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Stabilization of soils with lime and sodium silicate

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STABILIZATION OF SOILS
WITH LIME AND SODIUM SILICATE

by

Harold Bernard Ellis

A Dissertation Submitted to the
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Major Subject: Soil Engineering

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INTRODUCTION

Occasionally in major construction projects and frequently in military combat operations, vital roadways deteriorate under heavy traffic and become impassible, particularly during adverse weather. The failure of a vital roadway at a crucial time can be intolerable. In a civilian construction project such a delay may threaten the contractor with great financial loss. In a military operation the consequences may be more drastic, being measured in men's lives and in the prolongation of the entire conflict.

The usual solution to a problem of this type is to hastily haul in granular materials to replace or bury the soil in the failure area. If granular materials are not locally and conveniently available, a better solution may be sought in the stabilization of the existing soils. The requirements of the soil stabilization materials and method which would give satisfactory results under such adverse conditions are indeed great. They would need to be effective in a short period of time, would need to operate in a wide variety of soil types and under widely varying moisture conditions because time for elaborate testing would not be available. The stabilizing materials should be easy to obtain, store and transport. Cost might be a lesser consideration.

Experience in chemical grouting operations has indicated that sodium silicate when mixed with soluble calcium chloride reacts instantly to stabilize troublesome foundation soils such as quicksand, thereby correcting failures and arresting severe seepage conditions. Samples taken from soil masses stabilized in this manner have exhibited very high

compressive strengths. It was conjectured from these experiences that if such a reaction could be slowed down and controlled, a method might be developed that could be employed effectively in highway construction operations. This thesis discusses the results of laboratory tests of four widely different soil types which have been stabilized with admixtures of sodium silicate and lime under optimum conditions of moisture content and compaction.

Also discussed are the results of tests using the same admixtures as a stabilizer of a problem soil under adverse moisture conditions.

SODIUM SILICATE AND LIME: HISTORY, PRODUCTION AND USES

History of Sodium Silicate

The action of dissolved silicates in the stabilization or cementation of granular strata is a natural geologic process, for example, the cementation of siliceous sandstones and conglomerates¹. The first reported reference to sodium silicate appears in the writings of Pliny, the Elder. He tells of Phoenician mariners who, while cooking on a sand spit at the mouth of the Belus River in Syria, used blocks of sodium nitrate around their wood fire to support their cooking pots. The action of the heat on the sand in contact with the sodium salt produced a glass.

In about 1620, Basil Valentine reported that he produced a soluble silicate by melting powdered silica and "sal tartari" and used it for molding blocks of sand and for petrifying wood. In 1648, Glauber was able to cause the precipitation of a metallic-silica gel by mixing a metallic salt with "oleum silicium", as he termed sodium silicate. The real beginning of the industrial development of soluble silicates probably dates from about 1825 when Johann Von Fuchs published his treatise on the commercial development of water glass. He proposed many uses for soluble silicates, including their use as hardening agents for artificial stone². Since that time, many other uses have been developed for soluble silicates, including uses as detergents, deflocculating agents, films and

¹Iler, Ralph K. The colloid chemistry of silica and silicates. Ithaca, New York. Cornell University Press. 1955. p. 15.

²Vail, James G. and Wills, John H. Soluble silicates their properties and uses. American Chemical Society Monograph 116, Vol. 1: 4-6. 1952.

coatings, sizings, corrosion preventives, bleaching agents, adhesives and cements. The ability of soluble silicates to bond to many different substances has resulted in their widespread industrial use as adhesives, the most important of which, tonnage-wise, is in the manufacture of corrugated paper¹. Soluble silicates are also used extensively in the manufacture of wood veneers, asbestos board and grinding wheels².

Present Methods of Producing Sodium Silicates

The most commonly occurring natural form of silica is quartz, and is essentially insoluble in pure water at temperatures below 150° C. As temperature increases above 150° C or when the pH of the solvent exceeds 9, solubility increases. When combined with the oxides of the alkali metals, it becomes much more soluble but when combined with the oxides of the alkaline earth metals, it is less soluble. Even slight traces of the polyvalent metals have a profound effect on solubility of dissolved alkali metal silicates. This is particularly true of the more siliceous forms of sodium silicate because they are of a lower pH³. It is believed that these chemical properties make sodium silicate potentially useful in soil stabilization. For example, the introduction of a small proportion of an acid or of an alkaline earth metal ion will precipitate an aqueous solution of sodium silicate and form a gel. The

¹Ibid., p. 11.

²Philadelphia Quartz Company. PQ soluble silicates, properties, applications. Philadelphia Quartz Company Bul. 17-1. [ca. 1957]. p. 11.

³Iler, op. cit. pp. 4-13.

gelation speed will depend upon the SiO_2 concentration and the ratio of acid to silicate or the ratio of alkaline earth to silicate.

In the United States, sodium silicate is manufactured by the fusion of quartz sand with sodium carbonate at temperatures of 1200-1400° C. A preliminary heating at 700° C may be included in the process to drive off most of the CO_2 , thereby reducing the agitation caused by escaping gas when melting proceeds at the higher temperatures. The final product of fusion is a clear, soluble glass which is either the neutral glass ($\text{Na}_2\text{O} : 3.3 \text{ SiO}_2$) or the more alkaline glass ($\text{Na}_2\text{O} : 2.1 \text{ SiO}_2$). From these two products many other forms of sodium silicate are manufactured, depending upon the needs of industry¹. For example, in 1956 the Philadelphia Quartz Company listed some 37 different forms of sodium silicate plus several potassium silicates². Each of these products possesses different properties; therefore, any potential user of one of these products should assure himself that the sodium silicate to be used is the one with the most desirable properties and that the desired type is clearly specified.

The various other forms of sodium silicate are produced by dissolving the initially formed glass in water. This is usually done by passing the molten glass from the furnace into water held at 100° C in a slowly rotating drum. The quick cooling solidifies and fractures the glass. The granules then gradually dissolve. Fresh water is added

¹Vail and Wills, op. cit. pp. 17-22.

²Philadelphia Quartz Company. PQ silicates of soda. Philadelphia Quartz Company Bul. T-17-1A. [ca. 1957].

as the more viscous solution is drawn off as supernatant liquor. Stationary dissolvers are also used in which cakes of sodium silicate are submerged in water and subjected to a steam pressure of 90-100 pounds per square inch. When the solution reaches the desired concentration, it is drawn off and the dissolver replenished to repeat the process. The primary purpose of the pressure in this process is to be able to quickly eject the solution at the correct moment to avoid over-concentration. The crystalline hydrates are produced from the solutions by processes of recrystallization through controlled seeding¹.

History of Lime

Lime appears to have been used since prehistoric times. The ancient Egyptians had mastered the art of mortar and plaster, some of which is still in perfect condition today in the Pyramid Cheops. The ancient Greeks used lime in the construction of the Acropolis. The Romans made extensive use of lime as a mortar in their buildings and used it in the construction of their first and perhaps most famous road, the Appian way which is still in service today². They also found that when lime mortar was combined with a volcanic ash known as pozzuolani and found near Naples, a much stronger cement developed. The great cathedrals and castles of the Middle Ages were constructed with lime mortar³.

¹Vail and Wills, op. cit., pp. 25-29.

²Lazell, E. W. Hydrated lime. Pittsburgh. Jackson-Remlinger Ptg. Co. 1915. pp. 9-10.

³Eckel, E. C. Cements, limes and plasters. New York. John Wiley and Sons. 1928. pp. 1-2.

It appears that all civilizations have learned the value of lime as a construction material. The ancient Peruvians and Mexicans acquired the art of using lime mortar for their structures, presumably quite independently of the peoples of the Mediterranean basin.

Although the Romans used lime with sand and rock in their roads and the Chinese have used lime for many years as a soil stabilizer, modern man has only recently found it useful in road construction. In 1920 the University of Missouri in cooperation with the highway department of that state began experimentation with lime as a soil stabilizer. But true progress had to wait for the development of a better soil technology¹. Suffice it to say that extensive use is made of soil-lime stabilization techniques today.

Present Methods of Producing Limes

Lime is manufactured commercially by heating (or calcining) crushed limestone, which is predominately calcium carbonate, to temperatures of 900° C to drive off the CO₂. Until recent decades this was usually done in vertical kilns, so constructed that the limestone was fed in at the top to descend slowly through the rising hot gases that entered through ports in the wall of the kiln near the base. The lime was then removed from the bottom. This process can be operated continuously².

¹National Lime Association. Lime stabilization of roads. National Lime Association Bul. 323. 1954. p. 9.

²Lazell, op. cit., pp. 100-107.

Currently about 50% of the lime production is done with horizontal, rotary kilns similar to those used in the portland cement industry. The rotary kiln requires that the limestone be crushed to a finer and more uniform size before calcining than does the vertical kiln. Previously this was an objectionable feature of the rotary kiln, but with rising labor costs and the possibility for increased automation, instrumentation and quality control over the process, the rotary kiln appears to be gaining in favor despite the greater initial cost¹.

As with sodium silicates, lime is a loosely defined term which includes the oxides and the hydroxides of calcium and of these mixed with magnesia. The oxide is usually referred to as caustic lime or quick lime; the hydroxide as slaked lime. The magnesia content may vary from zero to 50% and the mixture still be referred to as lime. If in excess of 50% it is classified as magnesia. According to the National Lime Manufacturers' Association, limes are classified by magnesia content as follows²:

Classification	Magnesia Content
High-calcium lime	0-5%
Magnesian lime	5-25%
Dolomitic	25-45%
Super-dolomitic lime	over 45%

¹Boynton, R. S. and Jander, F. K. Lime and limestone. In Kirk, Raymond E. and Othmer, Donald F., eds. Encyclopedia of chemical technology. Vol. 8. New York. The Interscience Encyclopedia Inc. 1952. p. 362.

²Ries, H. and Watson, Thomas L. Engineering geology. New York. John Wiley and Sons, Inc. 1946. p. 549.

Although this classification defines limes with 5 to 25% MgO as a magnesian lime, limes in this class are rarely seen. Usually either the magnesia content is low or in excess of 30%.

If the magnesia in the lime is not hydrated while the calcium is, the lime is referred to as a monohydrate. If both are hydrated, it is a dihydrate¹.

Because limestone, the raw material from which lime is produced, occurs in nature in widely varying compositions and with impurities such as oxides of iron, aluminum, silicon and sulfur, lime can vary widely in its constituents.

Another type of lime not included in the above mentioned categories but of some importance is hydraulic lime, so called because of its ability to harden under water. Hydraulic lime is made from limestone which is high in silica, and/or alumina. During the calcining operation, which is carried on at a higher temperature, sintering takes place during which calcium silicates or aluminates are formed which are not unlike the compounds found in natural cements and portland cement².

The magnesia in a magnesian or dolomitic lime varies in properties depending on the calcining temperature. If the calcining is done quickly at the minimum temperature, the product will have a specific gravity of 3.00 to 3.07 and will readily slake with water, recarbonate in air and

¹Lu, L. W., Davidson, D. T., Handy, R. L. and Laguros, J. G. The calcium-magnesium ratio in soil lime stabilization. Iowa State University of Science and Technology Engr. Expt. Sta. Bul. 195. 1961. p. 17.

²Eckel, op. cit., pp. 172-177.

harden like calcitic lime. If calcining is done at a higher temperature or continued too long, the specific gravity is higher, at 3.61 to 3.80, and the magnesia will not slake with water nor recarbonate in air. Magnesite will decompose at about 750° C whereas calcium carbonate requires a temperature of 900° C. This would explain why the magnesia in dolomitic lime does not hydrate as readily as the CaO since the calcining must be done at the higher temperature¹.

Because of the numerous variations that can exist within the general term lime, a user of lime must be careful to determine the type of lime best suited to his purpose, and that the desired type is clearly specified.

Sodium Silicates in Construction

The construction industry has made only limited use of sodium silicate solutions, confining the applications to unusual foundation and construction problems and to road construction operations of a very minor extent.

The earliest reported use of sodium silicate in the construction industry appears in literature dating from about 1919. At that time, Albert Francois, a French engineer, found that the effectiveness of cement grout could be increased by preceding the grout with an injection of alum followed by sodium silicate. Apparently the slippery gel formed by the combination, coated the granular surfaces and permitted greater penetration of the cement grout².

¹Eckel, op. cit., p. 155.

²Vail, James G. and Wills, John H. Soluble silicates their properties and uses. American Chemical Society Monograph 116, Vol. 2: 485. 1952.

The Joosten process

A few years later, a Dutch engineer, Dr. Hugo Joosten, injected sodium silicate as a grout into deep foundations and followed it with an injection of calcium chloride. He found that when these two chemicals came into contact, an insoluble gel was immediately formed, filling the voids in the foundation material and preventing the seepage of water or the continuance of settlement. This method is still in use in the heavy construction industry¹.

An outstanding example of the use of the Joosten process for the stabilization of existing foundations undergoing dangerous settlement is the stabilization job done in Clewiston, Florida by the Chemical Soil Solidification Company of Chicago. In this case, the foundations under a heavy concrete base of the mill engine in a sugar mill of the U. S. Sugar Company were settling at a dangerous rate. Injection pipes were driven at varying angles, spacings and depths, into the sand under the concrete base. While driving the injection pipes, a solution of sodium silicate was injected under pressures as high as 400 psi. As the pipes were withdrawn, a calcium chloride solution was injected, and the subsoil was effectively solidified. The injection pipes were driven with pneumatic hammers and withdrawn with hydraulic jacking equipment. Some 22,900 gallons of sodium silicate and 16,600 gallons of calcium chloride solutions were used. The concentrations of these solutions were not reported. Approximately 660 cubic

¹Philadelphia Quartz Company. Silicate P's and Q's. Vol. 35, No. 5: 1-4. 1955.

yards of sand were solidified at a unit cost of \$23 per cubic yard. This project was completed in September 1943, and observations 18 months after the work was completed indicated that no additional settlement had occurred¹.

The Joosten process has also proven very effective in stopping the percolation and seepage of waters. Three instances may be cited which illustrate the sealing abilities of silica gel in permeable media. In the tunnels of the Pennsylvania Turnpike, severe damage was being caused by the seepage of water through construction joints and cracks. In winter, this condition was worsened by the freezing temperatures which caused exhaust ducts to fill with ice, restricting the flow of ventilating air. To eliminate the difficulty, the Manu-Mine Research and Development Company of Reading, Pennsylvania was engaged. Their recommendations were to drill through the lining of the tunnel, inject an expanded shale aggregate known as Lelite, into the void space between the lining and the tunnel roof, and then inject sodium silicate and calcium chloride. This reduced the leakage by approximately 90%².

Another example of the effectiveness of these chemicals in sealing-off seeping ground waters is the work done in a sloping tunnel driven by the Alpha Coal Company of Alpha, Illinois. Work had been stopped

¹Chemical injections solidify soil to stop foundation settlement. *Construction Methods* 27, No. 12: 78. 1945.

²Stickler, C. W., Jr. and Allan, A., Jr. Chemical sealing stops leakage in tunnels of Pennsylvania Turnpike. *Civil Engineering* 24, No. 11: 46-48. 1954.

because the tunnel had entered an aquifer which was bearing water under a 50-foot head at a depth of 110 feet below ground surface. The water-bearing stratum was a 4-foot layer of medium-fine sand which occurred under a layer of hard blue clay and over coal and slate. The Joosten process was employed under the supervision of the Chemical Soil Solidification Company of Chicago. In this case, the injection pipes were driven radially into the sand stratum and the two chemicals were injected to form an oblong ring of solidified sand around the tunnel, extending ahead of the tunnel for 12 feet. This effectively sealed off the leakage and the tunnel was advanced 8 feet. Then the injection pipes were again driven into the sand stratum and chemicals injected. This process was repeated seven times until the tunnel had progressed entirely through the aquifer and into the lower stratum, where no more leakage was observed. In this case, 8400 gallons of silicate solution and 5100 gallons of calcium chloride solution were used to advance the tunnel for a distance of 62 feet. The total job required 2 months¹.

A third example of the use of a silica gel to prevent leakage is the rehavilitation of an old water reservoir in Easton, Pennsylvania. The reservoir was 40 years old, constructed of concrete on a loose rock base. Cracking of the concrete, the freezing and thawing, and the loss of subsoil by the erosion had reduced the reservoir capacity by 30%. Repair of the slabs included removal of the disintegrated concrete, jacking of the slabs back into proper positions, and filling of voids behind the

¹Johnson, J. W. and Reidel, C. Martin. Water stopped in Alpha slope. Coal Age 54, No. 7: 78-80. 1949.

slabs by blowing sand through 2 inch drill holes. Sodium silicate and calcium chloride were then injected through the drill holes to stabilize the sand and the subsoil. Finally, the concrete slabs were coated with 3" of new concrete. When the work was completed, the reservoir was restored to its original 1,000,000 gallon capacity¹.

The single solution processes

Another method of injection, using a different admixture with sodium silicate has been developed by Charles Langer in which the precipitation reaction is much slower than in the Joosten method. He found that chemicals could be mixed prior to injection and injected as a single shot. At present, the most common admixture is sodium bicarbonate which lowers the pH and converts sodium silicate to a silicic acid gel. The gel, in this case, is not as stable as in the Joosten method and will gradually deteriorate. Nor does it attain the strength of the Joosten method. However, it has distinct advantages in excavations of loose, free-flowing sands, particularly tunneling operations, where permanent strength is not essential².

An excellent example of the successful use of a single solution process is the construction of footings for a power line across the sandy wastes of the Mojave Desert by the Los Angeles Department of Water

¹Johnson, William B. and Fulmer, John O. Subsoil stabilization saves 40 year old reservoir. The American City 69, No. 5: 122-124. 1954.

²Philadelphia Quartz Company. Soil solidification [by the] sodium silicate-bicarbonate method. Hectographed special bulletin. Philadelphia. Author. [ca. 1956].

and Power. Each tower of the power line required four concrete footings with diameters ranging from 20 to 48 inches. If formed footings had been constructed in this loose sand, it was estimated that it would have taken five days per tower per crew. By injecting a mixture of sodium silicate and bicarbonate of soda into the sand, the holes were drilled with an auger in as little as 35 minutes each, and the concrete placed without form work. The mixture injected was 12 parts Na_2O :3.22 SiO_2 at a viscosity of 41° Baume mixed with 60 parts water and with 28 parts of a saturated solution of sodium bicarbonate. This mixture set-up in 45 to 60 minutes and attained maximum hardness in about 3 days, although drilling was done as soon as 2 hours after injection. By stabilizing the sand, it was estimated that the work required to prepare the holes for placement of concrete was reduced to 1/40 of that required for formed footings, a substantial savings¹.

