SOIL STABILIZATION

By

CHEMICAL METHODS

MASSACHUSETTS INSTITUTE of TECHNOLOGY

For

CORPS OF ENGINEERS, WATERWAYS EXPERIMENT STATION

FINAL REPORT, PHASE XII

NOVEMBER, 1960

$7.60

SOIL STABILIZATION
by
CHEMICAL METHODS

by

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

for

CORPS OF ENGINEERS

WATERWAYS EXPERIMENT STATION

FINAL REPORT XII

NOVEMBER, 1960

INTRODUCTION

A. CONTRACT

Since 18 June 1946, the Massachusetts Institute of Technology Soil Stabilization Laboratory has been conducting research on soil stabilization under the sponsorship of the Corps of Engineers, U. S. Army. The work has been covered by the following contracts:

W-44009-ENG-408	18 June 1948 - 15 March 1950
DA-44-009-ENG-11	15 March 1950 - 15 March 1951
DA-44-009-ENG-924	15 October 1951 - 15 October 1952
DA-44-009-ENG-1494	21 October 1952 - 22 October 1953
DA-44-009-ENG-2002	22 October 1953 - 22 October 1954
DA-22-079-ENG-171	22 October 1954 - 22 October 1960

This report entitled, "Soil Stabilization by Chemical Methods", Final Report No. XII, describes work done under Contract No. DA-22-079-ENG-171 during the period 22 October 1959 to 22 October 1960.

B. SCOPE OF CONTRACT

Article 1 of Contract No. DA-22-079-ENG-171 states the work and services to be performed. Below are listed those parts of this report which describe work on contract items:

Item No. in Article 1	Report Section
2(a)	No specific section
2(b)	II, III, IV, V, VI
3(a)	No specific section
4(a)	No specific Section
5	This report completes fulfillment

In addition to funds from Contract No. DA-22-079-ENG-171, the Soil Stabilization Laboratory is sponsored by funds from industrial organizations. These funds support fundamental and applied soil stabilization research unrestricted in scope. When work supported by those non-Government funds is thought to be of value to the Army-sponsored work, it is included in the reports made to the Army.

C. ORGANIZATION

The soil stabilization research was conducted by the Massachusetts Institute of Technology Soil Stabilization Laboratory, which has the following organization and staff:

Director	Dr. T. William Lambe Professor of Soil Engineering
Associate Director	Dr. Alan S. Michaels Associate Professor of Chemical Engineering

The staff of the Laboratory consisted of many part-time workers and the following:

Dr. R. T. Martin	Research Associate
Mr. C. C. Ladd	Research Assistant in Civil Engineering, started July, 1955
Mr. C. G. C. Sonino	Research Assistant in Chemical Engineering, started September, 1958
Mr. A. Higinbotham	Research Assistant in Chemical Engineering, started September, 1959
Mr. B. Hill	Research Assistant in Chemical Engineering, started September, 1959

TABLE OF CONTENTS

I. SUMMARY

A. SOIL STABILIZATION WITH ACIDIC PHOSPHORUS COMPOUNDS

Improvement of the stabilization of Massachusetts Clayey Silt (M-21) and Vicksburg Buckshot Clay (VBC) with acidic phosphorus compounds by the use of secondary additives has been studied.

It was confirmed that traces of ortho-rhombic phosphoric anhydride (o-P_2O_5) increase the compacted density of M-21 stabilized with 2% phosphoric acid but the addition of sodium flousilicate nullifies this effect even while accelerating the stabilization reaction.

Octylamine (0.05%) improves the wet strength of M-21 stabilized with phosphate rock and sulfuric acid. (175 psi wet strength after one day humid cure.)

Sodium fluosilicate accelerates the stabilizing reaction of o-P_2O_5 with M-21.

A study of compressive strength of M-21 as a function of the amount of phosphoric acid added, in the presence of 0.5% Na_2SiF_6, was performed. It showed that the strength increases monotonically with the amount of phosphoric acid. To achieve 150 psi and 300 psi wet strength after seven days humid cure less than 1% and less than 2% phosphoric acid is needed, respectively.

Ferric chloride is a good waterproofer for VBC treated with 3.1% o-P_2O_5 when used in conjunction with sodium fluosilicate. Wet strengths of up to 140 psi were obtained after one day humid cure.

Hydrochloric acid, sulfuric acid and ferric chloride have been used to improve the stabilization of VBC by phosphoric acid and sodium fluosilicate. Promising results have been obtained and further research on this point is recommended.

It has been found that, contrary to earlier observations, sodium fluosilicate improves the acidic phosphorus stabilization of VBC. The results suggest that the action of the fluosilicate on VBC may be more than acceleration of the reaction between phosphoric acid and the soil.

B. SOIL STABILIZATION WITH CEMENT

The stabilization of VBC with cement and caustic soda was investigated in detail. With the additive system 5% cement and 1N NaOH, studies were made on the effect of the initial water content and compactive effort on the stabilization of VBC. It was found that, when the water content is varied at constant compactive effort, the wet strength goes through a maximum somewhat below the water content needed for total void saturation.

When the compactive effort is varied at constant water content, the wet strength rises at a decreasing rate: a considerable increase in strength is observed between 850 psi and 1000 psi compactive pressure, but thereafter wet strength does not rise significantly.

A study was made of the wet strength of VBC as a function of cement concentration, in the presence of 0.5 N and 1N NaOH in the pore fluid. In the range from 3% to 10% cement, wet strength increases monotonically with the amount of cement, 1N NaOH is in all cases superior to 0.5N NaOH but for ultimate strength the difference was not found to be significant. To achieve wet strength of 150 and 300 psi after seven days of cure , 4% and 6% cement with 0.5 N NaOH is estimated to be needed, respectively.

A number of organic and inorganic additives were investigated as secondary additives for VBC, 5% cement, and 1N NaOH. None was found to provide significant strength improvement.

C. SOIL STABILIZATION WITH LIME

The stabilization of soils M-21 and VBC with both calcium hydroxide (slaked lime) and calcium oxide (including quicklime, commercial grade) was investigated. In VBC calcium oxide was found to be more effective than calcium hydroxide. Some inorganic salts were used as secondary additives; substantial improvement in wet strengths being obtained with magnesium sulfate.

M-21 cannot be stabilized with 5% calcium oxide alone, but if in addition 1.25% magnesium sulfate is used, 100 psi wet strengths after one day humid cure are obtained. On the other hand, stabilization with slaked lime alone gives 80 psi wet strength after one day cure at equivalent calcium concentrations.

With VBC effective stabilization can be achieved with calcium oxide alone. As the amount of calcium oxide is increased, the strength reaches a maximum at about 5% CaO on dry soil, and then decreases, at least for the curing periods investigated. Two percent or less calcium oxide was inadequate for stabilizing VBC, probably because this amount is insufficient to form a continuous gel throughout the soil. Approximately 2.5%

quicklime alone is necessary to give a wet strength of 150 psi after 7 days humid cure. The maximum wet strength observed with calcium oxide alone was 260 psi after 28 days humid cure with 5% calcium oxide. In the case of calcium oxide and magnesium sulfate (4 to 1 ratio by weight) approximately 3% calcium oxide is needed to give 150 psi and 4% to give 300 psi wet strength after seven days cure.

D. SOIL STABILIZATION WITH SODIUM SILICATES

It was found that New Hampshire Silt and Vicksburg Loess can be stabilized with sodium silicates and basic Magnesium carbonate ("BMC": $4\,MgCO_3 \cdot Mg(OH)_2 \cdot 4\,H_2O$). The best results obtained after seven days humid cure and one day immersion are summarized in the table below:

Soil	Total additive content as % of dry soil weight	Weight ratio $Na_2O:SiO_2:BMC$	Wet Strength, psi
New Hampshire Silt	8.5	1 : 3.2 : 1.14	365
Vicksburg Loess	9.1	1 : 3.2 : 1.5	235
Massachusetts Clayey Silt	6.4	1 : 2.4 : 1.14	610

Unsuccessful attempts were made to stabilize Vicksburg Buckshot Clay with sodium silicate and basic magnesium carbonate.

The effect of silica, magnesium, and sodium contents in each of these soils was studied. In addition a similar study in Massachusetts Clayey Silt was completed. An explanation of the observations is advanced in terms of the mechanism of silicate stabilization previously proposed, and of the presence of polyvalent gel-forming cations in the soils.

Several inorganic salts were reacted with sodium silicate in the absence of soil to investigate their gel-forming characteristics. Only one, a reactive magnesium oxide, formed a gel with the silicate. This reactive magnesium oxide was found to stabilize Massachusetts Clayey Silt as well as an equivalent amount of basic magnesium carbonate. Stabilization with magnesium oxide, however, is considerably slower than stabilization with the carbonate.

Calcium hydroxide was found to be a much less successful precipitating agent than basic magnesium carbonate, when used with sodium silicates to stabilize Massachusetts Clayey Silt. Using $Ca(OH)_2$, seven day soaked strengths of 200 psi resulted, as compared with soaked strengths of 600 psi using BMC.

E. SOIL STABILIZATION WITH ASPHALT EMULSIONS

Asphalt emulsions for the topical treatment of soil surfaces have been developed which have satisfactory soil-penetration characteristics. These include cationic emulsions prepared using N-alkyl propylene diamines in conjunction with hydrochloric acid and trace amounts of chromium or ferric chloride, and nonionic emulsions using Nonic 218 as the emulsifying agent. Dilution of the asphalt to be emulsified with at least half as much gasoline is necessary to make the emulsified droplets sufficiently fluid to pass through the pores of a fine sand. At least ten per cent by weight of ortho-phosphoric acid may be incorporated into any of these emulsions without impairing the soil penetrating characteristics.

When compared at equal asphalt dosages per unit area of soil surface, these emulsions will penetrate soils to greater depths than cutbacks containing the same ratio of asphalt to gasoline; but, at low dosages, the average asphalt concentration in the penetrated layer will be greater for cutbacks. Increasing the dosage of emulsion per unit surface will result in a greater average asphalt concentration in the penetrated layer, even higher than that obtained with cutbacks. Blocking of soil pores by rapid deposition of asphalt droplets from the emulsions, however, limits the dosage of emulsion which will penetrate the soil. For cutbacks, the depth of penetration is directly proportional to the amount applied, the asphalt concentration in the treated layer remaining invariant.

The rate of water seepage through a soil is governed primarily by the fraction of the stabilized surface of the soil which is covered with a film of residual asphalt that has not penetrated the soil. A resistance to mechanical penetration of the surface of 465 psi has been obtained when the surface of a silty sand has been treated with the maximum dosage of a cutback emulsion prepared with Nonic 218 this emulsion contained ten per cent ortho-phosphoric acid by weight and was diluted with an equal weight of water before application to the soil surface.

Two types of water-erosion of surface-treated soil have been observed: (1) complete disruption of the stabilized surface and washing out of the underlying soil, and (2) gradual but slow erosion of individual soil particles from the surface. The erosion resistance is greatest, for any given phosphoric acid content and curing period, when the surface of the treated soil is only partially covered with residual asphalt, permitting the volatiles to escape from the soil and allowing the surface to cure.

Massachusetts Clayey Silt was successfully stabilized by uniformly blending the soil with an amine-stabilized, asphalt-cutback emulsion containing phosphoric acid and a trace of ferric chloride. At a concentration of 2% phosphoric acid and 4% asphalt on dry soil, soaked strengths of 265 psi after one day humid cure, and 455 psi after 7 days humid cure were obtained. These results are superior to those previously observed using asphalt cutback, amines, and P_2O_5.

Using the same emulsion with Vicksburg Buckshot Clay, at a concentration of 2% phosphoric acid and 10% asphalt, a soaked compression strength of 85 psi after one day humid cure was measured; this, also, is substantially higher than values previously obtained with this soil using asphalt cutback-phosphoric acid combinations.

II. SOIL STABILIZATION WITH ACIDIC PHOSPHORUS COMPOUNDS

A. Introduction

It has been shown (Refs. 1, 2) that stabilization of Massachusetts Clayey Silt (M-21) and Vicksburg Buckshot Clay (VBC) with acidic phosphorus compounds can be achieved with three different systems: ortho-phosphoric acid, ortho-rhombic phosphoric anhydride (o-P_2O_5), and phosphate rock with sulfuric acid.

These acidic phosphorus compounds are very effective with the lean soil studied (M-21) but progress is still needed in waterproofing heavy clays economically.

The work reported here and that in progress is aimed at improving the effectiveness of these systems for stabilization of M-21 and VBC by the use of certain secondary additives (a) to accelerate the stabilization reaction, and (b) to increase the resistance of soil so treated to immersion.

These secondary additives included sodium fluosilicate, o-P_2O_5, octylamine, ferric chloride, hydrochloric acid, and sulfuric acid.

In addition, the amount of phosphoric acid added to M-21 was varied in order to obtain a curve of compressive strength vs. % H_3PO_4 which indicates the amount of additive needed to achieve a given strength.

B. Experimental

1. Materials

The soils used in this investigation were Massachusetts Clayey Silt (M-21) and Vicksburg Buckshot Clay (VBC), the properties of which are reported in Table II-1. The phosphoric acid was Merck, reagent grade, analyzing 86.1% H_3PO_4 (by specific gravity). The sulfuric and the hydrochloric acids were du Pont, reagent grade (98% H_2SO_4 and 37% HCl respectively). The natural Florida phosphate rock (supplied by International Minerals and Chemicals Co.) was a fine powder (93% pass 60 mesh, 75% pass 100 mesh, 63% pass 200 mesh) which was reported to contain 71.4% by weight $Ca_3(PO_4)_2$ and about 3% fluorine, the remainder presumably consisting of silica and other components ineffective in soil stabilization. Previous investigation disclosed the presence of some carbonate in this material.

Anhydrous ferric chloride, sodium fluosilicate, and melamine were reagent grade. Octylamine was provided by Pennsalt Chemicals. Orthorhombic P_2O_5 was supplied by Victor Chemical Works; its properties have been discussed (Ref. 2). Rosin Amine D acetate was a 70% aqueous dispersion supplied by Hercules Powder Co.

Since concentrations of primary additives are usually referred to the equivalent amount of phosphoric acid, the following conversion table, giving the actual percentages on dry soil for an equivalent amount of phosphoric acid, should prove useful.

3.82% Ph Rock + 2.58% H_2SO_4 is equivalent to 2% (86% H_3PO_4)
9.55% Ph Rock + 6.45% H_2SO_4 " " " 5% "
1.25% o-P_2O_5 " " " 2% "
3.12% o-P_2O_5 " " " 5% "

Commercial reagent grade phosphoric acid is 86% H_3PO_4.

2. Procedure

The preparation and testing of samples were similar to those detailed in previous reports. o-P_2O_5 was generally dispersed in water and the mixture heated until a clear solution was obtained (approximately 30 minutes). Phosphate rock and sulfuric acid were preblended and the slurry added to the soil. Ferric chloride and sodium fluosilicate were dry-mixed with the soil. Octylamine was dissolved in the acidic phosphorus additive. Sulfuric acid and hydrochloric acid were mixed with phosphoric acid and the mixture added to the soil. Melamine and RADA were dispersed in the phosphoric acid.

Mechanical mixing was accomplished in a finger prong mixer for a few minutes. Samples were then prepared by two-end static compaction in a Harvard Miniature mold. Compaction pressure was always approximately 1000 psi. Samples were cured at 100% relative humidity for various periods of time (1, 7, or 14 days) then immersed in water for 1 day, and finally tested to failure in unconfined compression and dried at 110°C. They were weighed and measured after molding, at testing, and after drying.

C. Discussion of Results

1. Stabilization of M-21

a. Effect of Secondary Additives

In the previous period of investigation it was found that trace amounts of o-P_2O_5 increased significantly the strength and density of M-21 stabilized with phosphoric acid. The o-P_2O_5, a polymeric form of the anhydride, appears to act as a deflocculant of the clay through a mechanism analogous to the action of alkali polyphosphates on kaolinite (Ref. 3).

Those experiments had been performed in the absence of other secondary additives. It was decided to improve the performance of soil so treated by the use of secondary additives that had been previously found effective: sodium fluosilicate, which functions as an accelerator of the reaction of phosphoric acid with the soil, and octylamine, which improved considerably the immersed strength. Results of such experiments are shown in Table II-2.

