (from C_3S and C_2S) are the main reaction products. Taylor (22) points out that the silicate and aluminate phases are intimately mixed and that probably none of them is fully crystalline. Part of the $Ca(OH)_2$ may also be intimately mixed with the other hydrated phases and be only partly crystalline.

The composition of the cement hydration products is not uniquely defined by chemical formulas, but may vary considerably. For instance, the Ca:Si ratio of calcium silicate hydrate, CSH, which is formed from C_3S, and which is the main strength-producing compound, can vary between 0.8 and 1.33 (22). CSH type of compounds are similar to the natural mineral tobermorite. Taylor (22) reports the results of a study in which CSH was treated with water and lime solutions. A gain in the lime content of CSH in the presence of lime-rich solutions and a loss in the lime content of CSH in the presence of solutions of low lime concentration were observed. Thus in soil-cement the pore water could extract lime from the hydrating cement in the event that the lime content of the pore water was maintained at low level as a result of reaction with the clay.

Changes in the composition of hydrated calcium silicates are not necessarily accompanied by a change in their X-ray patterns. Taylor (22) found that irrespective of the Ca:Si ratio of CSH the X-ray pattern resembles that of tobermorite, but that only the strongest reflections can be observed.

SOIL PROPERTIES RELEVANT TO CLAY-CEMENT INTERACTION

One prerequisite for the formation of additional cementing materials in cement-clay interaction is the solubilization of silica and alumina from clay components. The solubility of clay minerals is not as well defined as that of pure chemical compounds. The amount of impurities present, the degree of crystallinity of the minerals and particle size distribution are some of the factors influencing solubility. In general, acids are effective in removing alumina and other metallic oxides from clay minerals, while alkalies dissolve mainly silica (9), although alumina may be attacked in a high pH environment as well.

Correns (4) reported the dissolution of small amounts of Al_2O_3 and SiO_2 from montmorillonite and kaolinite with distilled water. Hashimoto and Jackson (11) observed the dissolution of about 3 percent SiO_2 from oven dry kaolinite and approximately 8 percent SiO_2 from montmorillonite by boiling in 0.5 N NaOH. The solubility of the clay minerals would be expected to increase with decreasing particle size and decreasing degree of crystallinity because of a greater specific surface and less resistant crystal structure.

Minerals having a three-layer structure (e.g., montmorillonite) are usually more soluble than minerals having a 2-layer structure (e.g., kaolinite) because of the greater specific surface of the 3-layer minerals and because these minerals are less resistant to penetration of interlayer cations.

High cation exchange capacity could lead to the binding of cations that would otherwise participate in the formation of cementing material. Soil components other than clay minerals, particularly finely divided amorphous silica and alumina, are less resistant to chemical attack and represent a potential source for cementitious matter. Such components may play a very significant role in clay-cement stabilization.

LIME-CLAY MINERAL INTERACTION

Inasmuch as lime is generated during the hydration of portland cement, the interaction of lime and clay minerals would be expected to have an important influence on the overall clay-cement interaction process. The colloid chemistry of lime stabilization of soils has been reviewed by Anderson (2), and a comprehensive bibliography of lime stabilization was presented by Herrin and Mitchell (13). Reactions taking place between clay minerals and lime may be divided into two distinct types: those which are completed rapidly (ion exchange and flocculation) and reactions which proceed slowly (carbonation, pozzolanic reactions, and the formation of new materials).

Addition of lime to clays causes flocculation because of the increased electrolyte content of the pore water and also as a result of ion exchange by the clay to the calcium form. Although flocculation and ion exchange may be complete in a few days, the slower reactions producing cementitious material may proceed for years. Cementitious material in lime-clay mixtures may be formed by carbonation and by chemical reactions between clay components and lime. Carbonation normally is confined to surfaces exposed to air and involves the conversion of lime to calcium carbonate by carbon dioxide absorbed from the air. The calcium carbonate cements soil particles together and enhances their stability.

Pozzolanic chemical reactions between lime and soil materials have been utilized for centuries. Little was known about the composition of the reaction products before 1960 because of the probable amorphous nature of the initial products and the fact that crystalline products, often poorly defined, usually do not appear until after long curing periods, and then only in cases where a high percentage of lime was added. Pozzolanic materials are defined as silicious or silicious and aluminous materials, which themselves possess little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties (ASTM Designation C219-55). Clay minerals and some other soil components possess pozzolanic properties (2). The addition of lime to soil causes an instantaneous rise in the pH of the molding water (15) due to solution and dissociation of the $Ca(OH)_2$. The high pH increases the reactivity of surface silica and alumina. The calcium ions combine with the reactive hydrous silica and alumina and form gradually hardening cementitious material.

Goldberg and Klein (8) treated Wyoming bentonite with up to 8 percent calcium hydroxide. They reported the disappearance of $Ca(OH)_2$ lines from the X-ray spectrum, observed no other changes in the X-ray pattern of the clay, and suggested the conversion of calcium hydroxide to $CaCO_3$ and some other compound.

Eades and Grim (6) investigated the reaction of hydrated lime with pure clay minerals and noted the formation of new crystalline phases, probably calcium silicate hydrates, produced by lime-kaolinite interaction.

> This reaction appeared to take place by the lime eating into the kaolinite particles around the edges with a new phase forming around the core of kaolinite. In contrast, the reactions of lime with three layer minerals (illite, montmorillonite) begin by a replacement of existing cations between the silicate sheets with Ca^{++}. Following the saturation of the interlayer positions with Ca^{++} the whole clay mineral structure deteriorates without the formation of substantial new crystalline phases.

The X-ray diffraction pattern of well crystallized kaolinite showed some weakening of the prism reflections. The effect was accompanied by the weakening of the basal reflections and was more pronounced on less resistant, poorly crystallized material. Halloysite behaved in the same manner as kaolinite.

The formation of new minerals by lime stabilization under field conditions was proven by Eades, Nichols and Grim (7), who identified calcium silicate hydrates by X-ray methods on soils which were stabilized with lime in 1956-57 and sampled in 1960. Hilt and Davidson (14) isolated a crystalline calcium aluminate hydrate reaction product from a mixture of bentonite, 40 percent lime and 110 percent water after 7 months' curing.

Some of the more significant points emerging from the preceding review may be summarized by the following statements:

1. Hydrating cement is a partly crystalline, multiphase, alkaline system containing calcium hydroxide, calcium silicate hydrates and hydrated aluminates. The system is capable of releasing lime and may vary in composition without significant changes in its X-ray diffraction pattern.
2. Silica and alumina may be dissolved from clay minerals, and from amorphous material also present in most clays, by alkalies or calcium hydroxide.
3. Flocculation and ion exchange take place rapidly when lime is mixed with moist clay minerals. Lime in excess of the exchange requirement of the clay may react with clay minerals. This pozzolan-type reaction proceeds slowly. The initial reaction products are amorphous and may later change to a crystalline form. The X-ray diffraction patterns indicate a destruction of montorillonite mineral structure by lime, but only moderate attack on kaolinite.

A HYPOTHESIS FOR CLAY-CEMENT INTERACTION

Primary and secondary processes may be distinguished during the hardening of a clay-cement mixture. Hydrolysis and hydration of cement could be regarded as primary reactions. These processes would initially form usual cement hydration products and increase the pH of the pore water. The fresh calcium hydroxide formed during this period would be more reactive than ordinary lime.

Clay may participate in the secondary processes. The calcium ions produced by cement hydration first convert the clay to the calcium form and tend to intensify the flocculation initiated by the increase in total electrolyte content accompanying the addition of cement. The calcium hydroxide initiates attack of the clay particles and amorphous constituents. Dissolved silica and alumina combine with calcium ions and precipitate additional cementitious material.

Calcium hydroxide used up in the secondary processes could be replenished to some extent by the release of lime from the hydrating cement. Thus, products of the primary reaction provide a reactant necessary for the continuation of the secondary processes.

Since the secondary cementitious matter would be mainly formed on or near the surface of clay particles, the flocculated clay particles would be "glued" together at points of contact by the secondary cementitious material. Even stronger bonds may be expected to develop between the hydrating cement paste and clay particles coating cement grains. The structural similarity between some cement hydration products and clay minerals even suggests possible epitaxy; i.e., a direct propagation of a similar structure from clay crystal to cement particles.

The overall effect of the postulated cement-clay interaction would be the formation of primary and secondary cementitious matter. The primary products harden into a high strength "aggregate" and differ from normally hydrated cement in that their calcium content is lower. The secondary processes enhance the strength and stability of soil-cement by producing additional cementitious matter which increases interparticle bond strength.

EXPERIMENTAL STUDIES

The experimental work was carried out on compacted clay-cement specimens. Monomineral clays were used in preference to natural clays of more complex composition. Kaolinite and montmorillonite were selected to represent the least and most reactive type of clay minerals. Compacted clay-cement cylinders were cured at 100 percent relative humidity and 60 C for 1 day, and 1, 4 and 12 weeks. After each curing period the specimens and appropriate blank control materials were tested.

To test the hypothesis, experimental procedures were selected as follows:

Purpose of Test	Test Used
1. To index the formation and hardening of primary and secondary cementitious material	Unconfined compression test on soaked specimens
2. To follow the variation of water content and to assess dimensional stability during curing and soaking	Water content, linear shrinkage, and swelling
3. To identify crystalline products from primary and secondary reactions and to observe changes in the crystal structure of clay minerals	X-ray diffraction
4. To observe changes in the alkalinity of specimens after various curing periods	pH
5. To determine changes in the available (free) $Ca(OH)_2$ content of specimens with curing time	Free calcium hydroxide content
6. To determine changes in concentration of all water soluble electrolytes in specimens with curing time	Conductivity of water extract
7. To determine changes in the relative concentration of certain water soluble cations with curing time	Ca, Na and K content of water extract

Materials

Materials used in the investigation were as follows:

1. Georgia kaolin, representing a coarse-grained well crystallized kaolinite. This material has a cation exchange capacity of 5 meq/500g with calcium accounting for about 70 percent of the total exchange complex. The pH was 5.5, determined in a 1:5 clay-water suspension.

2. Wyoming bentonite, a commercially available Na montmorillonite with a cation exchange capacity of 85 meq/100g. Exchangeable cations included 70-meq Na^+, 8-meq Ca^{++} and 1-meq K^+ per 100-g montmorillonite. The pH of the montmorillonite was 8.6, determined in a 1:5 clay-water suspension.

3. Portland cement, a Type I low alkali cement from the Permanente Cement Co. of California. Chemical analysis of the cement is given in the Appendix.

4. Tricalcium silicate, C_3S. Type I portland cement contains about 50 percent tricalcium silicate, C_3S, which is the main contributor to the strength of hardened cement. It was hoped that by using pure C_3S as cementing material the effects of any clay mineral-cement interaction would be more readily detectable. The tricalcium silicate was a synthetic material of high purity, made at the University of California, Berkeley. Characteristics of this preparation have been described by Polivka, Klein and Best (19).

Methods

Preparation of Specimens.—Eight series of specimens were prepared, four with kaolinite (K) and four with montmorillonite (M). Each clay was stabilized with portland cement (P) or with tricalcium silicate (C_3S). For each clay-cement combination, 15 percent and 30 percent (by weight of dry clay) of cementing material was employed. The symbols used to identify each test series indicate the clay mineral (K or M), the cementing material (P or C_3S) and the percent cement (15 or 30) in the specimens. Thus, MP refers to specimens made from montmorillonite with 15 percent portland cement.

Clay and cement were blended together in the dry state to obtain a uniform mix. Distilled water was added from a burette and mixed by hand with the dry materials for 7 minutes. The samples were prepared by kneading compaction using a Harvard miniature compactor type apparatus. The hand-mixed material was compacted in 5 layers at a molding water content exceeding the optimum value by about 2 percent in order to insure adequate water for cement hydration. The resulting cylindrical samples were 0.75 in. in diameter and 1.5 in. high.

Figure 1 shows compaction curves of the clay-cement mixtures for the applied compactive effort. The compactive effort for the montmorillonite series was higher than that used for the kaolinite series in order to obtain about the same water/cement ratio and a similar dry density in each series.

Specimens were cured at 100 percent relative humidity in screw-top glass jars at 60 C for 1 day and 1, 4 and 12 weeks. The curing temperature of 60 C was selected to accelerate hydration reactions. Each batch consisted of at least four specimens of the same composition. Compositional data on specimens of all test series are given in Tables 1 and 2.

Linear Swelling and Moisture Content.—The length of each test cylinder was measured after compaction, after curing and after soaking. The moisture content of the material used during compaction and the fractured test cylinders was determined by oven drying at 110 C. Because some hydration of cement takes place during oven drying, some of the water originally present in the sample will become tied up in cement hydration; therefore, moisture content values are relative rather than absolute.

Unconfined Compression Tests.—At the end of each curing period the test cylinders were submerged in distilled water and soaked for 24 hours. The wet specimens were tested in unconfined compression, after the removal of surface moisture, at a strain rate of 1.6 percent per minute in a Universal testing machine.

X-Ray Tests.—Cured samples were ground in a mortar and packed into a bakelite holder. Care was taken to avoid particle orientation. The tests were carried out with a North American Phillips Geiger counter X-ray spectrometer, type No. 12021. Copper Kα radiation was used with a nickel filter. The scanning speed was 2 deg 2θ per min.

pH and Conductivity Tests.—Cured specimens not soaked in water were ground up in a mortar. Ten grams of the ground specimen were mixed with 50-cc distilled water with an electric stirrer for 10 minutes. The suspension was filtered immediately and

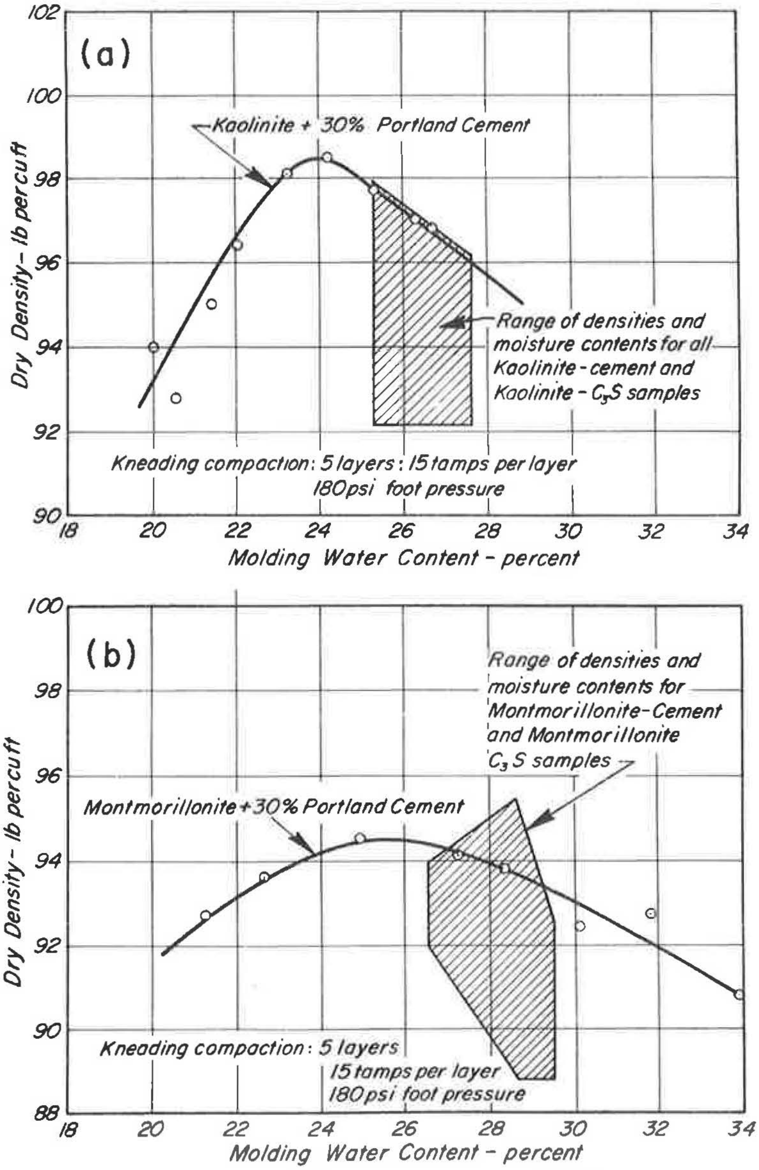


Figure 1. Compaction of mixtures: (a) kaolinite-cement; (b) montmorillonite-cement.

the filtrate collected in a polyethylene bottle. The pH of the filtrate was determined with a Beckman model H2 pH meter. The conductivity of the filtered extract was tested with a "Solu Bridge" type RD conductivity bridge.

The moisture content of the pulverized specimens was determined on a separate sample. Conductivity results are based on the oven dry weight of the ground material.

Calcium, Sodium and Potassium Content of Water Extract.—The filtrate from the previously described extraction was used for the determination of water soluble Ca^{++}, Na^{+}, and K^{+} content of the specimens. The tests were carried out with a Perkin-Elmer model 52 flame photometer. Results are presented on the same basis as conductivity data.

In an attempt to avoid the carbonation of the fresh lime and the hydrated calcium compounds in the specimens, the exposure of chemical test samples to open air was

TABLE 1

COMPOSITION AND MECHANICAL PROPERTY DATA[a] KAOLINITE-CEMENT SPECIMENS

Sample	Cement or C$_3$S Content (%)	Molding Water Content (%)	Dry Density (pcf)	Age at Test (days)[b]	Axial Swell During Soaking (%)	Water Absorption During Soaking (%)	Compressive Strength (psi)
K	0	26.4	92.2	0	—	—	134[c]
KP 15	PC 15	26.0	92.2	2	0.35	5.5	432
		26.5	92.4	8	0.09	4.4	704
		25.4	95.7	29	0.09	4.0	862
		26.3	92.2	85	0.19	2.4	1,305
KC$_3$S 15	C$_3$S 15	27.6	92.1	2	1.72	4.9	633
		26.8	92.5	8	0.17	3.9	832
		27.3	92.4	29	0.08	3.1	1,630
		27.0	92.2	85	0.07	1.3	1,830
KP 30	PC 30	25.6	97.6	2	0.10	3.3	1,030
		26.5	96.6	8	0.22	2.5	1,130
		26.4	97.0	29	0.21	4.8	1,830
		27.0	98.5	85	0.15	1.7	2,180
KC$_3$S 30	C$_3$S 30	25.3	97.6	2	1.48	3.1	1,130
		25.9	96.5	8	0.44	2.8	1,450
		25.6	97.4	29	0.04	2.7	2,550
		26.0	98.5	85	0.09	2.2	2,600

[a] Values represent the average of the values for a minimum of three samples.
[b] Samples cured at 60 C and 100 percent relative humidity and soaked without confinement for final 24 hours.
[c] Untreated samples disintegrated upon immersion; strength value is for unsoaked specimen.

TABLE 2

COMPOSITION AND MECHANICAL PROPERTY DATA[a] MONTMORILLONITE-CEMENT SPECIMENS

Sample	Cement or C$_3$S Content (%)	Molding Water Content (%)	Dry Density (pcf)	Age at Test (days)[b]	Axial Swell During Soaking (%)	Water Absorption During Soaking (%)	Compressive Strength (psi)
M	0	29.0	92.3	—	—	—	100[c]
MP 15	PC 15	28.6	95.5	2	1.01	5.0	237
		28.7	91.8	8	0.17	3.5	472
		28.4	95.1	29	0.00	1.5	475
		28.9	90.4	85	0.08	1.6	527
MC$_3$S 15	C$_3$S 15	27.9	90.3	2	1.06	7.1	427
		29.5	90.0	8	0.11	5.9	628
		28.7	88.9	29	0.65	1.0	631
		28.8	88.7	85	0.13	1.4	612
MP 30	PC 30	27.0	98.4	2	0.36	4.9	462
		27.7	94.1	8	1.47	5.3	581
		27.7	94.0	29	0.28	5.0	1,020
		27.5	93.5	85	0.00	1.0	1,470
MC$_3$S 30	C$_3$S 30	26.6	92.8	2	0.88	5.4	800
		28.0	93.1	8	0.61	4.6	950
		27.0	92.0	29	0.08	6.4	1,150
		27.0	91.4	85	0.13	1.0	1,570

[a] Values represent the average of the values for a minimum of three samples.
[b] Samples cured at 60 C and 100 percent relative humidity and soaked without confinement for final 24 hours.
[c] Untreated samples disintegrated upon immersion; strength value is for unsoaked specimen.

TABLE 3

CHEMICAL TEST RESULTS, KAOLINITE-CEMENT SPECIMENS

Sample[a]	Age at Test (days)	pH[b]	Soluble Salts by Conductivity (meq/100g)	Free $Ca(OH)_2$ (meq/100g)	Water Soluble Ca^{++} (meq/100g)	Water Soluble Na^+ (meq/100g)	Water Soluble K^+ (meq/100g)
K	0	5.50	0.80	0	—	—	—
P	1	12.60	60.5	260	56	0.91	0.85
	7	12.65	87.5	400	74	0.87	0.79
	28	12.63	79.0	420	72	0.83	0.76
	84	12.65	81.0	440	70	0.71	0.69
C_3S	1	12.65	36.1	280	35	0	0
	7	12.63	94.2	430	88	0	0
	28	12.64	91.0	460	79	0	0
	84	12.65	89.7	500	80	0	0
KP 15	1	11.80	5.8	2.8	4.0	0.4	0.3
	7	11.75	5.1	3.0	3.3	0.4	0.3
	28	11.65	4.9	2.8	2.9	0.3	0.3
	84	11.55	3.7	2.0	2.9	0.2	0.3
KC_3S 15	1	12.30	13.8	5.2	3.8	0.4	0
	7	12.10	6.4	5.2	3.0	0.3	0
	28	11.95	6.0	4.4	2.9	0.3	0
	84	11.60	3.8	2.2	2.8	0.2	0
KP 30	1	12.15	12.6	12.2	8.0	0.4	0.6
	7	12.00	8.1	11.2	5.0	0.5	0.6
	28	11.90	7.7	10.8	4.6	0.5	0.5
	84	11.58	5.8	4.2	4.1	0.5	0.4
KC_3S 30	1	12.50	20.7	15.2	12.1	0.5	0
	7	12.20	8.5	11.5	5.1	0.4	0
	28	12.00	7.1	9.8	4.8	0.3	0
	84	11.60	5.2	3.2	4.7	0.2	0

[a] P = portland cement, C_3S = tricalcium silicate, 15 = 15 percent cement or C_3S by weight of dry clay, 30 = 30 percent cement or C_3S by weight of dry clay.

[b] Determined in suspension of one part solid in five parts water.

prevented insofar as possible. These efforts are believed to have served their purpose but prevented the use of techniques (repeated grinding and sieving, multiple extraction and filtration, etc.) required for complete extraction. Thus data on conductivity and water soluble salts do not represent absolute values but serve as comparative figures for the interpretation of the processes during clay-cement interaction.

Free Calcium Hydroxide Content.—The method used for determination of free calcium hydroxide content is based on extraction with a nonaqueous solvent from dried specimens to prevent the formation of additional $Ca(OH)_2$ from unhydrated cement during extraction. Cement paste and clay-cement specimens were ground and dried to constant weight in vacuum over P_2O_5. The free $Ca(OH)_2$ content was determined by using several extraction times in accordance with the modified Franke method (20).

RESULTS

The results of the mechanical tests are given in Tables 1 and 2; the chemical test results in Tables 3 and 4.

Unconfined Compression Tests

Figure 2 shows compression test results for kaolinite-cement specimens, and Figure 3 for montmorillonite-cement specimens. Each point represents the average of at least 3 test results obtained on clay-cement samples soaked for 24 hours before testing. Because blank specimens containing no stabilizer disintegrated rapidly on

TABLE 4

CHEMICAL TEST RESULTS, MONTMORILLONITE-CEMENT SPECIMENS

Sample[a]	Age at Test (days)	pH[b]	Soluble Salts by Conductivity (meq/100g)	Free $Ca(OH)_2$ (meq/100g)	Water Soluble Ca^{++} (meq/100g)	Water Soluble Na^+ (meq/100g)	Water Soluble K^+ (meq/100g)
M	0	8.50	8.70	0	—	—	—
P	—	—	—	—[c]	—	—	—
C_3S	—	—	—	—[c]	—	—	—
MP 15	1	11.80	23.3	1.0	0.6	16.2	0.2
	7	11.70	22.2	4.6	5.3	16.4	0.2
	28	11.40	20.5	3.2	4.3	16.3	0.2
	84	10.90	17.2	2.8	2.0	15.0	0.1
MC_3S 15	1	12.15	32.7	1.0	0.9	17.0	0
	7	12.10	23.6	5.8	5.0	17.7	0
	28	11.55	18.0	5.0	4.3	13.3	0
	84	11.10	13.0	3.0	1.4	10.8	0
MP 30	1	12.25	27.3	1.6	2.7	16.8	0.2
	7	12.10	24.2	7.2	4.6	16.5	0.2
	28	11.95	21.0	6.2	4.3	16.2	0.2
	84	11.48	16.6	5.2	1.8	13.2	0.2
MC_3S 30	1	12.30	38.5	3.8	4.1	17.9	0
	7	12.25	29.7	10.0	5.2	17.6	0
	28	12.00	23.0	8.0	4.7	17.3	0
	84	11.65	14.2	5.4	1.6	13.0	0

[a] P = portland cement, C_3S = tricalcium silicate, 15 = 15 percent cement or C_3S by weight of dry clay, 30 = 30 percent cement or C_3S by weight of dry clay.

[b] Determined in suspension of one part solid in five parts water.

[c] See Table 3.

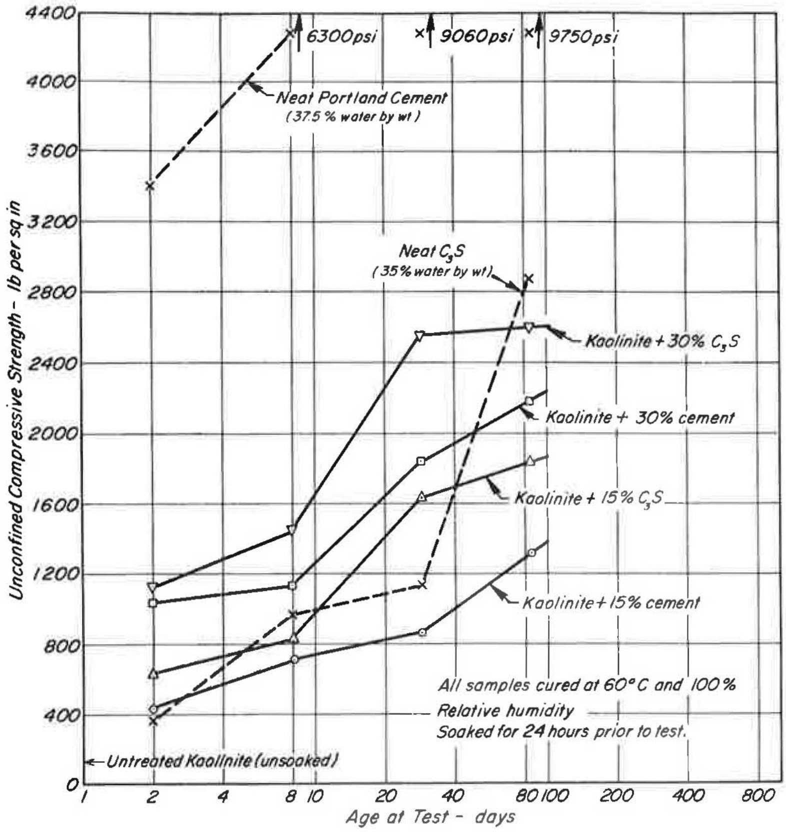


Figure 2. Strength of kaolinite-cement as a function of curing time.