Another example of the same method is a tunneling project for a storm sewer in San Francisco. In this job a loose, free-flowing dry sand was encountered that flowed into the tunnel about as fast as it could be removed. The tunnel was a ten foot, horseshoe-shaped section, 2700 feet in length. To prevent the sand from flowing into the tunnel, the silicate-bicarbonate mixture was injected into the tunnel face for a distance of 10 feet. After allowing about 40 to 80 minutes for the silica gel to form, digging was continued. This process was repeated

¹Gershoy, E. C. and Mueller, Carl. Soil stabilization speeds tower footing excavation. Electrical World 135, No. 3: 66. 1951.

as long as the tunnel was in sands. Pressure required for injection was 200 pounds per square inch, and 30 to 45 gallons of mixture were required for each cubic yard of sand stabilized¹.

Stabilization of shallow foundations

More recently, in Denver, the percolation of sodium silicate, followed by a calcium chloride solution, into the foundation soils, greatly increased their durability and strength for residential structures. In this instance a trench was dug along the line for the proposed foundation walls in soils which slaked badly when exposed to air and moisture. A silicate of a 1:3.22 soda-silicate ratio, diluted with about 3 parts water was poured into the trench to a depth of 2 1/4 inches. When this had seeped into the ground, a solution containing 2 pounds of calcium chloride in each 20 gallons of water with 3 ounces of sodium chloride was poured into the trench. Within 24 hours, this solution had soaked into the soil and the soil had developed strengths which supported loads of 7000 pounds per square foot without settlement. This practice is now in general use in the Denver Area².

Sodium silicates in road construction

Sodium silicate, despite the spectacular successes reported in foundation work, seems to have met with rather discouraging results

¹Wright, Roy E. Chemical stabilization of sand speeds driving of 10-foot tunnel. *Engineering News-Record* 143, No. 5: 42. 1949.

²Denver Fire Clay Company. Soil petrification with sodium silicate and calcium chloride. Mimeographed. Denver. Author. [ca. 1957].

as a soil stabilizer in highway research. Mainfort tried combining a highly siliceous sodium silicate with sodium aluminate, with cement, with a lime asphalt emulsion, as well as using the silicate alone, but he concluded that the results were generally unsatisfactory¹. In an earlier paper, he held open a promise by suggesting that "by using the powdered form of these chemicals instead of the solution, better results and simplified methods could be obtained"². Winterkorn was less optimistic. He concluded after considerable research that "it appears best, if sodium silicate is to be employed, use it as is, and not to attempt to improve it by other reactions" and that it was "hardly worthwhile to bypass the more promising avenues of research just for the sake of finding the ideal partner for sodium silicate"³. But some successes have been reported with sodium silicate in road work in which the solutions were applied to the surfaces and allowed to percolate into the soils. Prestlicka reported that an improved type of macadam roads was constructed in Europe and America, wherein a coarse graded base was cemented by a slurry composed of finely ground limestone and sodium silicate. Although in other methods, a high silica content is desired, when being

¹Mainfort, R. C. A summary report on soil stabilization by the use of chemical admixtures. Washington, D. C. U. S. Dept. of Commerce. 1951. p. 11.

²_____ A laboratory study of the effectiveness of various chemicals as soil stabilizing agents. Washington, D. C. U. S. Dept. of Commerce. 1945. p. 11.

³Winterkorn, Hans. F. Final report on beach sand stabilization research, 15 November 1947 to 15 November 1949. Washington, D. C. U. S. Navy Bureau of Yards and Docks. [ca. 1950]. p. 69.

combined with limestone, the more alkaline solution is preferable because of an increased reactivity with the calcium carbonate¹.

Preliminary Investigations

The great need for a rapid stabilizer in emergency and military road construction operations in the light of the rapid precipitation of silicate in the presence of a metallic ion offered a challenge, but preliminary investigation seemed to rule out the possibility of mixing the dry chemicals to the soil without water because the lack of moisture prevented the necessary compaction. When only one chemical was added with water to the soil, followed by compaction, the soil then became too impervious to permit the percolation of the other additive, even though it was highly soluble, such as calcium chloride. This suggested an alternative: the use of a calcium compound which was only slightly soluble. Calcium hydroxide, which in commercial grades is known as hydrated lime, seemed likely. It was hypothesized that since it has a low solubility, it might remain undissolved long enough to inhibit the precipitation of the sodium silicate even though some water were added to the soil to aid compaction. Preliminary molding of small specimens in this way produced sufficient strength to indicate that some degree of stabilization was achieved. At the conclusion of the preliminary experimentation, it was decided to pursue this avenue to

¹Great Britain Department of Scientific and Industrial Research, Road Research Laboratory, Soil mechanics for road engineers. London, Her Majesty's Stationery Office. 1954.

determine what type of soil could be stabilized in this manner, what forms of sodium silicate and lime were best and in what percentages, and to attempt to determine what the reaction products might be.

MATERIALS AND METHODS

Plan of the Investigation

The principle course of experimentation proceeded in several stages. The number of stages was determined in general by the number of variables which it was believed might affect the results. At the conclusion of each stage, a decision was made regarding the results. Thereafter the conditions which appeared to produce the best results were maintained while other conditions were varied. The several stages of the investigation were planned as follows:

1. Determination of the optimum ratio of selected sodium silicates with two principle types of lime (i.e. calcitic and dolomitic) at an estimated optimum moisture content for maximum dry density.

2. Determination of the effect of varying moisture content upon strength and dry density of specimens treated with most promising sodium silicate-lime combination.

3. Determination of the optimum curing conditions.

4. Determination of the effect of curing time on unconfined compressive strength.

5. Determination of the durability of the stabilized specimens when subjected to freeze-thaw and wet-dry tests.

6. Determination of the effectiveness of the most favorable combination of additives as a stabilizer of an extremely wet soil.

In addition to the above, an effort was made using the x-ray diffractometer analysis to determine the reaction products of sodium

silicate and lime. Also, incomplete but indicative tests were performed to ascertain the effect of mixing and compaction time on strength and density.

Description of Soils

Because the engineer must be prepared to cope with a wide variety of problem soils, four soils were used in this study, selected on the basis of their dissimilarity. They included a dune sand, a friable loess, a lean clay and a plastic clay. The soils are described as follows. The symbols shown in parentheses are the laboratory designations used to identify the samples.

The Dune Sand (S-6-2) is a fine, clean, uniformly graded soil believed to be aeolian in origin. This sample was taken from the Iowan drift plain in East-Central Iowa. When dry as it would be in an arid region, this soil would present particularly difficult stability problems because of its nearly complete lack of any clay fractions which could act as a binder.

The Friable Loess (20-2) was sampled in Harrison county in South-Western Iowa, adjacent to the Missouri River flood plain, and is believed to be aeolian. The sample is grayish-brown in color, oxidized and unleached, and from a deposit which exceeds 100 feet in thickness. It was laid down during the early Wisconsin Glacial Stage.

The Lean Clay (409-12c) is an oxidized, unleached glacial till of the Kansan ice age, sampled in Ringgold county in Southern Iowa. The sample is yellowish in color and was taken from the C horizon between 4 1/2 and 10 1/2 feet in depth.

The Plastic Clay (528-8) classified as a Kansan Gumbotil, has a reddish color and was found in Keokuk County, Iowa. It is believed to be a severely weathered loess of the Mahaska soil series. This soil presents a most difficult stability problem because of the large fraction finer than one micron, which is predominantly montmorillonite. The deposit varies in depth from a few feet to a few tens of feet.

The properties of the four soils are shown in Table I.

Description of Additives

Sodium silicate is not a compound with a fixed molecular structure, but exists in a wide variety of forms and compositions. The molar ratio of Na_2O and SiO_2 can vary from 2:1 to 1:4. Sodium silicate is marketed as a solution, as a powder, in lump form and as granules. So as to observe the effects of these variations, both solution and powder forms were used. The properties are listed in Table II.

The limes used are marketed by the U. S. Gypsum Company under the trade names of Kemical and Kemidol, being respectively calcitic lime and dolomitic lime. The properties are listed in Table III.

Preparation of Bulk Soils

The four soils used in this investigation were all air dried by spreading them over a large area and exposing them to air currents for at least one week. Each soil was then crushed in a small crushing plant, followed by pulverization by a motor driven-rubber pestle in a mortar until it passed a U. S. Standard No. 10 sieve. Each soil was then stored in large galvanized iron cans with covers until needed for

Table I. Properties of soils

	Dune sand (S-6-2)	Friable loess (20-2(VII))	Lean clay (409-12c)	Plastic clay (528-8)
Location	Benton County Eastern Iowa	Harrison County Western Iowa	Ringgold County Southern Iowa	Keokuk County South Central Iowa
Geological identification	Wisconsin aeolian sand uniformly graded fine grained clean, oxidized leached	Wisconsin oxidized loess	Kansan glacial till oxidized calcareous	Kansan glacial gumbotil severly weathered
Soil series	Carrington (?)	Hamburg	Burchand	Mahaska (?)
Great soil group	Brunizem		Brunizem	
Textural composition: (% by weight)				
Gravel (2 mm)	0.0	0.0	0.0	0.0
Sand (2.0-0.74 mm)	95.8	0.4	31.9	17.5
Silt (74-5 μ)	1.2	82.6	28.9	8.5
Clay (<5 μ)	3.0	17.0	40.0	74.0
Colloids (<2 μ)	2.9	12.3	36.0	71.0
Finer than 0.42 mm	94.2	96.0	93.5	95.0
Atterberg limits:				
Liquid limit, %	19	33	42	87
Plastic limit, %	no plasticity	21	20	35
Plasticity index	-	12	22	52

Table I (Continued)

	Dune sand (S-6-2)	Friable loess (20-2(VII))	Lean clay (409-12c)	Plastic clay (528-8)
Standard Proctor values:				
Density in pcf	109.9	105.0	115.4	95.2
Optimum moisture content	12.3	18.1	13.7	28.0
Engineering classification according to:				
Unified soil classification system	SP	CL-ML	CL	CH
AASHO system	A-3	A-4 (8)	A-7-6 (12)	A-7-6 (20)
Chemical properties:				
Organic content	0.04%	0.2%	0.17%	0.2%
Carbonates	0.02%	10.2%	2.10%	0.8%
pH	6.5	7.8	8.25	6.5
Cation exchange capacity m.e./ 100 grams	1.3	14.7	20.6	38.3

Table II. Properties of sodium silicates

Manufacturer's designation ^a	Ratio Na ₂ O/SiO ₂	% NaO ₂	% SiO ₂	% H ₂ O	Viscosity at 68° C	
					Baume	Centipoises
N-Silicate	1:3.22	8.90	28.7	62.4	41.0	180
D-Silicate	1:2.00	14.70	29.4	55.9	50.5	350
Metso Granular	1:1	29.00	28.7	41.7	-	- Powdered in hammer mill from granular form
SS:65 Pwd	1:3.22	23.10	74.4	1.5	-	- Passing 65 mesh sieve
Metso Anhydrous	1:1	51.00	45.5	0.5	-	-

^aThe silicates were provided by the Philadelphia Quartz Company, Philadelphia, Pa. manufacturer.

Table III. Properties of the limes^a

Chemical analysis percent by weight	Kemical ^b	Kemidol ^c
Calcium Oxide	73.82	49.60
Magnesium Oxide	0.59	32.00
R ₂ O ₃	0.60	0.17
SiO ₂	0.28	0.34
SO ₃	0.25	1.13
Loss on Ignition	24.10	16.97

^aBoth types of lime were produced by the U. S. Gypsum Company.

^bKemical is a calcitic, hydrated lime.

^cKemidol is a dolomitic, monohydrate (type N) lime.

experimentation, at which time only as much as was needed for a day's work was taken. The unused material remained in covered cans in the basement of the laboratory where temperature variations were small. In this way, the hygroscopic moisture content was held nearly constant.

Preparation of Samples

Proceeding from air dried soil, each sample was gravimetrically metered on a laboratory triple beam balance to within 1 gram of a predetermined weight which was sufficient to prepare the desired number of specimens for a particular series of tests. The trial amounts of lime and sodium silicate were weighed to an accuracy of 0.1 gram. The distilled water which was needed in a sample to provide the desired moisture content was measured volumetrically in a graduate to an accuracy of 0.1

milliliter. Distilled water was used throughout to eliminate the effects that the mineral content of tap water might have on test results.

When the chemical additives were to be used in a dry powder form, they were thoroughly blended with the soil sample prior to the addition of any water. This was done to prevent or at least inhibit any reaction between the lime and silicate prior to the completion of blending. It had been observed previously that the two compounds in dry powder form could be mixed without noticeable change, but when this mixture is brought in contact with water, an observable reaction occurs. Therefore the prescribed amount of water was not added until immediately prior to the mixing operation which preceded molding.

When the sodium silicate was added as a solution, it was combined with whatever additional water was needed for the desired moisture content. In computing the required water, the water of the solution was considered as being available to aid in compaction, and hence was included in the total water. Prior to the addition of the water and dissolved sodium silicate, the soil and dry lime were thoroughly blended.

Mixing of Samples

Mixing of the dry ingredients was always done by hand until the mix appeared homogeneous, primarily to prevent loss by dusting. The lowest speed of the available power mixer is too rapid and violent and was found to raise a cloud of fine dry material. This was objectionable from the point of view of the health of people in the laboratory as well as affecting the accuracy of results. When hand mixing, as well as when handling the finished specimens, it was necessary to wear

rubber gloves. The alkalinity of the admixtures is high and, in presence of moisture, will attack the skin; therefore, some protection is essential. The protection was particularly necessary with the sand specimens and to a progressively lesser degree with the finer soils presumably because the higher permeability of the coarser soils permitted the migration of the dissolved alkalis to the surface of the specimen.

After a dry batch was thoroughly mixed it was set aside, and smaller quantities, each large enough for about three specimens and two moisture content determinations, were taken from it. To each of these smaller portions the desired volume of distilled water was carefully added. Because dusting was no longer a problem, these were then mixed in a Hobart, electrically operated Model C-100 kitchen mixer for 30 seconds at the lowest speed. The mixer was then stopped and the mixing paddle and the sides of the mixing bowl scraped clean. The mixing was then continued for 30 seconds more. The mixer speed of the second phase was done at the second or next higher speed for sands and loess but continued at the lower speed for clays. The higher speed caused the clays to compact in the bottom of the bowl and prevented thorough mixing. The higher speed appeared to produce a more homogeneous mix for the coarser materials. After this second half minute of mixing, the soil was ready for molding into a three specimen set.

Molding of Specimens

All specimens were molded in a cylindrical mold, two inches in diameter and four inches in length as shown in Figure 1. The

cylindrical mold was placed in position on the pedestal and supported by temporary supports. A measured quantity of treated soil was inserted. The quantity of soil was weighed so as to produce a specimen 2.000 inches in diameter and 2.000 ± 0.050 inches in length. The hammer head was placed upon the soil mixture and the 5 pound weight dropped once through a 12" distance. The temporary supports were then removed from under the cylinder. The weight was dropped four more times and the molding cylinder inverted, the hammer replaced, and 5 more blows rendered upon the specimen. The specimen was then extruded with the extrusion apparatus, weighed to the nearest 0.1 gram, and the height measured to the nearest .001 inch. In the extremely moist mixtures, particularly in the more granular soils, it was found that the mixtures adhered to the hammer head. To prevent this, a single sheet of thin plastic (Saran wrap) was used to cover the molding base and hammer head. This plastic sheet could then be stripped off the specimen without causing irregularities in the surfaces. Otherwise the large chunks of soil adhering to the hammer head rendered the specimens unusable.

The number of specimens made from each batch depended upon the particular investigation, but in most series, 15 to 21 specimens were produced in sets of three. For example, if it were desired to produce a series in which only the moisture content was to be varied, sufficient soil, lime and sodium silicate for the entire series were mixed dry. Then smaller amounts of soil, sufficient for a set of three specimens of like moisture content, were weighed from the larger batch. The correct amount of water was determined for that amount of soil, added

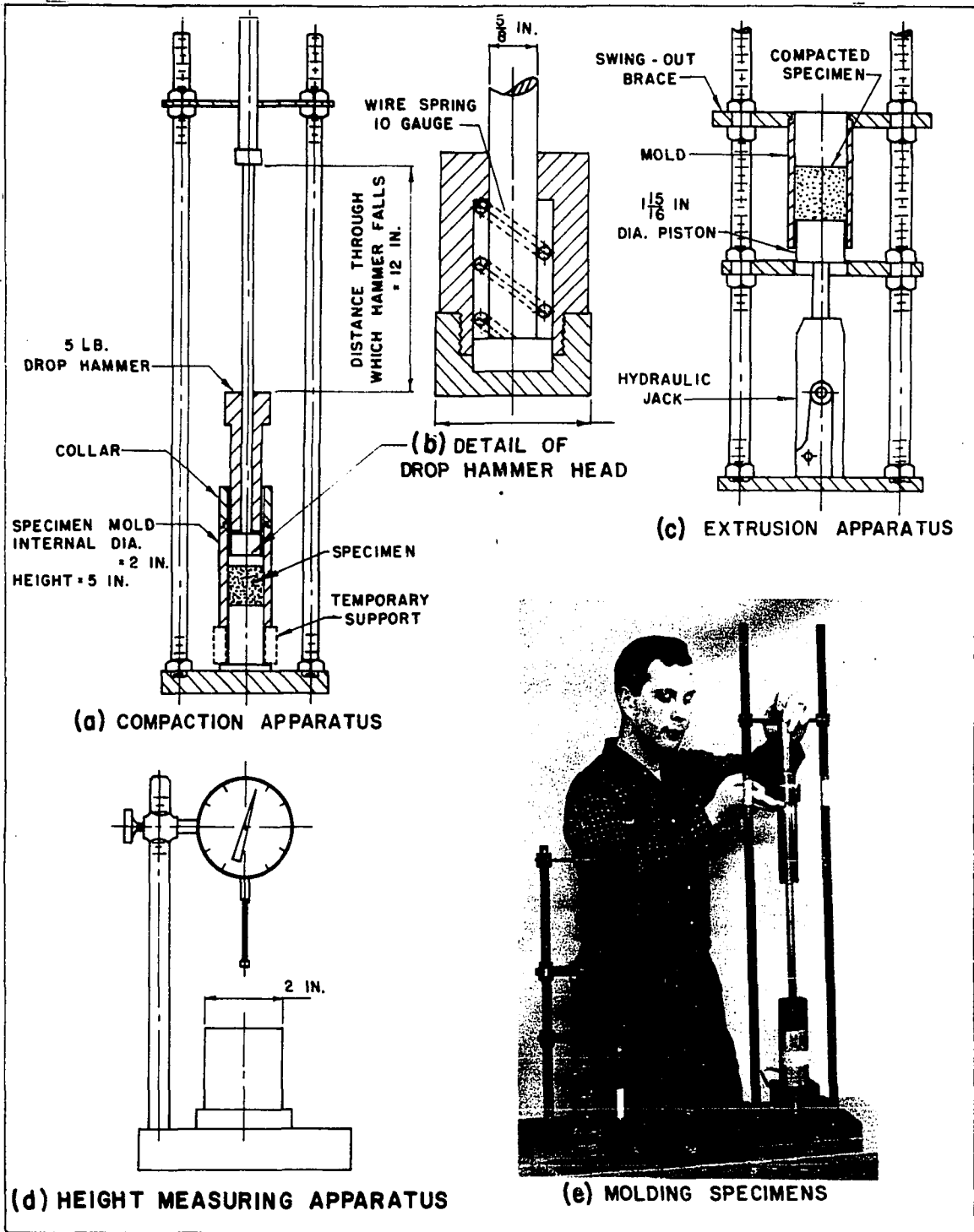


Figure 1. Apparatus for preparation of specimens

and mixed. The set of three specimens was molded as rapidly as accuracy would permit, usually a matter of 10 to 12 minutes. It was observed that in most cases the last specimen was less dense than the first, indicating that a reaction was taking place.