At first, varying amounts of o-P_2O_5 were used with the octylamine and Na_2SiF_6 + H_3PO_4 system. The amounts of Na_2SiF_6 and octylamine used (0.5% and 0.05%) were established on the basis of previous results. An increase in density was obtained which decreased as the amount of o-P_2O_5 was increased. As explained before (Ref. 2), more than trace amounts of o-P_2O_5 reverse the initial deflocculating effect.

The maximum density achieved (131.5 pcf) with this system was, however, noticeably below that which had been obtained previously (135 pcf).

At the same time the humid cure strength did not correlate well with density. Comparing the samples without o-P_2O_5 with those with the smallest amount of this substance, it is seen that the humid cure strength of the former is higher (510 psi vs. 450 psi) even while the density is lower (128.5 pcf vs. 131.4 pcf).

Investigation was then aimed at clarifying the interactions among the various secondary additives. The results are presented in Table II-2. The following can be noted:

(1) In the absence of sodium fluosilicate, o-P_2O_5 does cause an increase in compacted density of M-21 + 2% H_3PO_4 to 134-135 pcf and a corresponding increase in strength.

(2) Addition of the fluosilicate (0.5%) prevents the achievement of this high density.

(3) Octylamine, on the other hand, does not affect the action of the o-P_2O_5.

(4) If the amount of fluosilicate is reduced to 0.1% some improvement in density is obtained but most of the accelerating effect is lost.

(5) If aluminum fluoride is used instead of sodium fluosilicate both beneficial effects are lost.

(6) The order of addition of o-P_2O_5 and Na_2SiF_6 during mixing does not affect the results.

Thus it appears that the densification caused by o-P_2O_5 and the acceleration of the reaction caused by the Na_2SiF_6 cannot be obtained simultaneously. Under conditions where rapidity of cure is of secondary importance, however, the use of o-P_2O_5 as secondary additive for the phosphoric acid stabilization of M-21 is advantageous. In such cases sodium fluosilicate should not be used.

b. Strength of M-21 as a Function of Concentration of H_3PO_4

In order to determine the amount of phosphoric acid needed to achieve a given desired strength, a series of experiments was made varying the amount of phosphoric acid from 0.5% to 3%, keeping the amount of sodium fluosilicate constant at 0.5%. It should be noted that, at the lower levels of phosphoric acid, less fluosilicate may be adequate. The results of this study are shown in Table II-3 and graphically in Fig. II-1.

As expected, the strength under all curing conditions increases monotonically with the amount of phosphoric acid used. Less than 1% and less than 2% phosphoric acid is needed to achieve wet strengths of 150 psi and 300 psi, respectively, after 7 days humid cure.

2. Effect of Secondary Additives on the Stabilization of M-21 with Other Acidic Phosphorus Systems

In the stabilization of M-21, calcium phosphates + H_2SO_4 systems are less resistant to immersion than equivalent systems with phosphoric acid or ortho-rhombic P_2O_5 (see Table II-4). This has been attributed to the presence of calcium sulfate formed in the reaction of the phosphate with the acid (Ref. 2). With phosphate rock and sulfuric acid at the equivalent of 2% phosphoric acid, 0.5% octylamine improves the wet strength after 1 day cure from 125 psi to 175 psi. Upon immediate immersion samples with octylamine have considerable strength (105 psi) whereas those without it disintegrate.

It has been shown that orthorhombic P_2O_5 can be used with M-21 instead of phosphoric acid with the advantage of a reduction in the material requirements for stabilization (1.25% P_2O_5 is equivalent to 2% H_3PO_4). The effects of Na_2SiF_6 and octylamine have now been studied with 1.25% o-P_2O_5 and results are parallel to those obtained with phosphoric acid (see Table II-4). The fluosilicate accelerates the reaction as seen by the higher 1 day strength and similar 7 days strength. A small amount (0.05%) of octylamine provides substantial immediate immersion strength (115 psi) but does not improve the soaked strength after humid cure.

3. Stabilization of VBC with Orthorhombic Phosphoric Anhydride

a. Effect of Octylamine

On the basis of good humid cure strengths obtained previously (Ref. 2) with VBC and 1.25% o-P_2O_5, experiments were made to test the water resistance of these systems and improve it with secondary additives. The results obtained with 1% octylamine are shown on Table II-5. It can be seen that some soaked strength could be obtained only after seven days humid cure. Samples immersed after 1 day humid cure had negligible strength, although humid cure strengths were comparable to those obtained previously. As usual it was noted that increasing the water content decreased humid cure strength but also decreased the extent of disintegration of the soil on water immersion. Increasing the amount of o-P_2O_5 to 3.6% (3.1% free o-P_2O_5 equivalent to 5% free phosphoric acid) improves considerably the wet strength (from 65 to 155 psi after 7 days humid cure).

Preliminary results show that if P_2O_5 is dry mixed with the soil rather than added in solution, strength development is inferior. The practical convenience of dry-mixing method of addition warrants further investigation of this point.

b. Effect of Ferric Chloride

This substance had been found to impart some water resistance to VBC stabilized with H_3PO_4 (Ref. 2). Table II-5 shows that when this substance was used in conjunction with 3.1% o-P_2O_5 (equivalent to 5% phosphoric acid) excellent soaked strengths (200 psi) were obtained after one week humid cure, but the one day humid cure plus one day soaked strength was very low (20 psi). The addition of sodium fluosilicate, however, improved this strength considerably (up to 140 psi), possibly because of acceleration of the stabilization reaction. This is at variance with earlier work, where no accelerating effect of fluosilicate was reported with VBC.

4. Stabilization of VBC with Phosphoric Acid

a. Effect of Sodium Fluosilicate

It had been reported previously (Ref. 1) that sodium fluosilicate was a very effective accelerator for the phosphoric acid stabilization of M-21, but not of VBC. The experiments with o-P_2O_5 and $FeCl_3$ mentioned above seemed to contradict this conclusion. Remembering that the VBC now being used is from a different shipment from that used previously, it was hypothesized that perhaps the earlier batch contained a significant amount of some fluorine-bearing mineral which was not to be found in the subsequent batch. The two soils were analyzed for fluorine by the recommended A.O.A.C. procedure and both showed the presence of approximately 0.05% fluorine, a small amount in comparison with the amount of fluosilicate usually added to the soil. Furthermore, the difference between the two soils was not significant.

Tests were then made to ascertain the effect of sodium fluosilicate on VBC stabilized with 2% free phosphoric acid and 1% octylamine. The results (Table II-5) show that the humid cure strengths are essentially unaffected by the fluosilicate but that the soaked strengths are considerably improved. Apparently the amount of fluosilicate needed is higher for VBC than it was found to be for M-21. VBC, a heavy clay, has considerable humid cure strength *per se*, and therefore stabilization is difficult to detect through humid cure strength measurements alone.

A consideration of the results obtained with orthorhombic P_2O_5 and ferric chloride (see above) suggests that the action of the fluosilicate in VBC may be different from that observed in M-21. In the latter soil the effect is essentially an acceleration of the reaction of the phosphoric acid with the soil as demonstrated by the fact that the fluosilicate does not increase substantially the ultimate strength. On the other hand, in the case of VBC with 3.1% o-P_2O_5 and 1.6% FeCl, the fluosilicate about triples the wet strength after 1 day cure but also doubles the ultimate strength.* Thus it seems that the presence of fluosilicate either increases the amount of cementitious materials produced or improves their effectiveness. Further investigation may explain more satisfactorily the present results.

b. Effect of Mineral Acids and Ferric Chloride

It has been suggested that the addition of mineral acids (e.g., HCl and H_2SO_4) to the phosphoric acid system may improve the soil stabilization reaction.

A series of tests was run by adding 2% HCl or 2% H_2SO_4 or 2% H_3PO_4 with and without 1.6% $FeCl_3$ to the system VBC + 2% H_3PO_4 + 1% Na_2SiF_6. The results are shown in Table II-5. Both with and without $FeCl_3$ the additional phosphoric acid provides better wet strength improvement than the other acids. The humid cure strength, however, is highest when 2% sulfuric acid is used in addition to 2% H_3PO_4 and 1% $NaSiF_6$. The improvement is quite considerable and it seems reasonable to conclude that there is definitely a better cementation even though the wet strength is lower. Thus, when $FeCl_3$ is added, the wet strength obtained with the phosphoric acid-sulfuric acid system is higher than that with the phosphoric acid-hydrochloric acid system even though the latter gives better results without $FeCl_3$.

It is possible that the phosphoric acid, due to its relatively low acidity, does not solubilize as much soil alumina as could react to form the cementing alumino-phosphate gel. The effect of the increased amounts of acid may be that of making available sufficient alumina to allow the soil stabilizing reaction to proceed further. In addition (and this is especially true of HCl) the lower pH may ionize polyvalent metallic components of the soil (aluminum, iron) which then undergo cation exchange with the clay and thus reduce its swelling tendency. In fact, samples prepared with HCl alone do not develop high humid cure strength, but the low strength is completely retained upon immersion (see Table II-5).

It is interesting to note the parallel between the acidic stabilizing systems and the basic ones, i.e., cement and lime. Increasing the basicity of the latter through the addition of caustic increases the participation of the soil in the stabilizing reaction by solubilizing silica which can then react with cations in solution thus increasing the amount of cementitious materials. Similarly in acidic systems (in

---

* Comparison of the wet strengths after 7 and 14 days cure shows that essentially ultimate strength has been reached.

which alumina rather than silica is the active substance) a lower pH, obtained by addition of strong mineral acids, may liberate larger amounts of alumina which react with excess phosphate ions to produce better cementation.

Ferric chloride decreases the humid cure strength but increases the wet strength. The former effect may be ascribed to the removal of some of the acid as insoluble ferric salts: the percentage decrease in strength caused by the ferric chloride is greatest in the case of phosphoric acid and smallest for hydrochloric acid and, in fact, the solubility of the ferric salts increases in the order $FePO_4 > Fe_2(SO_4)_3 > FeCl_3$.

The waterproofing effect of $FeCl_3$ on the wet strength has been discussed previously (Ref. 2).

The low cost of sulfuric acid relative to that of phosphoric acid makes the possibility of using the former worthy of further investigation.

c. Two Organic Waterproofers

Attempts were made to waterproof VBC stabilized with 2% phosphoric acid through the use of 0.1% and 1% rosin amine D acetate and 1% melamine. No resistance to immersion was obtained (see Table II-5).

5. Stabilization of VBC with Phosphate Rock and Sulfuric Acid

With this stabilizer system at the equivalent of 2% phosphoric acid quite good humid cure strengths can be obtained but the samples disintegrate upon immersion. Neither 1.0% octylamine nor 1.6% $FeCl_3$ improved significantly the wet strength (se Table II-5).

D. Conclusions

1. Sodium fluosilicate interferes with the deflocculating effect on M-21 of trace amounts of orthorhombic phosphoric anhydride (o-P_2O_5), thus decreasing the high compacted density obtained with the use of the latter additive.

2. With M-21 stabilized with acidic phosphorus systems, octylamine does not increase significantly the soaked strength after humid cure, although it improves the resistance to immediate immersion.

3. Sodium fluosilicate accelerates the reaction of o-P_2O_5 with M-21. An increase of at least 40% in the one day humid cure strength is observed.

4. The degree of stabilization of M-21 with phosphoric acid (in the presence of 0.5% sodium fluosilicate) increases regularly with increasing concentration of phosphoric acid in the range 0.5% to 3%. Soaked strengths after seven days cure of 150 psi and 300 psi can be obtained with somewhat less than 1% and 2% phosphoric acid respectively.

5. While only very low soaked strengths can be obtained with VBC stabilized with 1.25% o-P_2O_5 and waterproofed with 1% octylamine, excellent results can be obtained with VBC using 3.1% o-P_2O_5, 1.6% FeCl and 0.5% Na_2SiF_6. Soaked strengths of up to 140 psi after one day humid cure were obtained with this latter system.

6. Contrary to what was reported previously, sodium fluosilicate improves considerably the phosphoric acid stabilization of VBC in the presence of 1% octylamine. The effect is not noticeable in humid cure strength but very obvious in wet strength.

7. Hydrochloric acid, sulfuric acid and ferric chloride all improve the wet strength of VBC + 2% H_3PO_4 + 1% Na_2SiF_6. For example the addition of 2% H_2SO_4 and 1.6% $FeCl_3$ improves the wet strength after 7 days humid cure from 0 to 135 psi. If instead of these two additives an additional 2% H_3PO_4 is used, the strengths are similar.

8. Stabilization of VBC with phosphate rock and sulfuric acid has not been successful.

E. Recommendations

1. Investigate the stabilization of VBC with phosphoric acid, sulfuric acid, ferric chloride, and sodium fluosilicate, attempting to find the optimum relative proportions of these additives.

2. Study the effect of sulfuric acid on the phosphoric acid stabilization of M-21.

3. Examine the stabilization of other soils with the acidic phosphorus systems.

F. References

1. "Soil Solidification by Chemical Methods", Final Report, Phase X, Soil Stabilization Laboratory, M.I.T., November, 1958

2. "Soil Stabilization by Chemical Methods", Final Report, Phase XI, Soil Stabilization Laboratory, M.I.T., November, 1959

3. Michaels, A. S., Deflocculation of Kaolinite by Alkali Polyphosphates, Ind. Eng. Chem. 50, 951 (1958)

TABLE II-1

PROPERTIES OF SOILS INVESTIGATED

PROPERTIES	NHS	1056	Mass. Clayey Silt	Vicksburg Loess	Vicksburg Buckshot Clay
Textural Composition[1] % by weight:					
Sand, 0.06 mm. to 2 mm.	3	82	47	5	0
Silt, 0.002 mm. to 0.06 mm.	90	18	41	74	65
Clay, < 0.002 mm.	7	0	12	21	35
Physical Properties:					
Liquid Limit, %	28	--	20	41	63-67
Plastic Limit, %	20	--	14	26	27-28
Plasticity Index, %	8	N.P.	6	15	35-40
Specific Gravity 20°C/20°C	2.72	2.64	2.7	2.67	2.67
Maximum Dry Density[2] lb/ft^3	99.5	102	122.0	105.0	105.0
Optimum Water Content, %	19.9	14.2	13.3	18.2	22.0
Chemical Properties:					
Organic matter, % by weight	0.1	1.9	--	0.2	1.1
pH	5.4	6.7	--	4.6	4.6
Total soluble salts, as m.eq. NaCl/100 gm.	--	0.1	--	0.2	0.3
Soluble Sulfates, m.eq. NaCl/100 gm.	--	--	--	--	--
Carbonates, % by weight	--	--	--	--	--
Mineralogical Compositions[3]:					
Clay Composition %	10	--	30	35	50
Illite:Montmorillonoid:Clay-Chlorite	1:0:0	--	1:0:1	1:1:0	1:1:0
Chlorite, nonclay, %	--	--	--	--	--
Calcite, %	--	--	--	--	--
Free Iron Oxide, % Fe_2O_3	1.0	--	2.9	1.6	1.9
Gypsum, %	--	--	--	--	--
Source:	N.H.	Wisc.	Mass.	Miss.	Miss.

---

[1] M.I.T. Classification System.

[2] Harvard Miniature Compaction, 40-lb. tamper, 3 layers, 25 blows per layer.

[3] For -0.074 mm. fraction.