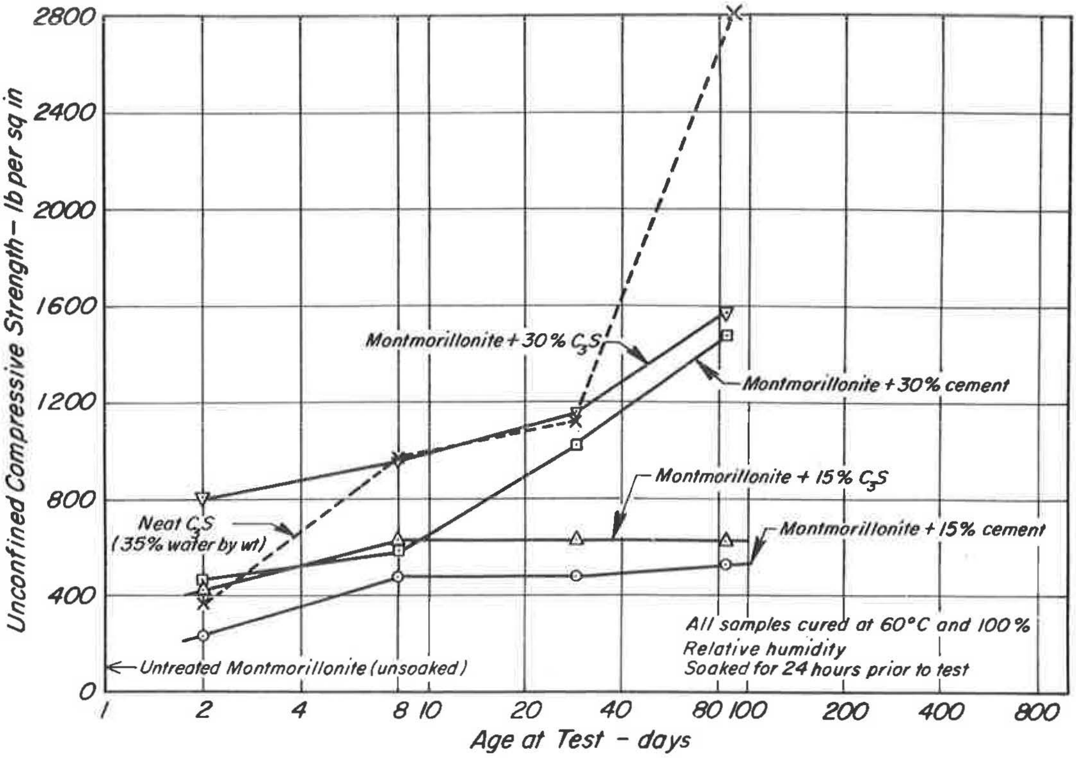


Figure 3. Strength of montmorillonite-cement as a function of curing time.

exposure to water, their strength was determined without previous soaking. Clay-cement specimens failed at axial strains ranging from 0.3 percent to 1.3 percent. Cement treatment led to a several-fold strength increase in all cases, indicating effective stabilization at the treatment levels used.

All KP and KC_3S specimens had a higher 1-day strength than the corresponding MP and MC_3S specimens. The rate of strength increase from 1 to 7 days was about the same for all series, but kaolinite samples gained strength more rapidly from 1 to 4 weeks than montmorillonite specimens. The strength of MP 15 and MC_3S 15 test cylinders remained practically unchanged for curing times exceeding 7 days.

Samples of the C_3S series were always stronger than the corresponding specimens in the portland cement series. Since alite, a C_3S type clinker component, is the major contributor to the strength of portland cement, stabilization with pure C_3S would be expected to increase the compressive strength above that obtained with ordinary cement.

Specimens made of neat C_3S paste were found to have only a fraction of the early strength of neat portland cement paste specimens of the same age (Fig. 2). The high purity of the C_3S preparation used in this work could possibly be responsible for this as a result of the formation of a low strength intermediate hydration product in a neat paste. All clay-C_3S specimens developed higher strengths after 24 hours than neat C_3S specimens cured for 1 day. This anomolous behavior could be explained by assuming an interaction between C_3S and clay which modifies the C_3S hydration process in such a manner that high strength hydration products are formed rapidly in the presence of clays. Finally, compression tests on specimens of kaolinite plus 30 percent cement and montmorillonite plus 30 percent cement cured at room temperature gave strengths approximately equal to the strengths of mixtures containing 15 percent cement but cured at 60 C, thus illustrating the acceleration in rate of strength gain effected by elevated temperatures during curing.

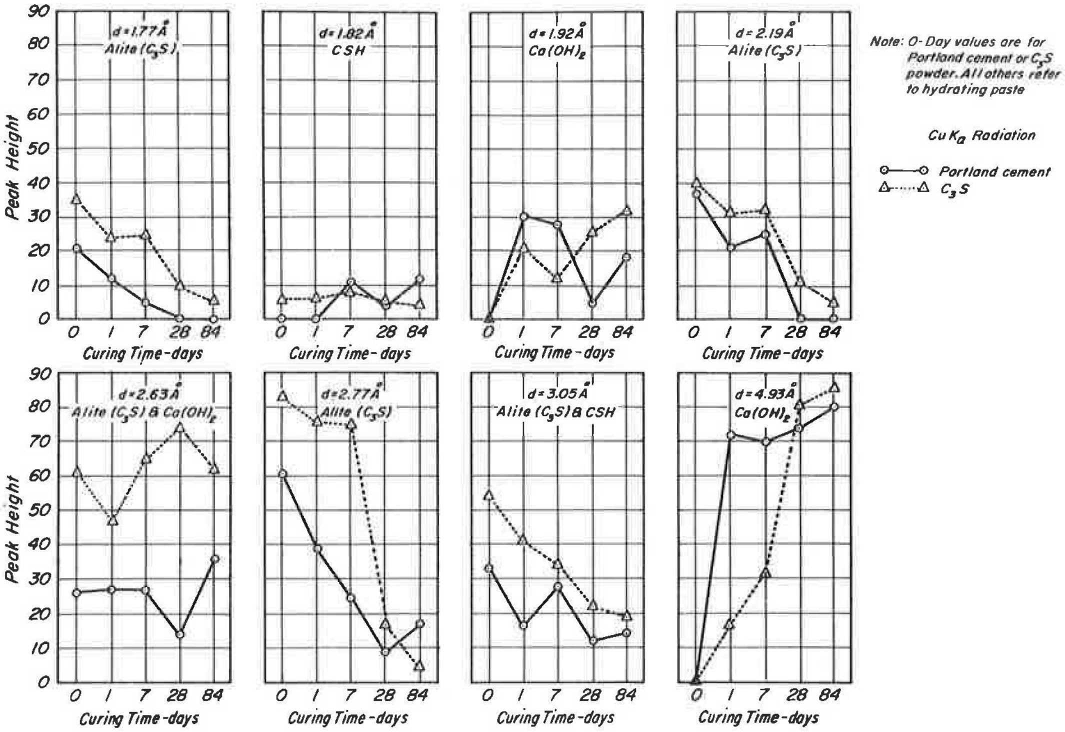


Figure 4. X-ray diffraction peaks for hydrating portland cement and hydrating tricalcium silicate.

X-Ray Diffraction Tests

Portland Cement.—Strong reflections at 1.77, 2.19, 2.60, 2.77 and 3.02 Å, characteristic of the principal clinker component alite prevail in the X-ray spectrum of unhydrated cement. As the hydration of the cement progresses the intensity of the alite lines decreases strongly (Fig. 4).

The spectra of hydrating cement samples are dominated by calcium hydroxide lines at 1.92, 2.63 and 4.93 Å (1). The intensity of the 2.63- and 4.93-Å lines increases with the curing age, indicating the accumulation of crystalline calcium hydroxide during hydration. Changes in the intensity of the 3.05-Å CSH line, which increases in intensity with time, are obscured by the initially present 3.02-Å alite line, which decreases in intensity with time, but strengthening of this reflection after 12 weeks is evidence for the presence of CSH.

C_3S.—The intensity of C_3S reflections at 1.77, 2.19, 2.77 and 3.05 Å decreases markedly with curing time (Fig. 4). Sharp peaks at 1.92, 2.63 and 4.93 Å increase with curing age and confirm the continuous formation of calcium hydroxide during hydration.

Only weak CSH lines can be observed at 1.82 Å and the intensity of the 3.05-Å reflection consistently decreases. This suggests the absence of a significant amount of crystalline CSH hydration products in the C_3S preparation used in this experimental work and may be responsible for the slow strength development in neat C_3S paste during hydration.

Kaolinite-Cement.—Figure 5 shows results of X-ray diffraction tests on kaolinite-cement and kaolinite-C_3S mixtures. (These tests were run at high amplification to accentuate detail; as a result, the first order basal reflections for the clay minerals were off scale and are not reported in Fig. 5.) Reflections at 1.82 Å and 3.05 Å appeared in kaolinite-cement specimens after 1-day curing, indicating formation of CSH. The strengthening of CSH reflections with time suggests normal cement hydration. No

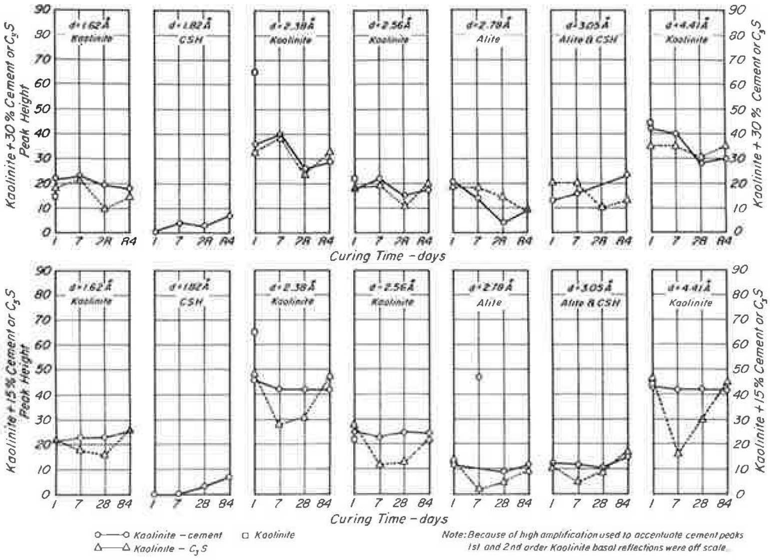


Figure 5. X-ray diffraction peaks for hydrating kaolinite-cement and kaolinite-C_3S mixtures.

$Ca(OH)_2$ lines were observed on cement or C_3S-treated samples of kaolinite except for KC_3S 30 specimens cured for 1 day. However, 4.87-Å and 2.62-Å $Ca(OH)_2$ lines appear on the X-ray trace of kaolinite powder blended with 30 percent (by weight) of 3-month-old ground, hydrated portland cement (Fig. 6). This indicates that the absence of $Ca(OH)_2$ lines on kaolinite cement X-ray traces for samples cured for more than 1 day is not due to masking effects by kaolinite. Since compression tests indicate large increases in the strength of stabilized specimens, the cement or C_3S can be assumed to hydrate normally and form $Ca(OH)_2$ during hydration. Because this $Ca(OH)_2$ is not present in a crystalline form in clay-cement, it must either be in an amorphous form or used up by a secondary process.

In both KP 15 and KP 30 specimens, the intensity of kaolinite lines generally decreased slightly with curing time. The weakening of kaolinite lines was more pronounced on KP 30 samples than on KP 15 specimens cured for the same periods. The increased weakening of kaolinite lines at higher cement content suggests the possibility of an attack on the kaolinite structure and/or a coating of clay particles and aggregates by reaction products.

The kaolinite lines in KC_3S 15 specimens (Fig. 5) show an initial weakening with subsequent recovery beginning after 1 week. The overall change in intensity during 3 months is about the same as in the KP 15 series. No 1.82-Å CSH line appeared, but in contrast to the C_3S paste trace, the 3.05-Å reflection became stronger from 1 week onwards.

Changes in reflection intensities of kaolinite and CSH lines in the KC_3S 30 series followed the same general trend as in the KC_3S 15 series. The increase of the 3.05-Å line after 1 month's curing of KC_3S 15 and KC_3S 30 is in contrast with the consistent weakening of this line on C_3S paste traces. It indicates that the hydration of C_3S is modified in the presence of kaolinite. As previously noted, KC_3S specimens are stronger at a given age than KP specimens, whereas neat cement is found to be many times stronger than neat C_3S paste of the age. Both of these observations support the hypothesis that kaolinite modifies the hydration of the C_3S material used.

Two $Ca(OH)_2$ lines, 2.63 Å and 4.93 Å, were noted on a KC_3S 30 specimen cured for 1 day but could not be observed after longer curing periods. This suggests the existence of a lime producing process and a simultaneous operation of a $Ca(OH)_2$ consuming mechanism. The rate of $Ca(OH)_2$ formation appears to exceed the rate of $Ca(OH)_2$ consumption only at high C_3S concentration and only during the first day or days of hydration. Only under these conditions would it be expected that $Ca(OH)_2$ reflections would appear on the X-ray pattern.

The gross effects of hydrating cement on kaolinite are shown by a comparison of X-ray diffraction traces of KP 30 cured for one day and for twelve weeks. Figure 7 shows a slight decrease in height of the 3.57-Å and 7.15-Å basal reflections after 12 weeks and a general broadening of some of the low-intensity peaks.

Montmorillonite-Cement.—Figure 8 shows the results of the X-ray diffraction tests on montmorillonite-cement and montmorillonite-C_3S mixtures. $Ca(OH)_2$ lines were missing from the diffraction patterns of all MP and MC_3S specimens. $Ca(OH)_2$ peaks were observed, however, on a mixture of montmorillonite and 30 percent (by weight) of ground hydrated cement (Fig. 9). Thus, the "dilution" of hydrated cement powder with montmorillonite could not have masked the appearance of $Ca(OH)_2$ lines.

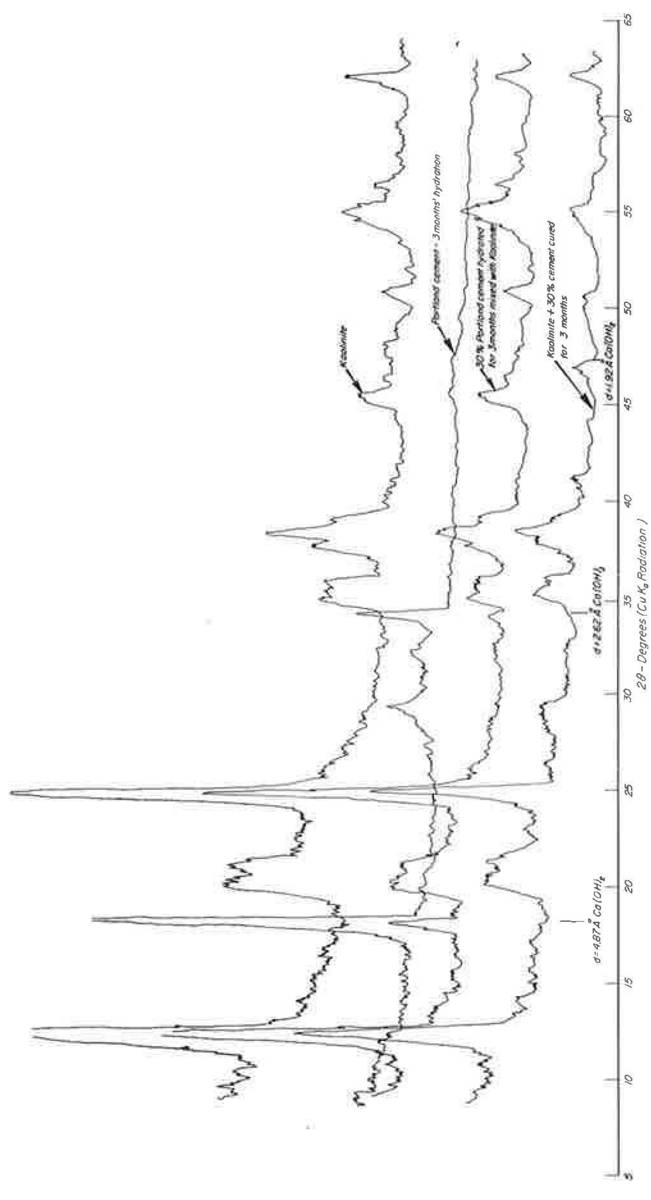


Figure 6. X-ray diffraction patterns for kaolinite, hydrated cement, hydrated cement mixed with kaolinite, and hardened kaolinite-cement.

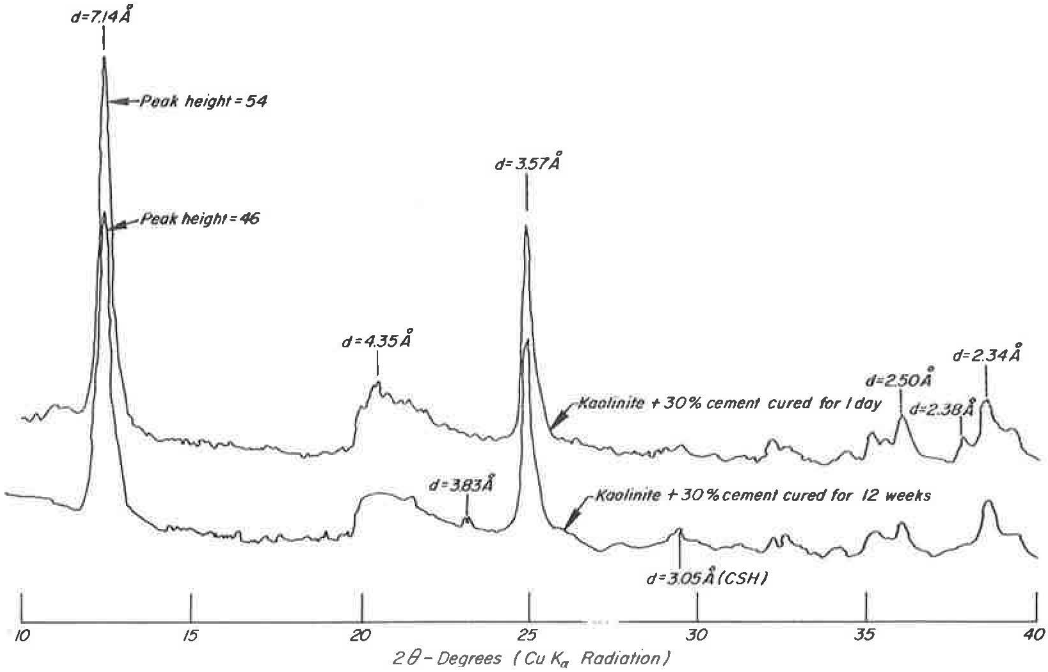


Figure 7. X-ray diffraction patterns for kaolinite + 30% cement samples cured for 1 day and 12 weeks.

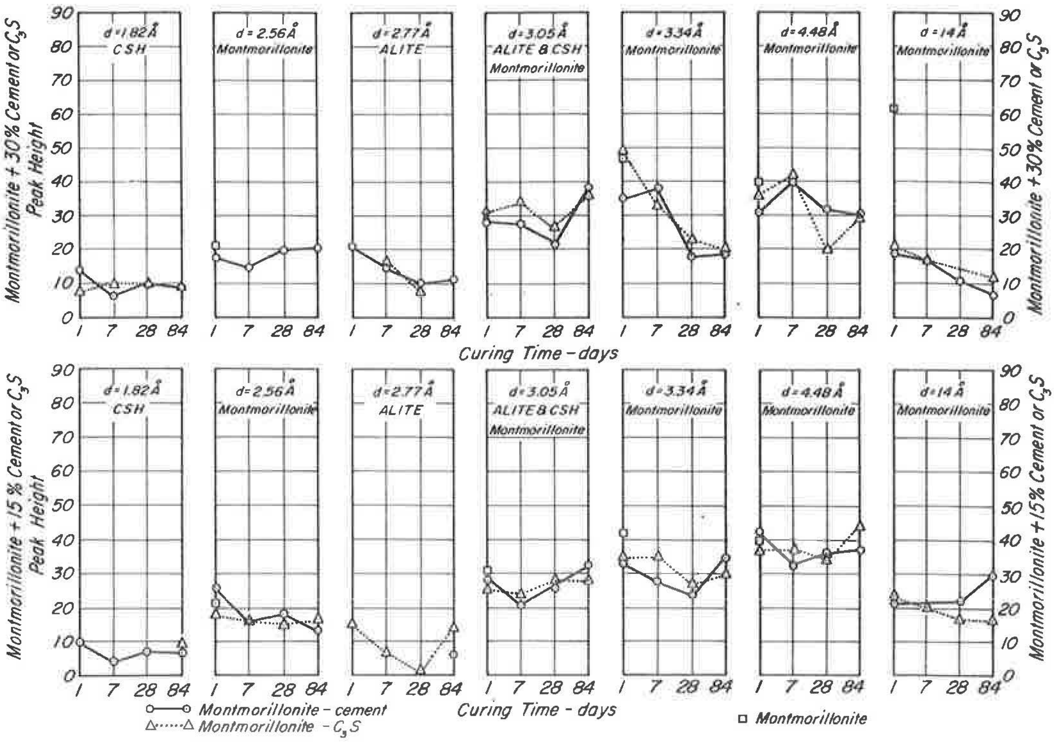


Figure 8. X-ray diffraction peaks for hydrating montmorillonite-cement and montmorillonite-C_3S mixtures.

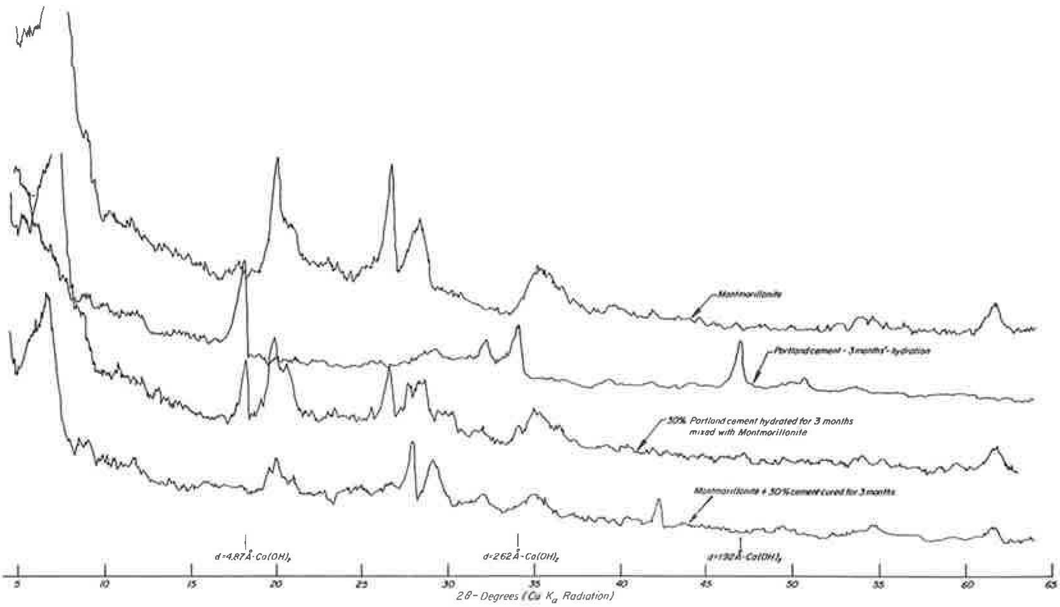


Figure 9. X-ray diffraction patterns for montmorillonite, hydrated cement, hydrated cement mixed with montmorillonite, and hardened montmorillonite-cement.

Montmorillonite lines interfere with the observation of CSH peaks at 1.82 Å and 3.05 Å for samples of the MP 15 series. Minor montmorillonite diffraction peaks at 2.56, 3.09, 3.34 and 4.48 Å weakened initially and became somewhat higher after 1 month. The 14-Å peak increased in height and sharpness during the last 8 weeks of curing for this series. An intensification of montmorillonite basal reflections caused as a result of the addition of small amounts of lime was found by Eades and Grim (6). The intensity of the 14-Å montmorillonite peak consistently decreased with increasing curing time for samples of the MC$_3$S 15 series, in which more lime would be generated than in the MP 15 series. Other montmorillonite lines showed a weakening tendency also. CSH reflections could not be clearly identified.

Degradation of the clay mineral structure was strongly indicated in MC$_3$S 30 specimens by the pronounced decrease of the 14-Å peak height, the reduction of the 3.34-Å line intensity by more than one-half and the decline of the 4.48-Å peak. CSH formation was indicated by the strengthening of the 1.82-Å and 3.05-Å lines with curing time.

Montmorillonite reflections weakened and the peaks became broader with increased curing time in mixtures of montmorillonite with 30 percent cement. Gross effects of hydrating cement on such mixtures are demonstrated by low amplification X-ray traces (Fig. 10). The considerable decrease of the 14-Å line intensity after 12 weeks suggests major structural breakdown of the montmorillonite.

During the preparation of extracts for chemical tests the separation of slowly settling, flocculent material from a rapidly settling, granular, dark sediment was observed in suspensions after 10 minutes of mechanical stirring. X-ray examinations of these two phases from specimens cured for 4 and 12 weeks showed that the 14-Å peak was higher and sharper on flocculent material than on the dark sediment. In the case of the specimens cured for 4 weeks, for example, the 14-Å peak height for the dark material was only 75 percent of the height of the corresponding peak for the light material. Upon further grinding, the dark sediment gave a 14-Å peak of intermediate intensity between that of the separated sediment and of the flocculated material.