Even for a series of specimens with identical moisture content, such as for curing and durability tests, it was necessary to follow this same procedure; that is, the water was added in the small three-specimen sets. By molding only three specimens from a small portion of the sample, it was believed that a reasonable compromise would obtain between the effects of time and the slight variations that would occur in moisture content between batches. The three specimens were then tested together and the results of the tests averaged. Hence each point on the curves usually represents an average of three specimens. An exception to this general rule was made in the tests to determine the effects of the time lapse from mixing to molding upon strength and density; in this series the points represent single specimens.

After molding, each specimen was weighed to the nearest 0.1 gram and the height measured to the nearest 0.001 inch. A tolerance of ± 0.050 inches was usually maintained for all specimens. If the height did not come within the tolerance, the specimen was usually crumbled, remixed, and a new specimen molded. Again an exception was made when reaction time was the independent variable since it was believed that it was more important to obtain a specimen at a particular time than to obtain one within the height tolerance at a later time.

Although the mix water was always carefully measured, as a check, two small samples were regularly taken from each batch for a moisture

determination. These were taken at about the same time the soil was taken for first and for the last molded specimens. These moisture content samples were retained in small aluminum cans with tightly fitting covers and were weighed on a torsion balance to the nearest 0.01 gram; then the covers were removed and the cans heated in a 105° C oven to a constant weight, and reweighed. The weight loss on heating was calculated to be the moisture content and is expressed as a per cent of the dry weight of solids. Because the sodium metasilicate pentahydrate contains some water which may be driven off during the drying period and conversely because the reaction between the silicate and the lime appears to immobilize water, it cannot be said with certainty that these moisture determinations represent the free water available for lubrication during compaction. The moisture content determinations were used merely as a check on procedure to assure that the moisture content in a series was either constant or had varied in the desired manner. Further investigation is believed to be necessary to determine the free moisture content at the time of molding and the effect on compaction.

Curing Methods

Except for studies on curing methods, all specimens were wrapped in a thin waxed paper and sealed with cellophane tape to reduce carbonation effects. The specimens were then stored in a specially constructed curing room, designed for a high relative humidity. Initially the humidity in this room was maintained by an atomizer which was very effective in adding moisture to the air under ordinary circumstances. However, to reduce

the high temperatures during the summer months, a refrigeration unit had been installed with the evaporating coils inserted into the curing room. This had been deemed necessary because earlier investigators had found that variations in the curing temperature had an appreciable effect on the strength of moist-cured specimens, making correlation of results difficult. Unfortunately, the cooling method selected had an undesirable effect upon humidity. The cooling coil, being operated at a temperature below the dew point in the room, condensed the moisture from the air as fast as the atomizers were able to replace it and the relative humidity was found to have dropped to as low as 80%. Therefore, test results of specimens cured in this room at the time are not satisfactory. During the course of the investigation, a new high-humidity curing room was constructed in which very close control of humidity and temperature is effected. The temperature is maintained at $75^{\circ} \pm 5^{\circ}$ F and the relative humidity is usually above 98%, seldom dropping below 95%. Later investigations making use of this new curing room found a much higher degree of reproducibility of results.

The new curing room was designed to control humidity and temperature by constant recirculation of the enclosed air past a small steam jet which raises the temperature and the moisture content of the air. After passing over several baffles, designed to cause turbulence and assure thorough mixing and evaporation, the air passes through a series of heat exchangers which are maintained at a constant temperature of 75° F. Thus the air is cooled to 75° F and any moisture which is in excess of the maximum that can be held by air at 75° F is removed. The air is then

forced back into the room at the correct temperature and at 100% relative humidity. Although the room is insulated with rock wool in all walls and the ceiling, on warm days there is some heat gain in the room, causing the actual temperature to be slightly about 75° F and the relative humidity slightly below 100%.

Unconfined Compressive Strength Testing

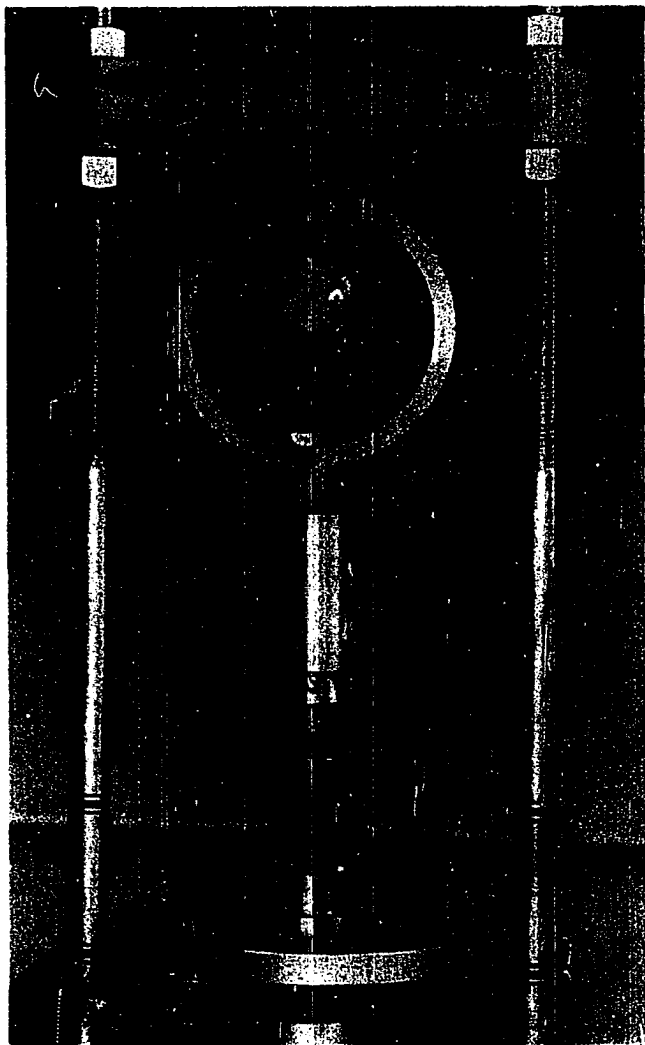
In this investigation, the principle criterion for evaluating the effectiveness of a soil stabilizer is the unconfined compressive strength or crushing resistance of a soil specimen. To measure this strength a specimen was placed in a testing machine (see Figures 2a and b), the platten or lower table of which may be made to rise at a rate of 0.10 inches per minute. The upper end of the specimen is restrained by a cylindrical block and proving ring. The deflection of the ring determined by a dial strain gage may be converted to pounds of force, and thence to pounds per square inch of pressure on the specimen. Since the maximum deflection of the proving ring was seldom in excess of 0.20 inches, the rate of applied strain was not appreciably affected by the deflection of the proving ring. The maximum reading of the proving ring dial was converted to pressure and recorded as the unconfined compressive strength.

Because excessive moisture is a principle cause of most instability, immersion was incorporated in the strength testing program. To determine the effect of immersion on specimen strength, double sets of specimens were molded in almost all cases so one set could be tested without immersion and one set tested after 24-hour immersion in

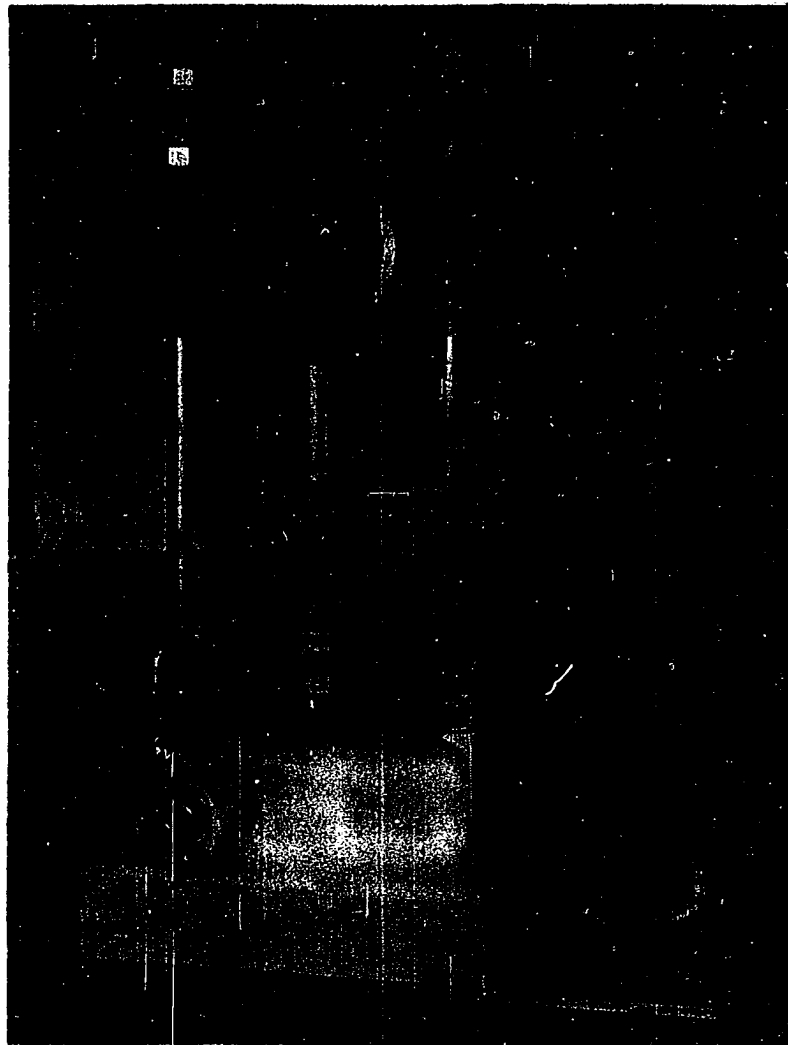
Figure 2. Unconfined compression testing apparatus

a) Close-up view of specimen being tested

b) General view of test apparatus



(a)



(b)

distilled water. For example, in the studies of curing time versus strength, after the two sets of specimens had cured for a predetermined period they were removed from the curing room and the heights and weights determined. Then one set was tested and the other immersed in distilled water; distilled water being used to avoid the possibility of minerals in tap water affecting results. After 24 hours of immersion, this second set was removed from water and allowed to drain; the heights and weights were again determined and the specimens then tested for unconfined compression strength. The results were plotted on the same graphs so comparisons can be made. It was noted that for the sand and loess there was often a strength gain as a result of immersion, whereas for the finer soils 24-hour immersion usually caused a loss of strength.

Presentation of Data

The results of the experimentation have been presented as graphs and each point on a graph represents an average of the test results of the three specimens of a set if all three were within $\pm 10\%$ of their average. If one specimen did not fall within these limits, it was rejected and a new average taken for the remaining specimens. If these did not fall within the limitations, the test was rerun.

In all stages of the experimentation except the first, test results have been plotted with the unconfined compressive strength as the ordinate and the independent variable, i.e., the curing time, freeze-thaw cycles, etc. as the abscissa. A smooth curve has been drawn where a trend seemed to be indicated.

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An exception to the above rule regarding the plotting of strength as the ordinate was made in the first stage because the purpose of this stage of the investigation was to ascertain the effects of changes in the lime-silicate ratio and to provide a comparison between the several types of sodium silicate. Inasmuch as the effectiveness of lime as a stabilizer was not the object of this research the strengths of specimens stabilized with lime alone were used only as a basis for comparison. It was believed that this would provide a more discernable comparison of the effects of different types of lime and sodium silicate and the proportions of each.

RESULTS

Determination of the Optimum Combinations of Silicates and Limes

In this stage of the investigation, only two of the soils were used, the glacial till and the loess. Because the plastic clay and the dune sand were at the two extremes as regards to grain size, and were the most difficult to stabilize, it was arbitrarily decided that if a particular combination did not stabilize the loess or lean clay it probably would not stabilize the plastic clay or sand. The additives used in this case included both calcitic hydrated lime and dolomitic monohydrated lime and three types of silicate, the "N", the "D", both in solution form, and the metasilicate in powder form. When the dissolved silicates were used, they were added with the mix water, and the water in the solution considered a part of the total water. Before proceeding with tests to determine the optimum ratio of lime to sodium silicate the optimum moisture content was estimated from a series of Standard Proctor compaction tests run with each of the additives that were to be used; i.e., the two types of lime and the three types of silicate. They were added singly. The tests were performed on the lean clay (409-12c) and friable silt (20-2(VII)) only, presuming that this would give sufficient indication of the general trend to permit estimation for the other soils. The results of the compaction tests are tabulated in Table IV.

It can be seen that the additives in every case reduced the maximum dry density and increased the optimum moisture content.

From the data of Table IV, the amount of water to be added was calculated on a pro-rata basis of each additive, for example if the

Table IV. Effect of lime and sodium silicate additives on the maximum dry density and optimum moisture content of friable silt and lean clay

Soil	Additive	Optimum moisture content	Maximum dry density in pcf	Increase in optimum moisture content	Decrease in dry density in pcf
Friable silt (20-2(VII))	none	18.1%	108.7	---	---
	6% Calcitic lime	20.2	99.1	2.1	9.6
	6% Dolomitic lime	19.2	104.1	1.3	4.6
	6% Sodium meta-silicate	19.0	104.1	0.9	4.6
	6% D-Silicate	21.7	100.4	3.6	8.3
	6% N-Silicate	26.1	92.1	8.0	16.6
Lean clay (409-12c)	none	15.2	118.2	---	---
	6% Calcitic lime	17.5	108.7	2.3	9.5
	6% Dolomitic lime	18.2	106.7	3.0	11.5
	6% Sodium meta-silicate	18.7	110.2	3.5	8.0
	6% D-Silicate	15.5	107.0	3.0	12.2
	6% N-Silicate	22.5	100.3	7.3	17.9

proportions were to be 6 parts calcitic lime and 4 parts sodium metasilicate in the friable silt, the moisture content would be

$$m + \frac{6}{10}\Delta C + \frac{4}{10}\Delta S = m'$$

where:

m = optimum moisture content for soil alone

ΔC = increase due to added calcium hydroxide

ΔS = increase due to added silicate

m' = estimated optimum moisture content with both additives

The dry weight of additives was maintained constant at 6% of the dry weight of soil in all these tests. The ratio of lime to silicate was 10:0, 8:2, 6:4, 2:8 and 0:10; i.e., both the lime and the silicate were used alone, as well as being in three combinations with each other. All specimens were cured for ten days, then immersed before testing. Results of these tests with strength expressed as a percentage of strength with lime alone are shown in Figures 3 and 4. It is seen that for dolomitic lime, the strength with silicate was never greater than when the lime was used alone. This perhaps should have been expected because magnesium oxide reacts very slowly with sodium silicate, even at 100° C. Magnesia which has been produced from $MgCO_3$ by calcining at high temperatures will not react with sodium silicate at all¹. Had the dolomitic lime been the dihydrate instead of the monohydrate, the results would probably have been quite different.

¹Wills, J. H. Soluble silicates and synthetic insoluble silicates. In Kirk, Raymond E. and Othmer, Donald F., eds. Encyclopedia of chemical technology. Vol. 12. New York. The Interscience Encyclopedia, Inc. 1954. p. 329.