TABLE II-2

EFFECT OF SECONDARY ADDITIVES ON M-21 STABILIZED WITH 2% PHOSPHORIC ACID

	Days Humid Cure		0			1			1			7			7		
	Days Immersion		1			0			1			1			0		
0.5% Na_2SiF_6	0.05% Octylamine	% oP_2O_5	C.S.[1] psi	Vol. %[2]	D.D.[3] pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf
No	No	No[6]				325 ± 15	10.1	132.1	280 ± 15	10.5	132.0	275 ± 15	10.5	132.1	330	10.1	132.6
Yes	No	No	190	11.0	132.2	540	9.8	131.8	325 ± 20	10.7	131.1	440 ± 5	10.4	131.0	780	9.6	
No	No	0.05	--	--	--	410 ± 10	7.9	133.6	340 ± 30	10.0	132.8	420 ± 5[4]	9.8	133.5	650[4]	9.5	133.4
Yes	No	0.05	--	--	--	540 ± 5	9.6	131.0	355 ± 50	10.4	131.0	400 ± 20[4]	10.5	130.8	690[4]	9.5	130.5
No	Yes	0.05	155 ± 5	10.8	131.8	375 ± 5	9.1	134.5	425 ± 5	9.5	133.7	430[4]	9.7	134.7	--	--	--
Yes[5]	No	0.05				475 ± 35	9.9	132.5	375 ± 5	10.1	133.1	470 ± 40	10.1	132.5	660	10.0	133.5
0.1%	No	0.05				370	9.8	133.2	285 ± 5	10.2	133.5	430 ± 10	9.8	133.5	550	9.7	135.2
AlF_3	No	0.05				330	10.2	131.5	305 ± 5	10.5	131.3	365 ± 25	10.3	131.6	580	9.8	132.0
Yes	Yes	No	165 ± 5	10.9	128.1	510	9.1	128.5	280 ± 10	9.5	127.6	290	9.7	128.5	670	8.6	129.0
Yes	Yes	0.05	175 ± 5	11.0	128.6	450 ± 20	9.6	131.4	295 ± 5	10.1	130.1	--	--	--	615	9.5	130.6
Yes	Yes	0.10	140 ± 5	11.4	126.9	400 ± 20	9.3	130.0	275 ± 5	10.2	129.1	--	--	--	610	9.0	129.0
Yes	Yes	0.25	120 ± 20	11.2	124.0	385 ± 15	9.2	125.9	190 ± 5	10.3	125.5	--	--	--	410	8.9	126.0
Yes	Armeen 8	No	120	12.4	125.2	300 ± 20	9.3	129.3	225 ± 5	11.7	127.7	--	--	--	570	9.7	128.5
Yes	Yes	No	210 ± 10	11.8	128.5	440	11.0	129.0	350 ± 20	11.1	129.8	--	--	--	830	--	--

[1] C. S. is actual compressive strength in psi

[2] % Volatiles at test, i.e., weight lost upon drying as a percentage of dry solids weight

[3] Dry density at test, i.e., pounds of dry solids per cubic foot

[4] These samples had 14 days humid cure

[5] The Na_2SiF_6 was mixed with the soil after the $o\text{-}P_2O_5$

[6] Other strengths: 14 days humid cure: 595 ± 5; 14 days humid cure and 1 day immersion, 345 ± 55

All percentages of additives are based on dry soil

TABLE II-3

STABILIZATION OF M-21 WITH PHOSPHORIC ACID

All samples contain 0.5% Na_2SiF_6

Days Humid Cure	1			1			7			7		
Days Immersion	0			1			0			1		
% H_3PO_4	C.S.[1] psi	Vol.[2] %	D.D.[3] pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf
0.5	250	9.2	129.2	85 ± 5	10.3	129.0	370	8.8	129.5	90	11.0	128.7
1.0	290	10.3	128.5	--	--	--	395 ± 5	9.6	127.8	175 ± 15	10.6	129.0
1.5	355 ± 5	9.9	129.5	170	11.2	128.9	470	9.7	128.3	240 ± 10	11.0	128.9
2.0	540	9.8	131.8	325 ± 20	10.7	131.1	780	9.6	--	440 ± 5	10.4	131.0
3.0	770	--	--	630 ± 20	10.4	130.8	1020 ± 50	9.1	130.1	610 ± 30	10.4	131.8

All percentages of additives are based on dry soil.

[1] Actual Compressive Strength

[2] % Volatiles at test, i.e., weight lost upon drying as a percentage of dry solids weight.

[3] Dry density at test, i.e., pounds of dry solids per cubic foot.

Table II- 4

STABILIZATION OF M-21 AT THE EQUIVALENT OF 2% PHOSPHORIC ACID

Days 100% RH Cure	0			1			1			7			7		
Days Immersion	1			0			1			0			1		
Formulation	C.S.[1] psi	Vol. %[2]	D.D.[3] pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf	C.S. psi	Vol. %	D.D. pcf
3.8% Phosphate Rock + 2.6% H_2SO_4	Disintegrated			270 ± 10	11.3	128.1	125 ± 5	11.7	127.2	340 ± 5	11.2	128.7	180 ± 5	12.1	127.6
Same + 0.5% Octylamine	105 ± 5	12.1	125.0	290 ± 10	10.6	127.2	175 ± 5	11.1	126.4						
1.25% o-P_2O_5 [5]	--	--	--	246 ± 4	10.6	133.3	--	--	--	570 ± 69	9.6	133.1			
Same + 0.5% Na_2SiF_6	Disintegrated			340 ± 5	9.6	129.4	225 ± 15	10.4	130.7						
Same + 0.5% Na_2SiF_6 + 0.05% Octylamine	115 ± 15	11.1	126.0	420 ± 5	9.2	127.7	220 ± 10	9.8	128.7	600	8.8	129.5			
2% H_3PO_4 + 0.5% Na_2SiF_6 (4)				305 ± 17	11.3	129.3	235 ± 15	12.0	128.8	357	11.3	129.0			
Same + 0.05% Octylamine[4]	70 ± 3	14.1	124.7	237 ± 23	12.1	127.3									

[1], [2], [3] See Table II-2

[4] Data from Ref. (1)

[5] Data from Ref. (2)

All percentages of additives are based on dry soil.

Table II-5

STABILIZATION OF VBC

ADDITIVES				1 Day Humid Cure			1 Day Immersion After		
				C.S.[1] psi	Vol.[2] %	D.D.[3] pcf	1	7	14
							Days Humid Cure, C.S., psi		
1.75% o-P_2O_5[4]			1% $C_8H_{17}NH_2$[10]	335±5	16.9	111.8	0	40±20	15±5
1.75% o-P_2O_5			1%$C_8H_{17}NH_2$	280±10	18.6	109.7	10	30	20
1.75% o-P_2O_5			1% $C_8H_{17}NH_2$	--	22.1	104.6[6]	25	65±10	55±10
1.25% o-P_2O_5[5]		0.75% H_3PO_4[4]	1% $C_8H_{17}NH_2$	270±5	17.2	109.2	0	0	0
3.6% o-P_2O_5			1% $C_8H_{17}NH_2$	--	22.0	105.0[6]	70±10	155	180±20
3.1% o-P_2O_5			1.6% $FeCl_3$	290±20	18.3	111.2	20±5	200±5	235±10
3.1% o-P_2O_5			1.6% $FeCl_3$	190±30	20.6	107.5	30±5	160±5	230±10
3.1% o-P_2O_5	0.5% Na_2SiF_6		1.6% $FeCl_3$	290±10	21.4	106.6	135±5	280±20	290
3.1% o-P_2O_5	2.0% Na_2SiF_6		1.6% $FeCl_3$	350±10	21.1	107.3	105±5	420±20	415±10
2.76% H_3PO_4[4]			1%$C_8H_{17}NH_2$	190±10	21.4	104.4	10	30±10	
2.76% H_3PO_4			1%$C_8H_{17}NH_2$	215±15	21.6	104.3	45±5		
2.76% H_3PO_4[7]	0.5% Na_2SiF_6		1% $C_8H_{17}NH_2$	290±20	15.1	113.5	110±40		
2.76% H_3PO_4	0.5% Na_2SiF_6		1% $C_8H_{17}NH_2$	205±5	23.3	104.0	45±5	50	
2.76% H_3PO_4	2.0% Na_2SiF_6		1%$C_8H_{17}NH_2$	215±5	23.9	101.7	135±25	140±10	
		11.5% HCl(aq.)		80±15	26.9	99.6		20±5[8]	
2% H_3PO_4		2% HCl(aq.)		255±5	21.2	106.9	0	45±5	90±5
2% H_3PO_4	1% Na_2SiF_6	2% H_3PO_4		330±20	21.0	107.2	55±5	105±5	
2% H_3PO_4	1% Na_2SiF_6	2% HCl(aq.)		285±5	19.6	108.8	10±5	65±5	
2% H_3PO_4	1% Na_2SiF_6	2% H_2SO_4		425±5	19.3	108.8	0	0	
2% H_3PO_4	1% Na_2SiF_6	2% H_3PO_4	1.6% $FeCl_3$	240±5	21.1	107.6	75±10	215±10	235±5
2% H_3PO_4	1% Na_2SiF_6	2% HCl(aq.)	1.6% $FeCl_3$	250±5	22.7	105.7	25±10	60±5	
2% H_3PO_4	1% Na_2SiF_6	2% H_2SO_4	1.6% $FeCl_3$	345±5	22.0	106.0	55±5	135±10	
2% H_3PO_4			0.1% RADA[9]	375±15	18.2	111.8	0	0	0
2% H_3PO_4			1.0% RADA	330±10	18.6	110.1	0	20±5	25±5
2% H_3PO_4			1.0% Melamine	315±5	17.8	110.0	0	20±20	0
3.8% Ph. Rock		2.6% H_2SO_4		310±20	17.1	110.6	0	0	
3.8% Ph. Rock		2.6% H_2SO_4	1% $C_8H_{17}NH_2$	325±10	20.3	106.6	10±10	25±25	
3.8% Ph. Rock		2.6% H_2SO_4	1.6% $FeCl_3$	325±15	22.5	104.9	0	0	0

All percentages of additives are based on dry soil

[1] Actual compressive strength

[2] % Volatiles at test, i.e., weight lost upon drying as a percentage of dry solids weight

[3] Dry density at test, i.e., pounds of dry solids per cubic foot

[4] 0.76% H_3PO_4 or 0.5% o-P_2O_5 are needed to neutralize 1% octylamine

[5] The o-P_2O_5 was dry mixed with the soil

[6] These data are "at molding" for the samples cured 14 days

[7] Data from Ref. (1)

[8] Humid cure strength after 7 days: 25±5 psi

[9] Rosin amine D acetate

[10] Octylamine

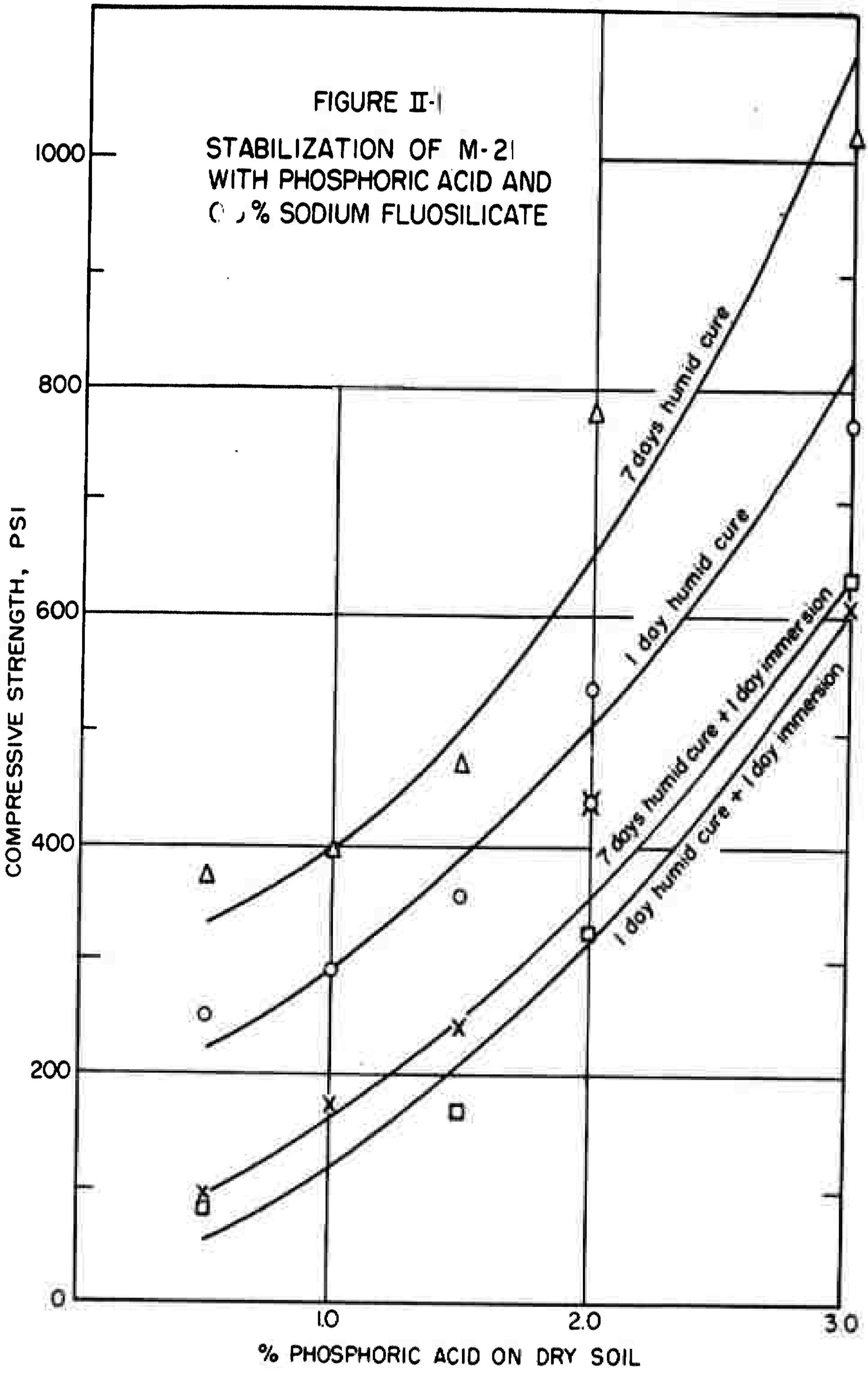


FIGURE II-1

STABILIZATION OF M-21 WITH PHOSPHORIC ACID AND 0.5% SODIUM FLUOSILICATE

III. SOIL STABILIZATION WITH CEMENT

A. Introduction

During the period September 1959 to June 1960 experiments were conducted to evaluate the effectiveness of various organic and inorganic additives as waterproofing agents for cement-stabilized Vicksburg Buckshot Clay (VBC). In view of the lack of reproducibility of some of these results, an effort was made to correlate the measured compressive strengths (following seven days cure and one day immersion), water absorption on immersion, and density at molding, with the independent variables of compactive effort and molding water content. The stabilizer system used for this study was 5% cement plus 1N NaOH in the molding water. In addition, tests were run with VBC where the cement content and the normality of the sodium hydroxide in the pore fluid were varied.

B. Experimental

1. Materials

The soil used in this investigation was Vicksburg Buckshot Clay (VBC). A description of this soil can be found in Table II-1.

The reagent grade chemicals used were sodium hydroxide, ferrous chloride, zinc nitrate, stannic chloride, ferric chloride, aniline and melamine. Rosin Amine D Acetate was supplied as a 70% solution in water by the Hercules Powder Company.

2. Procedure

The testing procedures were similar to those previously described. There was, however, one major change.

Air dried soil was equilibrated for 24 hours with half of the molding water and with the secondary additive when used. The secondary additives were incorporated as either suspensions or solutions except in the case of melamine which was mixed in as a dry powder. In some cases, where noted, the soil was not equilibrated overnight: the secondary additive was incorporated with half the molding water just prior to mixing and molding. After equilibration, cement and sodium hydroxide were added in that order and mixing was completed in a finger-blade mechanical mixer for 5 minutes.

The major change mentioned above is in the molding procedure. Samples were molded by two-end static compaction in a Harvard Miniature-size mold to a constant pressure of 1000 psi. In previous work samples were molded to constant density. It was felt that changing the procedure was justified and advisable because:

a. It is now possible to compare soil-cement results with the results of other systems studied in this laboratory. With this change, the procedure is standardized.

b. It is more practical and realistic to let the density be the dependent variable and the compactive effort the independent variable since this is what is done in the field.

c. Keeping compactive effort the same, we can observe the effect of various additives on the density of the sample. Since there appears to be, in general, a correlation between compacted density and strength, additives which increase the density of, as well as waterproof the soil are desirable.

All samples were cured under approximately 100% relative humidity at room temperature for various periods of time. Samples were then immersed in distilled water for one day prior to testing to failure in unconfined compression. Weights and dimensions of the samples were measured both after curing and after immersion.