The difference in 14-Å peak intensities between the flocculent and the rapidly settling fractions suggests the nonuniform degradation of montmorillonite. Clay particles

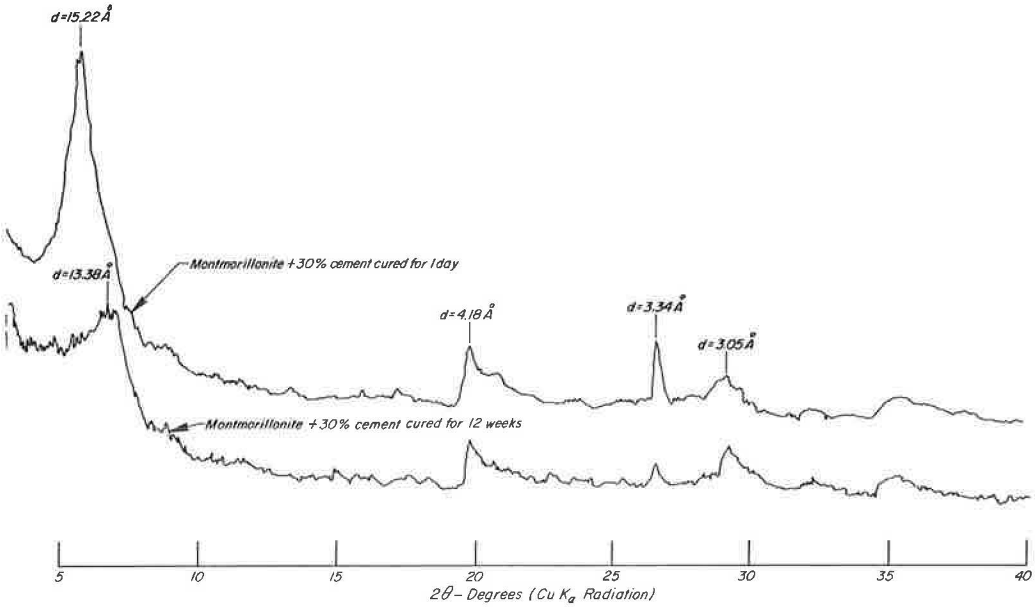


Figure 10. X-ray diffraction patterns for montmorillonite + 30% cement samples cured for 1 day and for 12 weeks.

in the immediate vicinity of cement grains are probably strongly attacked by the fresh lime from the hydrating cement and undergo greater structural degradation than clay particles located farther away from hydrating cement particles. It is also possible that clay particles close to cement grains become coated with reaction products leading to a decrease in peak intensities.

Recognizing that cement particles are very large (silt size) relative to the clay crystals, cement-stabilized montmorillonite could be regarded as composed of a clay-cement skeleton and a clay matrix. Units of the skeleton may be visualized as containing a core of hydrated cement gel to which layers of altered clay particles are attached. Such units may form larger size aggregates, trapping less altered clay in the intercellular space.

If the alteration of the clay mineral structure caused by $Ca(OH)_2$ is associated with the formation of cementitious material, severely altered particles would be strongly glued to each other and to cement grains. They could not be washed off during stirring with water from the large, heavy cement rich grains of the skeleton, and, after sedimentation, they would be part of the quickly settling sediment.

The least affected clay particles would be expected to be only weakly attached to each other and would readily go into suspension as flocculent matter. Thus, the less altered clay should be found in the suspended flocculent fraction; the more decomposed clay cemented to the heavier portland cement grains should be part of the sediment.

Further evidence for changes in montmorillonite character is given by the results of X-ray patterns of heat-treated and of glycolated specimens. Samples of MP 30 cured for 12 weeks were heated to 600 F, cooled in a dessicator and X-rayed. While untreated Ca montmorillonite exhibited a collapse of the basal spacing to 10.1 Å, the MP 30 samples showed only a partial collapse. The term "partial collapse" is used to describe a broad sawtooth-like peak with apexes at 10.4 Å and 12.06 Å, implying a nonhomogeneous material which had been altered to such a degree that basal spacings had become fixed at distances greater than 10 Å. Expansion as a result of ethylene glycol treatment could not be detected, indicating that the basal spacing had become fixed.

The most important results and conclusions that may be drawn from the X-ray phase of this investigation may be summarized as follows:

1. Portland cement hydrated normally in clay-cement and formed calcium hydroxide during hydration.
2. The hydration of the C_3S preparation was modified in the presence of clay.
3. Calcium hydroxide was identified in one set of clay-C_3S specimens after 1-day curing.
4. No calcium hydroxide lines were found in stabilized specimens after 1 week's curing time.
5. Small decreases in the intensity of kaolinite reflections and pronounced decreases in the intensities of montmorillonite reflections were observed after 12 weeks' curing of clay-cement mixtures.
6. Clay-cement appears to have a skeleton-matrix type structure.
7. New lines of low intensity were observed in some patterns for specimens containing 30 percent cement or C_3S at long curing times. This suggests the formation of new crystalline reaction products. Because of the low intensity of these peaks and the irregular patterns of their occurrence, the new compounds could not be identified specifically.

pH Determinations

Changes in pH accompanying the hardening of the clay-cement specimens are shown in Figures 11 and 12. The high pH values of all samples containing hydrating cement may be attributed to the dissociation of $(OH)^-$ ions from $Ca(OH)_2$ formed during cement hydration.

The pH did not change with time in neat portland cement, neat C_3S and blank clay specimens but decreased during curing in clay-cement specimens. This suggests the

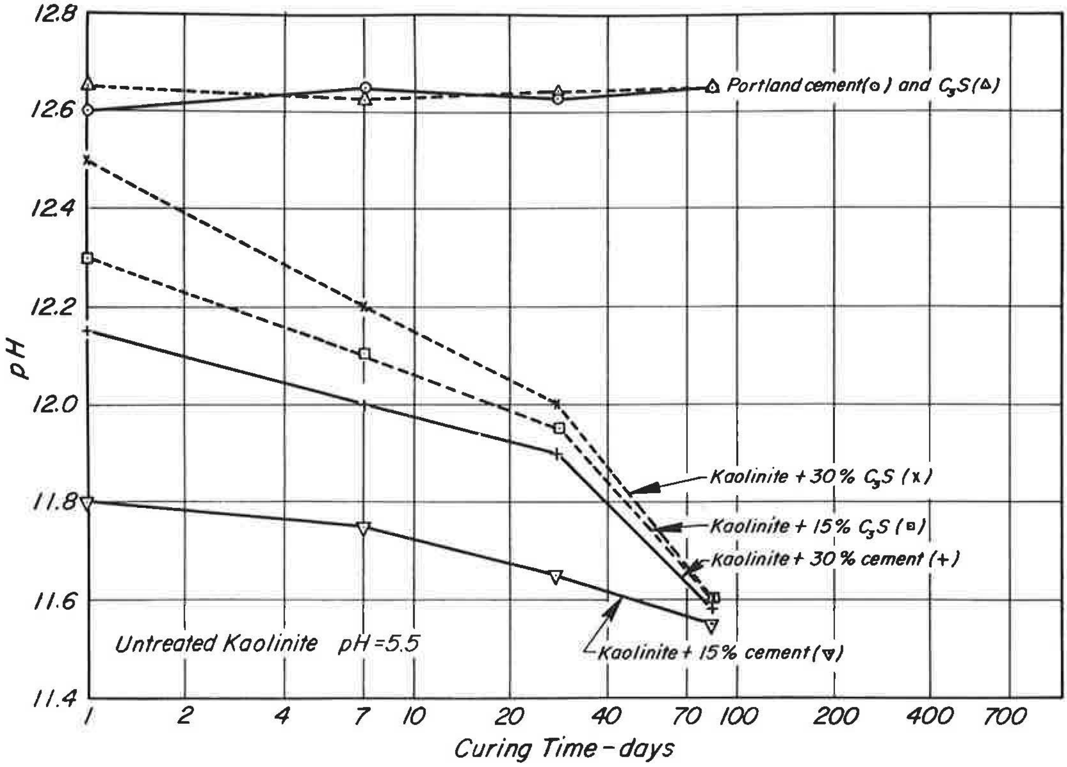


Figure 11. pH changes accompanying the hydration of kaolinite-cement and kaolinite-C_3S mixtures.

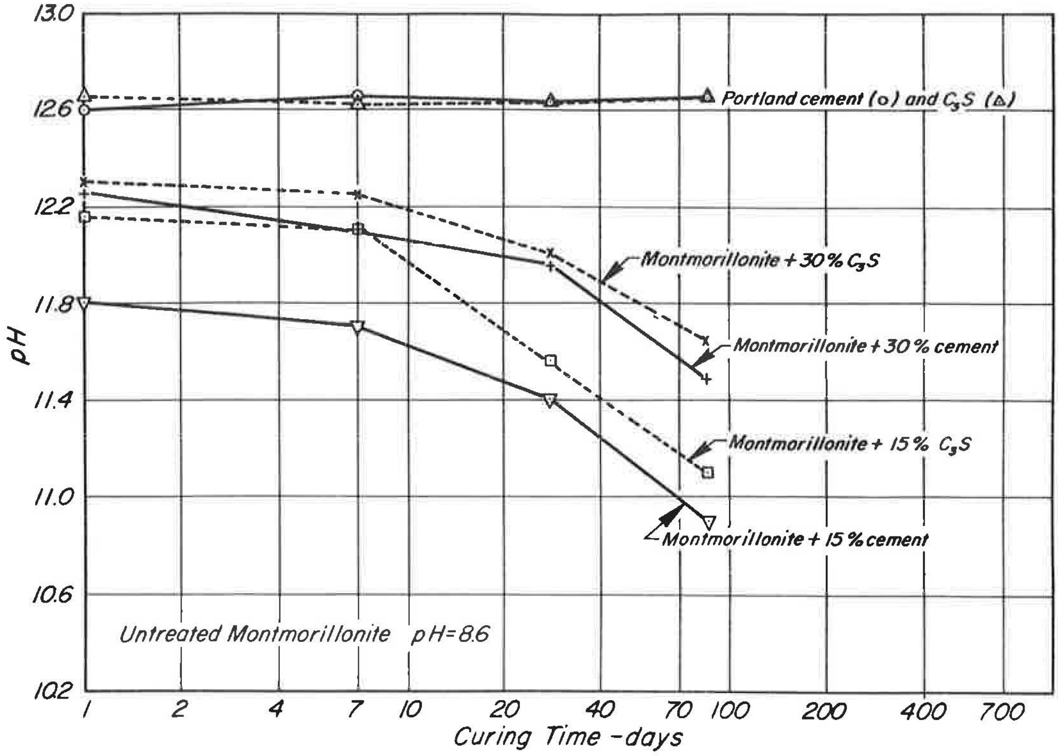


Figure 12. pH changes accompanying the hydration of montmorillonite-cement and montmorillonite-C_3S mixtures.

presence of an(OH)$^-$consuming process in hardening clay-cement. The existence of such a process is most evident in KC_3S 30 specimens (Fig. 11). Samples of this composition had the highest pH among all clay-cement specimens after 1-day curing and contained crystalline $Ca(OH)_2$ as previously noted. After longer curing periods the pH was always lower and no crystalline $Ca(OH)_2$ was detected in the X-ray patterns for the samples.

The pH values of montmorillonite-cement samples were lower than those of the corresponding kaolinite samples for curing times greater than 1 day. This is probably due to the greater solubility of montmorillonite in an alkaline environment than kaolinite and therefore higher (OH)$^-$ consumption of montmorillonites. Other factors could also contribute to this result. Montmorillonite has greater buffering capacity than kaolinite and therefore could be responsible for the greater pH drop. Montmorillonite would be expected to contain a greater amount of non-crystalline silica and alumina than kaolinite. If this material were involved in rapid reaction with (OH)$^-$ then the pH of the montmorillonite-cement should drop at a faster rate than the pH of the kaolinite-cement. The greatest decrease in pH with curing time was observed in the MP 15 and MC_3S 15 mixtures (Fig. 12). This large decrease in pH may have impeded the normal hydration of the cement and prevented strength increase after 1 week (Fig. 3).

It is also conceivable that a reduction in pH could be caused by adsorption of (OH)$^-$ If only adsorption and no chemical reaction took place, however, the mineral structure of the clay would remain unchanged. The pH in such a case would be the lowest after 1-day curing since adsorption reactions could be expected to be essentially complete after this time. $Ca(OH)_2$ generated by the hydrating cement after the first day could also be adsorbed and cause no change in the pH or would increase the pH after the adsorption capacity is satisfied.

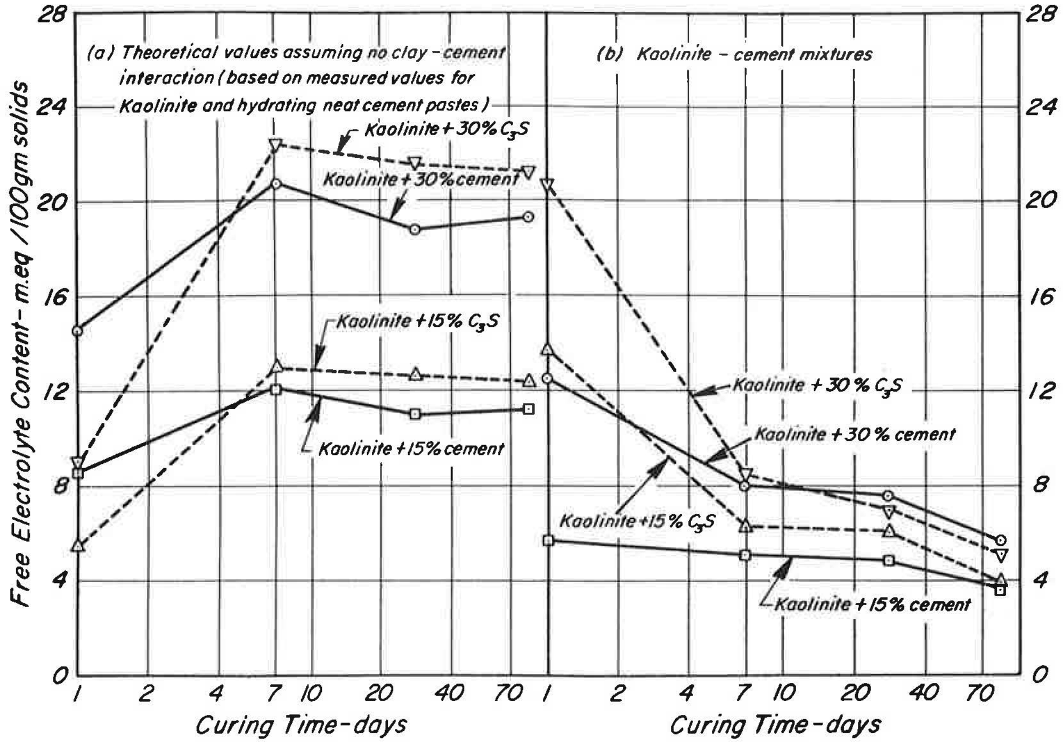


Figure 13. Free electrolyte content of hydrating kaolinite-cement and kaolinite-C_3S mixtures as indicated by conductivity of extract solutions.

The decrease of the pH of clay-cement with time, therefore, cannot be explained by adsorption. The data support the existence of a chemical reaction between $(OH)^-$ ions and the clay minerals or amorphous materials which leads to partial solublilization and the formation of secondary cementitious compounds.

Conductivity

Conductivity values of solutions prepared from specimens after each curing period are shown in Figures 13 and 14. Also shown are theoretical values that would be anticipated if there were no interaction by the clay and the hydrating cement. These values were obtained by taking the appropriate proportions of the conductivity values for neat cement and pure clay. The conductivity is indicative of the concentration of electrolytes readily extractable with water from the samples. The formation of water soluble salts, including $Ca(OH)_2$ during hydration would increase the electrolyte concentration. Conversely, the transformation of water soluble salts to compounds of lower solubility and/or the entrapping of soluble salts by the cementation of grains would decrease the electrolyte concentration of the water extract.

The conductivity of hydrating cement and C_3S extracts increases initially and decreases only slightly after 1 week, as do also the theoretical values for clay-cement mixtures (Figs. 13 and 14).

All clay-cement extracts indicate a significantly lower electrolyte content and a decrease of the electrolyte content with curing time. The observed decrease in conductivity suggests that in clay-cement the soluble salts formed by cement hydration are converted to less soluble compounds, perhaps precipitating with the mass and bonding particles together.

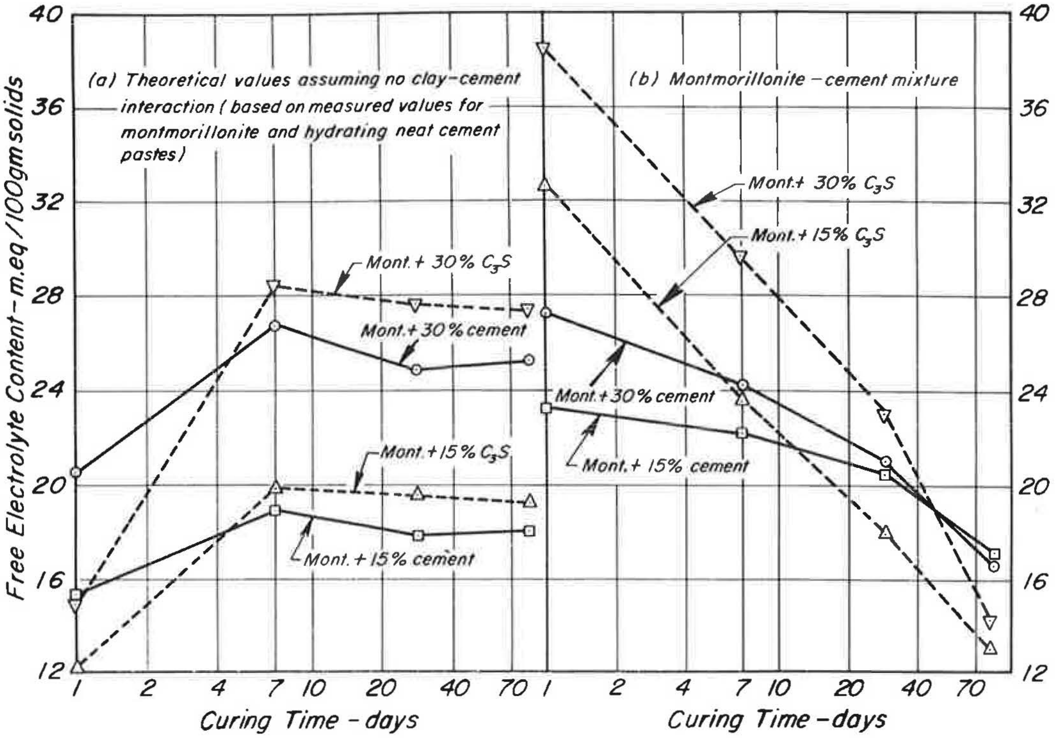


Figure 14. Free electrolyte content of hydrating montmorillonite-cement and montmorillonite C_3S mixtures as indicated by conductivity of extract solutions.

Calcium, Sodium and Potassium Content of Water Extracts

The extractable calcium content of all kaolinite-cement samples as a function of curing time is shown in Figure 15. Sodium and potassium contents are given in Table 3. If no interaction between the clay and hydrating cement took place, it would be anticipated that the extractable calcium content would be as given by the upper curves where values have been computed on the basis of a proportion of the values for neat cement. The observed values are significantly lower than those computed on this basis, suggesting that water soluble calcium ions liberated during cement hydration are to a considerable extent converted during the first day to a form not readily extractable with water.

The calcium content of water extracts of cement and C_3S sharply increased from 1 to 7 days, but declined in kaolinite-cement extracts, indicating further formation of insoluble compounds.

Montmorillonite-cement extracts were found to behave similarly except for a much higher sodium and lower calcium content than corresponding kaolinite samples at early ages (Fig. 16 and Table 4). The replacement of exchangeable sodium by calcium offers an explanation for this difference.

Free Calcium Hydroxide

Results of the free calcium hydroxide determinations using the nonaqueous extraction method are shown in Figures 17 and 18. The values of calcium content obtained for neat cement and C_3S were considerably higher than those determined with the flame photometer on water extracts. The longer extraction time and the higher temperature during extraction could be responsible for these higher values.

All clay-cement specimens contained only a fraction of the free calcium hydroxide that would be present if normal hydration of their cement content took place without

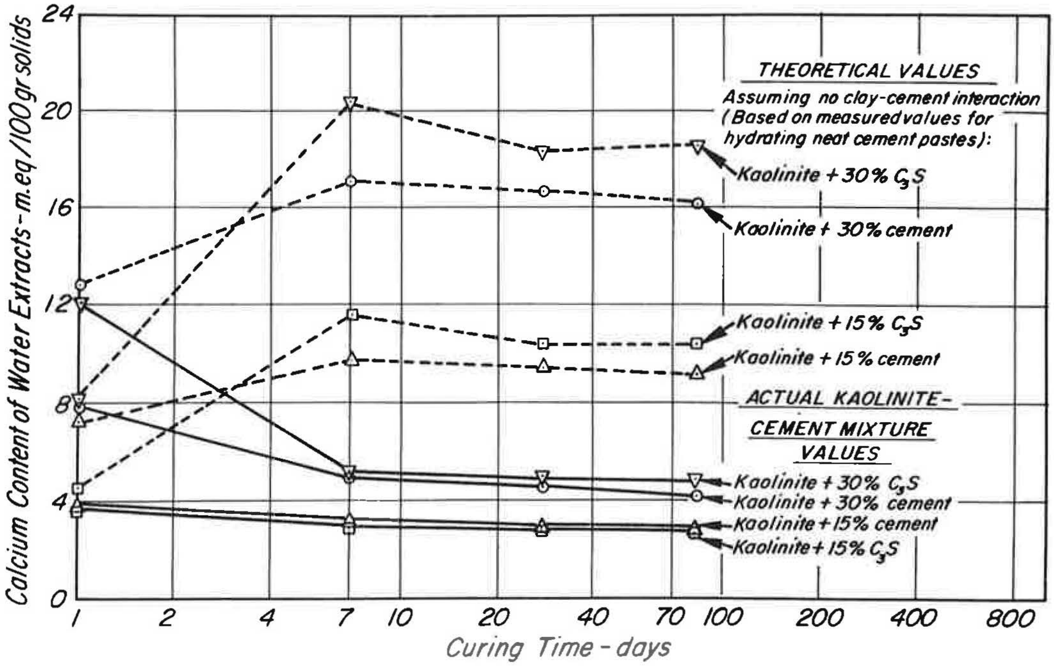


Figure 15. Calcium content of water extracts from kaolinite-cement and kaolinite-C_3S mixtures.

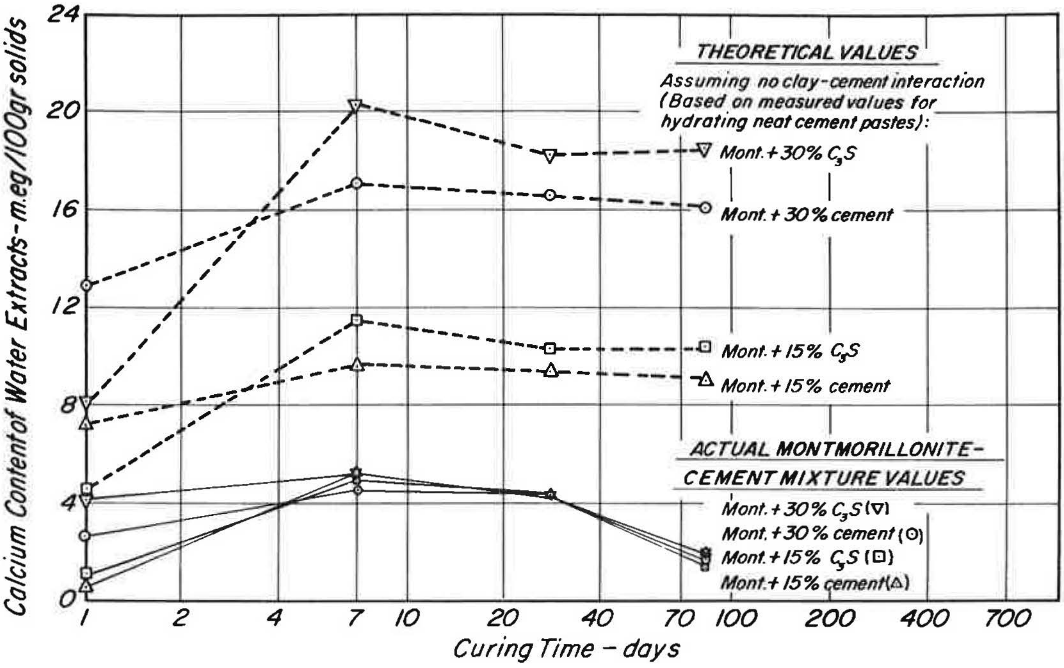


Figure 16. Calcium content of water extracts from hydrating montmorillonite-cement and montmorillonite-C_3S mixtures.

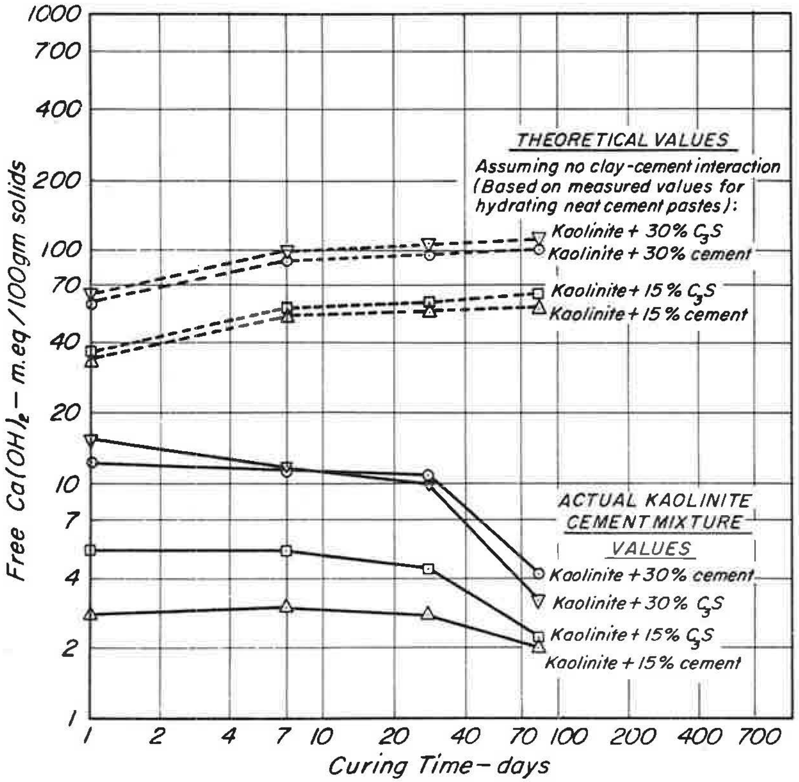


Figure 17. Free calcium hydroxide content of hydrating kaolinite-cement and kaolinite-C_3S mixtures.