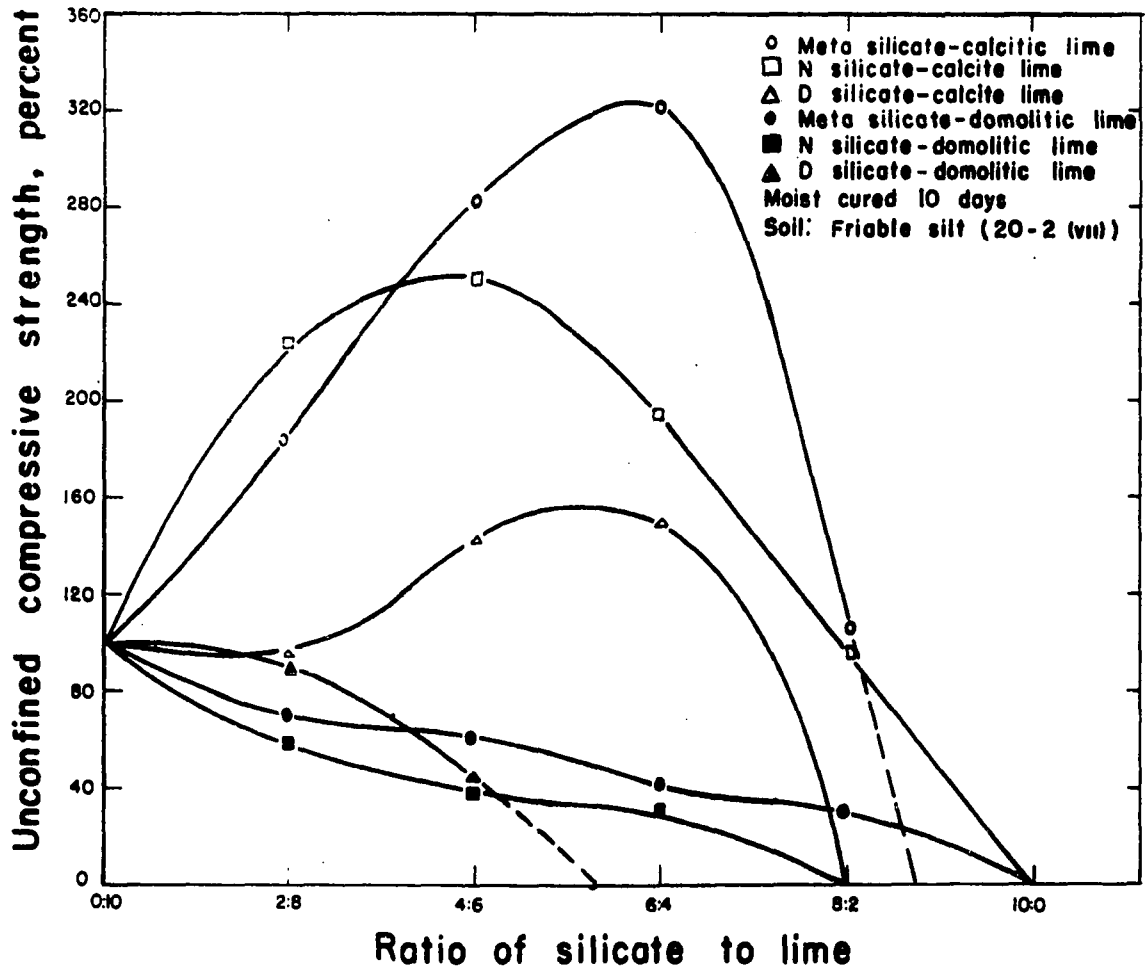


Figure 3. Effect of the lime-silicate ratio on the unconfined compressive strength of friable silt (20-2(VII)) (Strength of specimens stabilized with dolomitic lime alone averaged 268 ± 44 psi and with calcitic lime alone averaged 96.5 ± 10 psi)

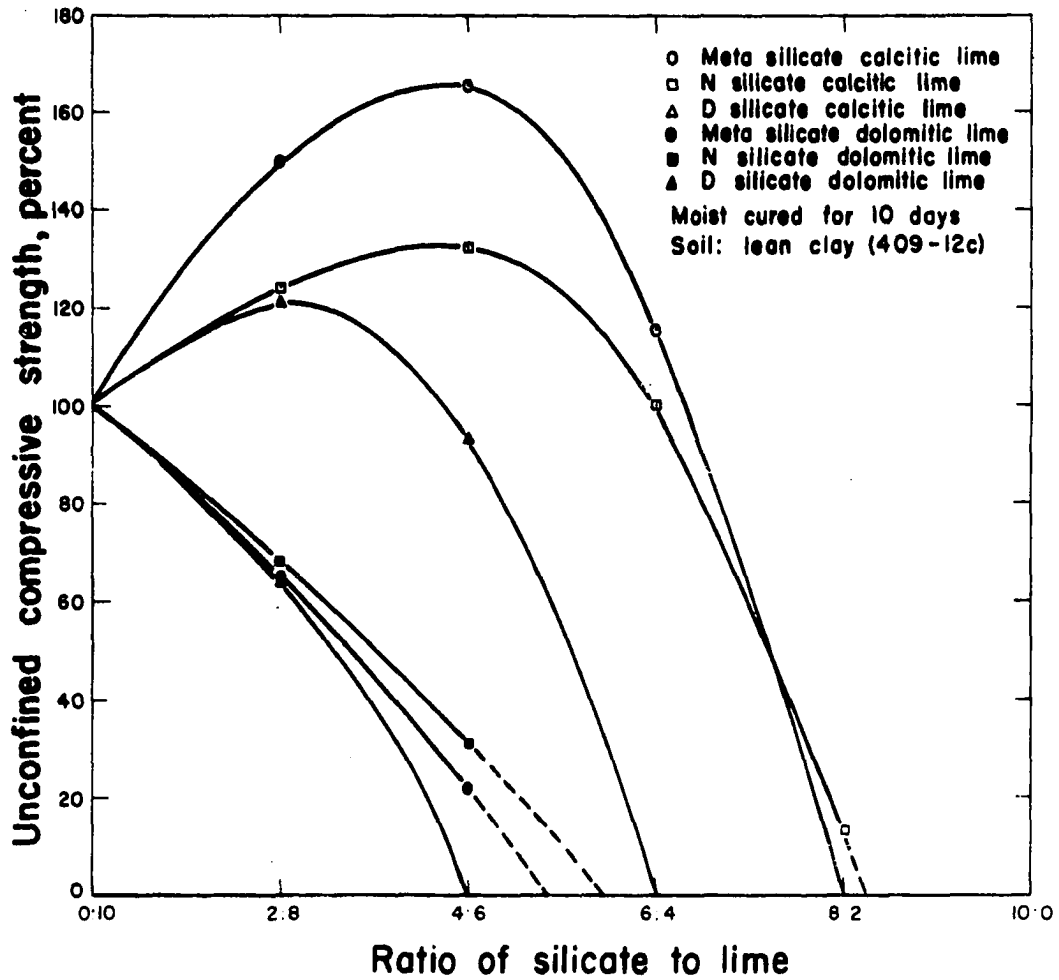


Figure 4. Effect of the lime-silicate ratio on the unconfined compressive strength of lean clay (409-12c) (Strength of specimens stabilized with dolomitic lime alone averaged 330 ± 30 psi and with calcitic lime alone averaged 174 ranging from 117 psi up to 213 psi)

When calcitic lime was used with the sodium silicate, strengths increased in all cases until the ratio of lime to sodium silicate was reduced to less than 4 parts lime to 6 parts silicate; then strengths reduced sharply.

As a result of these tests, it was decided to select sodium metasilicate and calcitic lime as the most promising combination.

Although the results of the previous tests were believed to be sufficiently qualitative for a decision regarding the most promising types of lime and of sodium silicate, further tests were necessary to determine quantitatively the optimum ratios for the selected types of additives and to include the sand and the plastic clay in the investigation. Therefore, in the next part of this stage of the investigation, a series of tests was performed for each soil in which the molding moisture content, as well as the percentage of sodium silicate, was varied. At this time the method of varying the ratio of silicate to lime, was calculated in a different manner than before. Because previous tests had indicated that silicates do not stabilize soil when used alone, the calcitic lime was maintained at 6% of the dry weight of soil, while the sodium silicate was varied from 0% to 10% in 2% increments. It was conjectured that this would give a better indication of the effect of the sodium silicate with lime in the stabilization of soil and provide a better basis for comparison. The addition of 6% lime was a somewhat arbitrary decision, chosen as one which might be economically feasible and also likely to produce satisfactory results for all four soils. Later this decision was modified in the case of the plastic clay when it

was found that because of the high colloidal fraction (about 70% finer than 2 micron), this soil could ionically retain and use about 6% lime¹ and thereby might not allow the sodium silicate to enter into the reaction. However, in the case of the other soils, the 6% addition of lime was used throughout the investigation.

Results of the tests in this stage of the investigation are shown in Figure 5. As before, except for the sand, the unconfined compressive strengths are expressed as a percentage of the strength with lime alone. Because lime alone did not stabilize sand and hence did not provide the basis for comparison as in other soils, strength with 6% lime with 2% sodium metasilicate was used as the basis for comparison.

This stage of the testing seems to indicate that the optimum ratio of lime to silicate varies with clay content, with the finer soils requiring a higher proportion of lime. The ratios decided upon for each soil are shown below:

Soil	Additive in % of dry weight of soil	
	Lime	Silicate
Dune Sand	6	8
Friable Loess	6	6
Lean Clay	6	6
Plastic Clay	12	4

These proportions seem to indicate a direct relationship between the ratio of lime to sodium silicate and the clay content of the soil

¹Hilt, G. H., Iowa State University of Science and Technology, Ames, Iowa. Data from research on the lime fixation in clayey soils. Private communication. 1959.

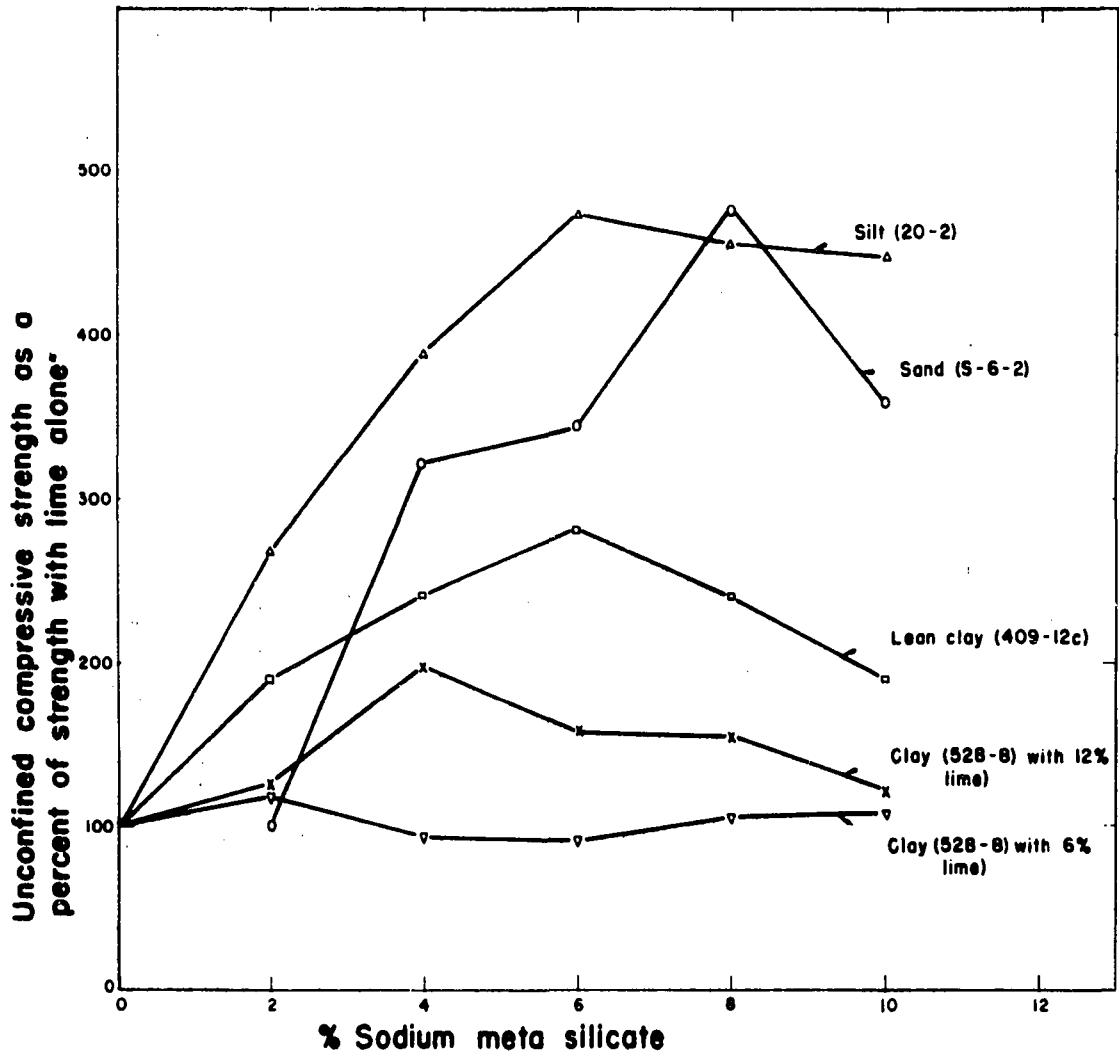


Figure 5. Changes in unconfined compressive strength of soils stabilized with calcitic lime (6% for sand, silt and lean clay; 6% and 12% for plastic clay) and 0 to 10% sodium metasilicate (*Note: Because lime does not stabilize sand when used alone, strength variations are expressed as a per cent of strength with 6% lime and 2% sodium metasilicate. 100% strength for each soil is as follows: silt, 95 psi; sand, 99 psi; lean clay, 145 psi; plastic clay with 12% lime, 165 psi; plastic clay with 6% lime, 92 psi.)

to be stabilized. Hilt observed in his work that the plastic limit of montmorillonitic soils increased with the addition of lime up to a point beyond which further additions of lime did not affect the plastic limit. He referred to this as the lime retention point. Although he expressed the lime retention point as a function of the amount of clay finer than 2μ in size, it appears better to equate the lime retention point to the cation exchange capacity of the whole soil specimen because the retention of the calcium ions in this case is probably similar to the cation exchange except that it is being done at a higher pH. When equated to the cation exchange capacity of the total soil we see that

$$L_{rp} = 0.08 c + 0.0127$$

where L_{rp} = lime retained in per cent of the dry weight
of the soil at the point where plastic limit
no longer increases

c = cation exchange capacity of the total soil

Assuming that lime thusly retained will be adsorbed by the clay and hence not reacting with sodium silicate, it is noted that the stoichiometric relationship between the remaining available CaO and SiO₂ lies between 1:2 and 3:2 except for the plastic clay (528-8) where 12% lime and 4% sodium silicate were used. It would appear that an excess of lime was used.

Further investigation would be appropriate in this regard to determine more precisely these ratios. However, the percentages listed above were decided upon and used throughout the remainder of the investigation, usually with quite satisfactory results.

Determination of Optimum Curing Conditions

The purpose of this stage of the investigation was to determine the curing condition which would produce the greatest unconfined compressive strength, all other conditions being constant. Four conditions of curing were selected for testing with two types of soil. Again, only the loess and the lean clay were tested. The conditions chosen for testing were:

1. Specimens wrapped in wax paper and stored in a room where relative humidity was maintained above 90% and the temperature at about 75° F.
2. Specimens unwrapped but stored as in Condition 1 above.
3. Specimens unwrapped and stored in a room of low humidity, but dipped in distilled water twice each day (to simulate sprinkling).
4. Same as Condition 3, but not dipped.

Results appeared to be quite conclusive. The specimens cured at low humidity but dipped (Condition 3) disintegrated during the curing period. The specimens cured at low humidity but not dipped failed during the immersion prior to testing. The specimens which were wrapped and cured in the humid atmosphere (Condition 1) developed far greater strength than the unwrapped specimen (Condition 2). Hence, all specimens thereafter were cured in this manner (Condition 1). However, it was later found during the durability tests that continuous immersion after a 14 day curing period further increased the strength of specimens.

Determination of the Effect of Curing Time

In this series of tests, 30 specimens of each soil were molded at the optimum moisture content with the predetermined percentages of the chemicals. The specimens were molded in sets of three. The procedure

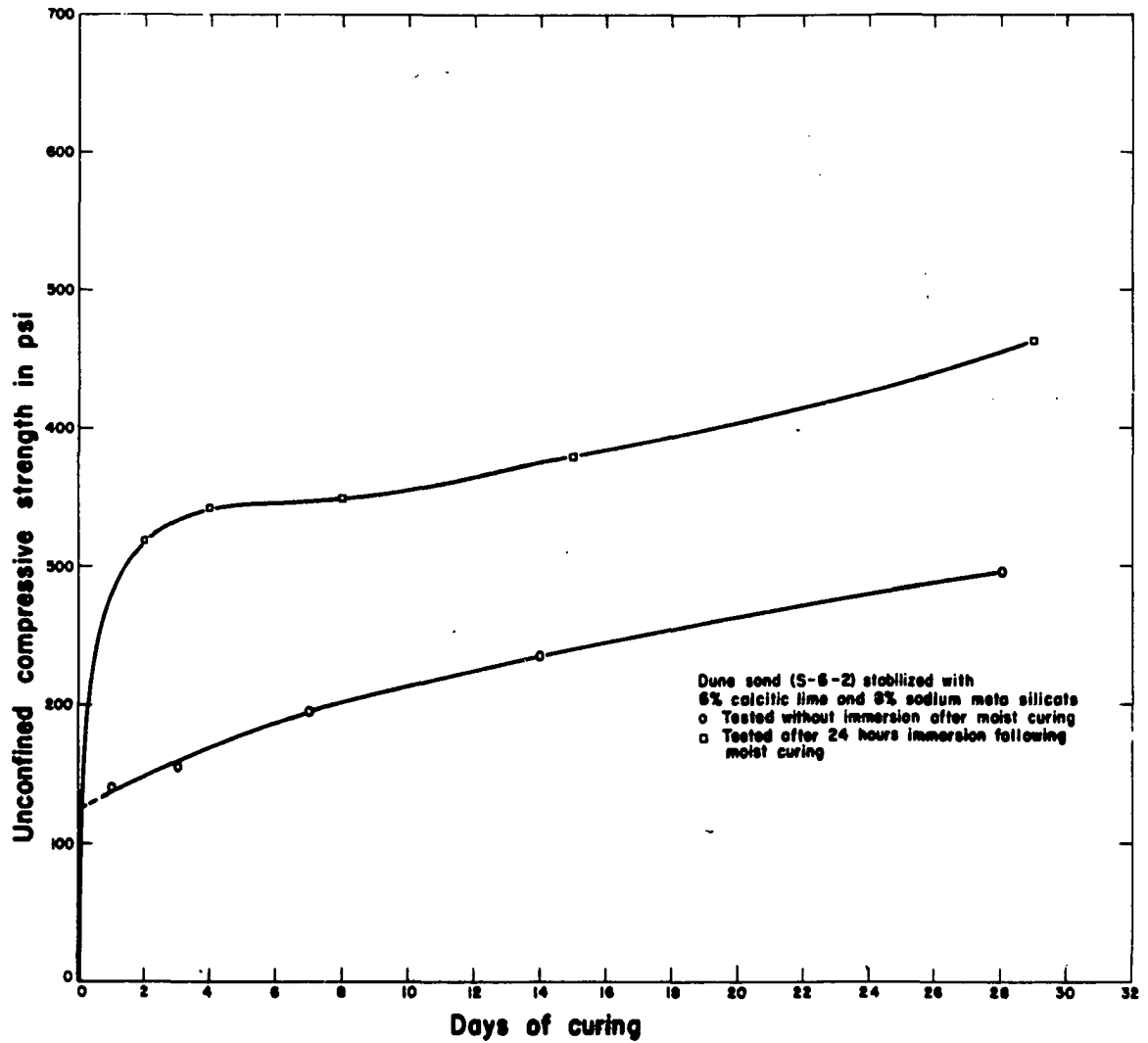


Figure 6. Variations of unconfined compressive strength with curing time for dune sand (S-6-2) stabilized with 6% calcitic lime and 8% sodium metasilicate

