

Development of a Test for Identifying Poorly Reacting Sandy Soils Encountered in Soil-Cement Construction

E. G. ROBBINS and P. E. MUELLER, respectively, Acting Manager; and Soils Engineer, Soil-Cement Bureau, Portland Cement Association, Chicago

In soil-cement construction, certain types of sandy soils are encountered that cannot be treated successfully with normal amounts of portland cement. While the use of additive soils, calcium chloride, and other chemicals, effectively neutralize this adverse reaction, it is impossible to detect the presence of this type of sandy soil until certain standard laboratory tests, which require approximately one week for completion, have been performed. The test method presented in this report provides a method of detecting this reaction in approximately one hour.

● SINCE THE ADVENT of soil-cement in the United States some 25 years ago, there have been encountered in some areas of the country certain sandy soils that require relatively high cement contents for successful treatment. Early research work by the Portland Cement Association (1) and others, resulted in the development of effective countermeasures that can be used when confronted with this type of material. These measures include the use of calcium chloride as an admixture, dilution with a normal acting soil, and under certain conditions, the use of salt water. Recent research has shown the addition of certain sodium compounds is very effective (2).

While this early work successfully gave a remedy to the situation, little was known of the chemical or physical cause of this poor reaction between the soil and cement. Until such research could be performed, little could be done to solve the rather vexing problem of identifying such soils during sampling operations in the field, or in the laboratory prior to running standardized soil-cement tests. This meant that in many cases valuable time was lost in testing a soil that could not be used for construction due to a high cement requirement. If it were possible to detect the poor reaction on the day sampling is performed, the use of borrow materials or other measures could be immediately decided on.

INITIAL TRIALS AND CORRELATIONS

The acidity or alkalinity of a soil was long thought to have a bearing on the manner in which it reacted with cement. Early research (3) showed that organic matter of an acidic nature had an adverse effect on the soil-cement system, but this work was confined to fine-grained soil materials. Little data were available on the effects of organic content and acidity on sandy soils, and this became the first phase of the investigation.

Samples of 27 different sand soils from Florida and Michigan (two areas where poorly-reacting sands occur) were studied. Values of pH for each soil were determined using a Model G-Beckman pH meter, and organic contents were measured colorimetrically (4). Unconfined compressive strength was chosen as the "yardstick" by which cement reaction could most readily be measured, and 7-day strengths were obtained for each soil at the cement content indicated by the short-cut test procedure for sandy soils (5).

Correlations were then made using these three factors (pH, organic content and 7-day compressive strength) but a definite relationship was not obtained (Figs. 1 and 2). The results of the correlation showed a trend of decreasing strengths with increasing organic contents, although some very low strengths were obtained on soils having organic contents as low as 2,000 parts per million (ppm).

From this work it was indicated that neither the pH nor the organic content is a direct measure of the cement reaction, in a soil-cement system using poorly-reacting soils (6). However, the following general conclusions can be drawn from the data:

1. Unsatisfactory strengths were obtained on all sandy soils having organic contents greater than 20,000 ppm. It is noted however, that equally poor strengths were found in some soils having values as low as 2,000 ppm.

2. Unsatisfactory strengths were obtained on all soils having pH values lower than 5.3, although higher values did not necessarily correspond to higher compressive strengths.

CONTRIBUTING RESEARCH

After analyzing these first unsuccessful correlations, it was apparent that efforts

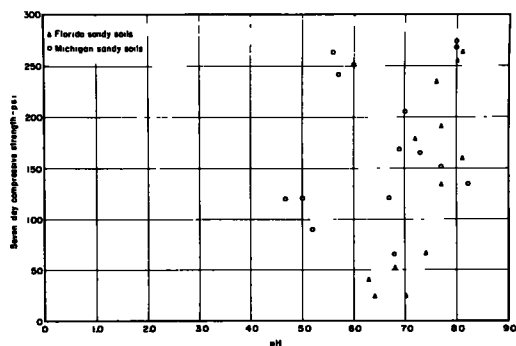


Figure 1. Effect of soil pH on the unconfined compressive strength of soil-cement mixtures.