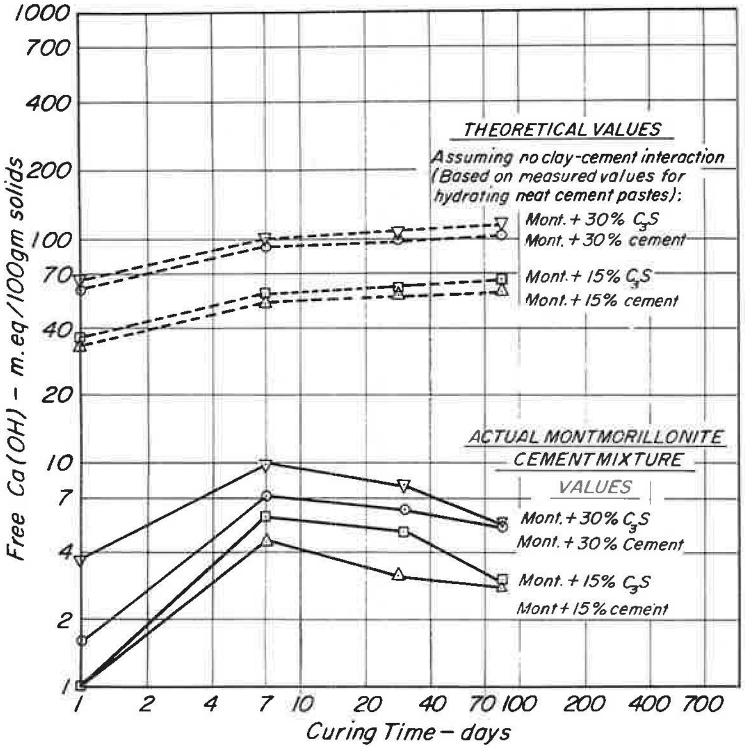


Figure 18. Free calcium hydroxide content of hydrating montmorillonite-cement and montmorillonite-C_3S mixtures.

interaction between cement and clay (Figs. 17 and 18). The calcium hydroxide formed by such hydration but missing from clay-cement could be assumed to have participated in a pozzolanic type reaction with the clay. Lime clay-mineral interactions producing cementitious material have been suggested, as previously noted. Inasmuch as freshly formed calcium hydroxide is more reactive than ordinary lime, the formation of cementitious material by a pozzolanic type reaction appears to be likely.

SUMMARY AND CONCLUSIONS

It has been postulated that the hardening of a clay-cement mixture involves alteration of clay mineral structures and amorphous constituents resulting in the formation of secondary cementitious material in addition to the usual hydrolysis and hydration of the cement, which may be regarded as primary stabilizing reactions. The results of mechanical, X-ray diffraction, and chemical tests all tend to support this hypothesis. The most significant results and conclusions that may be drawn from the experimental work are as follows:

1. Very effective stabilization of both kaolinite and montmorillonite was obtained using both normal portland cement and a pure tricalcium silicate compound, as reflected by the compressive strength of specimens soaked for 24 hours after curing for specified periods.
2. Specimens made from neat C_3S paste had only a fraction of the strength of specimens of neat portland cement; however, clay-C_3S specimens were always stronger than clay-portland cement samples, suggesting a modification of the C_3S hydration process in the presence of clay minerals. Hydrated C_3S is the main contributor to the strength of hardened portland cement.
3. X-ray diffraction studies of hydrating clay-cement mixtures showed that:
    a. Portland cement formed calcium hydroxide in hydrating clay-cement.
    b. The hydration of C_3S was modified in the presence of clay.
    c. After 1-week curing no crystalline calcium hydroxide was detectable in clay-cement specimens.
    d. Small decreases in the intensity of kaolinite reflections and large decreases in the intensity of montmorillonite reflections were observed after 12 weeks' curing of clay-cement.
    e. New lines of low intensity were observed in some clay-cement patterns after long curing times, suggesting the formation of new crystalline reaction products.
4. The pH of hydrating neat portland cement, hydrating neat C_3S, and blank clay specimens remained constant with time but decreased markedly during the curing of clay-cement mixtures indicating the existence of an $(OH)^-$ consuming process.
5. Conductivity values of clay-cement extracts indicate a significantly lower electrolyte content than would be expected if the cement hydrated without interaction with the clay, suggesting that in clay-cement the soluble salts formed by cement hydration are converted to less soluble compounds which may serve to bond particles together.
6. The actual extractable calcium contents of all clay-cement mixtures were significantly lower than values computed on the assumption of no clay-cement interaction.
7. All clay-cement specimens contained only a fraction of the free calcium hydroxide that would be present if normal hydration of the cement took place without interaction between cement and clay. This missing calcium hydroxide could have participated in pozzolanic reactions with the clay minerals.
8. Clay-cement cannot be regarded as a simple mixture of hydrated cement particles bonding together unaltered clay particles, but must be considered as a system in which both the clay and the hydrating cement combine through secondary reactions.

These results and conclusions, in conjunction with additional observations during the preparation of extracts for chemical tests, support the following clay-cement structure. Because cement particles are very large relative to clay particles, a clay-cement skeleton and a clay matrix are likely. Units of the skeleton contain a core of hydrated cement gel to which layers of altered clay particles are attached.

Alteration of the clay adjacent to the cement grains will be caused by the dissolution of silica and alumina from both clay particles and amorphous constituents in the high pH environment formed as a result of the generation of highly reactive $Ca(OH)_2$ by the hydrating cement. The dissolved material can combine with calcium ions and form additional cementitious material which bonds adjacent clay particles together. The material in the vicinity of cement grains could form aggregates which trap less altered clay in the intercellular space. In addition, in the case of montmorillonite the altered clay particles lose their expansive characteristics.

Although it is recognized that the stabilization of natural soils may involve the interaction of cement with all the soil components, only the interaction between cement and monomineral clay minerals was studied in this investigation in order to limit the number of variables. Once the behavior of such relatively pure systems is understood, investigation of more complex systems may be undertaken. It is hoped that improved understanding of soil-cement interaction will lead ultimately to improved techniques of soil stabilization.

ACKNOWLEDGMENTS

The authors wish to acknowledge with appreciation the assistance of the following: Alexander Klein, Clarence K. Chan, C. L. Monismith, H. B. Seed, and G. Dierking.

REFERENCES

1. "Alphabetical and Grouped Numerical Index of X-Ray Diffraction Data." ASTM (1950).
2. Anderson, L., "Clay Chemistry in Soil Stabilization." Geologiska Foreningens i Stockholm Förhandlingar, 8:2, 274-297 (1960).
3. Catton, M. D., "Soil-Cement Technology—A Resume." Jour. PCA, Research and Development Laboratories, 4:1 (Jan. 1962).
4. Correns, C. W., "The Experimental Chemical Weathering of Silicates." Clay Minerals Bull. 4:26 (1961).
5. Davidson, D. T., Mateos, M., and Barnes, H. F., "Improvement of Lime Stabilization of Montmorillonitic Clay Soils with Chemical Additives." HRB Bull. 262, 33-50 (1960).
6. Eades, J. L., and Grim, R. E., "Reaction of Hydrated Lime with Pure Clay Minerals in Soil Stabilization." HRB Bull. 262, 51-63 (1960).
7. Eades, J. L., Nichols, F. P. Jr., and Grim, R. E., "Formation of New Minerals with Lime Stabilization as Proven by Field Experiments in Virginia." HRB Bull. 335, 31-39 (1962).
8. Goldberg, I., and Klein, A., "Some Effects of Treating Expansive Clays with Calcium Hydroxide." Proc., 50th Annual Meeting, ASTM, Exchange Phenomena in Soils (1952).
9. Grim, R. E., "Clay Mineralogy." McGraw-Hill (1953).
10. Handy, R. L., "Cementation of Soil Minerals with Portland Cement or Alkalis." HRB Bull. 198, 55-64 (1958).
11. Hashimoto, I., and Jackson, M. L., "Rapid Dissolution of Allophane and Kaolinite-Halloysite after Dehydration." Proc., 7th Conf. on Clays and Clay Minerals. London: Pergamon Press (1960).
12. Heller, L., and Taylor, H. F. W., "Crystallographic Data for the Calcium Silicates." London: HMSO (1956).
13. Herrin, M., and Mitchell, H., "Lime Soil Mixtures." HRB Bull. 304, 99-138 (1961).
14. Hilt, G. H., and Davidson, D. T., "Isolation and Investigation of a Lime Montmorillonite Crystalline Reaction Product." HRB Bull. 304, 51-64 (1961).
15. Ladd, C. C., Moh, Z. C., and Lambe, T. W., "Recent Soil-Lime Research at the Massachusetts Institute of Technology." HRB Bull. 262, 64-85 (1960).
16. Lambe, T. W., Michaels, A. S., and Moh, Z. C., "Improvement of Soil-Cement with Alkali Metal Compounds." HRB Bull. 241, 67-108 (1960).

17. Lea, F. M., and Desch, C. H., "Chemistry of Cement and Concrete." 2nd ed., London: Arnold (1956).
18. Litvinov, I. M., Rzhanitzin, B. A., and Bezruk, V. R., "Stabilization for Soil for Constructional Purposes." Proc., 5th Int. Conf. on Soil Mech. and Foundation Eng., II:775-780, Paris (1961).
19. Polivka, M., Klein, A., and Best, C. H., "Laboratory Preparation of High-Purity Tricalcium Silicate." Materials Research and Standards, 1:7 (1961).
20. Pressler, E. E., Brunauer, S., Kantro, D. L., and Weise, C. H., "Determination of the Free Calcium Hydroxide Contents of Hydrated Portland Cements and Calcium Silicates." Analytical Chemistry, 33:7, 877-882 (1961).
21. Steinhour, H. H., "The Setting of Portland Cement." Bull. 98, PCA Research Dept. (1958).
22. Taylor, H. F. W., "The Chemistry of Cement Hydration." In Progress in Ceramic Science, J. E. Burke, ed., London: Pergamon Press (1961).

Appendix

PROPERTIES OF CEMENT

Chemical (% by wt.)

SiO_2	24.54
Fe_2O_3	2.89
Al_2O_3	2.97
CaO	66.38
MgO	0.36
SO_3	1.93
Ignition loss	0.73
Insoluble residue	0.08
C_3S	54.00
C_2S	29.70
C_3A	3.00
C_4AF	8.80
$CaSO_4$	3.30
Na_2O	0.14
K_2O	0.27

Physical

Surface area	3150 cm^2/gm
Initial set	2 hr 50 min
Final set	5 hr 0 min
Autoclave expansion	-.016%
Compressive strength	
1 day	943 psi
3 days	1,892 psi
7 days	2,220 psi

Effect of Chemicals on Soil-Cement Stabilization

JOAKIM G. LAGUROS, Assistant Professor of Civil Engineering, Ohio University, and
DONALD T. DAVIDSON, Late Professor of Civil Engineering, Iowa State University

The effects of certain chemicals on soil-cement mixtures were quantitatively measured by three tests: unconfined compressive strength, triaxial compressive strength, and Iowa freeze-thaw and wet-dry durability tests. The chemicals were compounds of sodium, calcium and magnesium, and also commercial lime. The eight soils used varied widely in properties due to differences in sampling location, depth, texture, and type of clay mineral.

The 7-day and 28-day cured, 1-day immersed, unconfined compressive strengths of specimens compacted to near standard Proctor density at optimum moisture content indicated that organic top soils benefited from the incorporation of sulfates when the soils were acidic and low in clay content. With increasing clay content and an alkaline environment, the addition of calcium and magnesium ions generally gave high strengths. The B and C horizon clay soils containing cement seemed to respond very favorably to additives of lime, sodium hydroxide, or sodium carbonate, the latter only with soils having near neutral pH.

Stabilized soil mixes that attained a 7-day unconfined compressive strength of 250 psi or more were further evaluated in the Iowa freeze-thaw and wet-dry durability tests. Results verified the strength beneficiation derived from adding the chemicals to soil-cement mixtures. Also, they provided data that suggested the establishment of a functional relationship between the 14-day unconfined compressive strength and the strength at the end of 10 cycles of freeze-thaw.

The triaxial compressive strength test on the natural soil, selected soil-cement, and soil-cement-chemical mixes indicated that for the sandy soil-cement the promising chemicals containing magnesium or sodium ions increased both the cohesion and angle of internal friction. For the clay soil-cement, however, the promising chemicals containing calcium or sodium ions increased the cohesion substantially but decreased the angle of internal friction slightly.

•THE EXPANSION of road nets, with the attendant obligation to use in-place soils with low load-carrying capacity, has forced engineers to look for methods or processes to enhance the effectiveness of cement. Recent research (2, 4, 7, 8, 10, 14) has shown that the incorporation of chemicals or lime in small amounts produces a strength gain in soil-cement. These recent advances have provided the foundation for the leading ideas of this investigation; namely, the extent of strength gain as related to the type of soil, the influence of the chemicals or lime on the cohesion and angle of internal friction of the soil-cement mass, and the effectiveness of chemicals or lime measured in terms of the durability of the stabilized soil.

Paper sponsored by Committee on Soil-Portland Cement Stabilization.

TABLE 1

PROPERTIES OF SOILS

Sample		Parent Material	Soil				Textural Composition^a (%)					Property							Non-Clay Minerals^h	Predom. Clay Mineral^h	Classification	
												Physical			Chemical							
Designation	Location		Group	Series	Horizon	Sampling Depth (in.)	Gravel (>2 mm)	Sand (2-0.074 mm)	Silt (0.074-0.005 mm)	Clay (<0.005 mm)	Clay (<0.002 mm)	LL^b (%)	PL^c (%)	PI (%)	C.E.C.^{d} (meq/100g)	Carbonates^e (%)	pH^f	Organic Matter^g (%)			Textural^i	Engg.^{j} AASHO
Iowa silt	Wayne Co.	Leached, fine-textured Wis.-age loess	Planosol	Edina	A	0-12	0.0	5.5	68.5	26.0	12.0	35	27	8	19.62	0.85	5.3	3.56	Q^k, F^l	M^m	Silty clay loam	A-4(8)
Iowa clay	Calhoun Co.	Surficial sediment derived from Wis.-age drift	Wiesenboden	Webster	A	0-12	0.0	13.5	39.5	47.0	28.0	58	31	27	40.00	1.50	7.5	7.00	Q, F	M	Clay	A-7-5(18)
Wis. sand	Racine Co.	Glacio-fluvial deposit	Gray-Brown Podzolic	Plainfield	A	0-120	0.0	77.0	13.0	10.0	4.0	NP	NP	NP	6.63	0.50	7.0	1.90	Q, F	Chl-M^n	Sandy loam	A-3
Ill. clay	Livingston Co.	Wis.-age glacial till	Humic-Gley	Clarence (Rowe)	C	46-56	0.0	10.0	38.0	52.0	34.0	36	18	18	10.80	22.50	8.3	0.70	Q, F	I^o	Clay	A-6(11)
Texas clay	Harris Co.	Coastal plain deposit largely deltaic	Grumusol	Lake Charles	C	39-144	0.0	3.0	36.0	61.0	40.0	65	18	47	27.30	16.60	8.2	0.13	Q, F	M	Clay	A-7-6(20)
Mich. clay	Ingham Co.	Wis.-age glacial till	Gray-Brown Podzolic	Miami	B	12-36	0.0	14.0	29.0	57.0	35.0	43	25	18	15.06	3.30	6.9	0.85	Q, F	Musc-Ill^p	Clay	A-7-6(12)
N.C. clay	Durham Co.	Triassic sediments	Red-Yellow Podzolic	White Store	B	18-21	0.0	13.0	22.0	65.0	50.0	74	26	48	36.20	0.94	5.4	0.27	Q, F	K-H^q	Clay	A-7-6(20)
Wash. sand	Snohomish Co.	Cemented gravelly till	Brown Podzolic	Alderwood	—	0-120	0.0	75.0	19.0	6.0	0.0	NP	NP	NP	3.32	0.20	6.0	1.80	Q, F	Chl-M	Sandy loam	A-3

[a] Textural gradation tests performed only on soil fraction passing No. 10 sieve, ASTM Method 422-54T.
[b] ASTM Method D423-54T.
[c] ASTM Method D424-54T.
[d] Cation exchange capacity determined by the ammonium acetate (pH = 7) method on soil fraction less than 0.42 mm.
[e] Versenate method for total 1N HCl soluble calcium.
[f] Glass electrode method using suspension of 15-g soil in 30-cc distilled water.
[g] Potassium dichromate method.
[h] X-ray diffraction analysis.
[i] Triangular chart, U. S. Bureau of Public Roads.
[j] AASHO Method M145-491.
[k] Q = quartz.
[l] F = feldspar.
[m] M = montmorillonite.
[n] Chl-M = chlorite-montmorillonite interlayer.
[o] I = illite.
[p] Musc-Ill = muscovite-illite interlayer.
[q] K-H = kaolinite-halloysite intermediate.

MATERIALS

Soils

The eight soils used in this investigation were so selected as to have a wide range of properties, which accrue not only from variation in sampling depth and particle size distribution, but also from differences in clay mineral type. Table 1 gives property information on the eight soils that were sampled from various parts of the country and had already been classified as difficult to stabilize or problem soils.

Cement

Type I portland cement was used throughout this study.

Hydrated Lime

Two types of commercial hydrated lime were used. One was hydrated high calcium, lime A, and the other was monohydrated dolomitic, lime B.

Chemicals

The selection of chemical compounds (Table 2) was dictated primarily by the consideration that there is a similarity between these compounds and the product resulting from the combination of cement and soil, and secondarily by the research findings of earlier studies (2, 7, 8, 10).

Preparation of Mixes

A measured amount of pulverized air-dry soil passing the No. 10 U.S. standard sieve was hand mixed with a measured amount of cement. The dry mixture was placed in the mixing bowl, some of the compaction water which had been calculated to give near standard Proctor density was added; the mixture was hand mixed again; and then it was mixed in a kitchen mixer, Hobart Model C-100, at low speed, for 1 minute. Following this, the rest of the compaction water, in which a measured amount of the chemical compound had been dissolved or dispersed, was added, hand mixed, and machine mixed for another minute. To insure uniform distribution, the bowl was scraped with a trowel, the mixture was hand mixed and finally machine mixed for an additional minute.

The amount of molding water used was that which gave maximum density and not maximum strength (Table 3) since the

TABLE 2

CHEMICALS USED

Chemical	Formula	Source
Sodium hydroxide	$NaOH$	—[a]
Calcium sulfate	$CaSO_4$	Reagent grade
Magnesium sulfate	$MgSO_4 \cdot 7H_2O$	Analytical reagent
Sodium sulfate	Na_2SO_4	Reagent grade
Calcium chloride	$CaCl_2$	—[a]
Magnesium oxide	MgO (C-1-60)	—[a]
Sodium orthosilicate	Na_4SiO_4	—[a]
Sodium carbonate	Na_2CO_3	—[a]

[a] Supplied by the Dow Chemical Co.

differences between the two molding water contents were very small.

Molding

When mixing was completed, the bowl was covered with a damp cloth to prevent evaporation. In a drop hammer molding apparatus, 2-in. diameter by 2-in. high specimens were molded to near standard Proctor density.

Curing

Immediately after weighing and measuring, the specimens were wrapped in waxed paper and sealed with Scotch tape to prevent loss of moisture and were cured in a chamber at 95 ± 5 percent humidity and 70 ± 5 F temperature. Two curing periods were used—7 days and 28 days.

Compressive Strength Testing

At the end of the specified curing period, the specimens were removed from the curing room, unwrapped, and immersed in distilled water for 24 hours following which they were tested to failure to determine their unconfined compressive strength.

METHODS OF EVALUATION

Preliminary Investigation

In the first phase of the investigation the 7-day and 28-day cured, 1-day immersed, unconfined compressive strengths of soil-cement specimens to which various amounts of a chemical had been added were determined. Although the 7-day strength was adopted as the criterion for successful stabilization, the 28-day strength provided not only an additional check, but also gave an indication of the strength potentiality of the mix and some measure of the rate of strength change with time. The cement contents used were 4, 8, and 12 percent; the chemical contents not more than 3 percent, the lime contents 1 and 3 percent, all based on the oven-dry weight of the soil.

From these strength data, the economically lowest optimum amount of chemical was chosen. In some mixtures, maximum strength was obtained with slightly varying amounts of chemicals for the three cement contents but the same soil. Where the variation was small, the adoption of one amount of chemical for all three cement contents with the same soil seemed justifiable.

Durability Tests

All stabilized mixtures which gave a 7-day cured, 1-day immersed strength of 250 psi or higher were further evaluated by the freeze-thaw and the wet-dry durability tests.

The procedure for the freeze-thaw test used in this work was developed in the Iowa Engineering Experiment Station Laboratory (6) to suit the climate in Iowa. (Apparatus is shown in Figs. 13, 17.) Two specimens, 2 in. in diameter by 2 in. high, were molded from the mixture to be evaluated and were moist cured for seven days. At the end of this curing period, the top of each specimen was sealed with a coat or resin-base paint (Plax) before the specimens were immersed in water for 24 hours. One specimen was removed from the water and

TABLE 3

OPTIMUM MOISTURE-MAXIMUM DRY DENSITY VALUES OF SOIL-CEMENT MIXES

Soil Type	Optimum Moisture Content (%)	Maximum Dry Density (pcf)
Iowa silt	22.5	95.0
Iowa clay	24.8	95.1
Wisconsin sand	17.0	100.5
Illinois clay	18.0	111.4
Texas clay	22.6	102.5
Michigan clay	19.5	105.5
North Carolina clay	25.7	97.2
Washington sand	12.0	122.0

placed in the specimen holder. The assembly was placed in a vacuum flask containing sufficient distilled water, at a temperature of 34 ± 1.6 F, to immerse the bottom ¼ in. of the specimen. The vacuum flask with the specimen in it was stored for 16 hours in a refrigerator, maintained at a temperature of 20 ± 2 F. Then the flask was removed and thawed for eight hours at a temperature of 77 ± 4 F. This was one cycle of freeze-thaw; the specimen was subjected to 10 cycles. At the beginning of the cycle, at the end of the freezing period and at the end of the thawing period, the height of the specimen was measured to calculate the amount of heave. The specimen was weighed at the beginning and at the end of the freeze-thaw cycles. The amount of moisture absorbed during the test could be determined from these weights. At the completion of the freeze-thaw cycles, the specimen was removed from the vacuum flask and specimen holder and tested for unconfined compressive strength. The other specimen called the "control" specimen, was left in water through all the freeze-thaw cycles. Its unconfined compressive strength was determined at the same time as that of the first specimen. The control specimen was weighed both at the end of the moist curing period and of the immersion period. The index of resistance (in percent) to the effect of freezing R_f was calculated by

$$R_f = \frac{p_f}{p_{c_f}} 100 \tag{1}$$

in which

p_f = unconfined compressive strength of freeze-thaw specimen, psi, and
p_{c_f} = unconfined compressive strength of control specimen, psi.

The preparation of two identical specimens for the wet-dry test was the same as for the freeze-thaw test; in fact, all four specimens were prepared from the same batch. One of the specimens was moist cured for 7 days, then it was immersed for 7 days. The control was moist cured for 14 days. At the end of this period, the two specimens were tested for unconfined compressive strength. The resistance to the effect of immersion was calculated from

$$R_i = \frac{p_i}{p_{c_i}} 100 \tag{2}$$

in which

p_i = unconfined compressive strength of immersed specimen, psi, and
p_{c_i} = unconfined compressive strength of control specimen, psi.

On the basis of the 7-day cured, 1-day immersed unconfined compressive strength and of the resistance to freeze-thaw and wet-dry tests, it was possible to select the best cement-chemical mix for each soil for further evaluation by the standard freeze-thaw test, ASTM Designation D560-57 (1, p, 1,182).

Triaxial Compression Tests

For each soil three different mix batches were prepared as explained previously. The first was the raw soil at its optimum moisture content for standard Proctor density; the second was a combination of soil and an amount of cement equal to that used in the third batch; the third was that combination of soil, cement, and chemical which gave the best mix based on 7-day strength and resistance to freeze-thaw.

From each batch 9 cylindrical specimens, 1.312 in. diameter by 2.816 in. high, were molded using the Harvard miniature compaction apparatus, which gives a compacted sample at approximately standard Proctor density. Samples were molded in 5 layers with 5 tamps per layer for sandy soils and 8 per layer for clayey soils.

All specimens were moist cured for 7 days in the same manner as the strength specimens.

At the end of the curing period the specimens (3 for each of the 3 lateral pressures of 10, 20 and 30 psi) were tested in a triaxial compression machine. The cohesion and angle of internal friction of any particular mix were determined graphically using the Coulomb-Mohr theory defined by

$$s = c + p \tan\phi \tag{3}$$

UNCONFINED COMPRESSIVE STRENGTH OF SOIL-CEMENT MIXTURES

Iowa Silt

The test results indicate that 4 percent cement added to Iowa silt produces low strength mixtures, whose strength cannot be increased by any of the chemicals incorporated in the mix (Fig. 1). At cement contents above 4 percent, the sulfates of sodium, calcium, and magnesium increased the 7-day strength. The 28-day strength data show the same trend. Although the sulfate data contradict the destructive effect of sulfates on concrete and clays stabilized with cement (13), they are in agreement with reported results on sandy soils and especially with the effectiveness of sodium sulfate (5). Paradoxically, no beneficiation is evident when either magnesium oxide, calcium chloride, sodium hydroxide, or limes are used. This would at first seem inconsistent with the expected

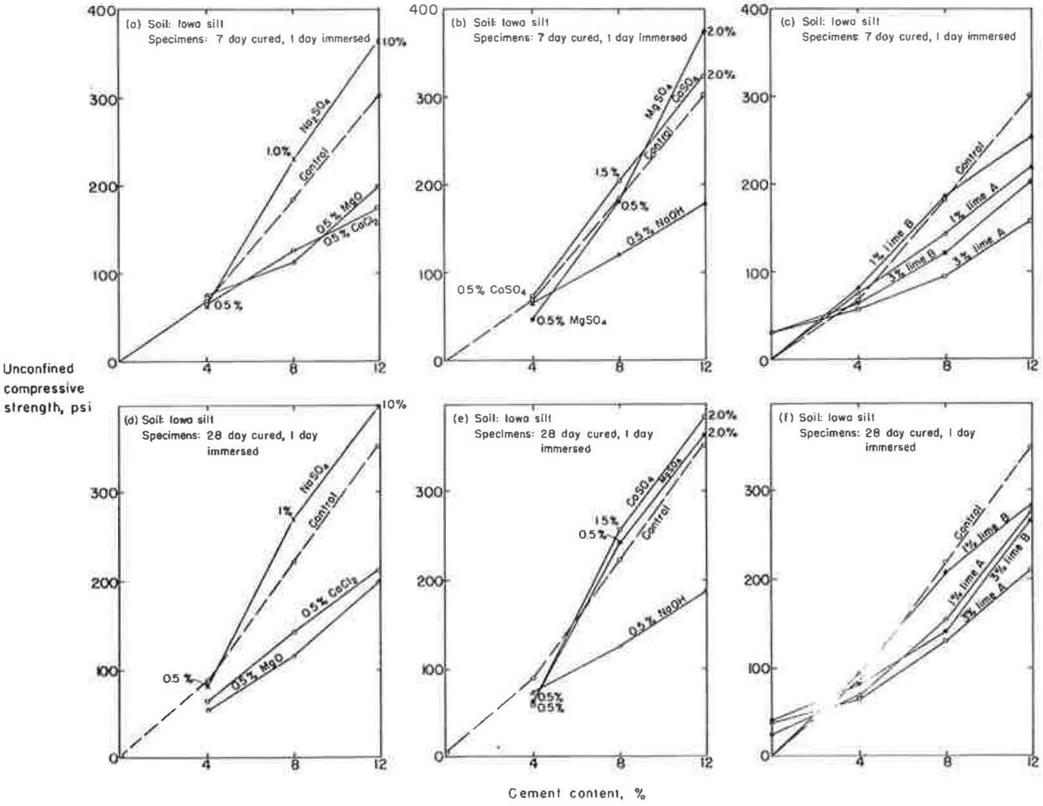


Figure 1. Unconfined compressive strengths of Iowa montmorillonitic, A horizon silt stabilized with optimum amount of chemical or hydrated lime at varying cement contents; lime A = $Ca(OH)_2$, lime B = $Ca(OH)_2$ + MgO.

stabilizing effect of magnesium, sodium, and calcium ions. The inconsistency may be traced to the type of compound which contains these ions and its reaction with the high organic matter content of the soil. An explanation may be based on the hypothesis that the basic compounds, such as sodium hydroxide and lime, increase the solubility of organic complexes with attendant uniform distribution through the water in the mix and interfere with the formation of silica gel. By decreasing the solubility, the sulfates reduce the interference of the organic complexes, the soil-cement reaction proceeds normally, and high strengths are attained.