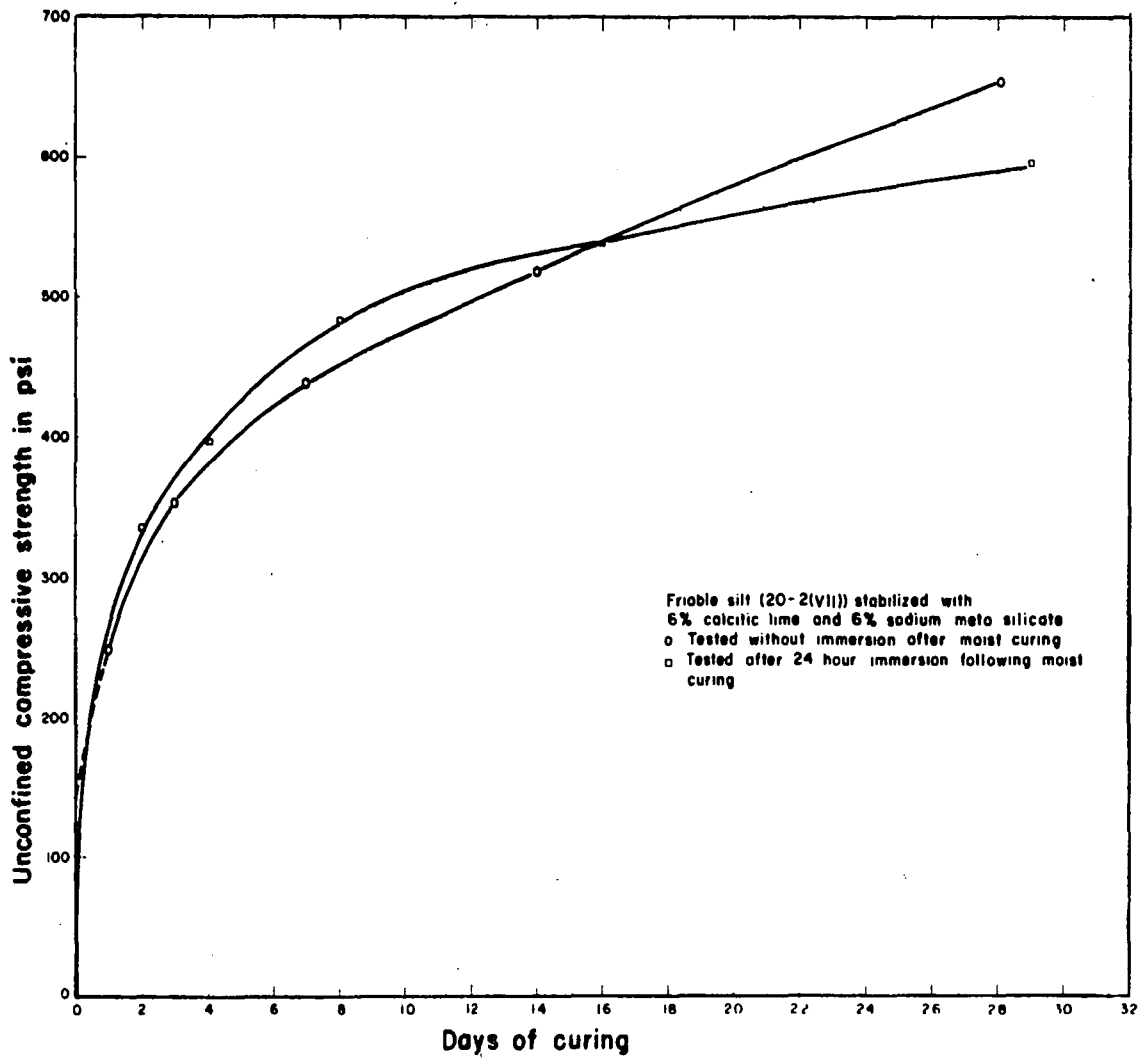


Figure 7. Variations of unconfined compressive strength with curing time of friable silt (20-2(VII)) stabilized with 6% calcitic lime and 6% sodium metasilicate

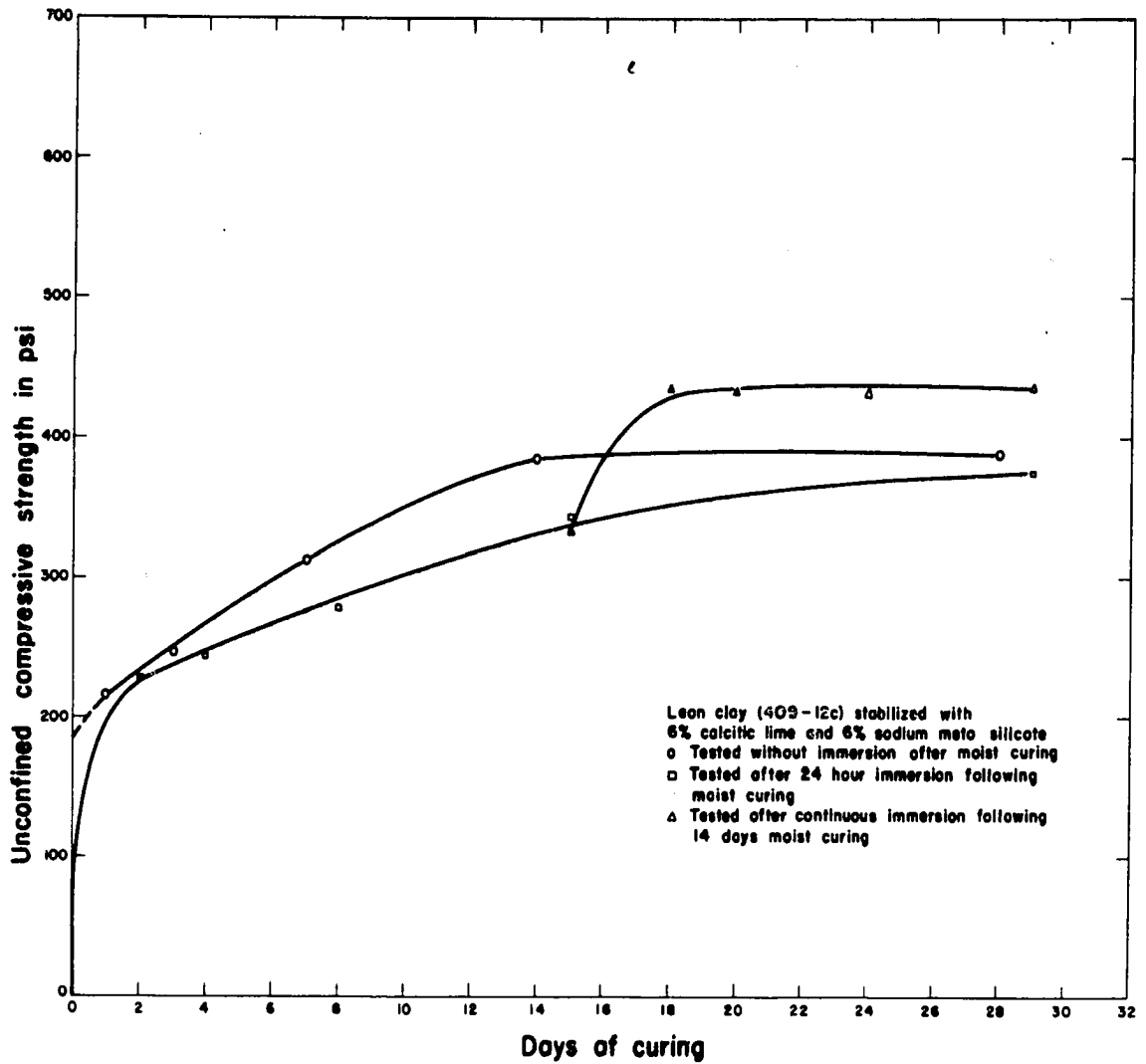
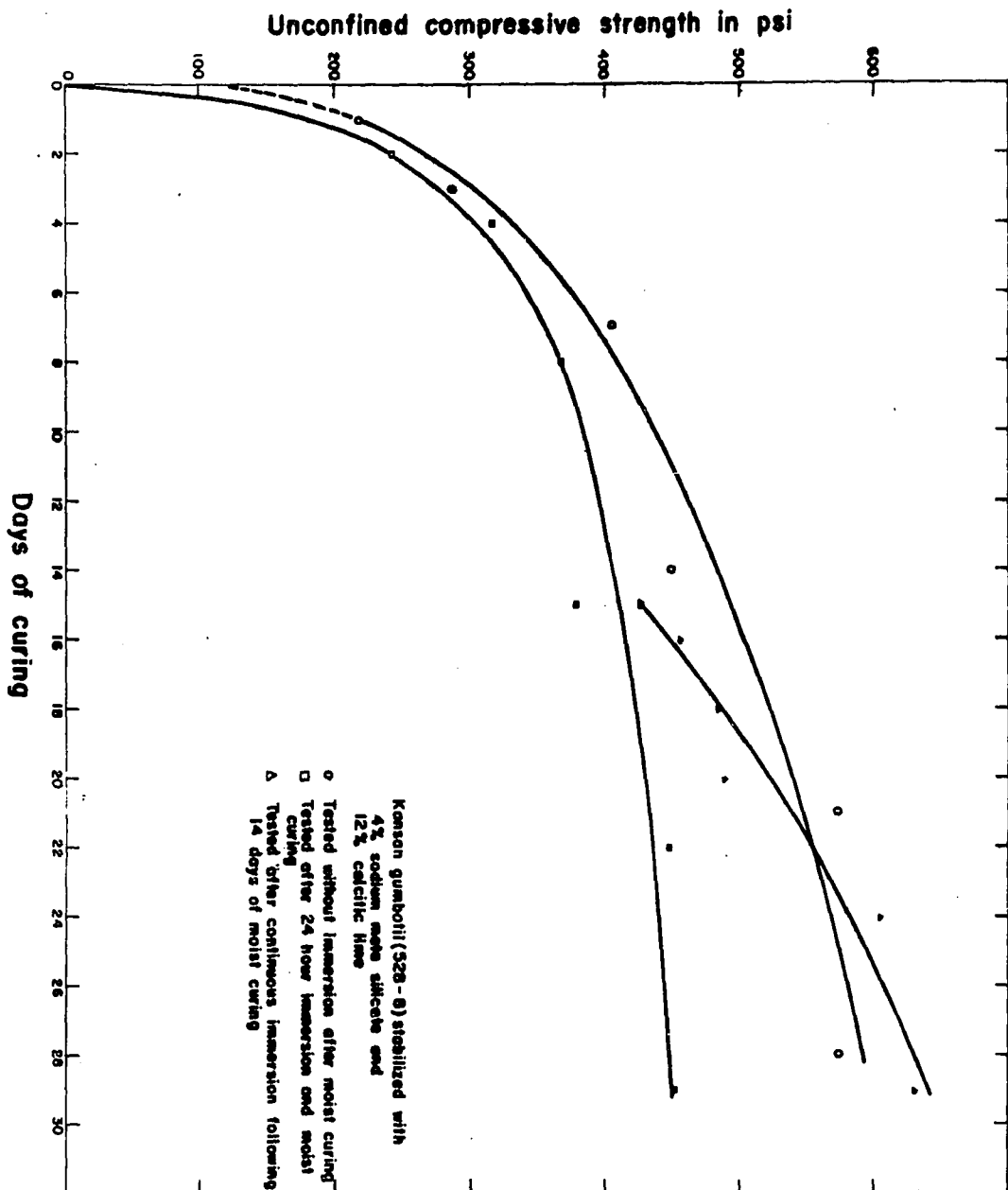


Figure 8. Variations of unconfined compressive strength with curing time of lean clay (409-12c) stabilized with 6% calcitic lime and 6% sodium metasilicate

Figure 9. Variations of unconfined compressive strength with curing time of plastic clay (528-8) stabilized with 12% calcitic lime and 4% sodium metasilicate



followed was to weigh out a batch of soil, lime, and silicate, large enough for all thirty specimens, then to mix the three ingredients in a dry state. From this large batch, a smaller portion was taken which was sufficient to mold a set of three specimens. An accurately measured volume of water calculated to produce the optimum moisture content for maximum strength was added and mixed. The three specimens of the set were then molded, weighed, measured and wrapped. This procedure was repeated until all 30 specimens were completed for each soil. All of the specimens were stored in the humid curing room. The following day, two sets for each soil were removed and each specimen weighed and measured. One set was immediately tested for unconfined compressive strength, while the other set was immersed in distilled water for 24 hours, then tested. This procedure was repeated at 3, 7, 14 and 28 days. Curves were plotted, showing the strength gain with curing time as well as the effect of the 24-hour immersion. The results are shown in Figures 6 through 9.

The entire series of tests had to be repeated because of a previously mentioned reduction in the relative humidity of the curing room after the specimens had been curing for about 2 or 3 days. A definite loss of strength was observed in those series which were being cured at time of reduction of the relative humidity. This affect on strength is another indication of the importance of a high humidity during the curing operation.

The effect of the 24-hour immersion is clearly shown in the graphs. The immersion causes a loss of strength in the finer soil and an increase of strength in the coarser soil, with lesser effects for the intermediate

soils. This would seem to indicate that a hydraulic cement is formed which improves in the presence of moisture, but in the soils that are high in clay, particularly montmorillonite, this strength gain is overcome by a strength loss probably due to adsorption of water by the clay particles.

Durability Testing

In order to determine the durability of stabilized soil when subjected to the rigors of freezing and thawing and of wetting and drying, specimens were exposed to varying numbers of cycles of freeze-thaw and wet-dry, believed to simulate field conditions, then tested for unfined compressive strength. In this stage of the investigation 54 specimens were molded from each soil using the moisture content and the combination of additives which had previously produced maximum strength. All specimens were wrapped and cured for 14 days in a humid atmosphere. One third of the specimens were then used for the freeze-thaw tests, one third for wet-dry tests and the final third for continuous immersion after the curing period for comparative purposes.

In the field, soil is subjected to a sub-freezing temperatures at the top surface only, while water is usually available to rise by capillarity from the free water table. In a test modeled after the British freeze-thaw test for soil cement¹, these field conditions were simulated

¹Davidson, D. T. and Bruns, B. W. Comparison of type I and type III portland cements for soil stabilization. Iowa State University of Science and Technology Engr. Expt. Sta. Bul. 194: 116. 1961.

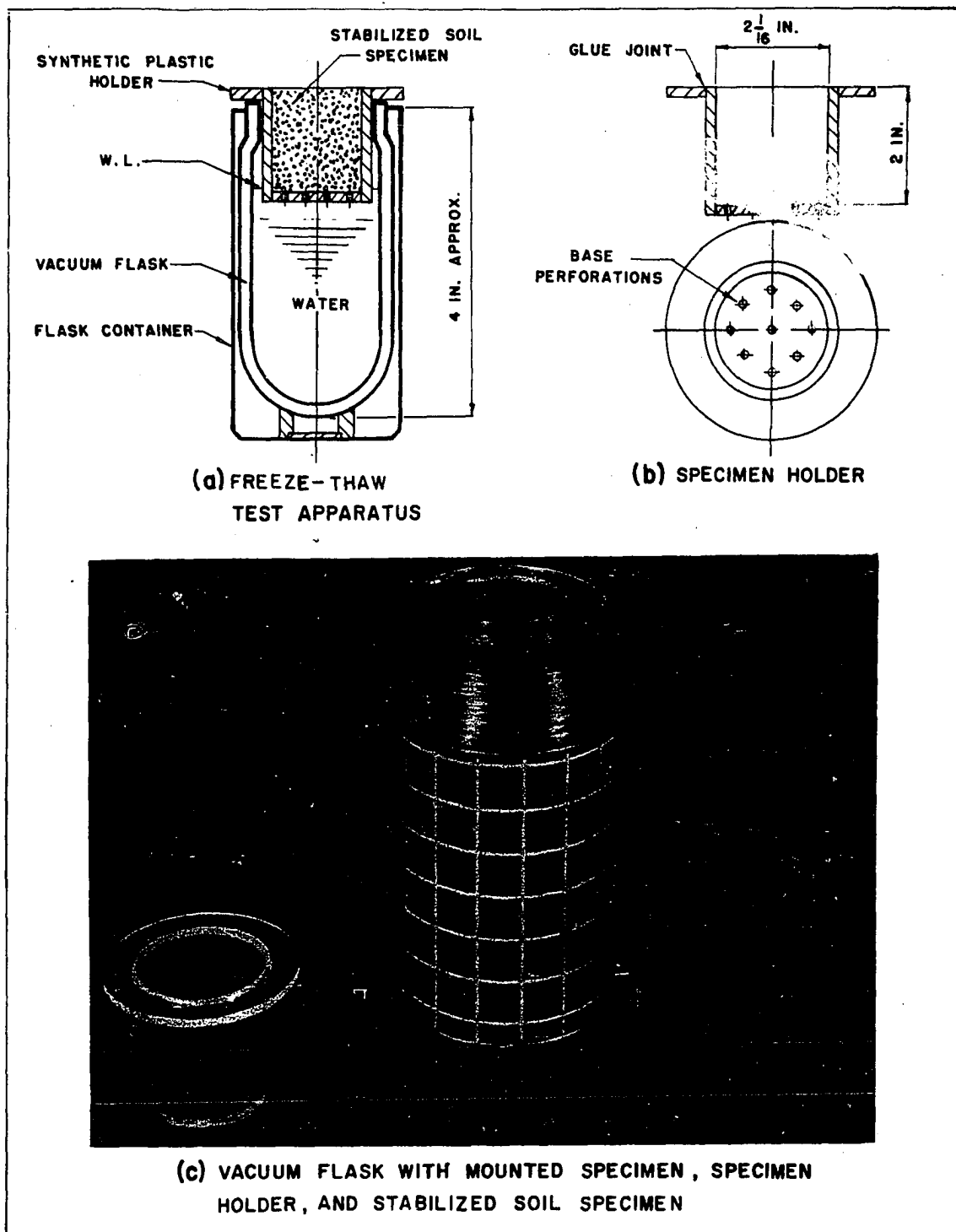


Figure 10. Apparatus for freeze-thaw testing

by treating the top surfaces of the stabilized soil specimens with an asphaltic compound, then wrapping them in a collar of self adhering plastic membrane which extended from the top surface to about 3/4" above the bottom surface. After curing, the specimens were placed in the lucite specimen holder shown in Figure 10. Each of the holders with its specimen was then placed in the mouth of a vacuum flask. Enough distilled water at 8° C had been put into each flask so that about 1/4" of each specimen was in water. The flasks were then placed in a refrigerator held at -5° C. After 16 hours the flasks were removed and allowed to stand at room temperature for 8 hours. This constituted one freeze-thaw cycle.