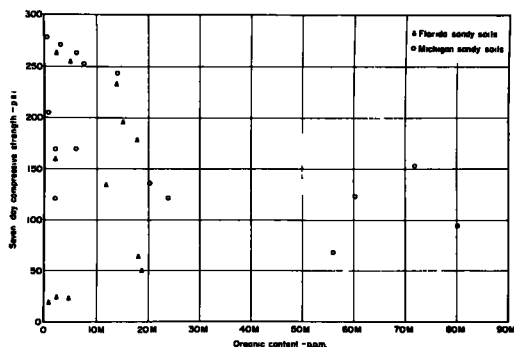


Figure 2. Effect of organic content of sandy soils on the unconfined compressive strength of soil-cement mixtures.

must be focused on the chemical process that was actually taking place when the cement came in contact with a poorly-reacting sand. A search for any published work on this subject drew attention to the extensive, and valuable, research conducted by the staff of D.J. Maclean of the Soil Section, Road Research Laboratory, Great Britain (7). The main finding of this work was that the compounds of a soil's organic matter that were most active in retarding the normal reaction of cement were of the hydroxy-quinone type; a type known to bind metal ions such as those of calcium, and in all probability a major cause in the activity of the soil toward cement. A test to measure this ability of the soil, to immobilize or absorb calcium, was therefore sought.

DEVELOPMENT OF A TEST TO MEASURE CALCIUM ABSORBED BY A SOIL

In order that the absorption of calcium by a soil might be measured, calcium ions must be placed in contact with the soil grains. A standard saturated and carbonate-free solution of calcium hydroxide was selected as the agent for this.

When such a standard solution of calcium hydroxide is mixed with a predetermined amount of soil, the amount of calcium actually absorbed by the soil can be readily determined by titrating the calcium remaining in solution. This amount of calcium,

when compared with that available in the standard solution, provides a direct measure of the activity (or calcium absorbing ability) of the organic matter in the soil.

Titration were performed using the di-sodium salt of (ethylenedinitrilo) tetraacetate dihydrate, commonly referred to as EDTA (8). Due to the presence of magnesium in most soils, an indicator was selected that allowed the titration of calcium in the presence of magnesium. Fisher Scientific's Calcien and HHSNN were investigated, and the latter chosen for its clear and unmistakable color-change (9).

Using these ingredients and standard titration procedures, the following test method was formulated:

CALCIUM ABSORPTION TEST METHOD

Reagents Used

EDTA Solution: Dissolve 3.72 grams of analytical reagent grade di-sodium (ethylenedinitrilo) tetraacetate dihydrate in distilled water and dilute to one (1) liter (0.01 M solution).

Calcium Indicator: Triturate 0.5 gram of 2-hydroxy-1-(2-hydroxy)-4-sulfo-1-naphthyl(azo)-3-naphthoic acid (referred to as HHSNN), and 50 grams of reagent grade sodium sulfate until the dye is thoroughly and evenly distributed.

Calcium Hydroxide: Dissolve 0.7408 grams of reagent grade calcium hydroxide in carbonate-free water and dilute to one (1) liter. Mix occasionally and allow to stand 24 hr.

Potassium Hydroxide, 8 Normal: Dissolve 448.8 grams reagent grade potassium hydroxide in distilled water and dilute to one (1) liter.

Procedure

Prepare soil on a No. 4 sieve and place 100 grams of dry Minus No. 4 material in a beaker. Add 150 ml of the standard calcium hydroxide solution. Allow to stand for 15 min with occasional swirling. Filter the resulting solution through Whatman No. 31 paper inside a Whatman No. 30 paper. Pipet 15.0 ml of the filtrate into a 125 ml Erlenmeyer flask and dilute with 30 ml of distilled water. Add 4ml of 8N potassium hydroxide to raise the pH of the solution to the required range of 12-14 (9), and allow to stand for 3 min with occasional swirling. Add 0.1 gram calcium indicator powder and titrate with the standard EDTA, the end point being attained when the wine color from the indicator turns to a vivid steel-like blue.