C. Results and Discussion

1. Effect of Changing Procedure

Strengths obtained using constant pressure are generally higher than those compacted to constant density because higher densities are being obtained . From Reference 2, the one day strength for 5% cement is 45 ± 5 psi and the dry density is 96.3 pcf. In this report the one day cure, one day immersed strength for 5% cement is 66 ± 9 psi and the dry density is 105.2 pcf.

2. Secondary Additives

The results with secondary additives showed that none of those tested effected any significant increase in the strength obtained with VBC, 5% cement, and 1N NaOH (See Table III-1). This conclusion supersedes a conclusion in an earlier report* that these waterproofers were effective, which was found to be incorrect because the blank for 5% cement and 1 N NaOH was in error.

---

*Sept. '59-Jan. '60 Periodic Report

3. Effect of Cement Content

In order to determine the amount of cement needed to achieve a given strength with VBC a series of experiments were made with varying cement concentrations.

It had been found previously* that at the higher concentrations of cement (10%) the improvement obtained by the addition of 0.5N NaOH was better than that obtained with 1N NaOH. It was therefore decided to vary the cement content at the two levels of caustic. Table III-2 and Figure III-1 show the results.

It can be seen that the strength increases with cement content first rapidly and then more slowly. Contrary to the previous results, the samples with 1N NaOH are consistently stronger over the whole range of cement concentration.

However, the advantage of using 1N NaOH rather than 0.5N decreases as the length of the curing period increases. The difference is considerable after one day cure but not significant after 28 days cure. Nevertheless the addition of 0.5N NaOH improves the strength of VBC-cement for all curing periods, although, again, the percentage increase decreases with time. The effect of 0.5N and 1N NaOH on VBC and 5% cement can be seen in the following table.

	5% Cement	5% Cement 0.5 N NaOH	5% Cement 1.0 N NaOH
1 day	60 ± 10	135 ± 15	190 ± 10
7 day	195 ± 5	255 ± 5	280 ± 10
28 day	310 ± 20	400 ± 20	425 ± 5

4. Relations Between Density, Water Absorption on Immersion, and Compressive Strength of Cement-Stabilized VBC

The effect of varying molding water content at constant compactive effort (1000 psi) is shown in Table III-3 and Figure III-2. As the molding water content increases in the range of 19.5% to 27.2% on solids weight, the as-molded density decreases. However, soaked compressive strength first increases, passes through a maximum in the vicinity of 22% water content, and then decreases. Density at

---

* Soil Solidification by Chemical Methods, Final Report Phase XI, November, 1959

test for the given water content values, vary between 98.2 and 105.8 pfc. The per cent water uptake upon immersion decreases with increasing molding water content. For the soil-stabilizer system studied, the minimum molding water content corresponding to maximum void saturation is estimated to be about 24%.

Table III-4 and Figure III-3 show the results obtained when as-molded density is controlled by adjusting the compactive effort at a constant molding water content of 22%. As-molded density increases with compactive effort in the range of 700 to 1000 psi; greater compactive effort has little further influence on density. Soaked compressive strength and density at test similarly increase with compactive effort, and show a corresponding leveling off in the vicinity of 1000 psi compaction pressure (see Figure III-3). (At the selected water content, the calculated density corresponding to maximum void saturation is approximately 108.2 pcf. This is greater than the measured as-molded density at 1000 psi compaction.) Water absorption on immersion (at constant molding water content) is observed to decrease as density at molding increases (see Figure III-3).

The above observations can, it is believed, be satisfactorily explained in terms of effects of density and water content at molding on:

a. The efficiency of interparticle cementation by the stabilizer, and

b. The capillary stresses imposed upon the stabilized soil structure upon immersion.

If a stabilizer such as cement is homogeneously distributed through soil, the degree of stabilization developed should increase with an increase in volume concentration of interparticle contacts; that is to say, the strength of the stabilized structrure should increase monotonically with the as-molded density. It is, however, well established that maximum density at compaction can be achieved only at a water-content somewhat below that corresponding to void saturation of the compacted soils. Since stabilizers such as cement appear to perform their interparticle cementing function only in the presence of water, it follows that, in any part of a consolidated soil mass where the voids are occupied by air, cementation cannot take place, or takes place only to a limited degree. In other words, a stabilizer such as cement will not be uniformly distributed through a soil which is below void saturation in water, and the lower the water-saturation of the soil as compacted (irrespective of density), the poorer will be the stabilizer distribution. Hence, it would be predicted that, at constant as-molded density, the lower the water content below saturation, the lower the strength of the cured stabilized soil. Thus, as a soil at constant molding water content is subjected to greater and greater compaction, both the density (which controls interparticle contacts) and the degree of saturation by water (which controls stabilizer distribution) increase (see Fig. III-3); both of these variables cooperate to yield a rapid increase in stabilized strength. Once complete void saturation by water is achieved, however, any further increase in water content will result in a reduction in as-molded density and a dilution of the stabilizer; hence an increase of water content in excess of that corresponding to saturation (at a specified compactive effort) can be expected to yield reduced strength (see Fig. III-2). It is, of course, entirely possible that a water content slightly below saturation may prove optimal with respect to strength, since the corresponding density increase may more than compensate for the loss in uniformity of stabilizer distribution.

When a compacted soil mass is totally immersed in water, absorption of water will take place by capillary imbibition, if the voids are not completely water-filled, and/or by interlaminar swelling of montmorillonoid minerals, if they are not at osmotic equilibrium with the immersion solution. With VBC, both of these imbibition mechanisms are likely to be operative. This imbibition produces rather large expansive stresses in the bonded soil skeleton, and if the magnitude of these stresses exceeds the interparticle bond-strength, the bonds will rupture, and the mass will undergo substantial expansion with a major loss in strength.

In all likelihood, those stresses produced on immersion of cement stabilized VBC which are attributable to interlaminar swelling are controlled primarily by the ionic composition of the pore fluid; for the series of experiments performed here, this contribution to expansion on immersion is probably nearly constant for all samples. Capillary stresses, however, are subject to control during molding, since both the void ratio and degree of saturation are dependent on compaction and molding water content. The magnitude of the capillary pressure developed on immersion will increase as:

a. The degree of water saturation at molding decreases, and

b. The molded density increases.

Since the lower the void ratio, the smaller is the mean pore size and the higher the imbibition pressure, of two soil samples of the same compacted density, the one of higher initial water content will be subjected to less capillary stress on immersion, depending upon how close to saturation it is prior to immersion.

It will be noted that, as the molding water content is reduced below that corresponding to void saturation, the capillary stresses developed on immersion increase, and the efficiency of interparticle bonds by the stabilizer decreases. Both of these factors cooperate to cause a major reduction in soaked strength with decreasing molding water content, and a corresponding increase in water absorption and swelling on immersion. These trends appear to be confirmed by the experimental observations (see Fig. III-2). Inspection of the data obtained at constant molding water content indicates that, as density at molding is increased, strength on immersion is increased, and water absorption decreased; this indicates that the increase in interparticle bonding efficiency, and/or the increase in void saturation resulting from greater densification, more than compensates for the increase in capillary pressure on immersion resulting from a reduction in pore size.

In summary, it appears that the immersed strengths of cement stabilized VBC are controlled by the compacted density, and the degree of saturation at compaction, and that these two variables affect strength in the opposite directions. Optimum immersed strength is developed at the highest density attainable at virtually complete void saturation rather than at the highest density attainable at the normal "optimum water content". Similar observations have been made with this soil stabilized with phosphoric acid; it appears likely that this may be a general characteristic of heavy clay soils stabilized with reactive, inorganic stabilizers.

D. Conclusions

1. A number of secondary organic and inorganic additives were tested, but no significant benefit was obtained over the VBC, cement, and NaOH system from them.

2. The ultimate wet strength of VBC-5% cement is improved as much by 0.5N NaOH as by 1.0 N NaOH. Strength development, however, is faster with 1.0N NaOH. The improvement made by 0.5N NaOH over soil cement is significant but the use of 1.0N NaOH does not seem to be justified.

3. As the amount of cement is varied in the range of 3 to 10% with 0.5N NaOH strength increases first rapidly and then more slowly. After seven day cure wet strengths of 150 psi and 300 psi can be achieved with 4 and 6 % cement respectively.

4. The immersed strength of cement stabilized VBC is controlled by the compacted density and the degree of saturation. These two variables affect strength in opposite directions. Optimum immersed strength is developed at the highest density attainable at virtually complete void saturation rather than at the highest density attainable at the normal "optimum water content".

E. Recommendations

Continue to investigate the effect of field variables on the strength of soil-cement.

1. Run a series of VBC samples with varying water content but no NaOH. Compare these to the results obtained with varying cement content and 0.5N and 1N NaOH.

2. Run a series of tests at different compactive efforts for samples having different water contents.

3. Investigate the relation between NaOH content and strength in VBC.

4. Investigate the effect of adverse curing conditions on the strength of soil-cement.

Table III-1

EFFECT OF SECONDARY ADDITIVES ON THE PROPERTIES OF SODIUM HYDROXIDE TREATED CEMENT-VICKSBURG BUCKSHOT CLAY (VBC)

Cement Content = 5% on Dry Soil Weight; 1N NaOH in Pore Water

				At Molding		After Curing		At Testing		
Secondary Additive	%	Equilibrated	Curing Days	Moisture[1] Content %	Dry[2] Density pcf	Moisture[1] Content %	Dry[2] Density pcf	Moisture[1] Content %	Dry[2] Density pcf	Wet Compressive Strength psi
*---	---	---	1	21.5	105.2	20.8	105.3	24.0	101.5	66 ± 9
			7	21.2	105.2	20.8	105.7	22.6	104.6	216 ± 9
			28	20.8	105.4	20.8	105.6	20.8	105.5	299 ± 11
---	---	---	1	21.4	106.1	21.6	106.2	23.5	104.3	185 ± 15
			7	22.0	105.2	21.8	105.3	23.3	105.0	315 ± 5
			28	21.3	105.5	21.3	105.6	22.3	105.5	420
---	---	---	1	22.0	104.9	21.8	104.9	23.9	103.6	190 ± 10
			7	22.0	104.9	21.5	105.0	23.0	104.8	240 ± 10
			28	21.2	105.8	21.0	106.0	21.8	105.8	430 ± 10
RADA[3]	0.7	Yes	1	23.0	102.2	23.3	102.4	25.4	100.9	204 ± 2
			7	21.4	104.1	21.5	104.8	22.6	103.7	292 ± 32
			28	21.5	103.4	22.5	103.1	23.0	103.5	365 ± 5
RADA	0.7	No	1	21.3	104.2	21.4	105.2	23.7	102.0	192 ± 6
			7	21.3	104.4	21.3	104.8	22.7	102.7	290 ± 5
			28	21.0	104.6	20.9	104.8	22.0	104.5	345 ± 15
RADA	0.2	Yes	1	22.3	103.5	22.3	103.7	24.9	102.4	183 ± 18
			7	22.2	103.8	22.0	103.6	23.7	103.2	300 ± 25
			28	22.4	103.5	22.6	103.8	23.4	103.7	415 ± 15
RADA	0.1	No	1	21.6	105.4	21.5	105.4	24.0	102.9	194 ± 13
			7	21.3	105.6	21.1	105.6	22.5	104.8	335 ± 5
			28	20.5	106.2	20.4	106.4	22.0	106.2	410 ± 0
RADA	0.025	No	1	22.2	104.4	22.0	104.4	24.0	102.5	225 ± 5
			7	22.4	104.2	21.8	104.3	23.5	103.6	355 ± 25
			28	21.0	105.3	21.4	105.8	22.6	105.3	400 ± 5
Melamine	1.0	Yes	1	22.0	104.0	22.3	104.8	24.1	101.7	177 ± 25
			7	21.9	104.0	22.0	104.9	22.7	104.0	320 ± 0
			28	21.6	104.0	21.4	105.0	22.3	103.7	428 ± 17
Melamine	1.0	No	1	22.2	104.1	22.0	104.6	24.9	101.8	134 ± 2
			7	21.3	104.6	21.2	105.0	22.3	103.4	279 ± 40
			28	21.7	104.4	21.5	104.7	22.4	104.5	425 ± 55
Aniline	1.0	Yes	1	21.6	104.2	21.0	105.3	24.4	102.3	152 ± 0
			7	21.4	104.6	21.2	105.3	22.3	102.8	255 ± 15
			28	21.6	104.4	21.5	104.4	22.0	103.5	400 ± 5
$Zn(NO_3)_2$	0.5	Yes	1	22.3	104.0	22.0	104.2	23.6	103.0	220 ± 15
			7	22.4	104.1	22.3	104.0	23.1	103.5	335 ± 5
			28	22.1	104.1	22.2	104.0	22.8	103.3	410 ± 30
$Zn(NO_3)_2$	0.1	Yes	1	21.5	105.1	21.5	105.2	24.2	103.3	155 ± 35
			7	21.3	105.2	21.3	105.2	22.8	104.2	385 ± 85
			28	21.3	105.2	21.6	105.2	22.4	105.2	465 ± 5
$Zn(NO_3)_2$	0.1	No	1	22.1	104.4	21.8	104.0	24.8	102.0	163 ± 23
			7	21.6	104.9	20.5	105.2	22.5	104.4	345 ± 45
			28	--	---	--	---	--	---	---
$Zn(NO_3)_2$	0.1	No	1	22.3	105.5	22.3	105.5	23.7	103.8	175 ± 15
			7	21.6	105.0	21.5	106.0	22.6	105.0	270
			28	21.3	105.5	21.1	105.8	22.1	105.6	330 ± 10
$SnCl_4$	0.1	Yes	1	22.0	104.5	22.0	104.5	23.2	104.5	225 ± 15
			7	21.0	104.8	21.9	104.9	22.6	104.9	290 ± 15
			28	21.5	105.0	21.5	105.1	22.6	105.0	320 ± 5
$FeCl_3$	0.1	No	1	22.1	105.1	22.1	105.3	23.8	104.8	230
			7	21.5	105.7	21.5	106.0	22.5	105.7	330 ± 5
			28	22.1	105.1	22.0	105.5	22.9	105.1	370 ± 10
$FeCl_2$	0.5	Yes	1	21.6	105.3	21.6	105.3	23.5	102.8	175 ± 1
			7	22.1	104.2	22.1	104.2	23.2	103.7	293 ± 7
			28	22.3	104.0	22.5	104.0	23.5	103.9	395 ± 15
$FeCl_2$	0.1	Yes	1	21.6	105.3	21.6	105.3	23.5	102.8	175 ± 1
			7	21.4	105.5	21.2	105.5	22.3	105.2	322 ± 28
			28	21.4	105.3	21.4	105.2	22.1	105.1	445 ± 35
**---	---	---	1	21.4	105.5	20.6	106.0	--	--	525 ± 5
			7	20.7	105.9	20.3	106.0	--	--	600 ± 20
			28	20.5	106.1	20.3	106.4	--	--	690 ± 10

---

[1] % moisture content is taken as the weight lost upon drying as a percentage of dry solids weight.

[2] Dry density is the pounds of dry solids per cubic foot.

[3] RADA refers to Rosin Amine D Acetate supplied by Hercules Powder Company.

* Does not contain NaOH.