After a set of specimens had been subjected to the desired cycles of freeze-thaw, each specimen was tested for unconfined compressive strength by crushing, as previously described. Tests were performed at the end of 1, 3, 5, 9 and 15 cycles. The results of the freeze-thaw tests with comparison with the continuous immersion are shown in Figures 11 through 14.

After the curing period the 18 specimens to be subjected to the wet-dry test were immersed in distilled water for 16 hours, then heated for 8 hours in an oven held at 60° C (140° F). This constituted one wet-dry cycle. After the same scheduled number of cycles as for the freeze-thaw test, a set of specimens was tested for unconfined compressive strength.

After curing, the remaining 18 specimens were continuously immersed in distilled water. One set of 3 specimens was then tested

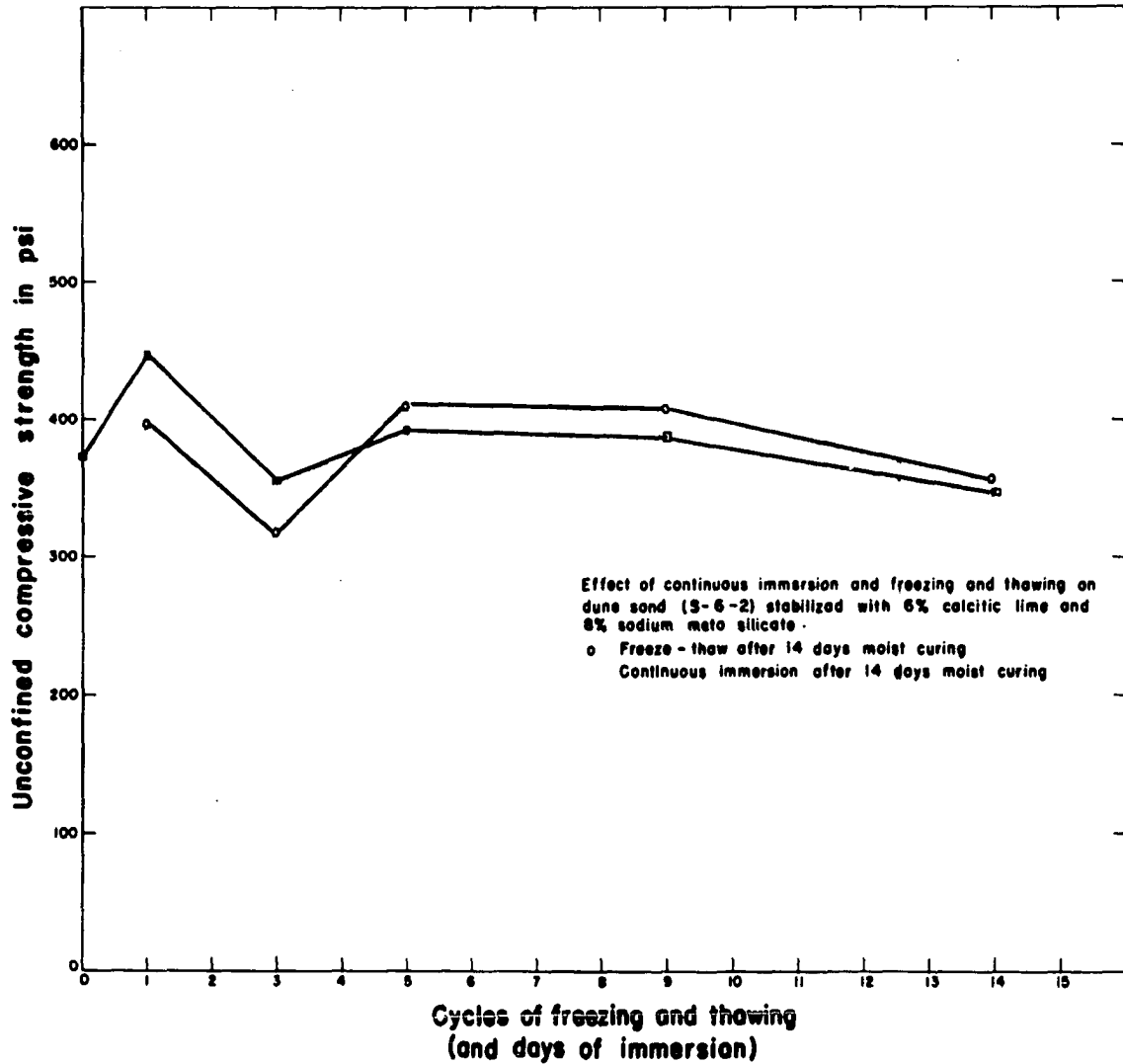


Figure 11. Effects of continuous immersion and of freezing and thawing cycles on the unconfined compressive strength of dune sand (S-6-2) stabilized with 6% calcitic lime and 8% sodium meta-silicate

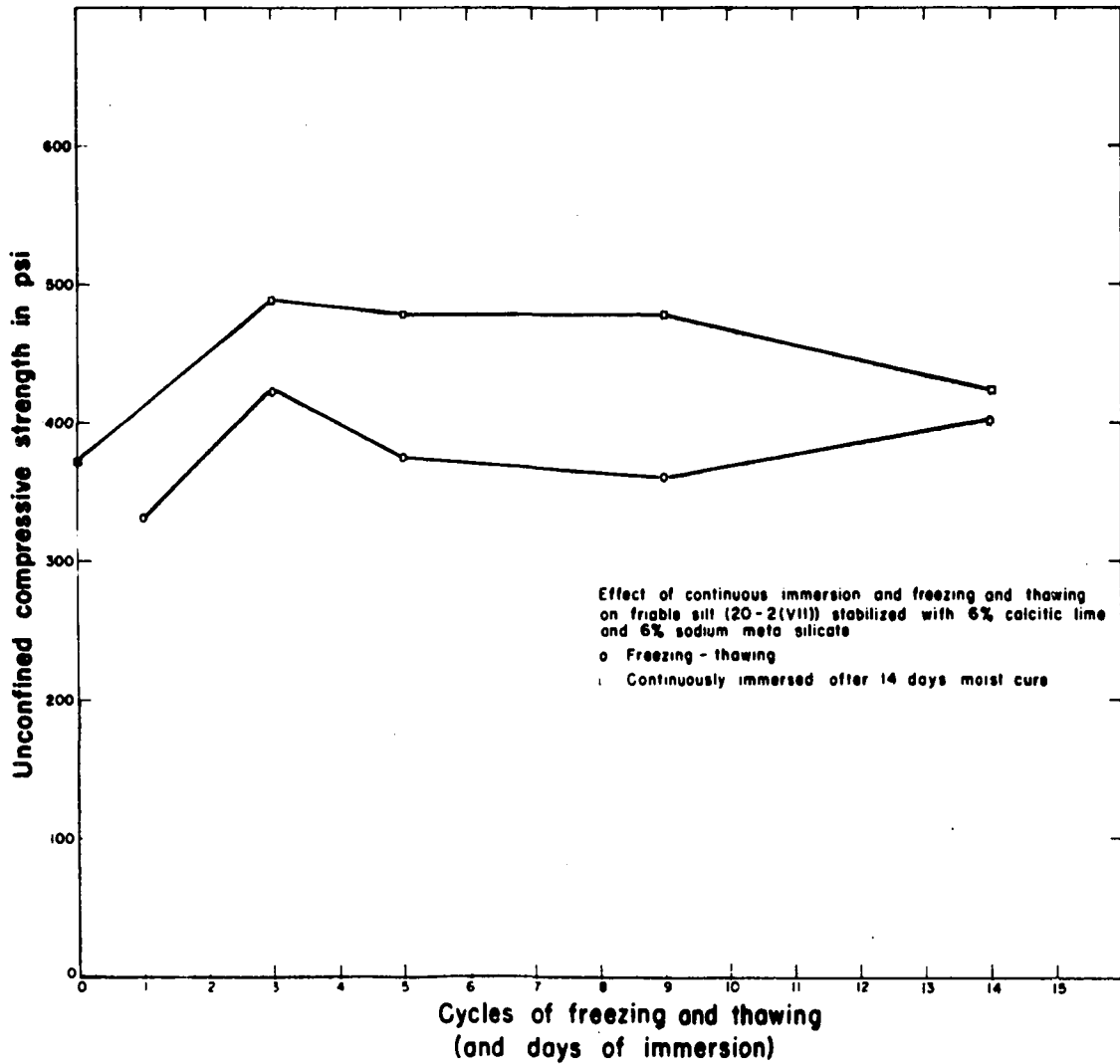


Figure 12. Effects of continuous immersion and of freezing and thawing cycles on the unconfined compressive strength of friable silt (20-2(VII)) stabilized with 6% calcitic lime and 6% sodium metasilicate

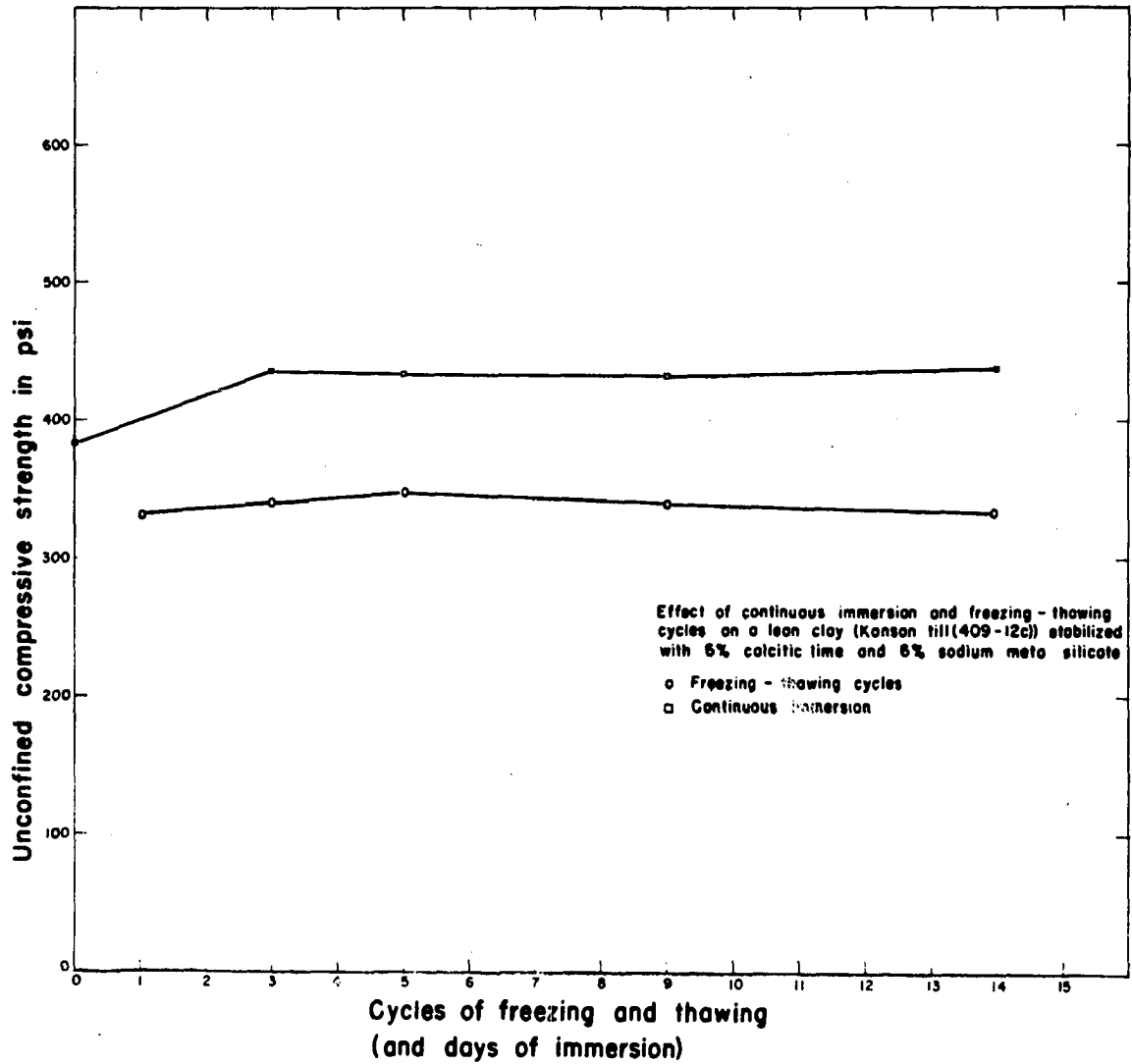


Figure 13. Effects of continuous immersion and of freezing and thawing cycles on the unconfined compressive strength of lean clay (409-12c) stabilized with 6% calcitic lime and 6% sodium metasilicate

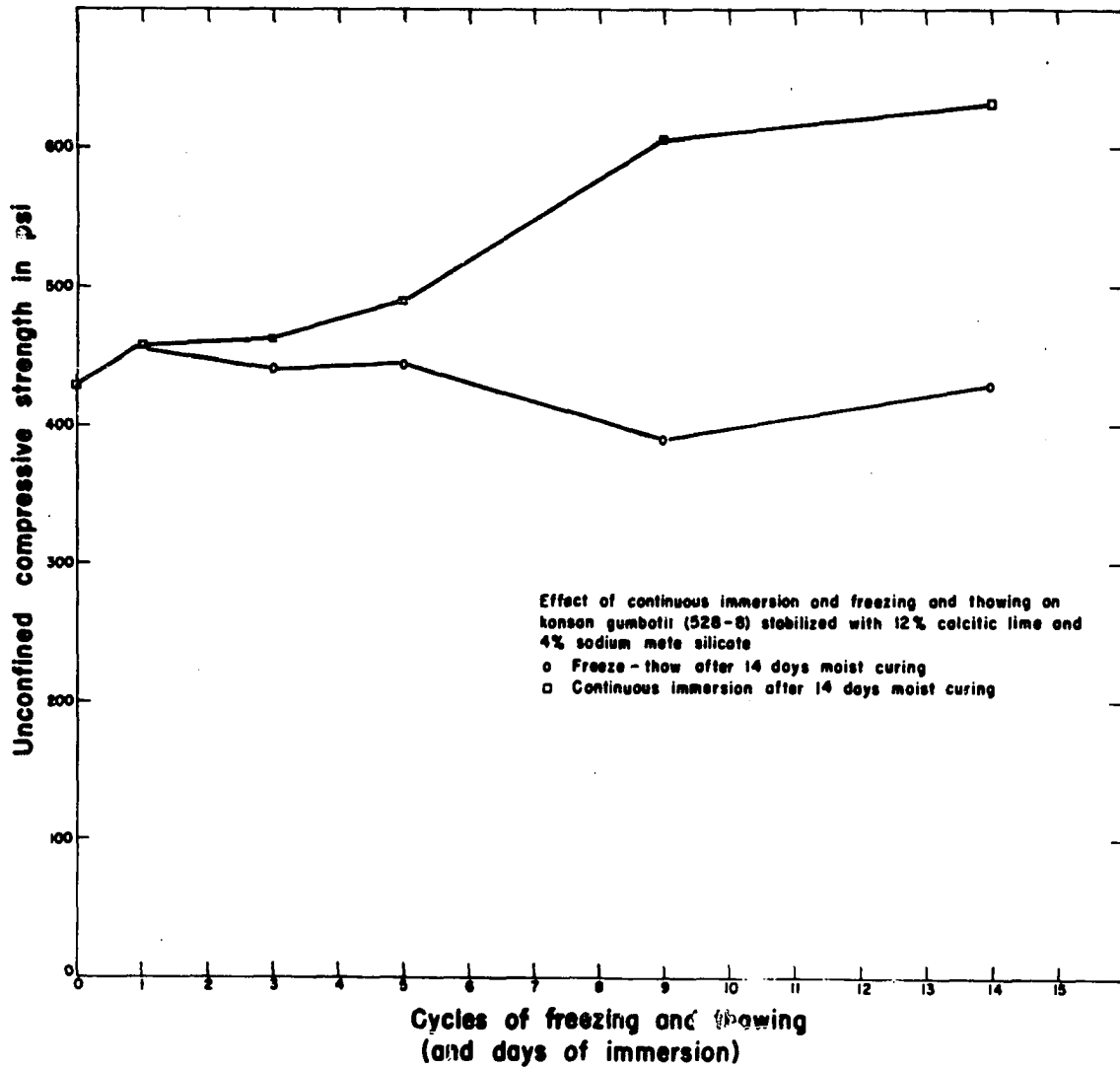


Figure 14. Effects of continuous immersion and of freezing and thawing cycles on the unconfined compressive strength of plastic clay (528-8) stabilized with 12% calcitic lime and 4% sodium metasilicate

along with the sets from the other series. This was done to provide a standard of comparison because both the freeze-thaw and the wet-dry cycles included immersion.

The results of all these tests, shown in Figure 15, indicate that the wet-dry cycles have a deleterious effect on the specimens, but that freeze-thaw cycles and the continuous immersion caused little or no loss of strength. For two soils the immersion appeared to be beneficial to strength. It is suggested that the desiccating action of the wet-dry cycles has an inhibiting effect on the curing process which was not compensated for by the subsequent immersion. In the case of the plastic clay, the strength loss caused by the wetting and drying occurred mainly during the initial cycles of the test. Subsequently the strength loss was diminished. In the friable silt the strength loss appears to continue with each cycle of wetting and drying. The strength of the sand specimens subjected to the wet-dry test was noticeably lower than the sand specimens in the other two tests.

Stabilization of Extremely Wet Soil

When roads fail because of excessive moisture, artificial mechanical or thermal drying is usually impossible to accomplish quickly or is economically impractical. Examples of such situations include roads which have failed during the season of heavy rainfall, or areas of perpetually high water table such as the Low Countries of Europe. Under conditions such as these, reduction of moisture content is impracticable. The capability of stabilizing a soil despite a high moisture content would find useful application in such circumstances.