A similar interpretation may be proposed for the ineffectiveness of the calcium chloride salt, which is supposed to have a catalytic effect on cement. Although the addition of calcium ions increases the rate of hydrolysis of the compounds in the clinker of cement, and thereby the transformation of the coagulated silicates and aluminates into crystalline bodies is enhanced and strength is attained, the organic complexes in Iowa silt seem to prevent this reaction. Inasmuch as the addition of 0.5 percent calcium chloride imparts relatively higher strength to the Iowa silt-cement mix than 1.5 percent, the theory that high concentrations of calcium chloride overcome the deleterious presence of the organic matter cannot be advanced.

The ineffectiveness of magnesium oxide seems to be derived from a poor solubility condition. Examination of the broken specimens after testing revealed distinct magnesium oxide agglomerations distributed throughout the specimen, an indication that magnesium oxide did not become part of the soil-cement structure.

Iowa Montmorillonite Clay

All the chemicals and limes used with mixes of Iowa clay-cement contributed to strength increase, indicating that they speeded up soil-cement reaction at all cement contents used (Fig. 2).

In comparison with the sulfates used, calcium chloride and magnesium oxide were very beneficial. This is in contrast with the results obtained with the organic Iowa silt soil. Although the two soils, Iowa silt and Iowa clay, are both top soils and highly organic, they respond differently to chemical stabilization. This difference may be due in part to their alkalinity or acidity. The Iowa silt is well leached and highly acid with a pH value of 5.3. The Iowa clay is not so well leached and is slightly alkaline, as indicated by a pH equal to 7.5. The low pH value might have caused a precipitation of a gel over the cement particles, thus delaying and possibly prohibiting the hydration of cement. The difference between the behavior of the two soils may also be explained by one's being silty and the other clayey. Although not fully elucidated for all chemicals, recent research (4) indicates that cement stabilization of friable loess does not benefit from the addition of lime; plastic loess does benefit.

Wisconsin Sand

Besides being organic, the nonplastic Wisconsin sand displays uniformity of particle size and relative absence of binder material. These properties make it very difficult to stabilize (Figs. 3 and 4). Cement contents up to 16 percent impart very little strength. Since molding and extrusion of specimens were difficult and specimens crumbled when handled immediately after compaction, it may be concluded that cement failed to give cohesive property to the soil. Except for magnesium sulfate, the other chemicals and lime in combination with cement did not contribute to the strength of the resulting mixture either. This supports the contention of the effectiveness of sulfates. The addition of sodium ions in the form of hydroxide, silicate, or carbonate was also ineffective. This leads to the conclusion that the formation of the rather stable Si-O-Na groups was prevented, possibly because the sodium ions preferentially attached themselves to the organic complexes.

The high strengths obtained with cement and magnesium sulfate merited further investigation. Combinations of 8, 12, and 16 percent cement with four different magnesium sulfate contents, the highest being 3 percent magnesium sulfate, revealed that 2 percent magnesium sulfate may be considered optimum (Fig. 4). The unique effectiveness of magnesium sulfate may be attributed to two factors: the incorporation of

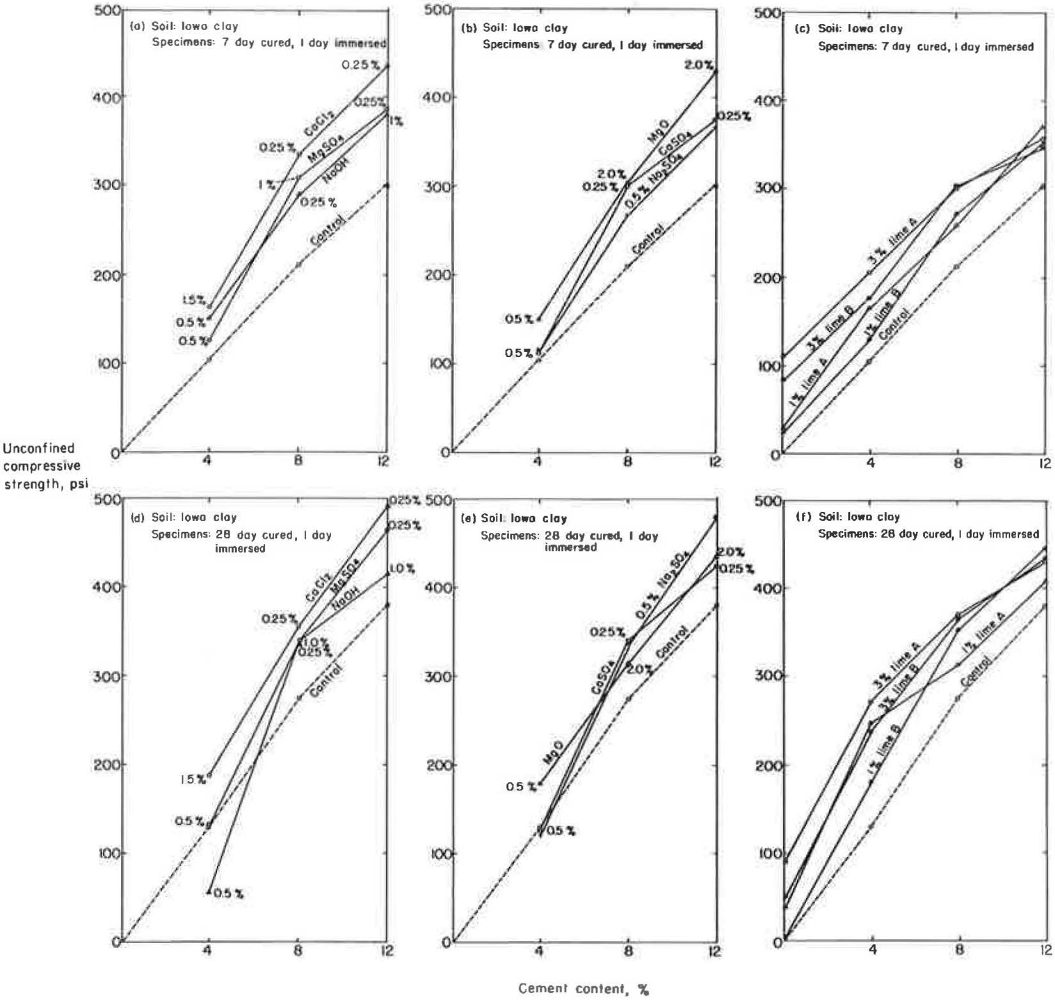


Figure 2. Unconfined compressive strengths of Iowa montmorillonitic, A horizon clay stabilized with optimum amount of chemical or hydrated lime at varying cement contents.

sulfate, the small ionic radius (0.78 Å) of magnesium as compared with the large ionic radii of sodium (0.98 Å) and calcium (0.99 Å).

Illinois Illite Clay

Although as low as 4 percent cement imparted a strength slightly higher than the minimum acceptable, 250 psi, the possibility of improving the mixture of Illinois clay-cement was investigated by incorporating some chemicals or lime (Fig. 5).

Sodium hydroxide and lime are the only additives which upgrade the quality of the Illinois clay-cement mixtures; the other chemicals investigated seem to have a destructive effect. Treatments with the silicate, carbonate and sulfate of sodium, and with the sulfate and chloride of calcium did not benefit the Illinois clay-cement mixtures, bearing out the thesis that the benefit derived from sodium hydroxide and lime is not due primarily to the addition of sodium and calcium ions. Rather, it seems to be because these ions are added in the form of hydroxide, which attacks the cement and produces an abundance of gel that eventually binds the soil particles. At the same time an interaction takes place between the silica surfaces of the quartz in the soil and the hydroxides, similar to the reaction between a weak acid and a strong base, assuming that the

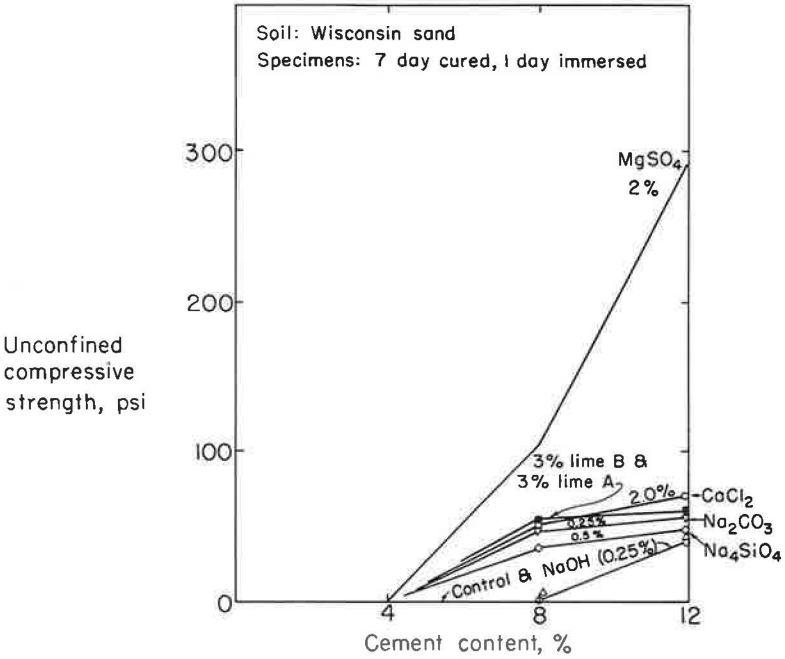


Figure 3. Unconfined compressive strengths of Wisconsin A-horizon pozzolanic sand stabilized with optimum amount of chemical or hydrated lime at varying cement contents.

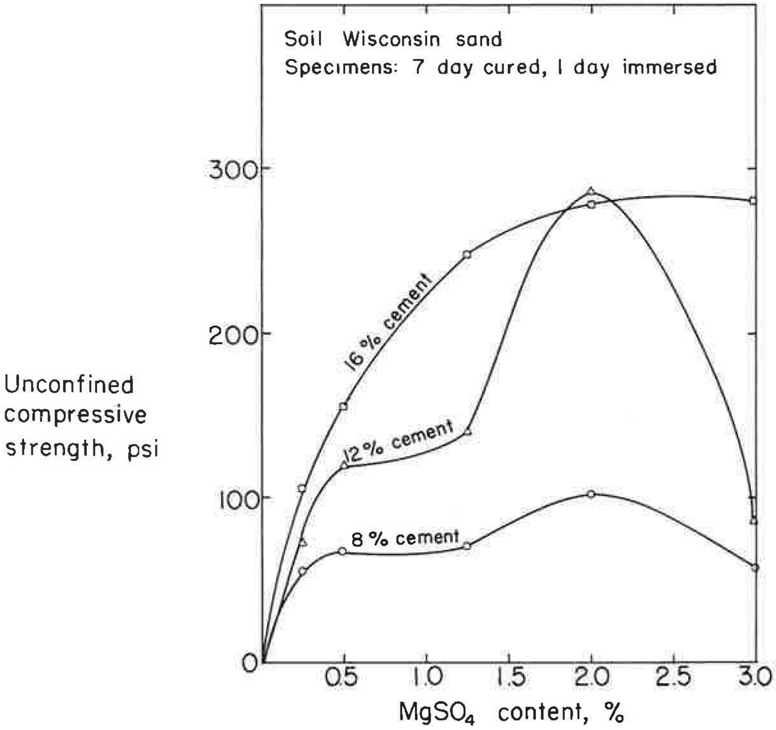


Figure 4. Effect of $MgSO_4$ concentration on the strength of Wisconsin sand stabilized with cement.

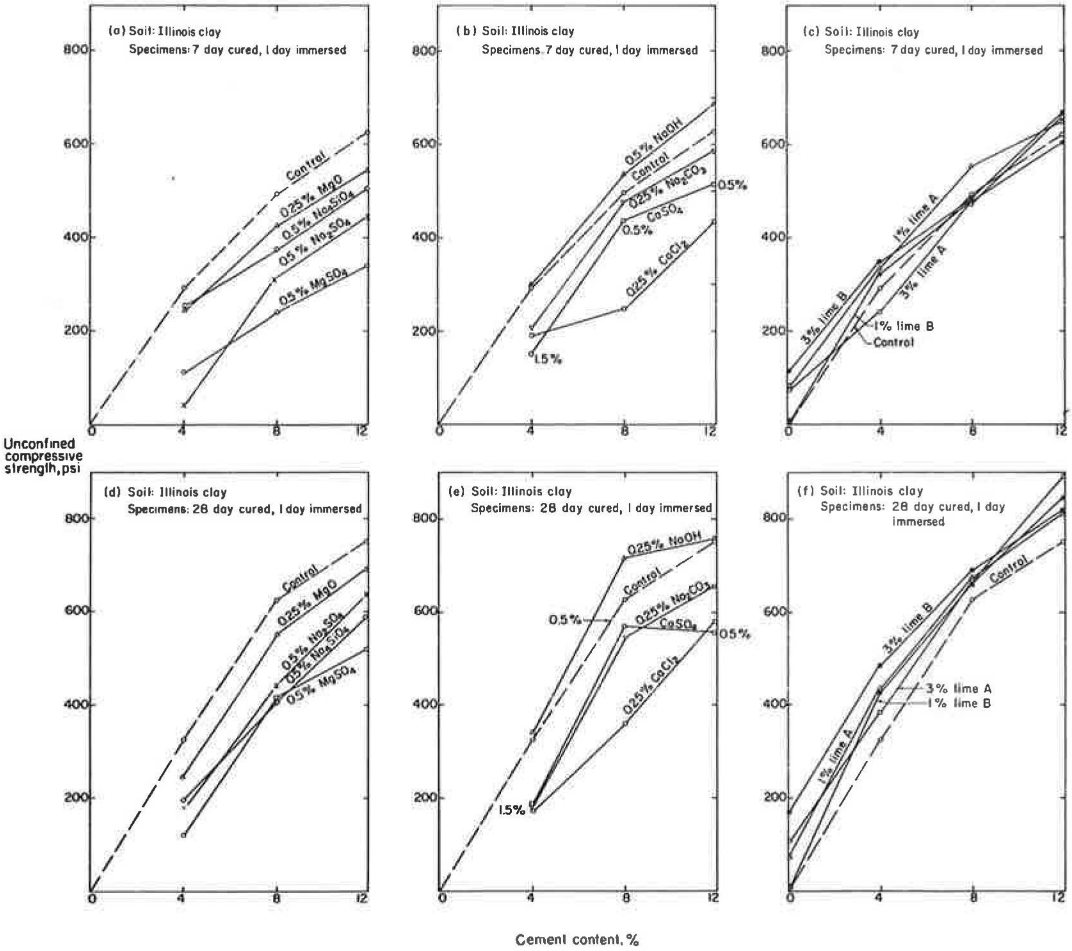


Figure 5. Unconfined compressive strengths of Illinois illitic clay stabilized with optimum amount of chemical or hydrated lime at varying cement contents.

silica surfaces and quartz are at least partly covered with acidic SiOH groups (9). This reaction may be presented by

$$-\overset{|}{\underset{|}{\mathrm{Si}}}\mathrm{OH} + \mathrm{M(OH)} \rightarrow (-\overset{|}{\underset{|}{\mathrm{Si}}}\mathrm{O})\mathrm{M} + \mathrm{H_2O}$$

in which M stands either for the sodium or calcium ion. Thus, the effects of these two chemicals are centered both in the clay fraction and the quartz surfaces.

Considering that the exchange capacity of the Illinois clay is only 10.8 meq/100g, it would be expected that addition of small amounts of the two effective chemicals would suffice for imparting high strength to Illinois clay-cement mixtures. The optimum amount for sodium hydroxide is 0.25 percent and for lime A (high calcium lime), 1 percent. When 3 percent lime B (dolomitic lime) was used, slightly higher strengths were obtained than with 1 percent. Excess of lime would be deposited as a separate crystalline solid phase (15). Mixtures containing 1.5 percent sodium hydroxide were weaker than those with 0.25 percent and weaker than the control Illinois clay-cement mixtures. It seems possible that the presence of excess cations on the mineral surface causes swelling in the presence of water as these cations tend to dissociate.

Texas Montmorillonite Clay

The response of mixtures of Texas clay-cement to chemical treatments differs slightly from Illinois clay-cement primarily because Texas clay is a heavy clay. The amount of clay-size material is higher, and the predominant clay mineral is montmorillonite; therefore, its exchange capacity is higher. Sodium hydroxide was beneficial at 12 percent cement and at 8 percent cement, the latter only after 28 days curing (Fig. 6) indicating a slower rate of reaction. Although calcitic lime A increased the strength of Texas clay-cement mixtures when used in amounts of 1 percent, it gave higher strengths at 3 percent. Again, the optimum amount of dolomitic lime B was 3 percent.

Of the other sodium compounds used, the orthosilicate improved the strengths when incorporated into mixes containing 8 and 12 percent cement; the carbonate proved effective only in those mixes containing 12 percent cement. The possibility cannot be excluded that these two sodium compounds were effective with Texas but not with Illinois clay because the fineness of the Texas soil provided more available surface area and hence a more extensive chemical reaction. The same reason may also be used to explain the slight benefit derived from mixing calcium chloride with the Texas clay-cement.

Michigan Muscovite-Illite Clay

Figure 7 shows that the mixtures of Michigan clay-cement have an unusual affinity for the chemicals studied. Although the strength improvement from some chemicals

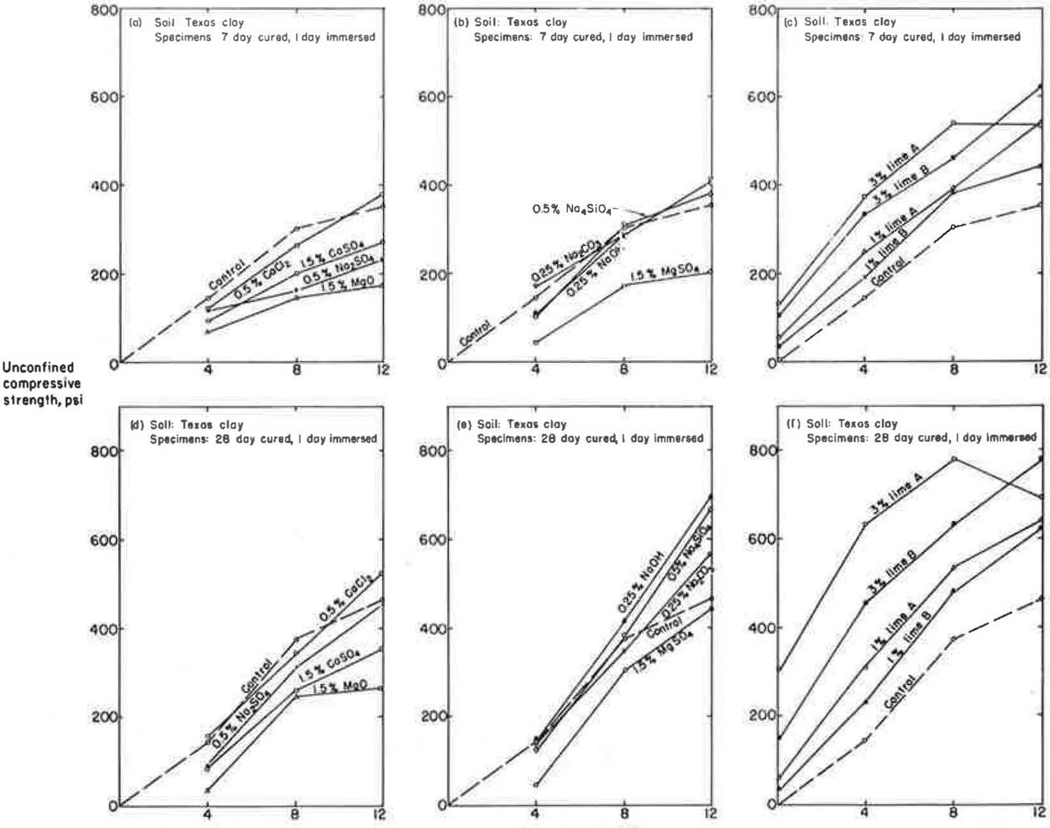


Figure 6. Unconfined compressive strengths of Texas montmorillonitic clay stabilized with optimum amount of chemical or hydrated lime at varying cement contents.

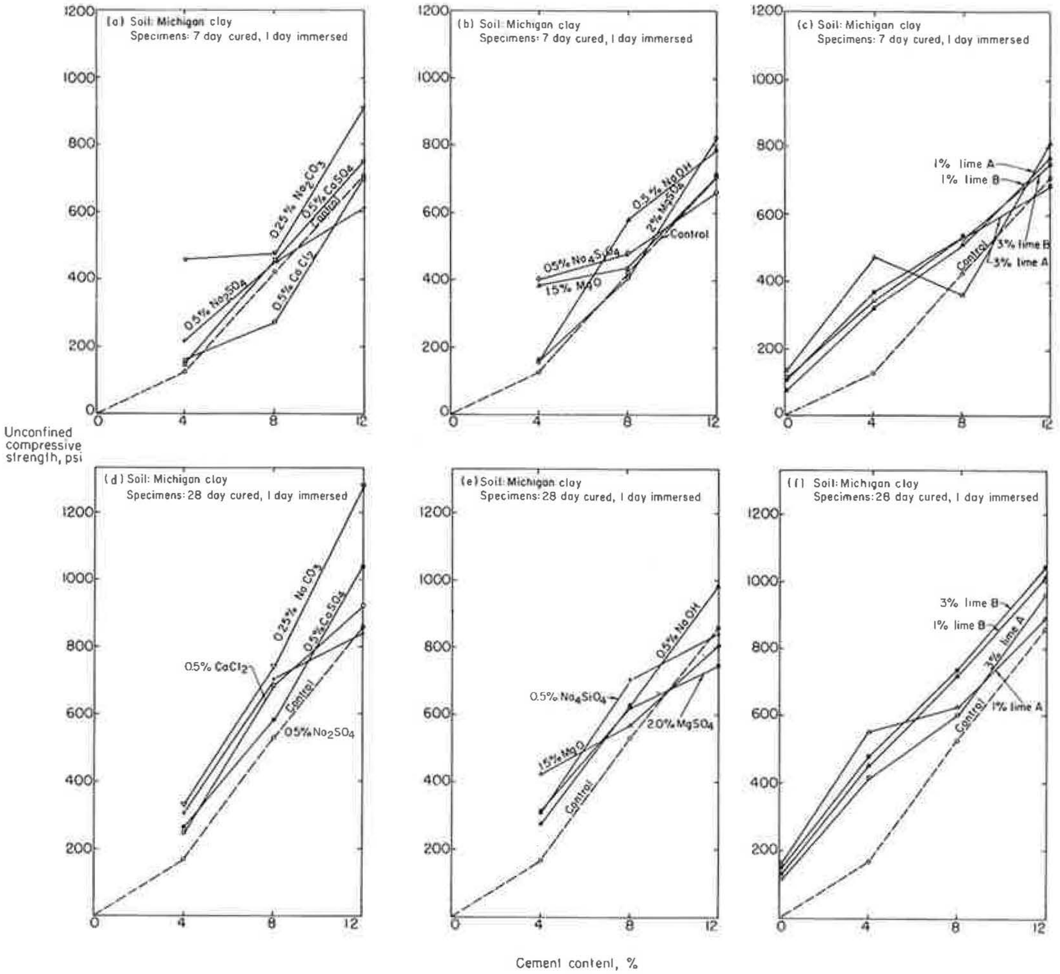


Figure 7. Unconfined compressive strengths of Michigan muscovite-illite clay stabilized with optimum amount of chemical or hydrated lime at varying cement contents.

may be called erratic, as were the benefits from calcium chloride because of the lack of a definite trend in strength increase, for other chemicals (sodium hydroxide and carbonite) and lime the improvement of the Michigan clay-cement mixtures is most notable. The validity of the suggestion that a low exchange capacity of the soil requires a low concentration of chemical additive to obtain optimum strength conditions is proved by the results with Michigan clay. For sodium hydroxide, calcium sulfate, and sodium carbonate, the optimum amounts were less than 0.5 percent and for lime 1 percent.

Addition of sodium carbonate to Michigan clay-cement mixtures offers a good possibility of producing extreme stability. This stability results from interaction between the calcium of cement and the sodium carbonate, and the subsequent formation of a skeletal calcium carbonate in the soil mass. Also, the release of sodium hydroxide acts in a way to accelerate the soil-cement reaction. There is fairly strong evidence (11) for this explanation. A point of interest emerges from comparing the sodium carbonate treatment of Michigan clay-cement with other clay-cement mixtures, as the latter show a marked decrease in strength. The explanation involves the assumption that the difference in response to sodium carbonate lies in the acidity of the soils. Michigan clay is a nearly neutral soil (pH = 6.9); the other soils are either acid ($pH < 6$) or alkaline ($pH > 8$).

North Carolina Kaolinite-Halloysite Clay

The type clay mineral (kaolinite-halloysite intermediate) in the North Carolina clay would seem to indicate that the addition of cement would impart high strength. But the high clay content (67 percent) and the high exchange capacity restrict the development of high strength so that a barely acceptable strength of 284 psi is attained with 12 percent cement (Fig. 8). On the basis of the simplified explanations given for other clay soils, a secondary function of the optimum 3 percent lime admixture seems to produce an electrical neutrality on the mineral surfaces of the soil particles—a neutrality not attained by the insufficient amount of cement originally added.