** Humid cure strengths, not immersed.

Table III-2

EFFECT OF VARYING CEMENT AND NaOH CONTENT

		At Molding		After Curing		At Testing		
Composition Soil: VBC	Curing Days	Moisture Content %	Dry Density pcf	Moisture Content %	Dry Density pcf	Moisture Content %	Dry Density pcf	Wet Compressive Strength psi
3% Cement 0.5N NaOH	1	--	--	--	--	--	--	Disintegrated
	7	21.8	104.9	21.5	105.0	23.1	104.3	100 ± 5
	28	21.4	105.6	21.5	105.8	22.8	105.0	165 ± 25
3% Cement 1N NaOH	1	--	--	--	--	--	--	30 ± 10
	7	--	--	--	--	--	--	90 ± 20
	28	23.5	103.4	23.3	103.5	24.2	103.4	170 ± 5
5% Cement 0.5N NaOH	1	23.0	103.9	22.8	104.3	25.0	102.0	135 ± 15
	7	21.8	105.0	21.5	104.2	22.5	105.0	255 ± 5
	28	21.1	105.2	21.0	105.5	21.8	105.5	400
5% Cement 1N NaOH	1	21.4	106.0	21.5	106.2	23.5	104.3	190 ± 10
	7	22.0	105.2	21.8	105.3	23.3	105.0	280 ± 10
	28	21.3	105.5	21.2	105.7	22.2	105.5	425 ± 5
6 1/2% Cement 0.5N NaOH	1	22.6	104.1	22.6	104.3	24.7	103.3	175 ± 25
	7	22.5	104.3	22.6	104.4	23.1	104.3	290 ± 30
	28	21.0	105.6	21.6	105.7	22.2	105.5	435 ± 5
6 1/2% Cement 1N NaOH	1	22.2	104.8	21.8	104.9	22.0	104.1	245 ± 15
	7	21.8	105.4	21.4	105.4	22.2	105.4	435 ± 5
	28	21.5	105.0	21.7	105.2	22.7	105.0	500 ± 10
8% Cement 0.5N NaOH	1	21.4	105.3	20.8	105.9	22.5	105.0	260 ± 20
	7	21.8	105.0	21.6	105.1	22.8	105.0	330 ± 10
	28	20.4	105.7	20.7	106.4	21.5	106.0	530 ± 10
8% Cement 1N NaOH	1	21.6	105.4	20.7	105.6	23.3	104.0	175 ± 15
	7	20.9	106.3	20.8	106.8	21.6	106.7	445 ± 5
	28	20.5	160.0	21.0	106.1	21.7	160.0	540 ± 10
10% Cement 0.5N NaOH	1	20.8	106.3	20.4	106.8	22.1	106.0	295 ± 25
	7	20.1	106.7	20.1	106.8	21.4	106.5	495 ± 5
	28	20.4	106.7	20.6	107.5	21.5	106.4	695 ± 35
10% Cement 1N NaOH	1	22.9	103.8	22.5	104.2	23.8	103.7	385 ± 15
	7	23.0	104.0	22.9	104.1	23.5	104.0	495 ± 25
	28	22.9	103.9	23.3	103.9	23.8	103.5	660 ± 5

Table III-3

EFFECTS OF VARYING WATER CONTENT AT A CONSTANT COMPACTIVE EFFORT OF 1000 psi FOR VBC, 5% CEMENT, AND 1N NaOH

Days of Cure	% Volatiles at Molding	Dry Density at Molding pcf	Dry Density at Testing pcf	Wet Comp. Strength psi	% Water Uptake upon Immersion	Degree Saturation at Molding
1	19.8	107.4	100.3	50	6.0	93.6
1	20.2	107.1	101.0	50	4.2	94.4
1	20.1	106.8	103.1	85	4.0	93.3
1	22.0	104.9	103.6	190	1.9	97.2
1	23.5	103.1	102.4	200	1.8	99.4
1	26.7	98.0	97.1	190	1.3	100
7	19.5	106.8	105.3	210	2.7	90.3
7	20.0	106.8	105.8	285	1.6	92.8
7	22.0	105.2	105.0	315	1.3	98.2
7	22.2	104.1	104.2	350	0.9	96.1
7	27.2	97.6	98.2	250	0.4	100

Note: A soil-cement density of 2.72 was used for degree-of-saturation calculations. A density of 2.76 would give more reasonable values for high water content. i.e. less than 100%.

Table III-4

EFFECTS OF VARYING COMPACTIVE EFFORT AT CONSTANT WATER CONTENT OF 22% FOR VBC, 5% CEMENT, AND 1N NaOH

Days of Cure	Compactive Effort in psi	Density at Molding pcf	Density at Testing pcf	Wet Comp. Strength psi	% Water Uptake upon Immersion	Degree Saturation at Molding
1	700	--	--	55 ± 5	--	--
1	850	103.0	101.6	110 ± 10	3.2	93.2
1	1000	104.9	104.9	190 ± 10	1.9	97.2
1	1150	104.8	103.7	195 ± 5	1.9	97.2
1	1250	104.3	103.0	175 ± 5	1.9	96.1
7	700	96.6	96.4	130 ± 20	3.0	79.3
7	850	103.0	102.0	160 ± 10	3.0	92.8
7	1000	104.9	104.8	280 ± 30	1.3	97.2
7	1150	105.3	105.3	315 ± 5	0.9	98.2
7	1250	105.2	105.2	310 ± 10	0.8	98.2

Note: A soil-cement density of 2.72 was used for degree of saturation calculations.

FIGURE III-1 % CEMENT vs WET COMPRESSIVE STRENGTH FOR VBC

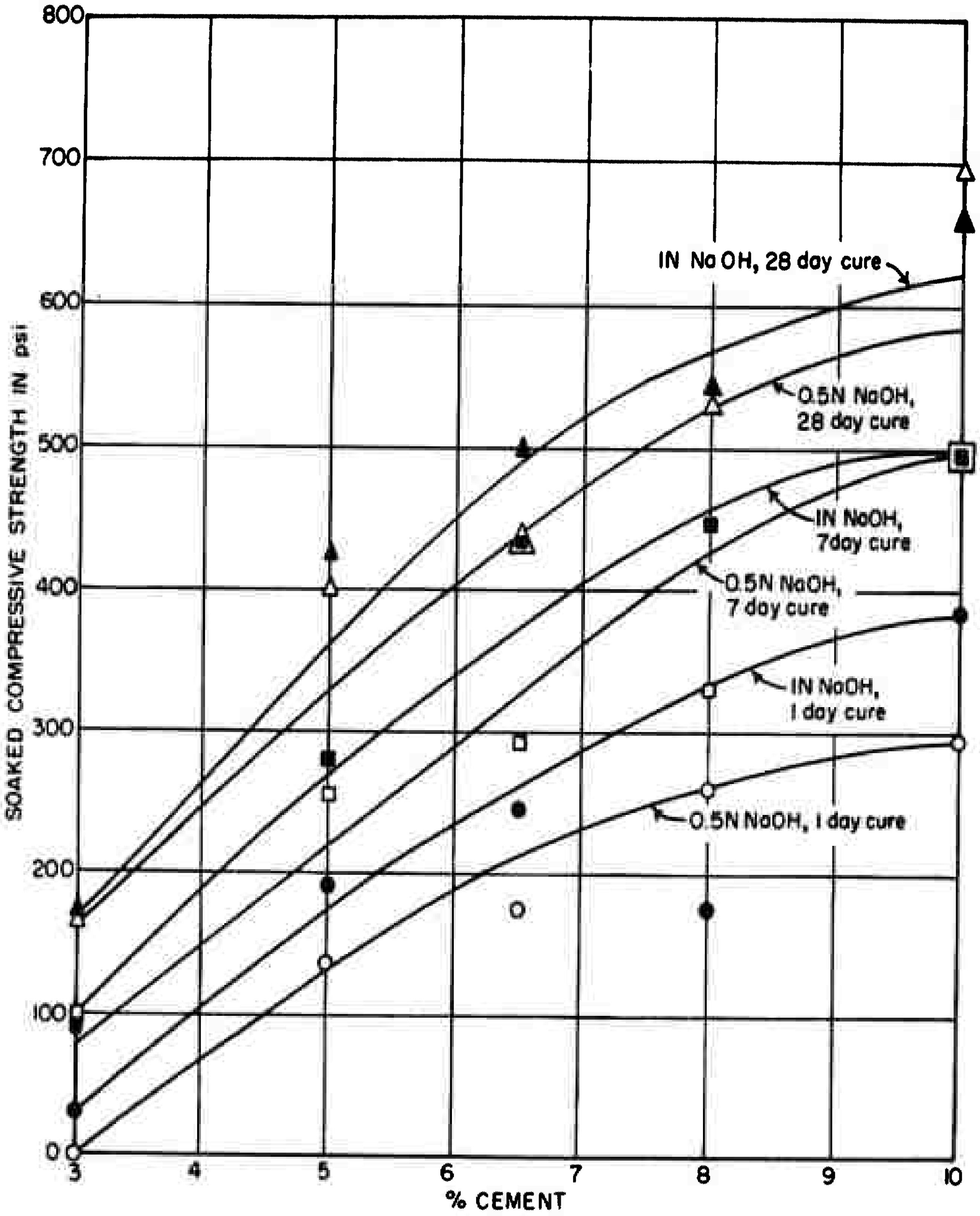


FIGURE III-2 CONSTANT COMPACTIVE EFFORT (1000 psi) FOR VBC, 5% CEMENT, IN NaOH (7 DAYS OF CURE)

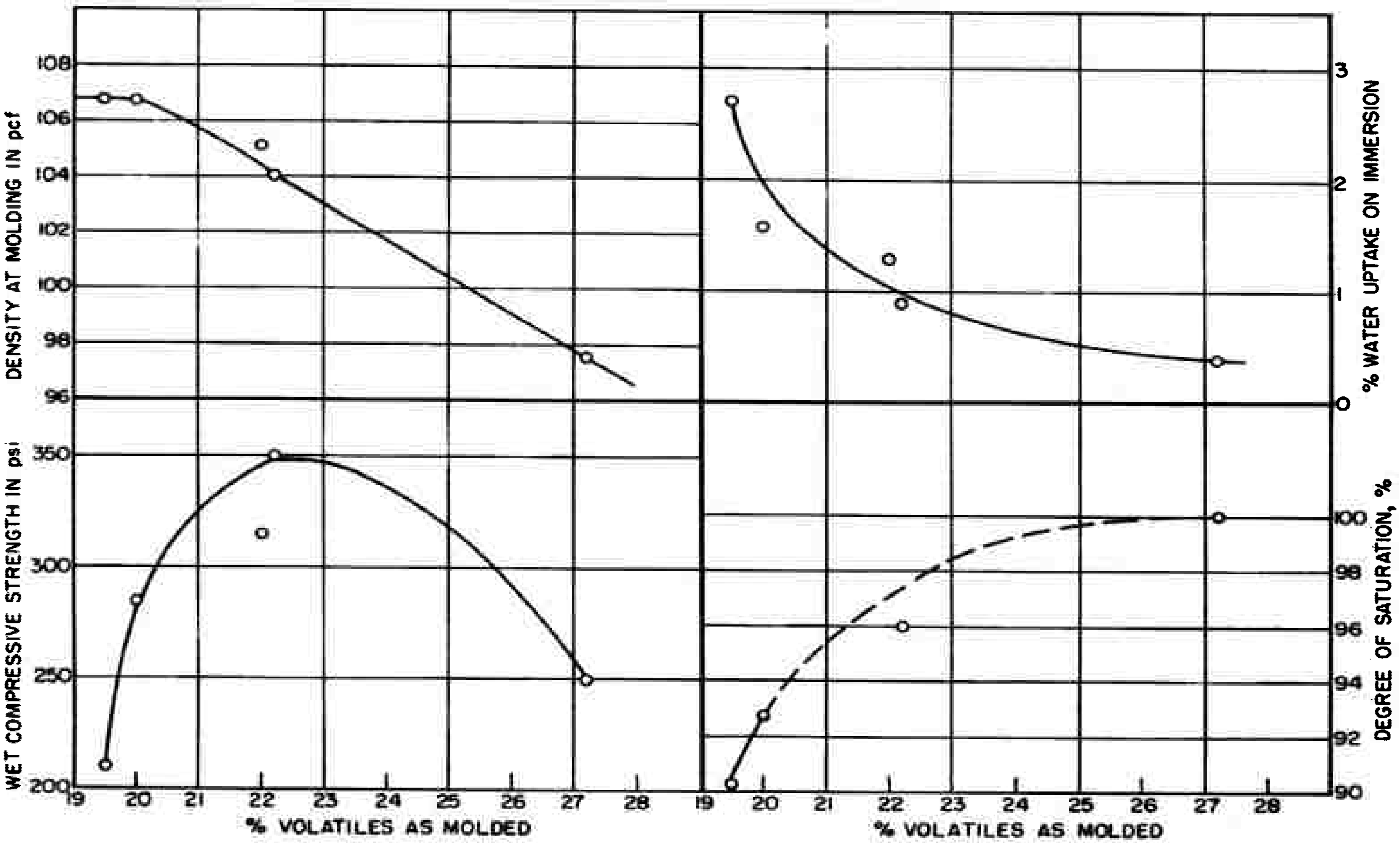
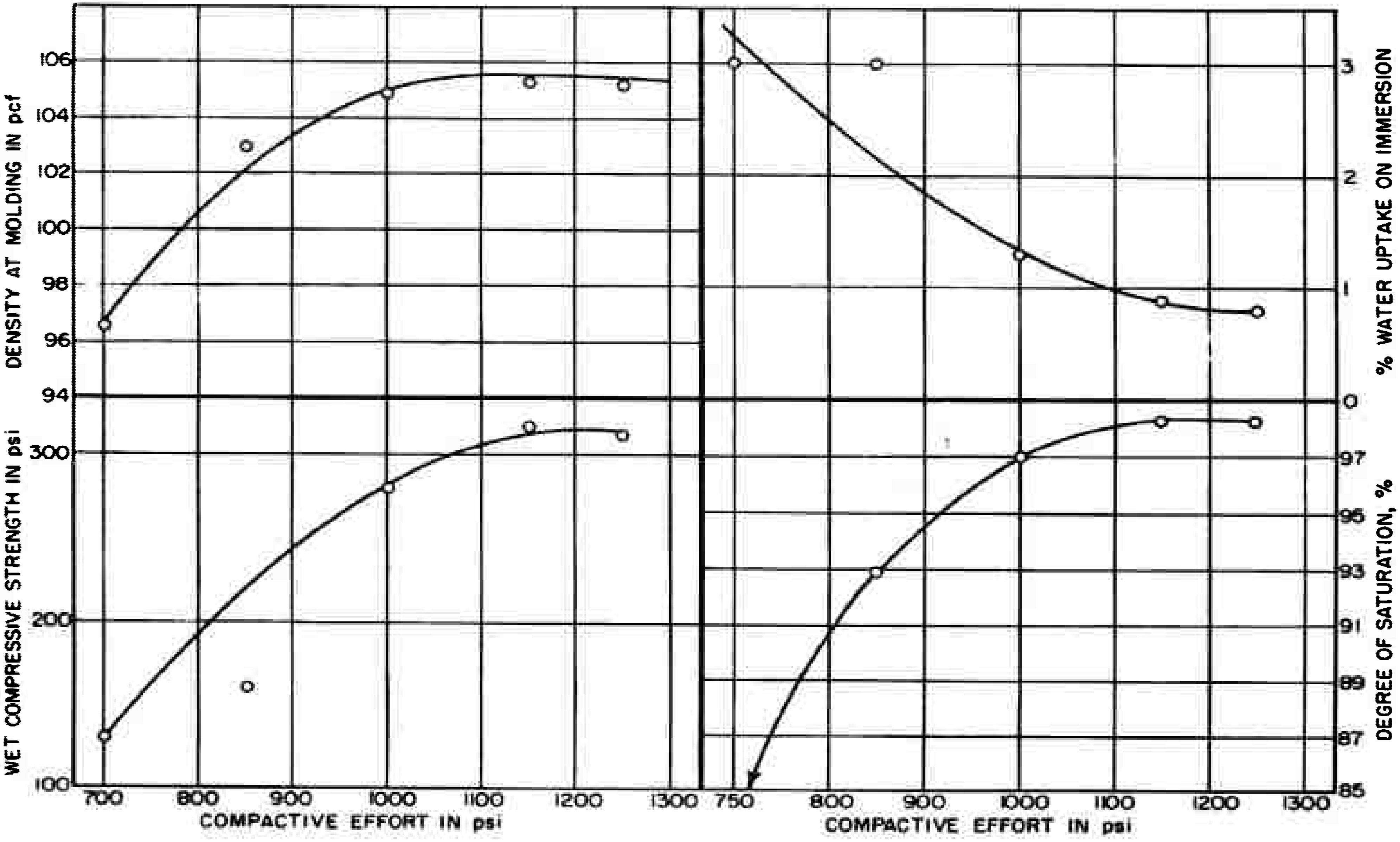


FIGURE III-3 CONSTANT WATER CONTENT 22% FOR VBC, 5% CEMENT, IN NaOH (7 DAYS OF CURE)



IV. SOIL STABILIZATION WITH LIME

A. INTRODUCTION

Much of the work done in the M.I.T. Soil Stabilization Laboratory has been directed toward the use of Portland cement, asphalt, phosphoric acid and various other chemicals as additives. During 1959-60 the stabilization of Massachusetts Clayey Silt (M-21) and Vicksburg Buckshot Clay (VBC) with clacium hydroxide, $Ca(OH)_2$, and calcium oxide, CaO, was investigated.