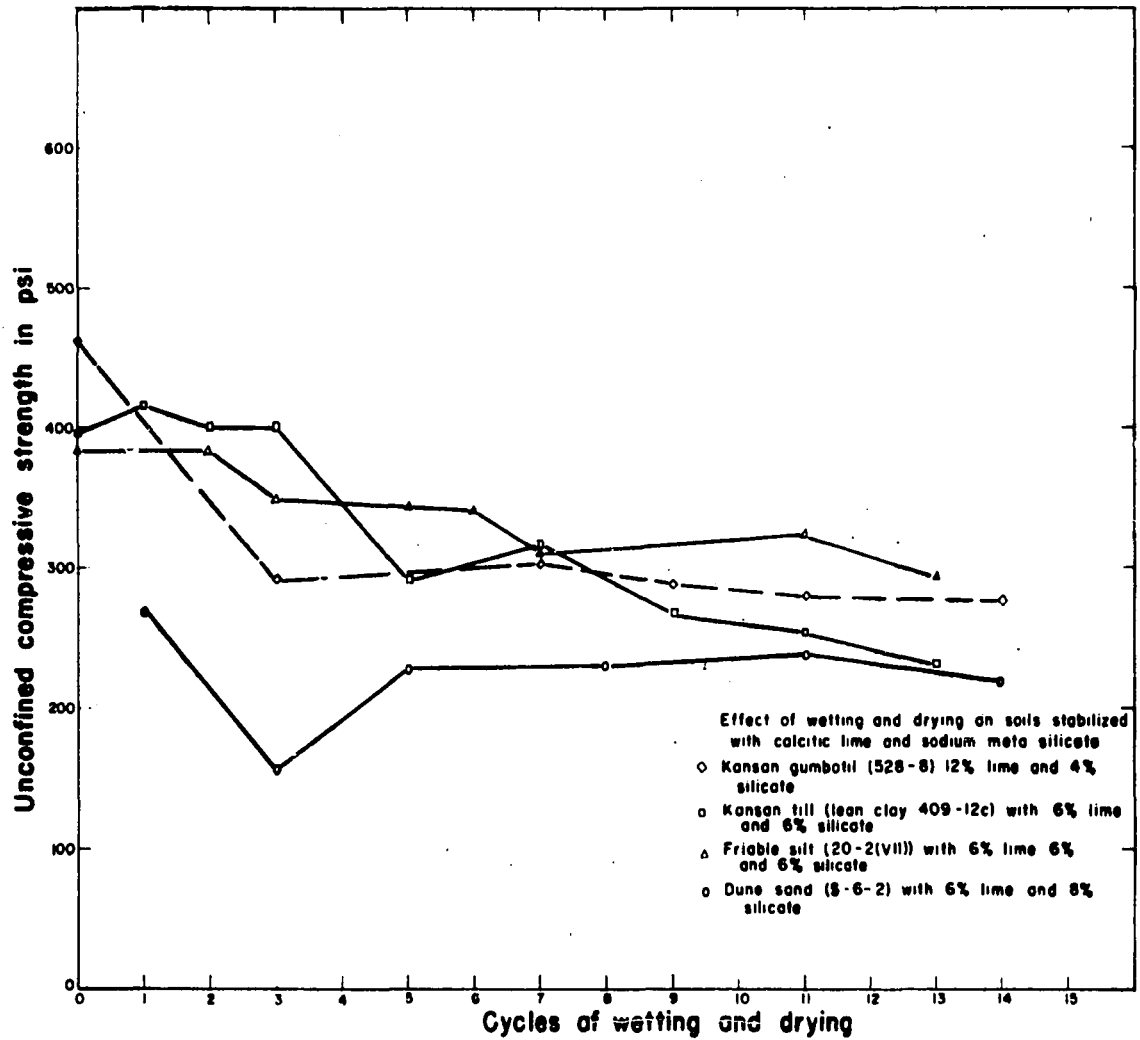


Figure 15. Effects of cycles of wetting and drying on the unconfined compressive strength of soils stabilized with calcitic lime and sodium metasilicate

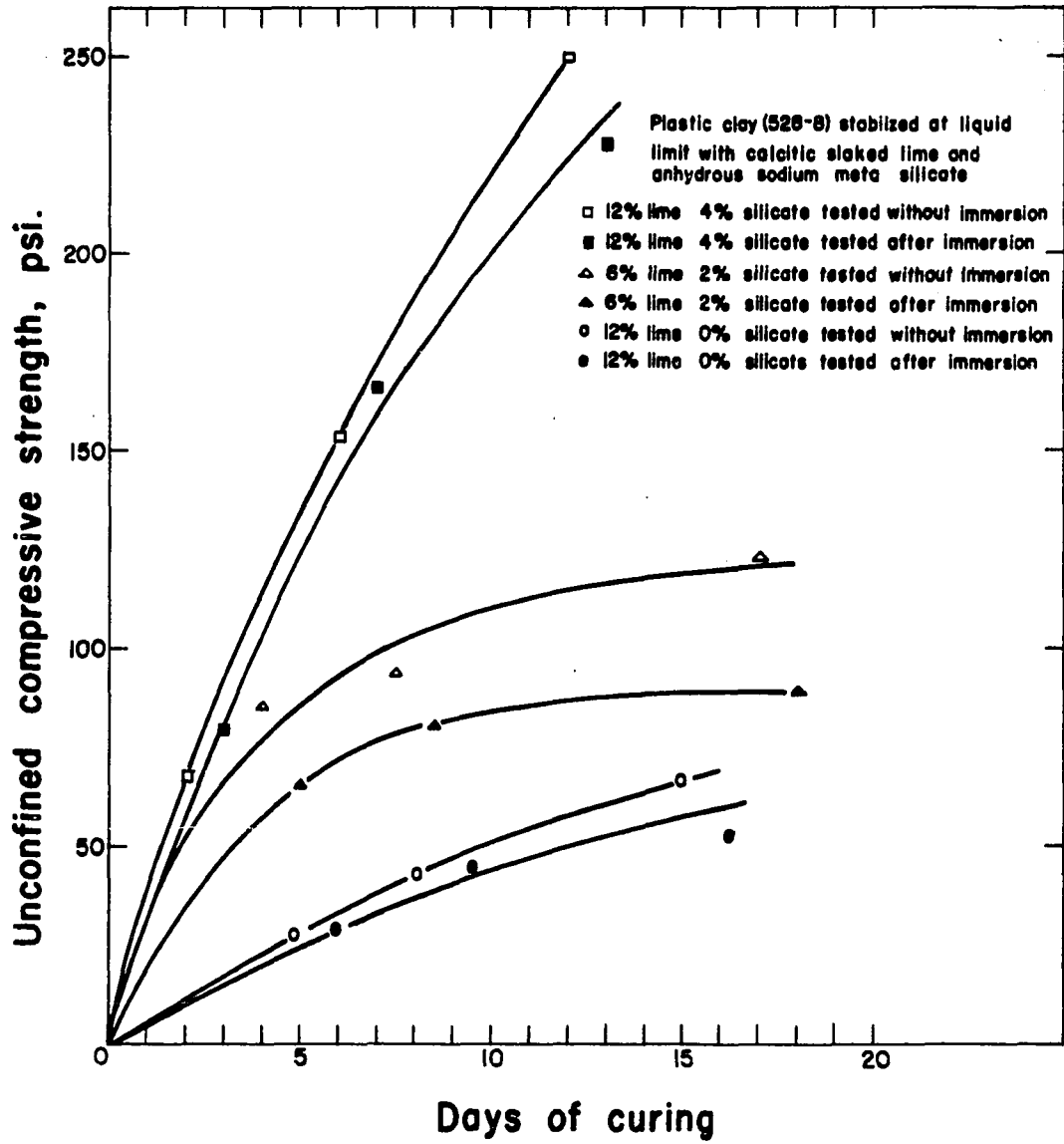


Figure 16. Variations in unconfined compressive strength of extremely wet plastic clay (528-8) stabilized with calcitic lime and sodium metasilicate

To determine the effectiveness of a combination of sodium metasilicate and calcitic lime as a stabilizer of soil with high moisture content, the plastic clay was selected for testing. This soil was chosen because of its extremely poor drainage characteristics and its high liquid limit. A sample of soil was prepared for testing by adding sufficient water to bring it to slightly above the liquid limit. Then the predetermined quantity of sodium silicate and lime were added simultaneously and mixed. Mixing was done with the model 100-C Hobart kitchen mixer. Mixing was continued until the sample appeared to be homogenous. Although mixing was relatively easy prior to the addition of the chemicals, the soil immediately changed from the liquid state to the plastic state, which made mixing very difficult thereafter. This would present a real problem in the practical application in that mixing will require powerful equipment with low ground contact pressures.

After mixing, specimens were molded by kneading in a 2" diameter tube, set over a pedestal so that the specimen could be struck-off at the 2" length. Because of the high moisture content and low air content, compaction by normal means was not feasible. After molding, the specimens were allowed to cure for a brief period until they had gained sufficient strength to permit extrusion. It was observed through trial and error that if extruded immediately after molding, the specimens were too soft and became deformed by handling. Some were allowed to remain in the curing room overnight, but this was objectionable because of the possibility of unknown changes that might occur due to carbonation or evaporation. It was later found that after about 45 minutes, an initial set gave sufficient strength to permit extruding, weighing and measuring.

The extruded specimens were wrapped and placed in the humid room for variable periods of curing. Testing for unconfined compressive strength was then done to determine the strength gain as a function of curing time. The testing for strength was performed in the same manner as heretofore. Some were tested after 24 hour period of immersion in distilled water while others were tested at the termination of the curing period.

The results, shown in Figure 16, appear to indicate considerable usefulness of sodium silicate and lime as a soil stabilizer under adverse conditions of moisture. The compressive strengths of the specimens molded at the liquid limit attained approximately 50% of the strength of specimens molded at a moisture content near the optimum for compaction.

Strength Losses Due to Delay in Molding

In the use of any soil additives a period of time must be provided for between initial mixing and final shaping and compacting. Because there appeared to be an immediate reaction when the lime and sodium silicate were combined in the presence of water, it was presumed that a reaction was occurring before the specimens could be compacted, hence the stabilizing effect might be partially destroyed by the subsequent manipulation.

To ascertain the effect, a large sample of plastic clay (528-8) was prepared including the addition of the water. Specimens were then molded, noting the time in each case, cured for 7 days and tested for unconfined compressive strength. The dry density and the strength were then plotted

as a function of molding time. Results are shown in Figure 17. It can be seen that there is a gradual reduction of both dry density and strength with time between mixing and molding.

This seems to indicate that the reaction between the lime and sodium silicate starts immediately but proceeds gradually as is also shown by the x-ray diffraction traces. Although there appears to be an attenuation of the reduction of compressive strength and density, it was not determined how long the loss would continue. Ruff ascertained that the strength loss for lean clay mixed with 6% calcitic lime and 6% sodium metasilicate (Metso Granular) and allowed to age for 5 hours before molding was about 30%¹. Five hours time should provide sufficient time to a practical application to allow for proper mixing and compacting by conventional equipment.

X-ray Analysis

During the course of experimentation it was observed that a physical change occurred in a paste formed of lime, powdered sodium silicate and water soon after the water was added. To study this reaction the two ingredients, in a stoichiometric proportion which would render a 1:1 ratio between CaO and SiO₂, were thoroughly mixed in dry, powder form, then an amount of water equal to 25% by weight of the dry chemicals was added to form a paste. Within a few minutes the paste became firm in a reaction liberating heat which may be accounted for by the

¹Ruff, C. G. Sodium silicate-lime stabilization of Kansan glacial till. Unpublished M.S. thesis. Ames, Iowa. Library, Iowa State University of Science and Technology. 1960.

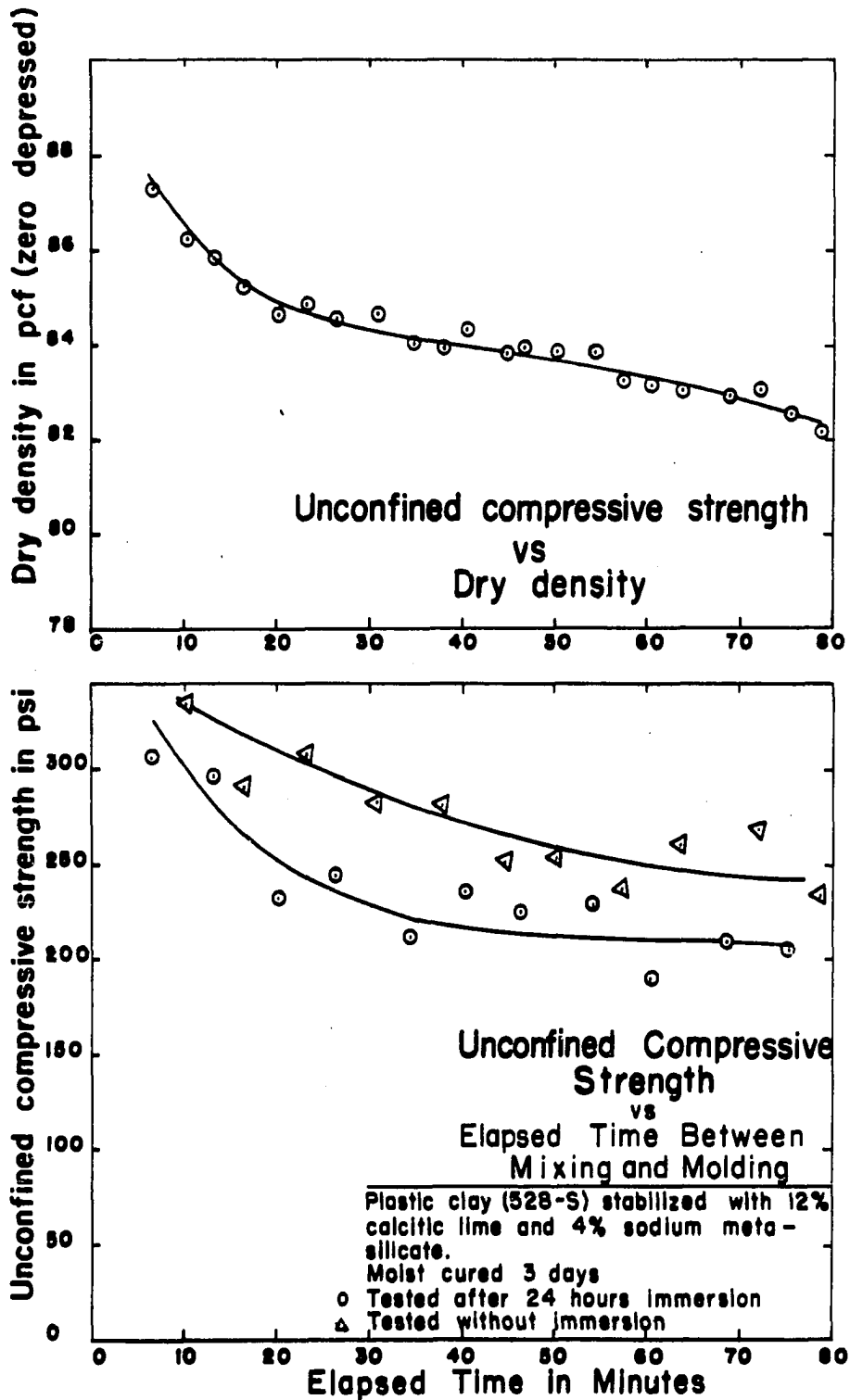


Figure 17. Effects of time between mixing and molding on the unconfined compressive strength and dry density of plastic clay (528-8) stabilized with calcitic lime and sodium metasilicate

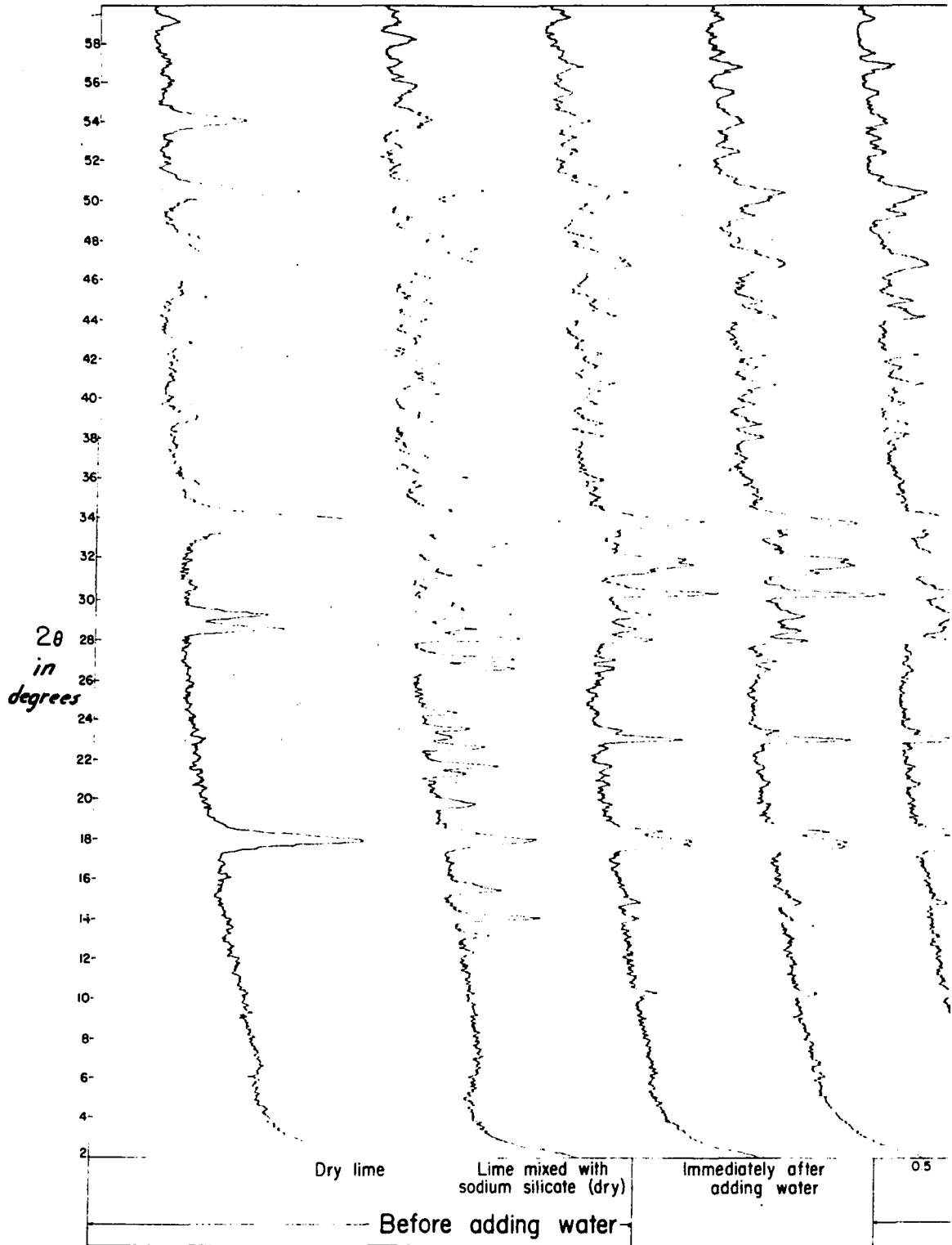
silicate passing into solution. To determine if a new crystalline substance was formed in this process, the mixed compounds were examined by x-ray diffractometer methods using $\text{CuK}\alpha$ radiation and recording intensities on a logarithmic scale with a full-scale deflection of 2000 counts per second. Tests were performed on dry lime, on dry lime mixed with dry sodium silicate, again immediately after adding water and then periodically thereafter for 15 days. When not being tested, the sample was stored in a humid atmosphere. The reaction is probably similar to the reaction that occurs when these two chemicals are mixed with a soil, except for the extent to which the soil enters into the reaction. The extent to which the soil reacts with lime and sodium silicate was not quantitatively determined.