The similarity of strength results, between the North Carolina clay-cement containing 1.5 percent sodium hydroxide, and that with 3 percent lime, helps explain the anomalous behavior of various sodium compounds and calcium compounds under acid conditions such as in the North Carolina clay (pH = 5.4). As long as the environment is acidic, the precipitation of calcium aluminates and silicates from the cement cannot take place fully because the cement-water solution does not reach saturation. However, the addition of either the sodium or calcium ions in the form of hydroxide produces near neutral solutions that promote the formation of the cementing gel. A somewhat less refined explanation and more remote possibility is the adsorption of hydroxyl ions from the alkaline medium (supplied by the sodium hydroxide and calcium hydroxide of the lime) onto the oxygens of the silica layer by dipolar bonds.

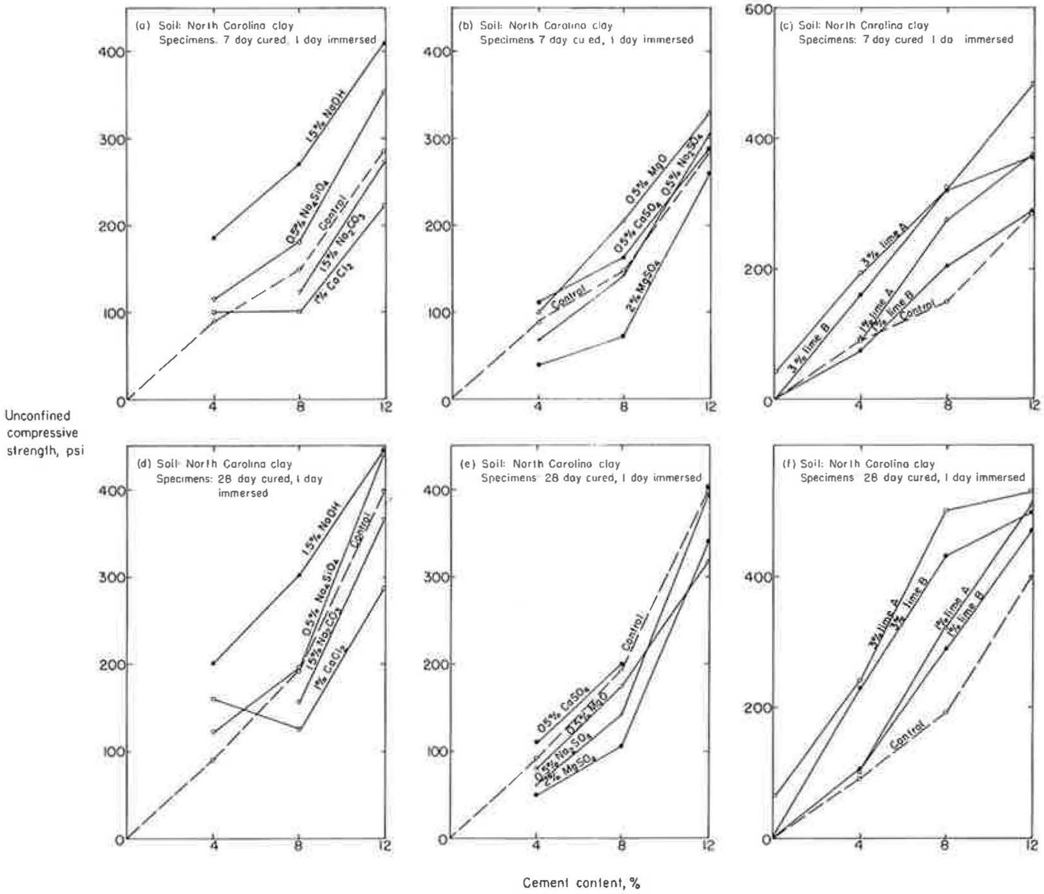


Figure 8. Unconfined compressive strengths of North Carolina kaolinite-halloysite clay stabilized with optimum amount of chemical or hydrated lime at varying cement contents.

Washington Sand

The high 7-day strengths and the marked difference in 28-day strengths (Fig. 9) between mixtures of Washington sand containing 4, 8, and 12 percent cement are indicative of the concrete-like nature of the mixtures.

That the optimum amount of lime was 3 percent instead of 1 percent shows two separate reactions, cement and sand. This, of course, does not exclude the possibility of a cement-lime synergism, typically occurring in concrete. The assumption that the product resulting from mixing Washington sand with cement is similar to concrete is further substantiated by examining qualitatively the effect of chlorides, hydroxides, and sulfates.

As is generally recognized, alkali chlorides, hydroxides, and sulfates are deleterious to concrete. In areas where, for economic reasons, concrete is made of deleteriously alkali-reactive aggregates the use of a pozzolan has become established practice.

Optimum conditions with the three types of compounds were reached at 0.5 percent for all cement contents. When 1.5 percent of the chemicals was used, the strength decreased. To account for increase in strength when supposedly deleterious chemicals are used the following is offered as an explanation. The extremely small amount of clay (2.5 percent) might have taken up the role of the pozzolan to offset the effect of the sodium and calcium compounds.

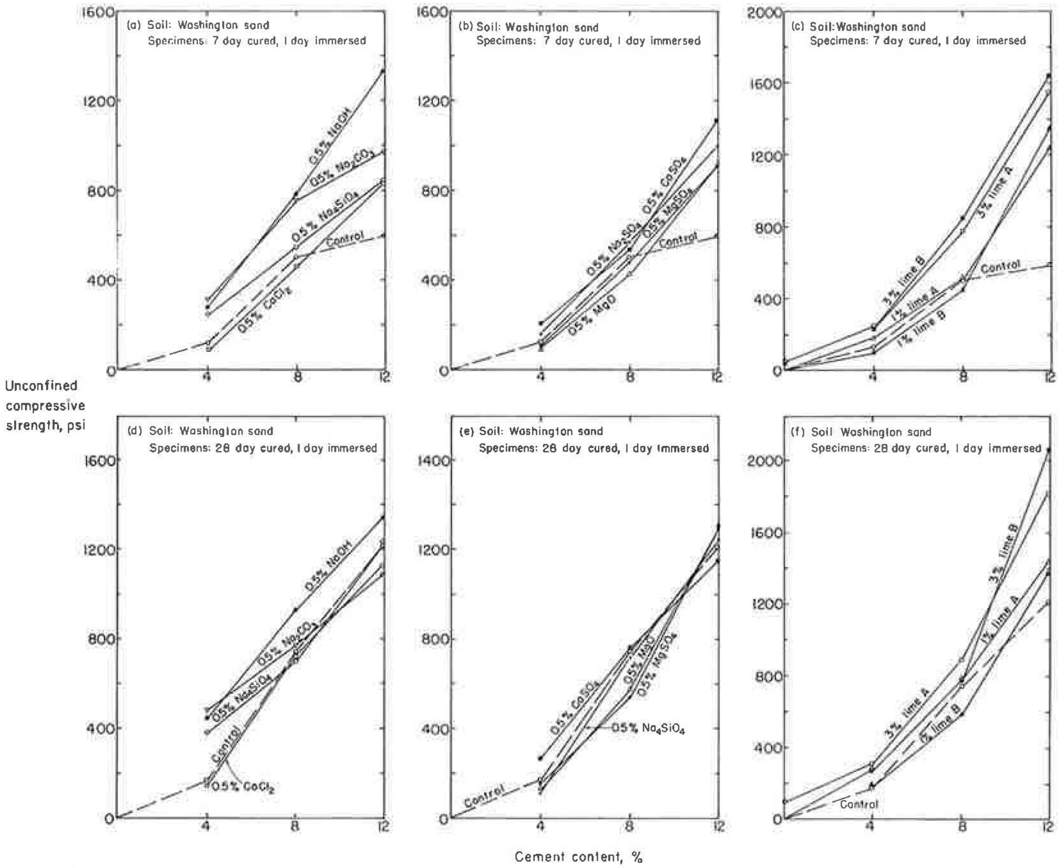


Figure 9. Unconfined compressive strengths of Washington sand stabilized with optimum amount of chemical or hydrated lime at varying cement contents.

DURABILITY TESTS

One of the most important observations regarding the performance of highways is the loss of strength of the base course and especially of the subgrade during spring thaws. There seems no doubt that strength loss originates because of the excessive amount of water absorbed by the soil mass either during a very wet season or during the freezing period. In the latter case the water changes to ice and then reverts to water during the thawing period. Therefore, it may be said that at its in-place moisture content the strength of the soil mass is reduced because its elastic character is partly lost or the soil reaches or tends to reach its plastic state.

The durability indices, R_f and R_i, and the unconfined compressive strengths (Appendix) show how the stabilized soils vary due to weather conditions. For the sake of simplicity in terminology R_f will be referred to as freezing index; it denotes the index of resistance to the effect of freezing, and it is not related to the cumulative degree-day plot (16, p. 126).

Freeze-Thaw Tests

Figures 10 and 11 plot p_{c_i} against p_f values for the stabilized soils. Although a quantitative evaluation of the resistance offered by the stabilized soil to the destructive influence of water or ice is given, the relationships established or deduced can be interpreted only as a general tendency of the particular soil to behave as it did. No attempt has been made to treat the data statistically because the experiment was not planned as such and the data are meager for such a treatment.

From the plots of p_{c_i} versus p_f an algebraic relation may be established

$$p_f = \frac{p_{c_i} - b}{m} + 250 \tag{4}$$

in which

p_f = unconfined compressive strength, psi, of specimen cured 7 days and subjected to 10 cycles of freeze-thaw;
p_{c_i} = unconfined compressive strength, psi, of the 14-day cured specimen;
b = the p_{c_i}-intercept at $p_f = 250$, psi; and
m = the slope of the p_{c_i}-p_f line.

This, of course, leads to the establishment of three different functional relationships, a different equation for each cement content (4, 8 and 12 percent). On the other hand, there is the establishment of an overall equation, irrespective of the variations in cement content. The equation may be called the "durability conversion equation." Its practical importance lies in obtaining from the 14-day strength the expected strength loss from freeze-thaw. Thus the same information is obtained as from the freeze-thaw test plus a simplification in equipment and technique. To avoid confusion between the p_f and p_{c_i} as used in the durability tests and in the 14-day strength determinations, the equation may be written

$$q_f = \frac{q_{14} - b}{m} + 250 \tag{5}$$

in which

q_f = expected strength after 10 cycles of freeze-thaw, psi; and
q_{14} = unconfined compressive strength, psi, of 14-day cured stabilized soil specimen.

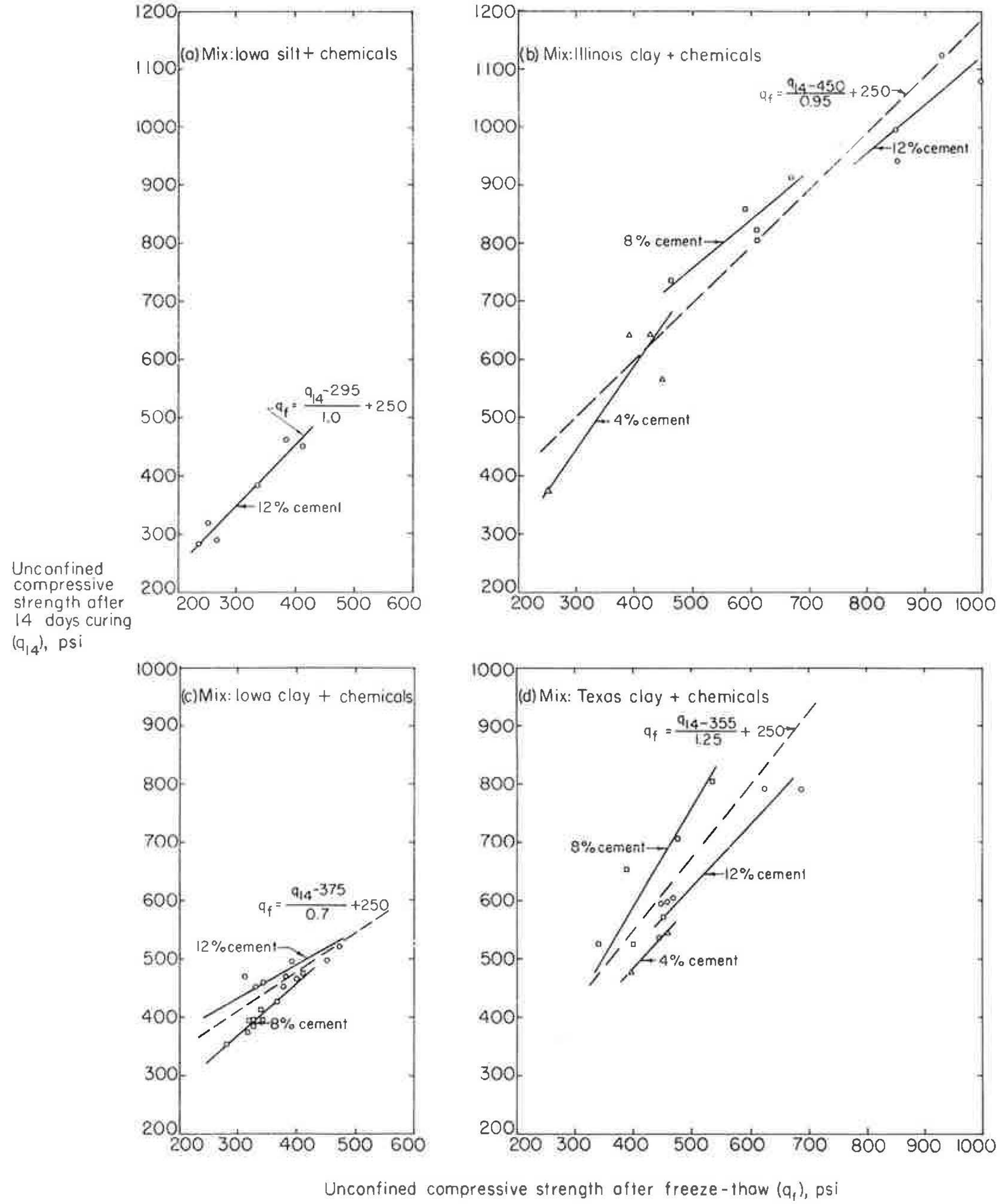


Figure 10. Strength-durability variations in soils stabilized with optimum amount of chemical at varying cement contents.

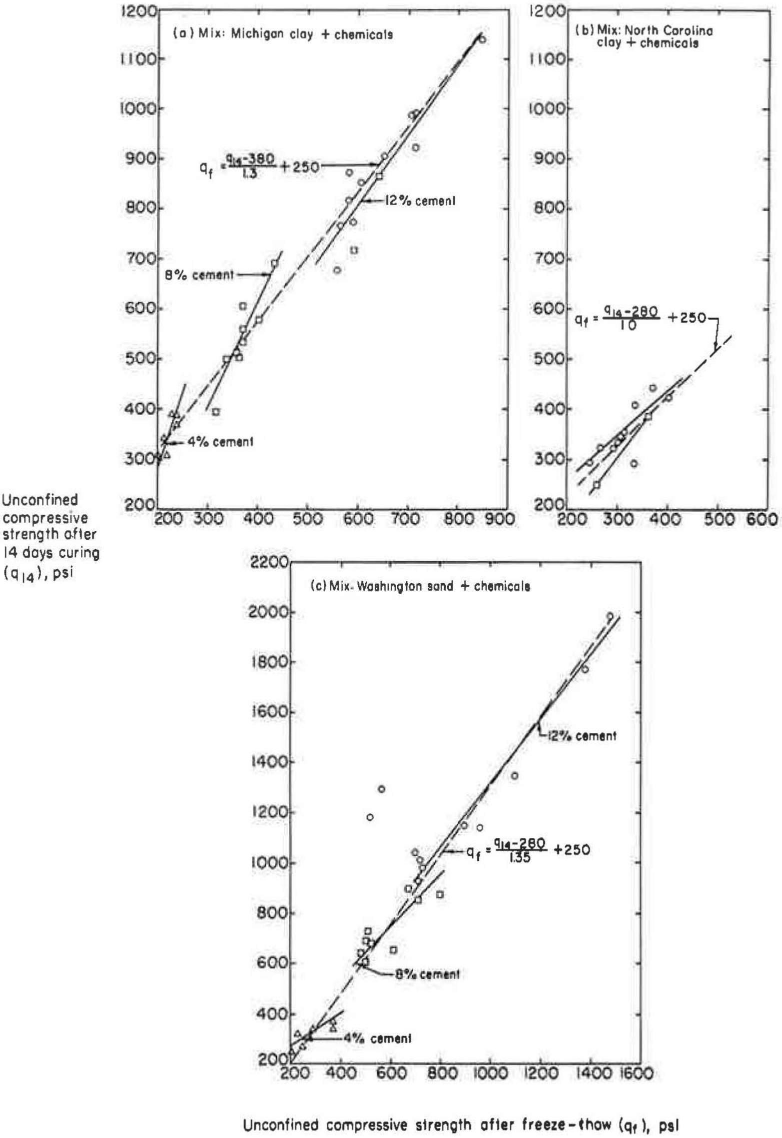


Figure 11. Strength-durability variations in soils stabilized with optimum amount of chemical at varying cement contents.

The equations in Table 4 may give rise to criticisms. Perhaps the most common will be that q_f is a strength value measured 18 days after molding the specimens, while q_{14} represents the strength 14 days after molding. In providing a satisfactory answer, it should be emphasized that the reason for establishing a relationship between freeze-thaw strength values and n-day strength values is to eliminate, if possible, the freeze-thaw test and to develop a means for predicting freeze-thaw reduction in strength.

Another point which needs clarification concerns durability indices less than the minimum acceptable value of 80 percent. When any of the p_f, p_{c_i} values are less than the minimum 250 psi, the soil is not adequately stabilized and that particular type of mix should not be used or should be improved. When the values surpass the minimum limit of 250 psi but the durability index is less than 80 percent, then the design value

TABLE 4
DURABILITY CONVERSION EQUATIONS

Stabilized soil type	Cement content, %	$q_f = \frac{q_{14} - b}{m} + 250$	Overall equation
Iowa silt	12	$q_f = \frac{q_{14} - 295}{1.0} + 250$	----
Iowa clay	8	$q_f = \frac{q_{14} - 325}{0.9} + 250$	
	12	$q_f = \frac{q_{14} - 405}{0.55} + 250$	$q_f = \frac{q_{14} - 375}{0.7} + 250$
Illinois clay	4	$q_f = \frac{q_{14} - 375}{1.45} + 250$	
	8	$q_f = \frac{q_{14} - 545}{0.8} + 250$	
	12	$q_f = \frac{q_{14} - 490}{0.8} + 250$	$q_f = \frac{q_{14} - 450}{0.95} + 250$
Texas clay	4	$q_f = \frac{q_{14} - 315}{1.0} + 250$	
	8	$q_f = \frac{q_{14} - 330}{1.7} + 250$	
	12	$q_f = \frac{q_{14} - 350}{1.0} + 250$	$q_f = \frac{q_{14} - 355}{1.25} + 250$
Michigan clay	4	$q_f = \frac{q_{14} - 430}{3.0} + 250$	
	8	$q_f = \frac{q_{14} - 295}{2.15} + 250$	
	12	$q_f = \frac{q_{14} - 320}{1.4} + 250$	$q_f = \frac{q_{14} - 380}{1.3} + 250$
North Carolina clay	8	$q_f = \frac{q_{14} - 240}{1.3} + 250$	
	12	$q_f = \frac{q_{14} - 305}{0.9} + 250$	$q_f = \frac{q_{14} - 280}{1.0} + 250$
Washington sand	4	$q_f = \frac{q_{14} - 305}{0.6} + 250$	
	8	$q_f = \frac{q_{14} - 390}{1.0} + 250$	
	12	$q_f = \frac{q_{14} - 330}{1.3} + 250$	$q_f = \frac{q_{14} - 280}{1.35} + 250$

of the mix is not the least of the 4 values as determined experimentally; the design value to be used is the one adjusted to give a durability index of 80 percent. The adjustment usually calls for a reduction of the experimentally determined strength value.

Some stabilized soil specimens gained strength during the freeze-thaw cycles. Although the resulting strength, p_f was never greater than p_{c_i}, it did give freezing index values R_f greater than 100 percent. The freeze-thaw cycles corresponded to a curing treatment. This is true with nearly all Iowa silt specimens, but an exception with clay and sand specimens, and contradicts previous results (13). Freezing in soils is largely a thermal conductivity controlled process, especially in view of the conditions of the Iowa freeze-thaw test where freezing is only from the top surface. To explain the unusual freezing behavior of the stabilized Iowa silt, the basic Stefan mechanism (16, p. 131) is used.

$$z = \sqrt{\frac{48\ kF}{L}} \tag{6}$$

Eq. 6 relates the depth of frost penetration z to the thermal conductivity k, the evironmental factor F, and the volumetric heat of latent fusion L. Because F is the same for all stabilized soil specimens, the calculated z-values (Table 5) may be used as a basis of comparison. Iowa silt gives the least value. This means that among the soils tested, Iowa silt, in the stabilized form in which it was used and under the conditions imposed by the Iowa freeze-thaw test, is the least affected by frost action. In fact, the depth of frost penetration is so small that the specimen, except for the bottom ¼ in., is in a humid atmosphere, which may account for the gain in strength as compared with that of the fully immersed specimens which reflected either loss of strength or a slower gain in strength. One would normally anticipate that the Wisconsin sand and Washington sand specimens, having high frost penetration depths, should not give freezing index values greater than 100 percent. Out of the 28 specimens of Washington sand tested, three displayed freezing indices greater than 100 percent: two were just above 100 percent, and one was 115 percent. With Wisconsin sand, one out of three specimens gave a freezing index of 110 percent.

TABLE 5

DEPTH OF FROST PENETRATION OF STABILIZED SOIL

Stabilized Soil	Frost Penetration (in.)
Iowa silt	1.98
Iowa clay	2.02
Wisconsin sand	2.72
Illinois clay	2.19
Texas clay	2.06
Michigan clay	2.10
North Carolina clay	2.00
Washington sand	3.42

Clays with few exceptions indicate freezing indices less than 100 percent.

Therefore, while the Stefan mechanism (Eq. 6) contributes to the understanding of the behavior of Iowa silt and Washington sand, it fails to cover the other soils where there may have been some interfering effects, possibly of a chemical nature.

In pavement design, the selection of a mix is not based entirely on strength loss due to freezing. Heaving also is taken into account because its extent is compared with that of the permissible deformation in the pavement. At the same time, the rate of heave reflects the permeability of the soil which may be used as an indication of the rate at which water is being moved to the frost line. Since freeze-thaw tests were not run on the natural soils, it is not possible to compare their frost heave with that of their stabilized form. However, measurements on the 2-in. stabilized-soil specimens indicate that the amount of frost heave was never greater than 0.07 in., which corresponds to a rate of heave of less than 0.2 mm per day—a value considered "negligible" according to the U. S. Corps of Engineers frost classification. The small increase in moisture content of the specimens also supports the observation that the specimens evaluated by the Iowa freeze-thaw test may be considered effectively stabilized.

TABLE 6

SUMMARY OF STANDARD FREEZE-THAW TESTS

Specimen	Density before freeze-thaw, pcf	Moisture before freeze-thaw, %	Number of cycles completed[a]	Conditions at completion of cycles indicated			
				Density, pcf	Moisture, %	Volume change, %	Stabilized soil loss, %
Iowa silt + 12% cement + 1% lime B	114.4	21.6	10	117.2	27.9	2.4	9.2
Iowa silt + 12% cement + 2% $CaSO_4$	114.8	21.5	12	117.1	27.4	2.3	8.7
Iowa clay + 8% cement + 3% lime A	112.2	25.5	8	114.8	29.9	3.1	7.0
Iowa clay + 12% cement + 1% lime B	112.9	25.9	12	114.9	29.9	2.1	7.3
Wisconsin sand + 12% cement + 2% $MgSO_4$	114.2	16.5	12	119.4	24.8	0.9	6.4
Illinois clay + 4% cement	131.7	17.9	8	137.9	24.1	2.8	8.9
Illinois clay + 12% cement + 1% lime A	132.7	18.2	12	137.4	23.6	1.1	7.3
Texas clay + 8% cement + 1% lime A	118.0	23.7	6	123.8	30.8	0.8	7.2
Texas clay + 12% cement + 3% lime A	118.5	23.4	12	122.9	27.6	1.2	8.1
Michigan clay + 8% cement + 2% $MgSO_4$	125.9	19.7	9	131.2	26.0	1.9	8.3
Michigan clay + 12% cement + 0.25% Na_2CO_3	126.8	19.1	12	130.3	24.9	1.2	8.0
North Carolina clay + 8% cement + 3% lime B	110.7	24.7	7	116.1	29.7	1.2	9.9
North Carolina clay + 12% cement + 3% lime A	110.0	24.9	9	115.8	29.1	1.3	8.4
Washington sand + 4% cement + 0.5% Na_4SiO_4	137.2	11.9	9	140.8	16.5	3.2	10.1
Washington sand + 12% cement + 3% lime A	138.1	12.3	12	140.7	16.6	2.5	9.1

[a] See reference (38).

Standard Freeze-Thaw Tests

In comparing the results of the Iowa freeze-thaw test with those of the standard test, (Table 6) it becomes obvious that those mixes which passed the standard ASTM test were considered successfully stabilized on the basis of the Iowa freeze-thaw test, except the North Carolina clay stabilized with 12 percent cement and 3 percent lime A. On the other hand, those mixes which could not withstand the severity of the 12 cycles of the standard freeze-thaw test are not considered as having failed in the Iowa freeze-thaw test.

Although it is unlikely that the results obtained from the two tests can be correlated, the following observation is pertinent. A few unconfined compressive strength tests were run on specimen No. 1 of the standard test at the end of the cycle when failure occurred or after it withstood 12 cycles of freeze-thaw. This specimen was not brushed and was used for volume change calculations. The unconfined compressive strengths determined by the standard test were very low in comparison with the p_f values of the Iowa freeze-thaw test. Perhaps the stabilized soil mixes benefited more, or lost less strength, from the curing nature of the Iowa freeze-thaw test than they did from the standard test. It is surprising that Iowa silt stabilized with 12 percent cement and 2 percent calcium sulfate demonstrated a slightly higher strength in the Iowa freeze-thaw test than after 12 cycles of the standard test. The previously given explanation regarding the depth of frost penetration may account for its unusual behavior.

Because the data collected from the Iowa freeze-thaw test indicate that, in general, those stabilized mixes with an unconfined compressive strength of 250 psi or more at the end of a 7-day humid curing period performed successfully under the imposed conditions, the original hypothesis that the 7-day strength may be used as a dependable guide becomes valid.

Wet-Dry Tests

The results of the wet-dry tests for the stabilized soil specimens (Appendix) may be used as additional assurance of the effectiveness of the stabilization methods used in the study, but not as a design criterion replacing that of the freeze-thaw tests. Such a replacement may be justifiable only in tropical climates which warrant the omission of freeze-thaw testing.