Soil-lime stabilization is thought to proceed by a mechanism similar in some respects to that of soil-cement. The high pH caused by the lime solubilizes part of the alumino-silicates in the soil, which in turn react with the calcium ions giving a cementitious calcium silicate gel. This is in contrast with cement stabilization, where the silicates are provided in part from the cement and in part from the soil itself.

Soil-lime stabilization is thus attractive both because lime (slaked and quicklime) is relatively inexpensive and because the total material requirements may be reduced below those required with cement. Soils were stabilized using calcium hydroxide (slaked lime) and calcium oxide (both reagent and commercial grades) with and without various inorganic salts, principally magnesium sulfate ($MgSO_4 \cdot 7\,H_2O$).

B. EXPERIMENTAL

1. Materials

The soils used in this investigation were Massachusetts Clayey Silt (M-21) and Vicksburg Buckshot Clay (VBC). The properties of these soils are given in Table II-1.

The following reagent grade chemicals were used: Calcium oxide (CaO), sodium hydroxide (NaOH), slaked lime ($Ca(OH)_2$), magnesium sulfate ($MgSO_4 \cdot 7\,H_2O$), potassium sulfate (K_2SO_4), and magnesium chloride ($MgCl_2$). A commercial grade CaO (quicklime) was also used. Rosin amine D acetate was a 70% solution in water supplied by the Hercules Powder Company.

2. Procedure

The testing procedures were the same as those used for soil-cement. The preparation of samples was as follows:

The soils were equilibrated for 24 hours with approximately half of the molding water content (8% in the case of M-21 and 12% for VBC). Mechanical mixing of the soils with the calcium oxide and calcium hydroxide, additives and the remaining water was accomplished in a finger prong mixer for about 10 minutes. The lime when used alone was mixed-in first, in powder form, and the water added subsequently. When used in conjunction with an additive, the additive powder was first mixed with the lime and the mixture then added to the soil in the mixer, followed by the required water. After mixing, samples were prepared by two-end static compaction in a Harvard Miniature mold. Compaction pressure was always approximately 1000 psi. Samples were cured at 100% relative humidity for various periods of time (1, 7, or 28 days) then immersed in water for 1 day, and finally tested in unconfined compression. The tested samples were dried at 110°C for at least 24 hours and weighed to determine water content. Samples were also weighed and measured after molding, curing and immersion.

C. RESULTS AND DISCUSSION OF RESULTS

1. Comparison of Calcium Oxide With Calcium Hydroxide

The results obtained with VBC using calcium hydroxide and calcium oxide are listed in Table IV-1 and those with M-21, in Table IV-2.

It is seen that CaO is more effective in VBC than $Ca(OH)_2$ at equivalent Ca concentrations (5% by weight CaO and 6.6% by weight $Ca(OH)_2$). Wet strengths of 215 psi and 50 psi respectively were obtained after 7 days cure. Also, an aged partially hydrated form of calcium oxide gave strengths higher than $Ca(OH)_2$ alone but lower than fresh, dry CaO.

An explanation of these results may follow from a consideration of these reactions:

(1) $CaO + H_2O \longrightarrow Ca^{++} + 2(OH^-)$

(2) $Ca^{++} + 2(OH^-) \longrightarrow \underline{Ca(OH)_2}$

(3) $2(OH^-) + SiO_2 \longrightarrow H_2SiO_4^=$

(4) $H_2SiO_4^= + Ca^{++} \longrightarrow \underline{CaH_2SiO_4}$ (gel)

Since reaction (1) is normally faster than reaction (2), when CaO is used there will be a high steady state concentration of OH^- ions, higher than that provided by the solubility product of $Ca(OH)_2$. Since at higher pH's larger amounts of silicates are solubilized, this explains why CaO is more effective in VBC than $Ca(OH)_2$.

From the results, it appears also that $Ca(OH)_2$ gives lower dry soil densities than CaO. It is expected therefore, that at higher densities achieved with CaO, greater strengths would be obtained. Results with soil-cement stabilization (see part III)show that an increase in density from 98 pcf to 102 pcf produces approximately a 40% increase in strength. Even if this correction were applied to soil-lime, the strengths obtained with $Ca(OH)_2$ would still be significantly lower than with CaO.

2. Effect of Inorganic Salts

It was suggested* that magnesium sulfate be used to improve the results obtained with lime. This was done and resulted in a significant improvement in strength both with CaO (wet strengths increased from 215 psi to 395 psi after 7 days cure) and $Ca(OH)_2$ (wet strengths increased from 50 psi to 135 psi at the same cures).

In soil M-21 (Table IV-2) stabilization with CaO was impossible without the use of magnesium sulfate. Samples prepared with 5% CaO expand and crack spontaneously upon humid cure alone.

The improvement in strength caused by the addition of magnesium sulfate both with CaO and $Ca(OH)_2$ may be due to the following reasons:

a.) Addition of $MgSO_4$ results in the formation of a $Mg(OH)_2$ protective gel on the CaO particles thus slowing the rate of hydration (reaction (1)). Under normal conditions (in the absence of magnesium salts) reaction (3) is probably slower than reaction (2), so that as reaction (1) proceeds, the ions produced crystallize as $Ca(OH)_2$ in addition to reacting by reactions (3) and (4). If $Ca(OH)_2$ is formed, then the advantages of using CaO is partially lost. If reaction (1) is slowed until it is no faster than reactions (3) and (4), then precipitation of $Ca(OH)_2$ is not possible.

b.) Formation of a Ca-Mg-Silicate gel. Replacement of Ca by Mg in the gel may improve its strength. This may be due to the greater hydration of the Mg ion, thereby forming a more expanded, less strained gel.

The dual effect of $MgSO_4$ is observed by comparing the results obtained with soils M-21 and VBC. In soil M-21 (a) seems to be the predominant effect of $MgSO_4$. Stabilization with CaO alone was impossible, but possible with $Ca(OH)_2$ alone. However, when $MgSO_4$ is incorporated, the strengths obtained with CaO and $Ca(OH)_2$ are comparable (wet strengths of 100 psi after one day cure were obtained for both, and 125 psi and 135 psi after 7 days cure respectively). In soil VBC, (b) seems to be the predominant effect of $MgSO_4$. The retardation of the hydration of CaO may not be needed as much as in the case of M-21, because the higher reactivity of the silicates in VBC itself produces a silicate protective layer which slows hydration. Thus in VBC the improvement in strength

---

* Based on preliminary results reported by G. R. Kozan, W.E.S., Vicksburg, Mississippi.

caused by $MgSO_4$ with both CaO and $Ca(OH)_2$ may be attributed to the formation of a Ca-Mg-Silicate gel.

Two other inorganic salts were used with VBC; K_2SO_4, used in conjunction with CaO and $Ca(OH)_2$, and $MgCl_2$, used only with CaO. The results obtained with these salts (see part a, Table IV-1) show that $MgCl_2$ gives no improvement in strength, the results obtained being comparable to those of CaO alone. Now, by the metathetical reaction

$$\underline{Ca(OH)_2} + Mg^{++} + 2(Cl^-) \longrightarrow Ca^{++} + 2(Cl^-) + \underline{Mg(OH)_2}$$

which is shifted to the right by the very low solubility of $Mg(OH)_2$, the Ca to Mg ionic concentration ratio is very high. This results in the formation of a silicate gel with a correspodingly high Ca to Mg ratio. As mentioned above, this will impair the strength of the soil.

K_2SO_4, on the other hand, caused a considerable improvement in strength in VBC both with CaO (from 50 psi to 160 psi for the same cures). The beneficial effect of K_2SO_4 may be due to the formation of KOH (causticization) caused by the precipitation of $CaSO_4$. The resulting free alkali can solubilize more soil silicates, and thereby produce better stabilization.

3. Relative Effectiveness of Commercial and Reagent Grade CaO

The effectiveness of commercial grade CaO was investigated. This grade of CaO is considerably coarser than the reagent grade and hence of lower reactivity. Consequently soil stabilization with it was expected to be less effective. The results obtained, however, with VBC showed little difference between the reagent and the commercial grade. On the other hand, the densities with the reagent grade were somewhat lower than with the commercial grade. It is probable that at equal densities, the commercial grade would give lower strengths.

The effectiveness of the commercial grade CaO (quicklime) would be expected to be improved by reducing its particle size. In fact, some samples made using ground quicklime (passing 40 mesh) which have densities comparable to those with commercial grade and higher than those with reagent grade, gave better strengths than the unground material. (See part b of Table IV-1.)

Comparison of the results obtained with reagent and commercial grade CaO together with magnesium sulfate show even more clearly the beneficial effect of grinding the commercial CaO. Wet strength after seven days cure is only 150 psi with the coarse quicklime but rises to 360 psi when it is ground, compared to 395 psi with the reagent grade CaO.

4. Effect of Calcium Oxide Concentration

Section c of Table IV-1 gives the results obtained with VBC using 2, 3, 4, 5, and 7% CaO with and without $MgSO_4$. The amount of $MgSO_4$ was always one fourth of the amount of CaO. The results obtained using 2% CaO were non-reproducible, probably because at this level the amount of CaO is insufficient to produce a uniform gel throughout the soil. The effect is more pronounced when $MgSO_4$ is incorporated, because the latter consumes part of the CaO leaving an even smaller amount of CaO for gel formation.

A plot (fig. IV-1 and IV-2) of strength of treated VBC versus percent CaO shows an optimum concentration of approximately 5% CaO. In all cases, except one, the curves are fairly flat for a range between 4 to 6% CaO.

The plot also shows that beyond the 5% CaO maximum, the strengths start to fall off. The reason for this may be the following: the soil can provide only a limited amount of reactive silica. If the Ca added is in excess of the amount which can react with the readily available soil silica, the resulting silicate gel will contain a high calcium-to-silica ratio, and may therefore be less satisfactory than the normal low-calcium gel as a cementitious binder.

Approximately 2.5% CaO alone is necessary to give a wet strength of 150 psi after 7 days humid cure. The maximum wet strength obtained with 5% CaO alone was 260 psi after 28 days humid cure. In the case of CaO and $MgSO_4$ (4 to 1) approximately 3% CaO is needed to give 150 psi and 4% to give 300 psi wet strengths after 7 days cure.

D. CONCLUSIONS

1.) Calcium oxide is more effective than calcium hydroxide in the stabilization of VBC. Seven-day-cure wet strengths are 215 psi and 50 psi respectively at equivalent Ca concentrations (5% CaO and 6.6% $Ca(OH)_2$).

2.) If commercial grade CaO is ground, it is as effective as reagent grade oxide.

3.) With both M-21 and VBC, use of magnesium sulfate in conjunction with lime produces a substantial improvement in strength.

4.) Calcium oxide by itself will not stabilize M-21 but if used in conjunction with magnesium sulfate (5% CaO and 1.25% $MgSO_4 \cdot 7\ H_2$)

soaked strengths of 100 psi are obtained after one day humid cure.

5.) A study of the variation of strength of lime treated VBC with concentration of additives showed that:

a.) Below 2% CaO there is insufficient stabilization, probably because the resulting gel is discontinuous.

b.) A maximum strength is observed at around 5% CaO for the curing periods investigated (up to 7 days curing).

c.) Approximately 2.5% CaO alone, with VBC, is necessary to give a wet strength of 150 psi after 7 days humid cure. The maximum wet strength observed with CaO alone was 260 psi after 28 days humid cure using 5% CaO. In the case of CaO and $MgSO_4$ (4 to 1) approximately 3% CaO is needed to give 150 psi and 4% to give 300 psi wet strength after seven days cure.

E. RECOMMENDATIONS

1.) Further investigate the CaO-$MgSO_4$ stabilization mechanism;

2.) Investigate inorganic salts other than $MgSO_4$ as additives for soil-lime stabilization;

3.) Vary the ratio of CaO to $MgSO_4$ or other additive.

TABLE IV-1

STABILIZATION OF VBC WITH LIME

	Days Humid Cure	1			1			7			7			28		
	Days Immersion	0			1			0			1			1		
I. Comparison of Calcium Oxide and Calcium Hydroxide, and Effect of Additives		C.S.[1] psi	D.D.[2] pcf	Vols[3] %	C.S. psi	D.D. pcf	Vols %	C.S. psi	D.D. pcf	Vols %	C.S. psi	D.D. pcf	Vols %	C.S. psi	D.D. pcf	Vols %
5.0% $Ca(OH)_2$		275±15	102.0	21.8	150±5	100.8	24.2	-	-	-	175±5	100.7	23.7	215±35	101.2	23.6
5.0% $Ca(OH)_2$	1.0 % NaOH	-	-	-	165±5	100.0	24.3	-	-	-	220±5	100.0	24.0	330±50	100.3	23.4
6.6% $Ca(OH)_2$		160±10	97.8	20.0	80±10	98.4	24.2	170	99.8	20.6	50±10	97.5	24.6	-	-	-
6.6% $Ca(OH)_2$	1.25% $MgSO_4$[5]	225±5	97.3	24.7	95±5	97.7	24.8	250	98.2	23.2	135±15	97.6	25.3	-	-	-
6.6% $Ca(OH)_2$	1.25% K_2SO_4	240±5	98.3	22.8	85±5	95.9	26.0	290	97.0	22.7	160±10	96.6	24.5	-	-	-
5.0% CaO		330	102.0	22.7	195±5	100.2	24.7	-	-	-	215±5	100.8	24.0	265±5	101.8	23.5
5.0% CaO	1.25% $MgSO_4$	395±15	102.0	22.9	210	99.0	25.4	-	-	-	395±5	101.0	23.7	410±5	102.2	24.2
5.0% CaO	1.25% K_2SO_4	395±5	103.1	22.1	155±5	99.1	26.5	-	-	-	285±5	101.1	25.3	340±10	102.8	23.2
5.0% CaO	1.25% $MgCl_2$	280	102.1	24.2	170	101.6	25.5	-	-	-	225±5	101.9	24.6	-	-	-
4.0% CaO	1.0 % $MgSO_4$[4]	300	99.0	24.8	170±5	97.5	25.6	310±20	103.2	23.2	375±5	98.9	25.4	365±5	98.5	25.5
II. Comparison of Reagent and Commercial Grade Calcium Oxide																
5.0% (reagent)		330	102.0	22.7	195±5	100.2	24.7	-	-	-	215±5	100.8	24.0	265±5	101.8	23.5
5.0% (aged)		290	103.5	21.6	170	103.0	23.5	-	-	-	185±5	103.0	23.2	245±5	103.0	23.3
5.0% (comm.)		235±25	102.2	22.6	190	103.0	23.5	270	102.5	22.4	210±25	102.4	23.2	240±10	103.0	22.1
5.0% (comm. ground)		335±15	103.9	21.6	215±5	103.6	22.8	-	-	-	255±5	104.8	21.2	320	104.8	21.6
5.0% (reagent)	1.25% $MgSO_4$	395±15	102.0	22.9	210	99.0	25.4	-	-	-	395±5	101.0	23.7	410±5	102.2	24.2
5.0% (comm.)	1.25% $MgSO_4$	270±5	102.7	22.2	70±5	98.4	26.8	-	-	-	150±10	102.5	23.5	170	103.5	23.1
5.0% (comm. ground)	1.25% $MgSO_4$	395±5	103.5	21.8	185±5	99.5	25.6	-	-	-	360±10	104.1	21.2	290	104.0	21.9
III. Effect of Calcium Oxide Concentration																
2.0% CaO		285±5	104.0	22.5	160	100.9	24.0	-	-	-	Disintegrated			Disintegrated		
2.0% CaO		295±5	102.4	23.9	130±20	101.2	25.6	-	-	-	185±15	101.5	24.7	Disintegrated		
2.0% CaO		235±5	105.1	22.6	70	102.0	25.5	-	-	-	150±20	104.5	24.8	180	103.9	23.9
3.0% CaO		270±10	103.1	23.2	155±5	103.5	23.3	310±20	104.6	22.1	205±5	103.7	22.7	-	-	-
5.0% CaO		330	102.0	22.7	195±5	101.2	24.7	-	-	-	215±5	101.8	24.0	260±5	101.8	23.5
7.0% CaO		250±10	101.6	23.4	130±10	102.2	24.5	325±15	103.1	21.6	190±10	102.5	23.6	-	-	-
2.0% CaO	0.50% $MgSO_4$	255±5	103.5	23.0	30	99.0	27.2	-	-	-	45±5	99.5	27.2	80±5	102.0	25.5
3.0% CaO	0.75% $MgSO_4$	350	102.8	23.7	170±20	100.0	26.3	410±5	104.3	23.0	205±5	102.1	25.3	-	-	-
4.0% CaO	1.00% $MgSO_4$	345±5	102.5	23.0	190±10	99.5	25.2	-	-	-	215±25	100.8	24.2	420	101.5	23.7
5.0% CaO	1.25% $MgSO_4$	395±15	102.0	22.9	210	99.0	25.4	-	-	-	395±5	101.0	23.7	410±5	102.2	24.2
7.0% CaO	1.75% $MgSO_4$	375±5	101.3	24.1	210	98.9	26.2	565±5	102.8	21.7	350±10	102.5	23.9	-	-	-

All percentages of additives are based on dry soil.