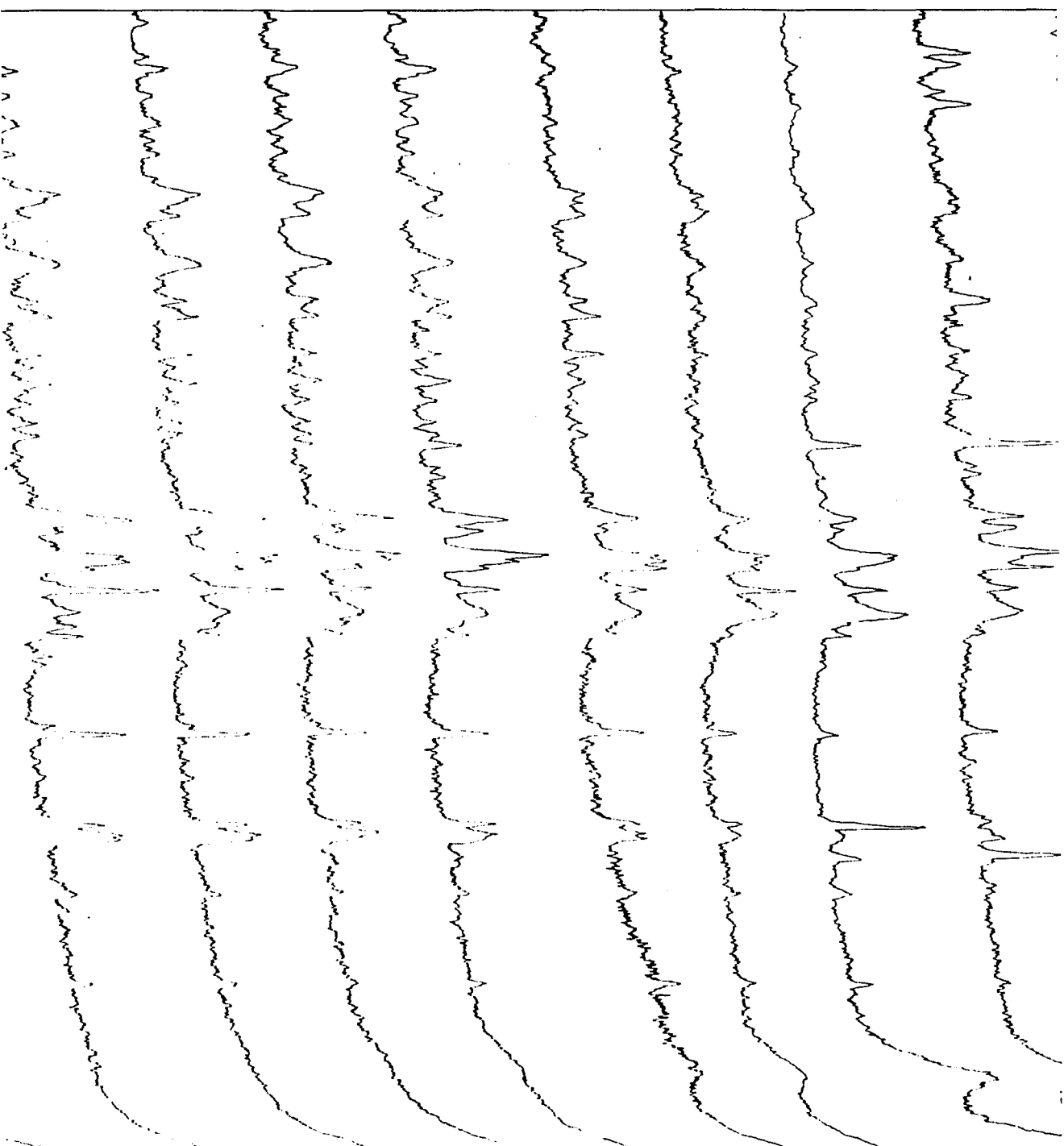
The x-ray diffraction traces in Figure 18 show that some crystalline changes do occur immediately, but that the lime is very gradually consumed. The strong Ca(OH)_2 peak at $2\theta = 18^\circ$ (4.92 \AA) continues to exist to a diminishing degree for about 6 days after the start of the tests, after which the peak disappears, indicating the lime has been consumed in the reaction. However, other peaks characteristic of the lime continue to show in later tests, suggesting that the new crystalline material may have spacings similar to the lime.

Unlike those for the lime, some peaks for powdered sodium silicate disappear immediately after water is added, such as the peaks at $2\theta = 13.2^\circ$ (6.70 \AA); 15.4° (5.75 \AA); 36.1° (2.48 \AA) and 58.3° (1.58 \AA). This is probably due to the silicate going into solution.

Some new peaks are formed or increased within a few minutes after wetting; for example, peaks at $2\theta = 10.3^\circ$ (8.58 \AA); 30.3° (2.95 \AA);

Figure 18. Diffractograms depicting crystalline changes in a mixture of calcitic lime, sodium metasilicate and water

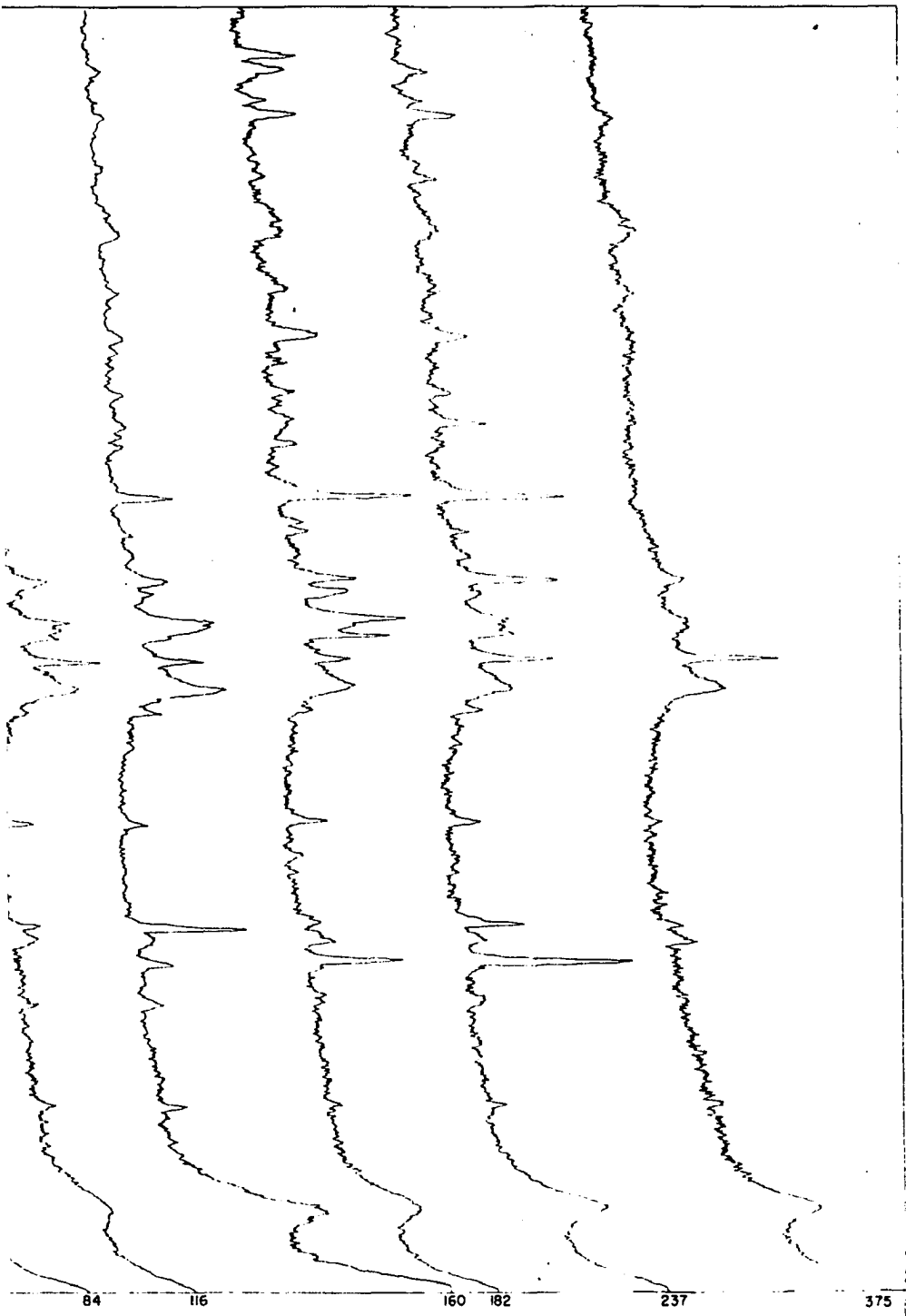




Immediately after adding water

0.5 12 38 62 84 116

Hours after adding water to mixture of lime and



84

116

160

182

237

375

water to mixture of lime and sodium silicate

31.8° (2.80 Å) and 32.1° (2.78 Å) indicating some early crystallization. Some peaks appear after several days of curing only to later vanish; such as, the peaks at $2\theta = 16.8^\circ$ (5.273 Å) and 37.8° (2.378 Å), suggesting that a transient compound is formed only to disappear later. Comparison with the ASTM Diffraction Data File (8-448) indicates the transient compound may be sodium carbonate monohydrate. Calcium carbonate evident in the original lime, may combine with the sodium which is probably being released by the reaction of lime with sodium silicate. Sodium carbonate monohydrate shows strong peaks at $d = 5.35$ Å; 2.768 Å; 2.372 Å; and 2.004 Å. The disappearance of this intermediate compound might be accounted for by its passing into solution because the specimen was stored in a humid environment.

After 15 days of curing, the x-ray diffraction trace appears similar to a calcium silicate hydrate tobermorite, but with a first order basal spacing of 15.36 Å instead of the 9 Å to 14 Å usually evidenced, perhaps as a result of interlayer Na^+ , analogous to expansion of clay minerals. Comparison with the tobermorite from near Tobermory pier on the island of Mull, Scotland and with a less well crystallized synthetic compound called calcium silicate hydrate (I) are shown in Table V. The spacings which have been underlined are those which, according to Heller and Taylor¹ are diagnostic of the tobermorite group.

The diffractogram made 15 days after the start of the tests shows changes from the previous run and would suggest that the final form still

¹Heller, L. and Taylor, H. F. W. Crystallographic data for the calcium silicates. London. Her Majesty's Stationery Office. 1956. pp. 31-32.

may not have been reached. This is corroborated by the strength tests of the specimens, because the strengths were still increasing with time after 28 days. Further experimentation will be required to determine the final product.

Table V. Comparison of x-ray powder data of reaction products with tobermorite and with calcium silicate hydrate (I)

Tobermorite from Tobermory Pier, Mull, Scotland ^a		Calcium silicate Hydrate (I) ^a		Reaction product (cured 15 days)		Diagnostic spacings ^b	
dÅ	I	dÅ	I	dÅ	I	dÅ	I
-	-	-	-	15.40	vsb		
11.4	vvs	11.0	s	-	-	14 to 9	vs
-	-	-	-	8.50	vw	-	-
5.47	mw	5.6	ms	-	-	-	-
-	-	-	-	5.006	w	-	-
-	-	-	-	4.82	w	-	-
3.50	mw	3.55	vvw	-	-	-	-
3.34	vw	3.28	vvw	-	-	-	-
3.10	s	3.07	vvs	3.056	ms	3.06	vs
2.99	m	2.97	s	2.95	s	-	-
2.82	ms	2.80	s	2.80	m	2.81	s
2.74	vvw	-	-	2.77	m	-	-
-	-	-	-	2.63	m	-	-
2.52	vw	2.52	vw	-	-	-	-
2.45	vw	2.48	vw	-	-	-	-
2.30	wb	2.28	ms	-	-	-	-
2.15	w	2.15	m	-	-	-	-
2.09	vw	2.07	m	-	-	-	-
2.01	mw	2.00	m	-	-	-	-
-	-	1.93	vvw	1.93	w	-	-
1.847	m	1.83	vs	1.83	w	1.83	s
-	-	1.81	vvw	-	-	-	-
-	-	1.76	vvw	-	-	-	-
-	-	1.71	vvw	-	-	-	-
1.673	mw	1.67	s	1.67	vw	1.67	m
1.622	vw	1.61	vw	-	-	-	-

^aClaringbull, G. F. and Hey, M. H. A re-examination of tobermorite. Mineralogical Magazine 29: 960-963. 1952.

^bHeller and Taylor, op. cit. p. 32.

DISCUSSION AND CONCLUSIONS

Discussion

Preliminary investigation had shown that sodium silicate and calcium chloride, the two ingredients of the Joosten process for strengthening deep foundations, were unsatisfactory for the stabilization of fine-grained soils, particularly when the two were added as solutions. Specimens were observed to swell excessively and to crack when immersed, indicating the soil thus treated developed or continued to have an affinity for water. Subsequent x-ray diffraction analysis of the precipitate of these two materials when combined as solutions indicated that very tiny crystallites on the order of 10° Å size were formed. The size is probably associated with the speed of precipitation, which was very nearly instantaneous. Because the crystallites are so small, they would have an extremely high specific surface with expected high volume change properties. The by-product of the reaction is probably sodium chloride, a deliquescent compound.

Sodium metasilicate and slaked lime effectively stabilized the variety of soils selected for study, causing sufficient strength in one or two days to be useful in emergency situations. Strength continued to increase thereafter. Because a sodium silicate solution precipitates in the presence of the calcium ion, it is suggested that the first reaction when lime and sodium silicate are combined as powders with water is the dissolving and immediate precipitation of the sodium silicate to form a matrix of silicic acid gel, acting as an adhesive holding the soil particles together. Later and more gradually the slowly dissolving

calcium combines with the silica to form a colloidal, hydrophillic calcium silicate hydrate microcrystalline gel.

Since the alkalinity of both sodium silicate and lime is very high, it is believed that some silica may be dissolved from the soil surfaces and enter into the reaction, improving the qualities of the mixture as a soil stabilizer. Additional research, possibly using x-ray diffraction methods, would be necessary to determine the extent to which soil particles enter into the reaction.

The hydraulic properties of the reaction product are discernable. Curing in a humid atmosphere appears to be essential. After an initial moist curing period, immersion seems to be additionally beneficial. Because most soil stability failures may be attributed to excessive moisture, this property would be highly advantageous.

If a practical method can be devised whereby lime and sodium silicate can be mixed into an excessively wet soil, it is believed that these materials could be used to effectively "dry up" a wet soil and stabilize it. Frequently conditions are such that an attempt at stabilizing extremely wet soils would be justified. If early results are not so important, greater strengths with less additive can be attained by reducing the moisture content of the soil. However, an occasional emergency will arise when the builder cannot afford the time required to dry the soil, or conditions are such that the soil cannot be dried by conventional methods. These occasions might justify the additional expense and the reduced effectiveness. Subsequently, soils thus stabilized and allowed to dry out probably possess a much lower affinity

for water and would be much less plastic. Considerable aggregation can also be expected so that the soil could then be readily stabilized with normal compaction methods.

A combination of sodium metasilicate and calcitic lime appear promising as a soil stabilizer in military situations where economy of time has become the primary consideration and economy of cost secondary. In an area of combat operations these chemicals would provide considerable advantage. Unlike portland cement, both of these materials are easily stored without deterioration due to moisture. Although the lime will gradually carbonate this does not affect the activity of the remaining calcium hydroxide. Although there may be soils which could not be effectively stabilized with calcitic lime and sodium metasilicate, it is believed that such soils would be of unusual composition and of limited extent.

If the relationship between the optimum lime-silicate ratio and the clay content can be determined, it is believed that a simple design formula can be developed which could be readily applied by troops without extensive training.

Although the use of sodium silicate and lime would not be economical in the usual soil stability problems, it does offer some promise as solution to unusual problems and also offers interesting possibilities as a method of producing calcium silicates for laboratory studies.

Conclusions

Recognizing that the findings of this research are subject to and in need of additional refinements and development, it is believed that the following conclusions may be drawn.

1. Calcitic lime and sodium metasilicate pentahydrate when employed under optimum conditions are effective soil stabilizers of a variety of soils ranging from a clean, uniformly graded dune sand to a montmorillonitic clay of high plasticity.

2. Calcitic lime and sodium metasilicate pentahydrate when mixed as dry powders and combined with water to form a thick paste, react to form a colloidal, hydrophillic calcium silicate hydrate microcrystalline gel similar to an expanded calcium silicate hydrate (I) or poorly crystallize tobermorite.

3. The compaction of soil stabilized with sodium metasilicate and calcitic lime should follow immediately after mixing so as to obtain maximum effectiveness.

4. Calcitic lime and anhydrous sodium metasilicate will effectively reduce the plasticity of a montmorillonitic clay under extremely wet moisture conditions and stabilize the soil for use under emergency situations.

RECOMMENDATIONS

In addition to recommending that some field testing be performed to ascertain if methods can be devised so that sodium silicate and lime can be employed in a prototype situation, four areas of further laboratory study are recommended. These are:

1. Research to determine more precisely the reaction products of lime and sodium silicate.
2. Investigate to determine whether clay minerals enter into the reaction between lime and sodium silicate, and if so to what extent.
3. Determine more precisely the effects of moisture and the hydraulic properties of soil stabilized with sodium silicate and lime.
4. Continue work to develop methods by which lime and sodium silicate could be mixed with extremely wet soil by conventional construction equipment.

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