With a few exceptions, the stabilized soil specimens indicated R_i values greater than 80 percent. But even with R_i values less than 80 percent, the strengths of the specimens as reflected by the p_i and p_{c_i} values are greater than the strengths of the corresponding specimens after freeze-thaw testing. Therefore, the wet-dry test results lead to an underdesign, but a design based on the freeze-thaw test results will be safe. Again, the specimens of stabilized Iowa silt showed strengths less than their freeze-thaw counterparts. This should be attributed to the curing rather than the destructive effect of the freeze-thaw test on Iowa silt.

No particular trend was observed in regard to increases of moisture contents of the specimens or was there any tendency of the specimens to absorb too much water during the wet-dry test.

TRIAXIAL COMPRESSION TESTS

In this study, failure is considered to be the loss of shearing resistance at an advanced state of stress to which a soil or a stabilized soil, unsaturated and compacted to near standard Proctor density at optimum moisture content, has been subjected to an exterior load.

Inasmuch as no attempt was made to measure the pore water pressures, the values of the two shear components, cohesion and angle of internal friction, as determined from the Mohr diagrams (Figs. 12 to 16) and given in Table 7 are not the "effective" values.

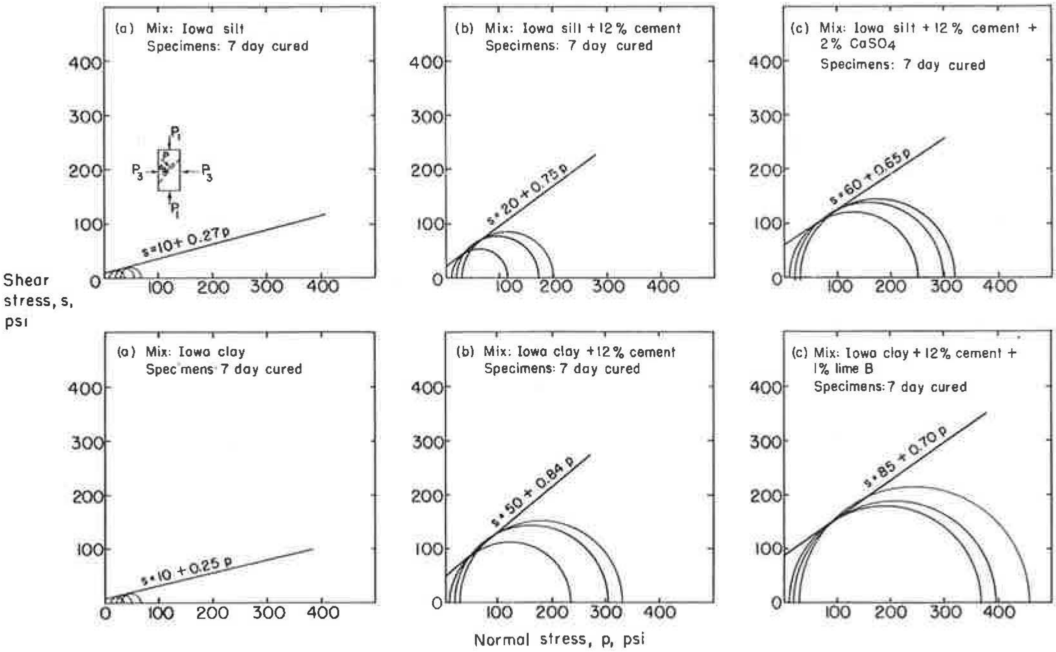


Figure 12. Mohr diagrams for tests on Iowa silt and clay mixes.

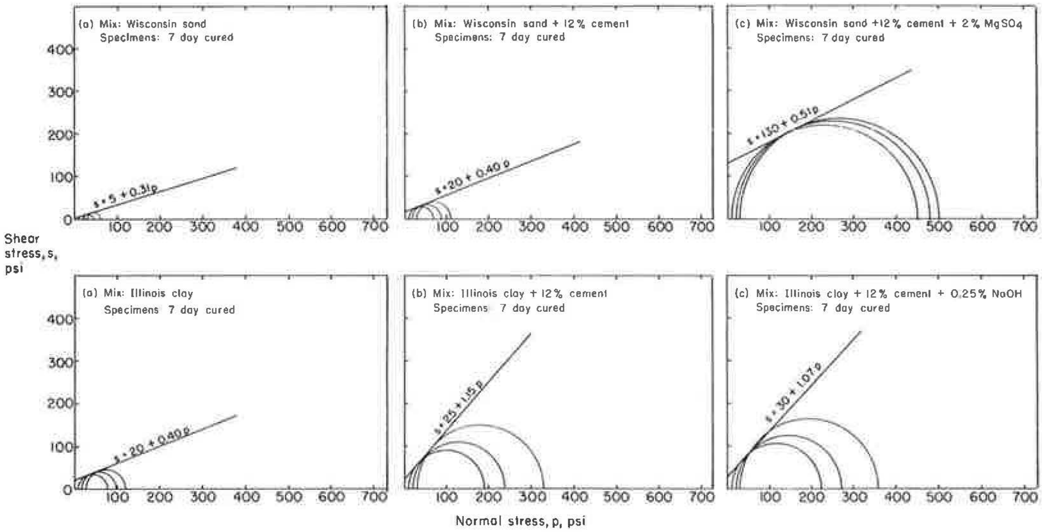


Figure 13. Mohr diagrams for tests on Wisconsin sand and Illinois clay mixes.

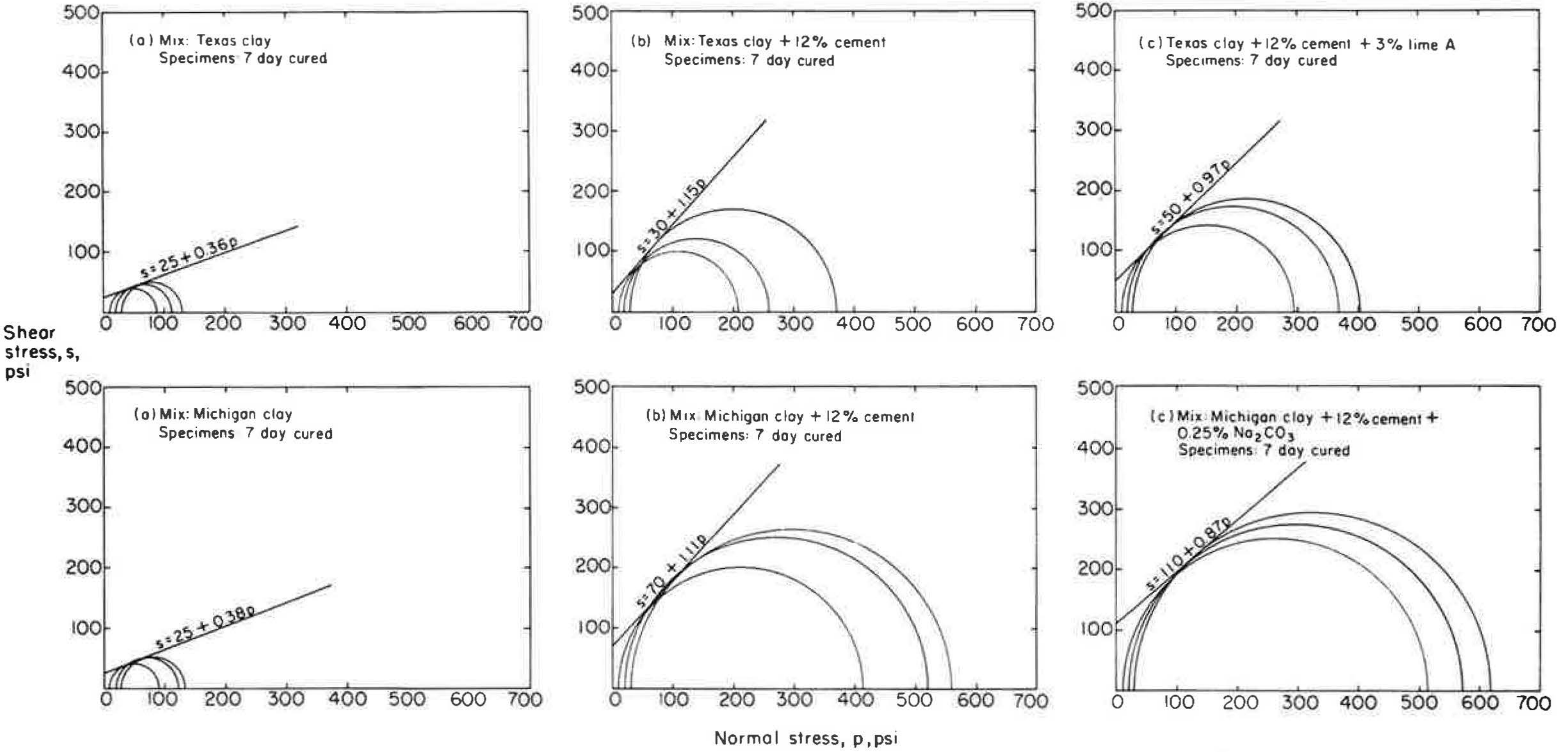


Figure 14. Mohr diagrams for tests on Texas and Michigan clay mixes.

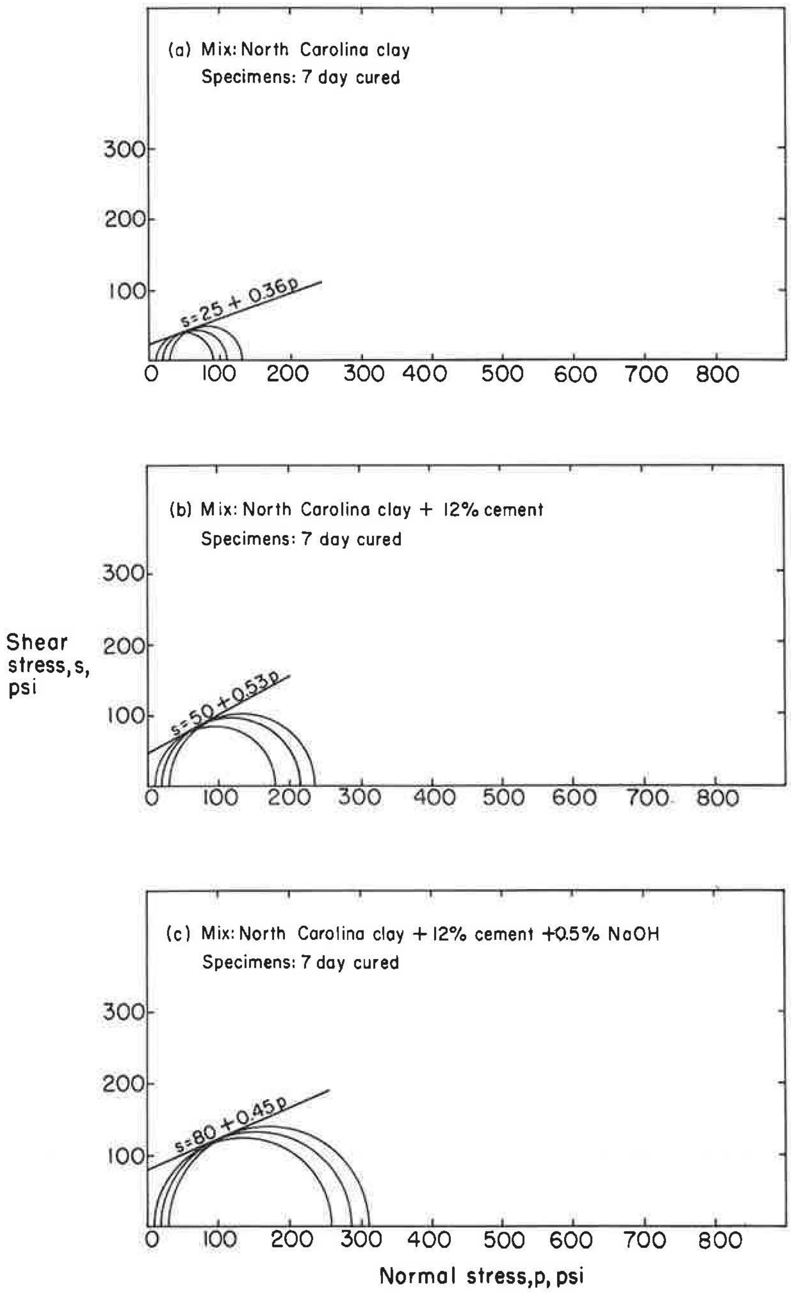


Figure 15. Mohr diagrams for tests on North Carolina clay mixes.

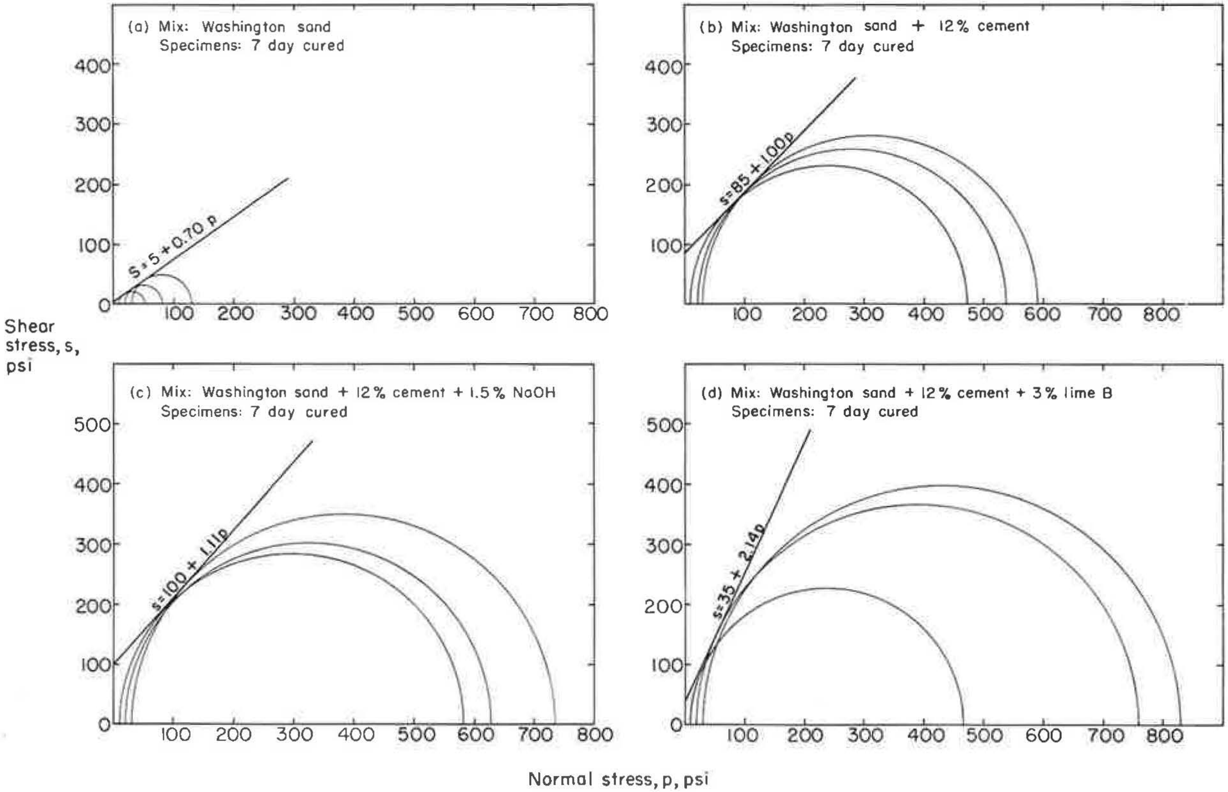


Figure 16. Mohr diagrams for tests on Washington sand mixes.

TABLE 7

SHEAR COMPONENTS OF RAW AND STABILIZED SOILS

Sample designation	Cohesion c, psi	Angle of internal friction ϕ, degrees	Shear equation $s = c + p \tan \phi$
Iowa silt	10	15	s = 10 + 0.27p
Iowa silt + 12% cement	20	37	s = 20 + 0.75p
Iowa silt + 12% cement + 2% $CaSO_4$	60	33	s = 60 + 0.65p
Iowa clay	10	14	s = 10 + 0.25p
Iowa clay + 12% cement	50	40	s = 85 + 0.84p
Iowa clay + 12% cement + 1% lime B	85	35	s = 85 + 0.70p
Wisconsin sand	5	17	s = 5 + 0.31p
Wisconsin sand + 12% cement	20	22	s = 20 + 0.40p
Wisconsin sand + 12% cement + 2% $MgSO_4$	130	27	s =130 + 0.51p
Illinois clay	20	22	s = 20 + 0.40p
Illinois clay + 12% cement	25	49	s = 25 + 1.15p
Illinois clay + 12% cement + 0.25% NaOH	30	47	s = 30 + 1.07p
Texas clay	25	20	s = 25 + 0.36p
Texas clay + 12% cement	30	49	s = 30 + 1.15p
Texas clay + 12% cement + 3% lime B	50	44	s = 50 + 0.97p
Michigan clay	25	21	s = 25 + 0.38p
Michigan clay + 12% cement	70	48	s = 70 + 1.11p
Michigan clay + 12% cement + 0.25% Na_2CO_3	110	41	s = 110 + 0.87p
North Carolina clay	25	20	s = 20 + 0.36p
North Carolina clay + 12% cement	50	28	s = 50 + 0.53p
North Carolina clay + 12% cement + 1.5% NaOH	80	24	s = 80 + 0.45p
Washington sand	5	35	s = 5 + 0.70p
Washington sand + 12% cement	85	45	s = 85 + 1.00p
Washington sand + 12% cement + 1.5% NaOH	100	48	s =100 + 1.11p
Washington sand + 12% cement + 3% lime B	35	65	s = 35 + 2.14p

Sandy Soils

The Wisconsin and the Washington sands have very low cohesive strengths of 5 psi in their unstabilized condition. Also, the Washington sand has a 35° angle of internal friction, as expected, but the Wisconsin sand has an unexpected low angle of 17°. The angular variation found between two like soils may be related to their composition. The clay content in the Wisconsin sand is higher by 4 percent. Coupled with a higher molding moisture, this produced a lubricant matrix, a condition not established in the Washington sand. The addition of cement has the same overall effect on both soils. It increased both the cohesion and angle of internal friction. The addition seemed to improve the coarser Washington sand more than it did the finer Wisconsin sand. Apparently cement fills in a large part of the voids in the Washington sand, producing a dense structure where the individual particles are more firmly embedded in a finer cementitious material which restricts their relative movement.

The addition of 2 percent magnesium sulfate and 0.5 percent sodium hydroxide to the Washington sand stabilized with 12 percent cement proved beneficial on the basis of the unconfined compressive strength. The chemicals further improved the shearing strength by increasing the cohesion and the angle of internal friction. This is the natural result of the hypothesis of cementation of granular soils. The chemicals in combination with cement produced a greater amount of gel which either encases the granular particles or acts as a void filler. This has two interpretations: the extent of cementation between particles is greater, and it tends to provide the soil mass with agglomerated finer particles. The first is conducive to greater cohesion, the second to larger values of internal friction.

Clayey Soils

All other soils in this study have been grouped for convenience into the "clayey" category, although they are not classified as clays texturally.

The addition of cement to clayey soils seemed to have the same effect as it had on sandy soils. The cohesive strength of clayey soils improved simultaneously with an increase in the angle of internal friction. The amount of increase varies from soil to soil, but no definite relationship could be established between the soil type and the change in the values of the two shearing components (Table 7). However, when the four inorganic clayey soils, Illinois clay, Texas clay, Michigan clay, and North Carolina clay are considered, the slightly inclined PI-ϕ and PI-c curves of the untreated soil (Fig. 17) become nearly vertical lines when depicting the same soils stabilized with 12 percent cement. In fact, the lines fall in a band limited by P.I. 8 to 19. A decrease in the plasticity index is a manifestation of aggregation, which leads to a larger internal friction angle. The cementation accruing from the cement gel was the factor that led to a higher cohesive strength.

The first and most important observation of the effect of chemical additive on soil-cement when the soil is nongranular is the tendency of the mix to gain in shearing resistance; yet, as Table 7 and Fig. 18 indicate, the increase in cohesive strength is masked somewhat by a slight decrease in the angle of internal friction when chemicals are added to soil-cement. The decrease in internal friction could not be explained in terms of a possible reduced agglomeration of particles at least not so far as the plastic properties were concerned. The soil-cement mixes did not seem to have plasticity indices very different from those of soil-cement modified with chemicals. The reason, then, should lie with the chemical, and the following is a possible explanation. The addition of the chemical gives a higher concentration of electrolyte in the pore fluid with attendant increase in the interparticle attractive forces. This chemical cementation is manifested as an increase in cohesion. But those ions, which are supplied to the soil-cement mass as a chemical, take their place between already aggregated clay particles resulting in a slight increase in spacing between particles. Accommodating the hydrated ion means that the interparticle contact is numerically reduced. It may be reasonable to accept the validity of the proposed hypothesis because internal friction arises from interparticle proximity.

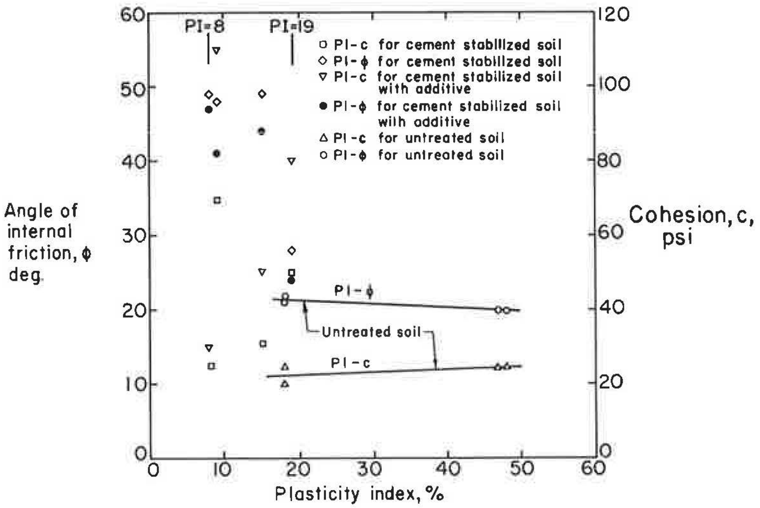


Figure 17. Variation of plasticity index with cohesion and angle of internal friction of untreated and stabilized soils.

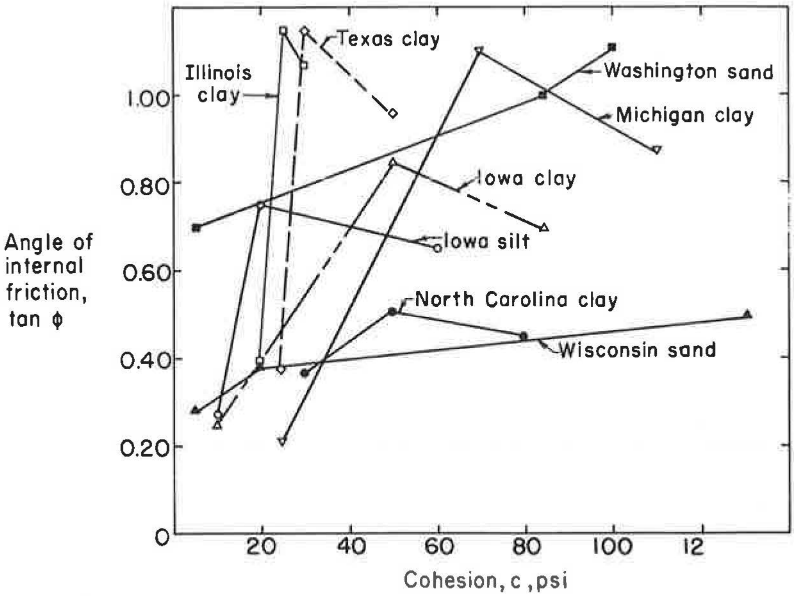


Figure 18. Effect of stabilization on c-tan ϕ relationship of soils.

CONCLUSIONS

Although the following conclusions apply specifically to the 8 soils used in this investigation, the variation in the properties of the soils is such that the conclusions may be considered more widely applicable.

1. The addition of selected chemicals to soil-cement in amounts of not more than 2 percent often results in significant strength increases over that of the soil-cement with equal cement content but no chemical additive. Whether or not the use of chemicals is economical and competitive depends primarily on the local conditions.
2. The type and exact percentage of chemical most beneficial to the strength mixtures of soil-cement are unique for each soil and depend on the texture of the soil, the type of clay mineral, and partly on the acidity of the soil.
3. Organic top soils that are low in clay content and not alkaline (Iowa silt, Wisconsin sand, and Washington sand soil-cement mixtures) responded to treatment with the sulfates of calcium, sodium, or magnesium. Wisconsin sand soil-cement showed a limited favorable reaction to magnesium sulfate only. Sodium hydroxide, and high calcium lime, and monohydrated dolomitic lime were beneficial to soil-cement containing Washington sand but not to Iowa silt or Wisconsin sand soil-cement.
4. The alkaline organic top soil (Iowa clay) in soil-cement did not seem to reflect any special preference for sulfates or hydroxides. The additives increased the strength of the mixtures of Iowa clay-cement but not as much as calcium chloride.
5. Soil-cement mixtures containing the B- and C-horizon soils from Illinois, Texas, Michigan, and North Carolina that are heavy clays texturally, were significantly upgraded strengthwise by additives of lime or sodium hydroxide. Sodium orthosilicate seemed to accelerate the soil-cement reaction in mixtures with all soils except Illinois clay. The beneficial effect of sodium carbonate was limited to mixtures with Texas clay and Michigan clay. Lime seemed especially effective with the montmorillonitic and kaolinitic clays.

These conclusions drawn on the basis of 7-day and 28-day unconfined compressive strength data were verified by the Iowa freeze-thaw and wet-dry test strengths. In almost all cases the assumed minimum acceptable 7-day strength of 250 psi indicated dependability in that mixtures having attained this or higher strength met the strength after freeze-thaw and the freezing index requirements of the test. The same can be said for the wet-dry test results. Therefore, the following can safely be proposed:

1. The Iowa freeze-thaw test is as dependable as the standard ASTM-AASHO freeze-thaw test; in fact, it appears to be a desirable alternate method because of its relative simplicity.
2. The establishment of a functional relationship between the 14-day unconfined compressive strength of the mixture of soil-cement-chemical and the strength of the same mixture after 7 days' humid curing and a full 10-cycle subjection to the Iowa freeze-thaw test further validates the contention that stabilizing agent requirements of soils may be eventually determined by means of simple strength tests.