On rare occasion, a soil may be encountered that will cause the operator great difficulty in finding a clear end-point in the titration. This interference is usually caused by the presence of certain metals such as copper, iron, nickel, cobalt, and zinc. The addition of 30 mg each of potassium cyanide and hydroxylamine hydrochloride prior to the introduction of the calcium indicator powder will eliminate this interference.

Knowing that the standard solution of calcium hydroxide requires a certain amount of EDTA to satisfy the amount of calcium in it, the difference between this EDTA requirement and the amount needed to satisfy the soil- $\text{Ca}(\text{OH})_2$ aliquot, becomes a direct measure of the amount of calcium taken up by the soil. This is referred to as the calcium absorption factor.

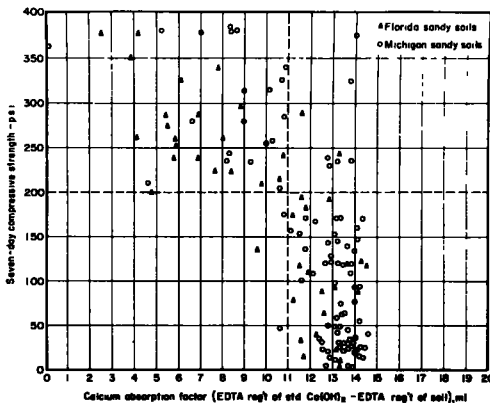


Figure 3. Unconfined compressive strengths of sandy soils with various calcium absorption factors.

Application and Accuracy of Calcium Absorption Test Method

To show the application and accuracy of the calcium absorption test method, it was then performed on 68 samples from Florida and Michigan, and the results were correlated with the 7-day unconfined compressive strength of each soil-cement mixture. The results of this correlation were quite promising, and an additional 70 samples, mostly from Michigan, were procured to further expand the correlation. A plot of the total 138 tests (Fig. 3) showed that in almost every case those soils with low absorption factors had 7-day compressive strengths in excess of 200 psi. Conversely, the majority of the high absorption factors were associated with strengths less than 200 psi. This value was therefore selected as a criterion to distinguish between the normally acting and poorly-reacting sandy soils, and is in agreement with the experience of the Portland Cement Association's Soil-Cement Laboratory in testing these types of soils over the past 25 years.

A line was then drawn at a calcium absorption factor of 11 ml (the difference between the EDTA requirement of the standard $\text{Ca}(\text{OH})_2$ solution and the EDTA requirement of the soil- $\text{Ca}(\text{OH})_2$ aliquot) and chosen as a dividing line; thus, those soils having calcium absorption factors less than 11 ml may be considered normal; those with factors greater than 11 ml, as poorly reacting.

By setting one value as the dividing line, the test is in no way quantitative; that is, it cannot predict just how "bad" a soil will react or how much additional cement will be needed to attain the hardness and durability required to pass the ASTM tests for soil-cement. Rather, in following this procedure, it is possible to determine, in advance of any routine soil-cement testing, only whether or not a soil will react normally with cement. If a soil is noted to be normal acting, tests must then be performed in order to establish a proper cement content for construction.

During the testing of these sandy soils it was noted that a few soils containing more than 15 percent silt and clay gave erroneous results. This was probably due to the much greater amount of surface area available for absorbing calcium. A maximum of 15 percent silt and clay was therefore set for soils to be tested by this procedure. This limitation will not detract from the usefulness of the test, however, for a great majority of the soils involved are always lacking in silt and clay material.

The accuracy of the test method was determined by plotting the calcium absorption values for the 138 soils against their 7-day compressive strength (Fig. 3) and determining the location of the points in regard to the 200 psi line and the 11.0 ml calcium absorption line.