[1] C.S. is actual compressive strength in psi.

[2] D.D. is dry density at test, i.e., pounds of dry solids per cubic foot

[3] % Volatiles at test, i.e., weight lost upon drying as a percentage of dry solids weight.

[4] 1% Rosin Amine D Acetate also added.

[5] Hydrated form of magnesium sulfate always used, i.e., $MgSO_4 \cdot 7H_2O$

TABLE IV-2

STABILIZATION OF M-21 WITH LIME

Days Humid Cure		1			1			7			7			28		
Days Immersion		0			1			0			1			1		
		C.S. psi	D.D. pcf	Vols %	C.S. psi	D.D. pcf	Vols %	C.S. psi	D.D. pcf	Vols %	C.S. psi	D.D. pcf	Vols %	C.S. psi	D.D. pcf	Vols %
5.0% CaO																
4.0% CaO	1.0 % $MgSO_4$	240	118.3	7.7	100	117.9	14.6	-	-	-	175±5	118.0	14.9	155±5	118.6	14.0
5.3% $Ca(OH)_2$		110±5	115.9	11.6	30±5	114.4	15.7	200	115.8	11.6	[illegible]±5	114.7	15.8	-	-	-
5.3% $Ca(OH)_2$	1.0 % $MgSO_4$	210±10	114.8	12.3	100±10	115.0	12.8	350	115.0	11.1	135±5	115.2	12.9	-	-	-
5.0% Cement*		-	-	-	225	130.0	10.4	-	-	-	328	130	10.4	536	130.0	10.0
		-	-	-	-	-	-	-	-	-	300±20	119	12.8	435±25	119	12.8

* Caro, G., Personal communication, Department of Civil Engineering, M.I.T., July 1960.

FIGURE IV-1

EFFECT OF MAGNESIUM SULFATE AND CONCENTRATION OF CALCIUM OXIDE ON THE STRENGTH OF VBC (ONE-DAY CURE)

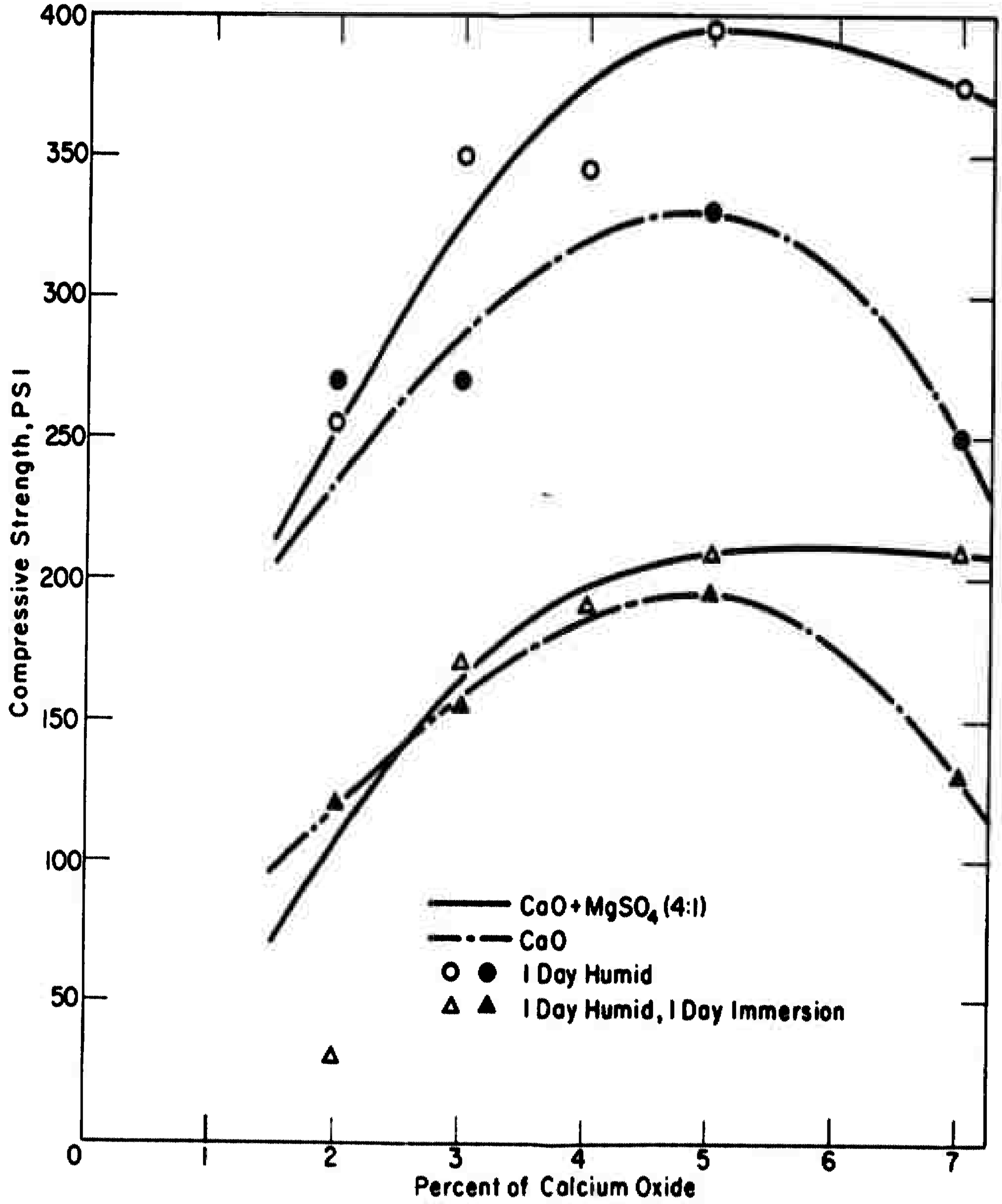
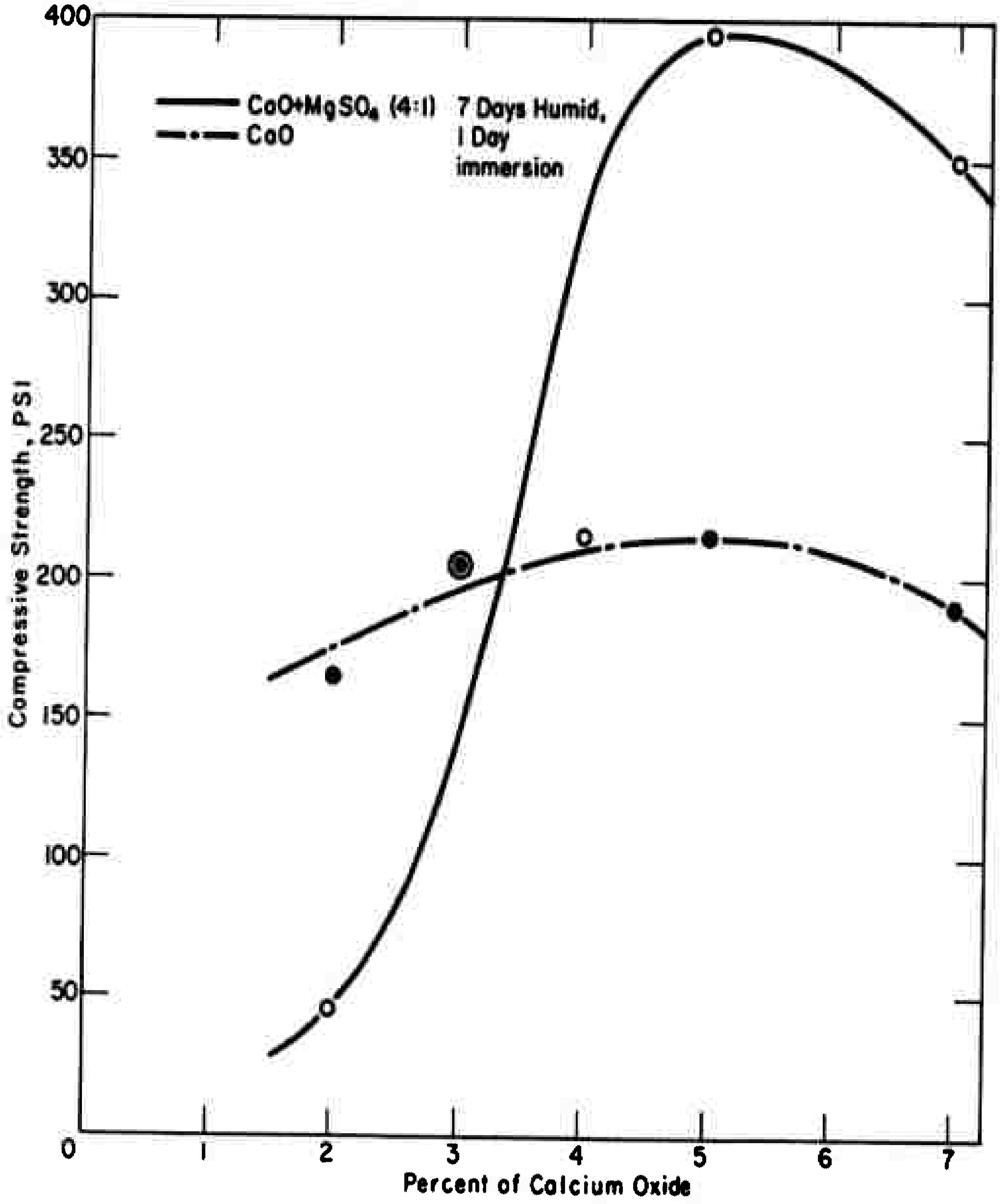


FIGURE IV-2

EFFECT OF MAGNESIUM SULFATE AND CONCENTRATION OF CALCIUM OXIDE ON THE STRENGTH OF VBC (SEVEN-DAY CURES)



V. SOIL STABILIZATION WITH SODIUM SILICATES

A. Introduction

Research during the period September 1958-June 1959 established that sodium silicate with basic magnesium carbonate effectively stabilizes Massachusetts Clayey Silt. In some cases, compressive strengths of more than 600 psi after 24 hour humid cure and 24 hour immersion in water were obtained. The effect of the content of silica, sodium, and magnesium was studied, and a mechanism of stabilization was proposed. Several recommendations were made for further research.

Three of these recommendations were as follows:

1. Further work should be directed toward determining the effectiveness of the sodium silicates as stabilizers for soils other than Massachusetts Clayey Silt.
2. Precipitants other than $MgCO_3$ should be investigated.
3. The effect of SiO_2 content on strength of soil stabilized with silicates should be further investigated.

Most of the work done during the period September 1959-June 1960 is concerned with these three recommendations.

New Hampshire Silt, Vicksburg Loess, and Vicksburg Buckshot Clay were treated with basic magnesium carbonate and sodium silicate in varying ratios and amounts. The effects of sodium, magnesium, and silica contents were studied in these soils.

Calcium hydroxide, reactive magnesium oxide, aluminum hydroxide, and zinc carbonate were also studied as precipitating agents with sodium silicate. Each was first screened in a test in the absence of soil; then those which appeared promising were tested in Massachusetts Clayey Silt.

The study of the effect of silica content on sodium silicate-magnesium carbonate stabilized Massachusetts Clayey Silt, begun during the previous research period, was completed.

In addition, after the optimum ratios of magnesium and sodium to silica were determined, the effect of total additive content at these ratios on immersed strengths of Massachusetts Clayey Silt was studied.

B. Procedure

1. Materials

The soils used in this work were Massachusetts Clayey Silt (M-21), New Hampshire Silt (NHS), Vicksburg Loess (VL), and Vicksburg Buckshot Clay (VBC).

The sodium silicates were aqueous solutions provided by the Diamond Alkali Company, the compositions of which are listed in the table below.

Grade	Weight per cent solids	Weight ratio $Na_2O:SiO_2$
58	49.8	1:1.58
52	46.6	1:2.40
40	37.5	1:3.22

The magnesium oxide was of three grades. The descriptions of each are listed in the table below.

Number	Description
210	C-874, Westvaco's 2667 MgO
211	C-878, Dow MgO (Texas Div.) Ball Milled and Air Classified
213	L-927, Dow MgO (Ludington Div.) Fast Setting

Rosin amine D acetate, a 70% solution in water supplied by the Hercules Powder Company, and Arquad 16, a quaternary ammonium salt supplied by Armour Industrial Chemical Company, were used.

Reagent grade chemicals used were basic magnesium carbonate ($4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$), calcium hydroxide, sodium hydroxide, potassium hydroxide, zinc carbonate, and aluminum hydroxide.

2. Preparation and Testing of Soil Samples

The same mixing procedure was maintained for most samples. The soil and any solid additives were first dry-mixed, then the silicate solution and sufficient water to give the desired water content were added. Mixing was carried out in a finger-prong mixer and was continued until the composition was visibly homogeneous (about four or five minutes). In some samples, one or more additives and some water was mixed with the soil by hand and allowed to sit for twenty-four hours, after which the remaining additives and water were mixed in the finger-prong mixer. After mixing the soil was compacted by two-end static compaction into cylindrical samples, in a Harvard miniature mold, under a pressure of 900 to 1000 psi.

Curing periods and conditions included: (a) 1 day at 100% relative humidity; (b) 1 day at 100% relative humidity followed by 1 day immersion in water; (c) one week at 100% relative humidity followed by 1 day immersion in water; (d) 2 weeks at 100% relative humidity followed by 1 day immersion in water; and (e) 4 weeks at 100% relative humidity followed by 1 day immersion in water. After curing, samples were tested to failure in unconfined compression. After testing, samples were dried at 105°C for at least 24 hours to determine the water content. The weight and dimensions of each sample were taken when compacted and just before testing. From the data recorded, density (lb dry solids/cu. ft.), and per cent volatiles (based on dry solids) at test were calculated.

3. Gel Formation in the Absence of Soil

In preparing gels the dry salt was added to a diluted sodium silicate solution. A basis of one part of water-free sodium silicate to four parts of water was maintained. One equivalent of precipitating salt per equivalent of sodium was added. The mixtures were stirred for a short time. The gels were evaluated qualitatively on the basis of visual and tangible characteristics.

C. Results and Discussion of Results

1. Proposed Mechanism of Soil Stabilization with Sodium Silicates

In previous work on the stabilization of M-21 with sodium silicates, it was proposed that stabilization resulted from the formation of a strong, cementitious gel. This gel is made up of chains of silica cross-linked by the cation of the precipitating salt. In most of this work basic magnesium carbonate has been used as the precipitating salt, hence the silica chains are cross-linked by magnesium. The sodium silicate initially is made up of long silica chains, to which sodium is bonded. The magnesium replaces the sodium on these chains, and, since it is divalent, can bond to two separate chains, forming a rigid gel. In addition the Mg can attack the silica chains directly, but this process is probably slower.