Triaxial shear tests run on samples compacted to near standard Proctor density at optimum moisture content indicated:

1. The addition of cement increased the cohesion and internal friction angle of the Wisconsin and Washington sands. Magnesium sulfate in the former and sodium hydroxide in the latter further increased the two shear strength components.
2. The other six soils, which fall in the silt-clay textural group, had their angle of internal friction greatly increased and their cohesion only slightly increased upon addition of cement. Chemicals or lime (calcium sulfate with Iowa silt, sodium hydroxide with Illinois clay, monohydrated dolomitic lime with Texas clay or with Iowa clay, sodium carbonate with Michigan clay, sodium hydroxide with North Carolina clay) giving optimum conditions substantially increased cohesion of soil-cement mixtures, but the angle of internal friction decreased slightly, the net result being an overall increase of shearing strength.

ACKNOWLEDGMENT

This investigation was conducted in conjunction with Projects 383-S and 449-S of the Iowa Engineering Experiment Station. Project 383-S is under contract with the Dow Chemical Company and Project 449-S is under contract with the Highway Research Board of the Iowa State Highway Commission.

Special acknowledgment is given to the members of the Soil Research Laboratory of the Engineering Experiment Station for their assistance during the various phases of this investigation; to the Dow Chemical Company for supplying some of the chemicals; the various State Highway Departments for supplying soil samples; the Penn-Dixie Company for supplying the cement and its analysis; and the U.S. Gypsum Company for supplying the hydrated lime and its analysis.

REFERENCES

1. "1958 Book of Standards: Part 4." American Society for Testing Materials, Philadelphia (1958).
2. Budnikov, P. P., and Azelitskaya, R. D. "Effect of Electrolyte Additions on the Hardening of 3 $CaO \cdot Al_2O_3$." (In English.) J. Appl. Chem. of the USSR, 32:1215-1219 (1959).
3. "Soil Stabilization with Portland Cement." HRB Bull. 292, 212 pp. (1961).
4. de Sousa Pinto, C., Davidson, D. T., and Laguros, J. G., "Effect of Lime on Cement Stabilization of Montmorillonitic Soils." HRB Bull. 253, 64-83 (1962).
5. Dutron, M. M. R., and Confyn, W., "Resistances et Portance CBR des Sols-Ciment. Leur Relation, Leur Estimation par un Essai Accelere." 5th International Conf. on Soil Mech. and Foundation Engg. Proc., 2:221-226 (1961).
6. George, K. P., "Development of a Freeze-Thaw Test for Evaluating Stabilized Soil." M.S. thesis, Iowa State Univ. (1960).
7. Handy, R. L., "Cementation of Soil Minerals with Portland Cement or Alkalis." HRB Bull. 198, 55-64 (1958).
8. Handy, R. L., Jordan, J. L., Manfre, L. E., and Davidson, D. T., "Chemical Treatments for Surface Hardening of Soil-Cement and Soil-Lime-Fly Ash." HRB Bull. 241, 49-66 (1960).
9. Iler, R. K., "The Colloid Chemistry of Silica and Silicates." Cornell Univ. Press (1955).
10. Lambe, W. T., Michaels, A. S., and Moh, Z. C., "Improvement of Soil-Cement with Alkali Metal Compounds". HRB Bull. 241, 67-108 (1960).
11. Leadabrand, J. S., Norling, L. T., and Hurless, A. C., "Soil Series as a Basis for Determining Cement Requirements for Soil-Cement Construction." HRB Bull. 148, 17 pp. (1959).
12. "Soil-Cement Laboratory Handbook." Portland Cement Association (1959).
13. Sherwood, P. T., "The Stabilization with Cement of Weathered and Sulphate Bearing Clays." Geotechnique, 7:179-191 (1957).
14. Tawes, R. H., "Improvement of Soil-Cement with Chemical Additives." M.S. thesis, Iowa State Univ. (1959).
15. Winterkorn, H. F., Gibbs, H. J., and Fehrman, R. G., "Surface Chemical Factors of Importance in the Hardening of Soils by Means of Portland Cement." HRB Proc., 22:385-414 (1942).
16. Yoder, E. J., "Principles of Pavement Design." Wiley (1959).
17. George, K. P., and Davidson, D. T., "Development of a Freeze-Thaw Test for the Design of Soil-Cement." Highway Research Record 36 (1963).

Appendix

TABLE 8

DURABILITY INDICES FOR THE IOWA SILT STABILIZED WITH 12 PERCENT CEMENT

Additive							
Type	Content (%)	p_f (psi)	p_{cf} (psi)	R_f (%)	p_i (psi)	p_{c_i} (psi)	R_i (%)
—	—	267	228	117	241	290	83
Calcium chloride	0.50	238	208	114	198	284	70
Calcium sulfate	2.00	412	379	109	409	452	91
Magnesium sulfate	2.00	383	304	126	350	465	75
Sodium sulfate	1.00	337	323	104	313	386	81
Lime B	1.00	251	261	96	254	320	79

TABLE 9

DURABILITY INDICES FOR STABILIZED IOWA CLAY

Additive							
Type	Content (%)	p_f (psi)	p_{cf} (psi)	R_f (%)	p_i (psi)	p_{c_i} (psi)	R_i (%)
(a) 8 Percent Cement							
—	—	337	340	99	337	415	81
Calcium chloride	0.25	323	363	89	392	396	99
Calcium sulfate	0.25	323	369	88	366	396	92
Magnesium oxide	2.00	340	353	96	356	396	90
Magnesium sulfate	1.00	277	297	93	304	353	86
Sodium hydroxide	0.25	313	337	93	343	373	92
Sodium sulfate	0.50	327	337	97	337	383	88
Lime A	3.00	406	409	99	419	475	88
Lime B	3.00	363	389	93	438	429	102
(b) 12 Percent Cement							
—	—	341	340	101	450	460	98
Calcium chloride	0.25	390	397	98	470	496	96
Calcium sulfate	0.25	400	410	98	450	465	97
Magnesium oxide	2.00	376	379	99	360	352	102
Magnesium sulfate	0.25	310	320	97	404	470	86
Sodium hydroxide	1.00	380	385	99	470	470	100
Sodium sulfate	0.50	330	350	94	390	452	86
Lime A	1.00	450	468	96	471	498	95
Lime B	1.00	470	490	96	500	520	96

TABLE 10

DURABILITY INDICES FOR STABILIZED WISCONSIN SAND

Additive		p_f	p_{cf}	R_f	p_i	p_{ci}	R_i
Type	Content (%)	(psi)	(psi)	(%)	(psi)	(psi)	(%)
(a) 12 Percent Cement							
—	—	64	80	80	73	73	100
Calcium chloride	2.00	83	96	87	87	77	113
Magnesium sulfate	2.00	406	369	110	379	396	96
(b) 16 Percent Cement							
—	—	70	92	96	78	93	84
Calcium chloride	2.00	90	100	90	93	89	105
Magnesium sulfate	2.00	400	408	98	400	400	100

TABLE 11

DURABILITY INDICES FOR STABILIZED ILLINOIS CLAY

Additive		p_f	p_{cf}	R_f	p_i	p_{ci}	R_i
Type	Content (%)	(psi)	(psi)	(%)	(psi)	(psi)	(%)
(a) 4 Percent Cement							
—	—	251	238	106	313	376	83
Calcium chloride	0.25	40	590	7	258	373	69
Sodium hydroxide	0.25	389	455	86	425	639	67
Lime A	1.00	425	419	101	386	642	60
Lime B	3.00	448	455	98	458	567	81
(b) 8 Percent Cement							
—	—	586	531	113	540	860	63
Calcium chloride	0.25	461	534	86	508	735	69
Sodium hydroxide	0.25	609	646	94	692	821	84
Lime A	1.00	665	656	101	686	913	75
Lime B	3.00	610	593	103	686	807	85
(c) 12 Percent Cement							
—	—	850	900	95	870	945	92
Calcium chloride	0.25	752	817	92	784	972	81
Sodium hydroxide	0.25	847	870	98	942	997	95
Lime A	1.00	924	1,092	85	918	1,122	82
Lime B	3.00	1,000	1,058	95	860	1,070	80

TABLE 12

DURABILITY INDICES FOR STABILIZED TEXAS CLAY

Additive		p_f	p_{cf}	R_f	p_i	p_{ci}	R_i
Type	Content (%)	(psi)	(psi)	(%)	(psi)	(psi)	(%)
(a) 4 Percent Cement							
—	—	113	156	73	139	261	53
Lime A	3.00	399	360	111	363	478	76
Lime B	3.00	471	461	102	415	544	76
(b) 8 Percent Cement							
—	—	402	432	93	458	521	88
Sodium hydroxide	0.25	389	409	95	498	652	77
Sodium orthosilicate	0.50	343	415	83	396	524	76
Lime A	3.00	537	606	89	649	804	81
Lime B	3.00	475	494	96	475	705	67
(c) 12 Percent Cement							
—	—	455	501	92	492	570	86
Calcium chloride	0.50	455	523	86	452	528	86
Sodium carbonate	0.25	471	468	101	508	604	84
Sodium hydroxide	0.25	450	447	102	483	594	82
Sodium orthosilicate	0.50	460	500	92	503	598	84
Lime A	3.00	687	734	93	674	792	85
Lime B	3.00	623	778	80	653	798	82

TABLE 13

DURABILITY INDICES FOR STABILIZED MICHIGAN CLAY

Additive		p_f	p_{cf}	R_f	p_i	p_{ci}	R_i
Type	Content (%)	(psi)	(psi)	(%)	(psi)	(psi)	(%)
(a) 4 Percent Cement							
—	—	138	193	72	158	202	78
Calcium chloride	0.50	123	113	109	139	208	67
Magnesium oxide	1.50	240	254	95	316	370	87
Sodium carbonate	0.25	175	156	106	165	281	59
Sodium hydroxide	0.50	241	241	100	228	389	59
Sodium orthosilicate	0.50	228	188	121	201	307	66
Sodium sulfate	0.50	192	182	106	201	307	66
Lime A	1.00	218	225	97	231	340	68
Lime B	1.00	238	267	89	251	389	65
(b) 8 Percent Cement							
—	—	364	410	89	408	503	81
Calcium chloride	0.50	320	377	85	380	393	97
Calcium sulfate	0.50	368	412	89	400	510	79
Magnesium oxide	1.50	373	454	82	475	535	89
Magnesium sulfate	2.00	340	378	90	410	500	82
Sodium carbonate	0.25	438	478	92	517	691	75
Sodium hydroxide	0.50	405	498	81	513	579	89
Sodium orthosilicate	0.50	376	421	90	467	559	84
Sodium sulfate	0.50	373	424	88	448	603	74
Lime A	1.00	593	702	85	610	718	85
Lime B	1.00	644	748	86	752	867	88
(c) 12 Percent Cement							
—	—	584	617	95	700	873	80
Calcium chloride	0.50	592	615	96	622	778	80
Calcium sulfate	0.50	717	748	96	900	993	91
Magnesium oxide	1.50	565	694	82	734	768	96
Magnesium sulfate	2.00	712	795	90	865	992	87
Sodium carbonate	0.25	848	962	88	1,020	1,140	98
Sodium hydroxide	0.50	717	737	97	807	922	88
Sodium orthosilicate	0.50	563	594	95	657	678	97
Sodium sulfate	0.50	581	620	94	700	819	86
Lime A	1.00	608	716	85	715	853	84
Lime B	1.00	657	810	81	822	907	91

TABLE 14

DURABILITY INDICES FOR STABILIZED NORTH CAROLINA CLAY

Additive		p_f	p_{ci}	R_f	p_i	p_{ci}	R_i
Type	Content (%)	(psi)	(psi)	(%)	(psi)	(psi)	(%)
(a) 4 Percent Cement							
—	—	0	0	0	61	78	78
Calcium chloride	1.00	55	88	63	78	90	87
Sodium hydroxide	1.50	72	90	80	93	120	78
(b) 8 Percent Cement							
—	—	104	133	78	140	184	76
Calcium chloride	1.00	58	85	68	75	93	81
Sodium hydroxide	1.50	188	175	103	165	192	86
Lime A	3.00	366	353	104	337	383	88
Lime B	3.00	261	277	94	205	251	82
(c) 12 Percent Cement							
—	—	337	320	105	343	294	117
Calcium chloride	1.00	248	294	85	263	291	90
Calcium sulfate	0.50	292	295	99	316	320	99
Magnesium oxide	0.50	272	309	88	303	320	95
Sodium carbonate	1.50	310	317	98	337	348	97
Sodium hydroxide	1.50	317	313	101	313	363	86
Sodium orthosilicate	0.50	340	340	100	398	410	96
Sodium sulfate	0.50	305	310	99	284	338	84
Lime A	3.00	406	422	96	327	412	79
Lime B	3.00	373	399	94	287	442	65

TABLE 15

DURABILITY INDICES FOR STABILIZED WASHINGTON SAND

Additive		p_f	p_{ci}	R_f	p_i	p_{ci}	R_i
Type	Content (%)	(psi)	(psi)	(%)	(psi)	(psi)	(%)
(a) 4 Percent Cement							
—	—	102	133	77	136	158	86
Calcium chloride	0.50	97	100	97	128	161	80
Sodium carbonate	0.50	396	412	90	389	337	115
Sodium hydroxide	0.50	369	461	80	419	369	114
Sodium orthosilicate	0.50	287	294	98	300	304	99
Sodium sulfate	0.50	198	294	67	235	251	94
Lime A	3.00	244	212	115	169	264	64
Lime B	3.00	231	248	93	287	320	90
(b) 8 Percent Cement							
—	—	501	620	81	702	730	96
Calcium chloride	0.50	504	588	86	512	607	84
Calcium sulfate	0.50	497	523	95	640	690	93
Sodium carbonate	0.50	610	666	92	687	652	105
Sodium hydroxide	0.50	800	780	103	792	874	91
Sodium orthosilicate	0.50	480	592	81	534	642	83
Sodium sulfate	0.50	512	592	87	687	687	100
Lime A	3.00	684	773	89	790	901	88
Lime B	3.00	710	768	93	800	852	94
(c) 12 Percent Cement							
—	—	522	784	67	942	1,183	80
Calcium chloride	0.50	697	840	83	886	1,043	85
Calcium sulfate	0.50	963	1,048	92	1,039	1,142	91
Magnesium oxide	0.50	717	850	84	783	920	85
Magnesium sulfate	0.50	573	628	91	1,282	1,296	99
Sodium carbonate	0.50	723	872	83	824	984	84
Sodium hydroxide	0.50	1,109	1,224	91	1,271	1,352	94
Sodium orthosilicate	0.50	719	958	75	1,008	1,008	100
Sodium sulfate	0.50	902	894	101	912	1,153	79
Lime A	3.00	1,382	1,595	87	1,545	1,770	87
Lime B	3.00	1,472	1,667	89	1,709	1,978	86

General Discussion

MILES D. CATTON, Consultant, Park Ridge, Ill.

•THE excellent papers on soil-cement mixtures cover a wide range of interests and viewpoints regarding this unique construction material. More than a quarter century has passed since the first scientifically controlled soil-cement road was built in South Carolina in 1935 (1). Incidentally, this road is still giving excellent service, carrying traffic loads many times greater than anticipated.

The summary of soil-cement test procedures by Norling gives an excellent review of the subject which can well serve as a standard reference of the basic principles governing soil-cement mixtures (also see 2).

The mix design for cement-treated bases presented by Hveem and Zube summarizes the eminently successful procedure used by California for many years. They have used these granular cement-treated bases for concrete pavements to prevent pumping and joint displacement due to consolidation. One of the specifications for cement-treated bases, which is the same as for California Class 2 aggregate base, includes the following gradations:

Sieve Size	Percent Passing
1 in.	100
¾ in.	90-100
No. 4	35-55
No. 30	10-30
No. 200	3-9

This gradation specification, together with strength requirements and other tests, classifies the California cement-treated base as one in which concrete and cement technology govern most aspects of mix design requirements. An exception is the recognition of influence of degree of compaction as judged by optimum moisture and maximum density. Therefore, these test procedures are not comparable to those required for materials that contain substantial quantities of fine-grain materials passing the No. 50 and No. 200 sieve which also possess significant test constants and which are classified as soil-cement mixtures.

The British practice summarized by Maclean and Lewis of the Road Research Laboratory shows a considerable difference between British and American basic thinking and philosophies. Some of these differences are explained as being due to specific soil and climatic conditions in Great Britain. For example, there are relatively few soil categories in this geographical area and conditions of rather continuous rain and high humidities prevail. These conditions led to rather simple, straightforward strength determinations and criteria. However, experience has brought forward modifications of these criteria, as pointed out in the subject paper.

Several phases of British practice have been of interest through the years. One phase is the generality of considering a cement content of 10 percent as a maximum and a tendency to emphasize lower cement contents. A common practice in the United States is to add sufficient cement to meet mix design criteria after conducting the standard ASTM wet-dry and freeze-thaw tests. These criteria include brushing losses, gain in strength with increases in age and cement contents, volume changes, and moisture changes. After this cement requirement is determined, the next step is to prepare a construction cost estimate to determine the economic feasibility of the project.

This same approach is used in much of the research involved. For example, some of the black, organic surface soils of the United States may require 16 to 18 percent cement to meet the usual criteria. This presents the need of additional work to evolve a more economical answer. The cement requirements of underlying horizons are determined and they are usually less. Can a soil borrow pit be established nearby in one of the favorable horizons and this soil used for a soil-cement pavement on top of the black, organic surface soil? Another possibility often present in the general geology of the United States is the nearby location of a more favorable surface soil that may be used for a soil-cement pavement on top of the black, organic surface soil. Suitable economic studies are made of the various possibilities. There are cases, of course, where sufficient suitable gravel or crushed stone is available on the site to provide the most economical construction. However, just because the surface soil is black with a high organic content, it is not rejected as an economic possibility. An early 1936 pioneering project by the Missouri State Highway Department, using a black, heavy clay, illustrates practical construction procedures as well as economical results (3).

In the case of high organic content of sandy soils, occurring in coniferous tree bearing areas, practical and economical answers have been found in the addition of calcium chloride or clay (4, 5).

Whether or not the British climate will permit the use of moisture-density control as practiced in the United States is a question that can only be answered by an on-the-job study. However, the northwestern United States has much rain and high humidities and moisture-density control is entirely practical there. With suitable construction specifications, moisture-density control can be used successfully in England for most of the normal paving construction season applying to other types of paving.

George and Davidson present interesting data for one construction project in Iowa. Because only one major soil is involved, the conclusions drawn regarding cement contents cannot be extended to general criteria. The procedures used in analyzing data are important, however.

Packard and Chapman compare freeze-thaw and wet-dry tests and durability and, as justification and authority for such comparisons, cite early papers on the subject (6, 7), the ASTM Standard wet-dry and freeze-thaw tests (8), and the PCA Laboratory Handbook (9) giving criteria for cement contents required for construction.

This is an unintentional but most unfortunate interpretation and understanding of these references and the basic principles involved in soil-cement mixtures and their testing. The writer was chairman of the sectional committee that wrote and sponsored the ASTM test procedures. He was therefore familiar with all details of the procedures and their intent. Further, he is the author of the test criteria cited and of their purpose and uses. He also is the author of two papers (2, 10) that discuss all these details, having been involved in the development of soil-cement mixtures from their inception in 1935 until 1951, when he was assigned other responsibilities.

Introduction of the durability concept contradicts directly what is later stated as the purpose of the investigation and subsequent presentation of data. It also contradicts the facts presented in the Norling paper.

Packard and Chapman state that the purpose of the investigation covered two areas of interest, as follows:

1. Measuring techniques that might be developed and evaluated to replace the brushing technique and accompanying soil-cement loss criteria used in connection with the Standard ASTM wet-dry and freeze-thaw procedures.
2. A shorter series of alternate wet-dry and freeze-thaw procedures to lessen time of testing.

Investigations in these two areas are highly desirable. One of the original criteria established concerned itself with volume change during testing. This criterion was evidence that the cement and soil had reacted together to form a structural material that was not disrupted by the high expansive or shrinkage forces of clay.

The data presented by Packard and Chapman on length (volume) change give preliminary evidence that it may be possible to establish criteria for length change that can replace the brushing technique and criteria. This would be worthwhile, as the measuring technique is more sophisticated and accurate. However, comparison must be made of test results on many more soils and by using the ASTM Standard procedures for wetting and drying and freezing and thawing. Also, as emphasized by Packard and Chapman, the adequacy of cement contents so determined must be verified by field performance over a considerable period of time. In no case does the writer consider the wet-dry and freeze-thaw tests as durability tests. Durability must be demonstrated by long-time field exposure. No laboratory procedure can duplicate weather for any location—the variables are too great. The expansion and shrinkage forces that may be produced by clays can be, and often are, many times those produced by freezing water. Also, the procedures evaluate soil and cement reactions, soil clay minerals, pozzolanic reactions, soil and cement hydration products, and other surface chemistry phenomena.

The other data presented on compressive strength, pulse velocity, and rate of weight loss as measures for developing criteria present many ambiguities and reversals. They appear to hold little promise of practical use.

The other item of interest, a shortened wet-dry and freeze-thaw period, presents many problems. Concrete technicians have worked with such procedures for many years; there are several recognized ASTM procedures. The concrete technician has learned that any small change in the procedure changes results so they cannot be compared. It would seem that with soil-cement mixtures having thousands of variations in physical and chemical composition, as compared to a few in concrete, the experience of the concrete technicians could well be used to demonstrate that changes in the soil-cement procedures could best be left alone. However, appreciably more data are needed on clay mineral composition, pozzolanic reactions, hydration products, classification of soils in terms of length change differences, criteria for length change for various soils, and the reproducibility of test procedures and results before changes in the test procedures can be considered and evaluated.

The extensive discussions and explanations of freezing and thawing, durability, etc., are confusing and contradictory. Extensive data in the literature demonstrate that soil-cement technology is unique and entirely different from the concrete technology repeatedly cited and compared by the authors. The important finding of the paper relates to the specific data presented on length change and the simple, rational, short, accompanying explanation. It permits a conclusion that with further, careful, extensive work, length change criteria might be developed as a replacement for the brushing technique in the present, widely accepted and most successful ASTM procedures.

The paper by Herzog and Mitchell is a valuable contribution to soil-cement literature. They include this important comment:

> Consideration of the nature of cement hydration, the physico-chemical characteristics of clays, and lime-clay interaction, leads to the hypothesis that during the hydration of a clay-cement mixture hydrolysis and hydration of cement could be regarded as primary reactions which form usual hydration products....

Their recognition of clay minerals and the reaction products of these clay minerals and cement is a most important, fundamental approach to a study and evaluation of soil-cement mixtures. Continued, extensive research in these areas is justified by the data obtained and analyzed by Herzog and Mitchell. This can be the most valuable research area for soil-cement mixtures which will result in an evaluation and delineation of the basic principles governing soil-cement mixtures.

The work reported by Laguros and Davidson also deals with reaction products of soils, cement, and compounds of sodium, calcium and magnesium and commercial lime. Their physical test results show that extensive chemical reactions take place. Investigation by X-ray diffraction to determine the hydration products produced would be most valuable. Inclusion of various clay minerals would also be valuable.

In the past, other investigators have explored these areas and reported on their importance and significance (11, 12, 13, 14).

The soils engineer and the cement chemist can combine their respective fields most effectively to make a productive team for research of this nature. Physical tests and results will no longer suffice to evaluate soil and cement reactions. As shown by Herzog and Mitchell, a new approach is now possible.

REFERENCES

1. Mills, W.H., Jr., "Stabilizing Soils with Portland Cement, Experiments by the South Carolina Highway Department." HRB Proc., 16:322-348 (1936).
2. Catton, Miles D., "Soil-Cement Technology—A Resume." Jour. PCA Research and Development Lab., 4:1, 13-21 (Jan. 1962).
3. Reagel, F.V., "Soil-Cement Stabilization in Missouri." HRB Proc., 17, Part II: 66-78 (1937).
4. Larson, G.H., "Experimental Soil-Cement Road in Wisconsin." HRB Proc., 17, Part II:83-91 (1937).
5. Catton, Miles D., and Felt, E.J., "Effect of Soil and Calcium Chloride Admixtures on Soil-Cement Mixtures." HRB Proc., 23:497-529 (1943).
6. Catton, Miles D., "Basic Principles of Soil-Cement Mixtures and Exploratory Laboratory Results." HRB Proc., 17, Part II:7-31 (1937).
7. Catton, Miles D., "Research on Physical Relations of Soil and Soil-Cement Mixtures." HRB Proc., 20:821-855 (1940).
8. "Standard Method of Wetting-and-Drying Test of Compacted Soil-Cement Mixtures." ASTM Designation: D 559; "Standard Method of Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures." ASTM Designation: D 560.
9. "Soil-Cement Laboratory Handbook." Portland Cement Association.
10. Catton, Miles D., "Early Soil-Cement Research and Development." Paper No. 1899, Jour. of the Highway Division, ASCE Proc., 85:HW 1, 1-16 (Jan. 1959); 85:HW 3, 77-93 (Sept. 1959); 86:HW 1, 67-76 (Mar. 1960).
11. Winterkorn, H.F., Gibbs, H.J., and Fehrman, R.E., "Surface Chemical Factors of Importance in the Hardening of Soils by Means of Portland Cement." HRB Proc., 22:385 (1942).
12. Lambe, T.W., "Physico-Chemical Properties of Soils: Role of Soil Technology." Jour. of the Soil Mech. and Foundation Division, ASCE Proc., 85:55-70, SM 2 (Apr. 1959).
13. Carroll, Dorothy, "Ion-Exchange in Clays and Other Minerals." Bull. Geological Soc. of America, 70:6, 749-780 (June 1959).
14. Anderson, L., "Clay Chemistry in Soil Stabilization." Geologiska forëningen Förhandlingar, Stockholm (In English), 82:2, 274-297 (1960).

Discussion

R.G. PACKARD and G.A. CHAPMAN—Mr. Catton's remarks are primarily concerned with principles involved in the development and use of the standard soil-cement testing procedures. His discussion of these principles appears to be based on his interpretation of the term "durability testing" as "climate simulation testing" only.

This definition is not the intent of the authors. As used by them, the term "durability testing" does not imply climate simulation except where specifically stated, but refers to a general classification of tests employed to evaluate various physico-chemical properties that may not be completely evaluated by "strength tests." Because these properties may influence the quality of field performance, their evaluation is stated as a major objective in mix design procedures. It is recognized that the standard tests are not climate simulation tests, and the associated PCA criteria do not relate to any specific climatic conditions. The paper is certainly ambiguous if these points are not clarified.

Mr. Catton also states that the reliability of a new procedure depends on verification by long-term field performance. This is true. Verification of a procedure can be established directly by comparison with field performance; or, it can be established indirectly, but more quickly, by correlation of results with the reliable standard tests and PCA criteria for a large number and variety of soils.

The authors agree that development of accelerated procedures for obtaining the same cement factor determined by standard tests presents many difficulties. However, the need for faster procedures justifies the attempt.

Items such as the determination of compressive strength and pulse velocity, discussion of reactions during freezing-thawing and wetting-drying, and notations of similarity of some reactions to those of concrete, are worthy of reporting and useful to the objectives of the study.