The lower right-hand quadrant represents the area where the poorly-reacting soils should fall; normally-reacting soils should fall in the upper left-hand quadrant. Out of 138 soils tested, 127 or 92 percent of the samples fell in these quadrants and were thus successfully predicted as being either normally or poorly reacting. Of the remaining eight percent or eleven soils, three were predicted to be normal, inasmuch as they had low calcium absorption values, when actually they were poorly reacting because their strengths were below 200 psi. The poor reaction of these soils would be detected on testing using normal ASTM standard procedures for soil-cement. The other eight soils were predicted to be poorly reacting because they had high calcium absorption values; however, they were normally reacting because their strengths were more than 200 psi. These eight soils would have been eliminated without further testing consideration, but the elimination of these few soils is thought to be sufficiently small to make the test and results acceptable.

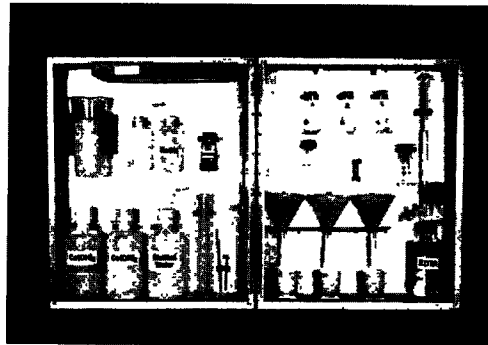


Figure 4. Equipment for calcium absorption test.

CONCLUSIONS

1. Organic content and pH do not in themselves constitute an indication of a poorly-reacting sand. However, a sandy soil with an organic content greater than 20,000 ppm (2 percent) or having a pH lower than 5.3, will in all probability not react normally with cement.

2. The calcium absorption test provides a method of quickly determining, either in the laboratory or in the field, whether or not a sandy soil will react normally with cement in soil-cement construction. Thus, steps may be taken immediately to either (a) improve the reaction of the sandy soil with admixtures, or (b) to find locations of suitable borrow materials. The 92 percent accuracy of the test method is considered acceptable for this work.

ACKNOWLEDGMENTS

The authors wish to thank W. G. Hime and his staff of the Portland Cement Association's Analytical Laboratories for the guidance and counsel they provided throughout the preparation of this work.

REFERENCES

1. Catton, Miles D., and Felt, E. J., "Effect of Soil and Calcium Chloride Admixtures on Soil-Cement Mixtures." HRB Proc. (1943).
2. Lambe, T. W., Michael, A. S., and Moh, Za-Chieh, "Improvement of Soil-Cement with Alkali Metal Compounds." HRB Bul. 241, pp. 67-108 (1960).
3. Winterkorn, H. F., et al., "Surface Chemical Factors of Importance in the Hardening of Soils by Means of Portland Cement." HRB Proc., 22:385 (1942).
4. A modification of ASTM Designation C40-48, "Standard Method of Test for Organic Impurities in Sand for Concrete." based on data contained in "Comparison of Standards for Colorimetric Test for Sand." ASTM Proc., 37th Annual Meeting, Vol. 34, Part I.
5. Norling, L. T., and Packard, R. G., "Expanded Short-Cut Test Procedures for Determining Cement Factors for Sand Soils." HRB Bul. 198, pp. 20-31 (1958).
6. Catton, M. D., "Research on Physical Relations of Soil and Soil-Cement Mixtures." HRB Proc. 20:821-855 (1940).
7. Arkley, V., "An Investigation of Soil Organic Matter on the Setting of Ordinary Portland Cement-Chemical Studies on the Active Fraction." Research Note No. RM/3193/VA, Dept. of Scientific and Industrial Research, Road Research Laboratory, Harmondsworth, Middlesex, England (Feb. 1958).
8. Welcher, F. J., "The Analytical Uses of Ethylenediamine Tetraacetic Acid." D. Van Norstrand Co., Inc., Princeton, N.J. (1957).
9. Patton, J., and Reeder, W., "Indicator for Titration of Calcium with (ethylenedinitrilo) Tetraacetate." Analytical Chemistry, 28:1026-8 (1956).