From this proposed mechanism, several conclusions were deduced. With any given silicate (i.e., constant soda-silica ratio), providing the other components of the system are not present in critical amounts, the greater the concentration of silica (from the silicate) added to the soil, the higher the resultant strength and the more effective the stabilization. For a given amount of silica, however, an optimum soda content exists, below which gel cross-linking sites are too infrequent, and the apparently diffusion-controlled gelation is too slow to stabilize the soil within a reasonable time. Above this optimum, the pH of the pore fluid is very high and the precipitating ion is removed in the form of a basic complex. (In M-21, since it has a low optimum water content, crystallization of Na_2CO_3 is also a significant factor.) In addition, an optimum magnesium content also exists. Below this optimum insufficient gelation results, while above the optimum either the gelation rate may be too rapid for best results, or excessive cross-linking may occur. A proper combination of all the above factors should yield the optimum stabilizing system.

2. Stabilization of New Hampshire Silt with Sodium Silicates and Basic Magnesium Carbonate

In an attempt to determine the optimum sodium silicate system for stabilizing NHS, the optimum formulation for stabilization of M-21 was tried in NHS. When it was found that this formulation successfully stabilized the silt, each of the four variables, silica content, magnesium content, sodium content, and water content, were varied independently. The effects of each additive are shown graphically in Figure V -1, where they are compared with the effects of each additive in M-21 and VL.

a. Effect of Silica Content (Constant Sodium and Magnesium Content

The first variable to be investigated was silica content. The results of this study are presented in Table V -1a. It is found that the silica content for best results varies with curing period.

At all cures, the low silica samples are inferior to those with the intermediate and high silica contents. This can be explained by the fact that the silica chains for the low silica samples are so short that the gel formed cannot compare in strength with either of the other two sets of samples.

After one day humid cure, the intermediate silica content gives the highest strength. After the first day, the magnesium added has attacked the silica chain at the points to which the sodium was originally bonded. Since the sodium and magnesium contents are constant, the high silica content samples have formed less compact gels and are therefore weaker.

As curing time increases,the strength of the samples with high silica content increases considerably more than the strength of the samples with intermediate or low silica contents (see Figure V -1-ii-A). This may be due to any of three causes:

1.) The rate of gelation is slower at the high silica content. There does not appear to be justification for such a difference in gelation rate since all of the other conditions (including pH which is determined by the amount of sodium) are similar. In any case, one would expect the rate of reaction to be increased by an increase in the concentration of one of the reactants (silica).

2.) There is present in the soil a certain amount of polyvalent metal cations in a rather unreactive form which belatedly react with the silica to form a more closely knit gel, thus increasing the strength of the sample. This effect is of greater advantage at the high silica content, since at lower silica levels the cross-linking may be excessive.

3.) Direct bonding between silica chains occurs. At high silica contents, this possibility is increased.

A consideration of the relation between wet strength and magnesium content seems to confirm the second hypothesis (see below).

b. Effect of Magnesium Content (Constant Sodium and Magnesium Contents)

After the effect of silica content had been determined, the effect of magnesium content was next investigated. Table V -1b shows these results.

Magnesium content showed the same effect on strength development at both silica contents studied. Increased magnesium gave higher strengths after one day humid cure and after one day humid cure and one day immersion. However, after seven day cure an optimum magnesium content was apparent.

After one day humid cure the compressive strength was found to increase with magnesium content over the range investigated. In the range of Mg contents investigated, the amount of magnesium is a measure of the amount of cross-linkage in the stabilizing gel after one day humid cure.

After one day humid cure and one day immersion, the same argument applies. At the low magnesium content (1.2% BMC, 0.5 equiv. Mg/equiv. Na) the cross-linkage is not sufficient to stabilize the soil to immersion in water, even though there is enough sodium present to permit rapid formation of a gel by the cations present in the soil. This seems to indicate that at low magnesium contents the rate at which the cations inherent in the silt can unite with the silica chains, even at the reactive sites, is low. At magnesium contents at which stabilization does result, higher magnesium content increases the amount of bonding between silica chains, resulting in a stronger gel.

After seven day humid cure and one day immersion, however, an optimum magnesium content at 1.8% BMC (0.75 equiv. Mg/equiv. Na) is observed. It will be noted here that the strength developed at the low magnesium content (0.5 equiv. Mg/equiv. Na) is 305 psi, which is high considering that similar samples disintegrated on immersion after only one day cure. This seems to confirm the presence of polyvalent inorganic cations in the soil, other than the magnesium added when the soil was compacted. This strength, however, is not quite as great as that of the next higher magnesium content, which is 335 psi. This discrepancy is due to a difference in the amount of cross-linkage between silica chains. The difference in cross-linkage can be explained in either of two ways. If equilibrium has essentially been reached after seven days, the explanation is the fact that the cation concentration in the low magnesium samples is lower than that in the intermediate magnesium samples. If equilibrium has not been reached, the higher magnesium content only serves to increase the rate of bond formation. If the latter is the case, longer cure periods should lessen the difference in strength observed after seven days. At the high magnesium content (1.0 equiv. Mg/equiv. Na) the strength is less than either of the other two samples. This is probably due to shrinkage of the gel formed, either due to excessive cross-linkage or to an overly rapid formation of the cross-linkages. Thus, apparently, the optimum amount of magnesium is larger at the shorter cure times: At the longer cures the slower reacting cations from the soil come into play and make up for the difference.

As a further test of the presence of additional gel-forming cations in NHS, samples without $MgCO_3$ should be tested. Such samples would be expected to disintegrate upon immersion after one day cure, but to show fairly good wet strengths after longer cure periods.

c. Effect of Sodium Content (Constant Silica and Magnesium Contents)

The results of this study are shown in Table V -1c. From this table it is seen that for one day humid cure and for one day humid cure followed by one day immersion, an optimum sodium content exists at about 2.14% Na_2O, but that after seven day cure-one day immersion, lower sodium content is slightly preferable.

In all cases, if the sodium content is too high poor strengths result. This can be charged to the higher pH in the pore fluid, which causes the precipitation of basic complexes with the magnesium and other cations, inhibiting stabilization, as has been explained above. Apparently, the maximum pH allowable is sharply defined. The change in sodium concentration investigated was only a little over twofold, which would change the pH by only 0.3. It is noteworthy also that the samples compacted at the high sodium content were quite plastic in comparison with other samples. (These samples showed a strain of 3% after one day humid cure, as compared to approximately 1% for normal samples. Immediately after compaction the plasticity was even more noticeable.) This high plasticity was previously noted at high sodium contents in M-21. The higher plasticity at high sodium contents may be due to the established dispersing effect of sodium. This effect also tends to increase the density. In fact, 1.59% Na_2O gave densities of about 102 lb. dry solid per cu. ft., while 3.24% Na_2O gave densities of about about 111 lb. dry solid per cu. ft. An alternative explanation for the plasticity may be the cation exchange between the sodium and hydrogen ions in the soil resulting in the combination of the hydrogen ions with hydroxyl ions in the basic pore fluid to form water.

The existence of an optimum soda content after the two short cure periods may be attributed to the availability of an excess of sites at which cross-linking can be rapidly accomplished, thereby increasing the rate of this cross-linking. After longer cure periods the cations have had time to form bonds with the silica whether or not the sodium is present.

The approximate optimum additive formulation for stabilization of New Hampshire Silt after seven day cure, found by investigating all three of the above-mentioned effects, is a soda to silica weight ratio of 1:3.22, and a ratio of equivalents of magnesium to sodium of 0.75. Using this formulation, strengths of 365 psi are obtained at a total additive content of 8.5% of dry soil weight. This formulation is only approximate as the effect of each additive was investigated by determining only three points. The optimum formulation can be more accurately ascertained by further investigation at ratios in the vicinity of the optimum given above.

d. Effect of Water Content

The optimum water content for stabilizing NHS with sodium silicate was not known when the effect of each additive was determined. Twenty-one per cent water was chosen from previous work with cement as the molding water content to be used.

However, after the optimum additive formulation was determined, it was decided that the effect of water content should be studied. This study was not completed, but partial results are given in Table V -1d. The table shows that nineteen per cent water at molding gives better strengths than twenty-one per cent after all cures used (365 psi as compared with 335 psi after 7 day cure, 1 day immersion).

3. Stabilization of Vicksburg Loess with Sodium Silicates and Basic Magnesium Carbonate

In an attempt to determine the optimum sodium silicate system for stabilizing Vicksburg Loess, the same procedure as previously described for determining the optimum formulation for NHS stabilization was followed. However, time did not permit completion of the study and only the effects of silica and magnesium were investigated. These effects are shown graphically in Figure V -1.

a. Effect of Silica Content (Constant Sodium and Magnesium Contents)

The results obtained from the study of the effect of silica content in VL are shown in Table V -2a. As for NHS, it can be seen that the best silica content depends on the cure time at which the samples are being evaluated. It is believed that the results obtained in this study are explainable in almost the same manner as those for the study of the effect of silica in NHS.

After one day humid cure the magnesium added has attacked the silica chains at their active sites (i.e. where sodium was initially bonded). The longer the silica chains, the less dense the cross-linkage that has resulted. However, in each case, the less dense cross-linkage of the higher silica content samples has been almost exactly counter-balanced by the longer silica chain lengths; hence the one day humid cure strengths are all approximately equal.

After one day humid cure and one day immersion the attack of the silica chains by other cations present in the soil is beginning. This "secondary attack" appears to be less rapid in VL than in NHS. This is illustrated by the fact that the strength developed by the high silica samples has not yet equalled that developed by the intermediate silica samples. However, the short chain lengths of the low silica samples has limited the strength that they can develop.

After a week of humid cure and one day immersion, the silica chains have been bonded together. At this time the absolute length of the silica chains makes itself felt, and the strength developed is seen to increase with increasing silica content.

b. Effect of Magnesium Content (Constant Sodium and Silica Contents)

Table V -2b shows the results of the investigation of the effect of magnesium content in Vicksburg Loess. This effect can be explained by the mechanism of silicate stabilization proposed above, together with the existence of the soil of other cations capable of forming a rigid gel with sodium silicate.

After one day humid cure the strengths shown increase with magnesium content up to a point, after which the strengths begin to level off. After one day only the active sites of the silica chains have been attacked, and only the magnesium equivalent to the amount of sodium can react.

After one day humid cure and one day immersion the magnesium along with other cations from the soil has begun to attack the silica chains. The rate of this attack is increased by increasing the magnesium added to the soil; hence, the strengths developed are found to increase with magnesium content.

After seven days cure and one day immersion an optimum magnesium content is found at 2.4% BMC (1.0 equiv. Mg/equiv. Na). Above this point, the rate of attack of the silica chains may have been too rapid and the gel has shrunk on aging, thereby becoming weaker. Below the optimum, the gel does not have as much cross-linking, either because of an insufficient supply of potential cross-linking cations, or because the cross-linking reaction has proceeded too slowly to develop the maximum strength at this time. If the latter is the case, longer cure periods might reveal a lower optimum magnesium content.

On the basis of the results obtained thus far, it appears that the optimum soda-silica ratio is 1:3.22, and the optimum magnesium to sodium equivalent ratio is 1.0; this optimum is shown from the wet strengths obtained after seven days humid cure. At a total additive content of 9.1% of dry soil weight, strengths of 235 psi are obtained.

It might also be expected that higher sodium contents would give better strengths after short cures, but that after seven days or more the sodium content would have almost no bearing on the strength developed. Of course, if the sodium content is too high, the maximum pH for strong gel formation would be exceeded and very low strengths should result.

4. Stabilization of Vicksburg Buckshot Clay with Sodium Silicates and Basic Magnesium Carbonate

In attempting to stabilize Vicksburg Buckshot Clay with sodium silicates, one formulation of soda, silica, and magnesium carbonate was tried. Even though this did not succeed in stabilizing VBC to immersion, the amounts of each additive, as well as the molding water content, were varied in the hope of finding a successful formulation.

a. Effect of Water Content

In an effort to determine the optimum water content of VBC stabilization with sodium silicates and basic magnesium carbonate, several samples were tested which contained the same amounts of additives, but varying amounts of water. The compressive strengths after 24 hour humid cure were then compared. This comparison, which is presented in Table V -3, indicated that a molding water content of about 23.5 per cent yields the best humid cure strengths.

At the same time each of the samples was subjected to 24 hour immersion following humid cures of 24 hours and one week. Although all of the samples disintegrated upon immersion, the rate of disintegration was observed to be significantly slower for the samples with higher water contents. This further confirmed the fact that the optimum water content for developing high humid cure strengths is not necessarily the same as that for developing good immersion strengths.

b. Effect of Silica Content

Table V -4 presents the results of a study of the effect of silica content on humid cure strength of VBC. It was found that for a given magnesium and sodium content, an optimum silica content existed. This seems to indicate that the humid cure strength developed in this soil is more dependent on the "cross-linkage density", that is, the number of cross-linking bonds between silica chains per unit length of chain, rather than on the absolute chain length. The sodium and magnesium to silica ratio must be high enough to form dense cross-linkage between silica chains. Hence, an increase in silica without a corresponding increase in sodium and magnesium reduces the strength developed. However, if the three components, sodium, magnesium, and silica, are increased in the same proportion, the resultant humid cure strength is greater, as is shown in Table V -4. The results of this table are presented graphically in Figure V -2. From this, it appears that the optimum weight ratio of SiO_2 to Na_2O should lie between 0.75 and 1.75.

c. Effect of Sodium Content

As shown in Table V -5a, a decrease in the sodium content of VBC at constant silica and magnesium contents results in poor humid cure strengths. This fortifies the conclusion that the magnesium cannot combine rapidly with silica except at points where sodium is present.

However, it is known that when calcium or magnesium montmorillonite, which is present in large amounts in VBC, undergoes cation exchange with sodium, it swells greatly when contacted with water. This swelling can be expected to cause breakage of the gel bonds; hence the samples disintegrate when immersed in water. If one observes the disintegration of a silicate-stabilized VBC sample during immersion, he will notice that it proceeds rather slowly, with an outer particle of the clay swelling, then breaking off and falling to the bottom of the container. In some of the weaker samples the entire sample will swell to 1 1/2 times its original diameter and height before disintegrating. If at any time during this process the immersed sample is cut in two, the center of the sample will be found to be dry, a clear indication that capillary uptake of water is not a cause of disintegration. This shows that the swelling of sodium montmorillonite may be an important cause of the disintegration of stabilized soils under water.

The above two facts present a dilemma: Small amounts of sodium (approximately less than 1% Na_2O) result in very little bond formation, and consequently poor stabilization; and sufficient amounts for stabilization result in a considerable amount of sodium montmorillonite which swells when wet, breaking any gel bonds which may have been formed.

One possible way of overcoming this situation was thought to be the use of a low sodium content silicate, with additional potassium hydroxide to provide cross-linking sites. Previous work had shown that addition of sodium hydroxide to a silicate which is low in sodium results in a solution with equal gel-forming abilities to the ready-made silicate solution with the desired soda content. Potassium hydroxide was expected to act in the same manner, and the potassium montmorillonite was expected to swell less than sodium montmorillonite. However, as is shown in Table V -5a, the humid cure strength obtained using KOH was no greater than if the potash had been absent. Apparently the KOH does not attack the silica chains rapidly enough to provide active cross-linking sites for the magnesium to attack. On immersion the samples disintegrated, but the swelling was noticeably less than in the other samples.

d. Effect of Magnesium Content

Table V -5b presents the effects of magnesium content on the stabilization of VBC. The data show that the optimum magnesium content for humid cure strength development in VBC is about 1.0. This is to be expected since, if the magnesium content is too high, the excess magnesium causes the reaction to proceed much too rapidly, and subsequent gel shrinkage significantly weakens the sample. In M-21 stabilization, good strengths were developed at magnesium-sodium equivalent ratios as low as 0.5; however, in VBC, a more difficultly stabilized soil, observable humid cure strength improvement does not develop until the ratio is much nearer unity. This may be due to the fact that cross-linking sites must be more frequent to realize this effect.

e. Effect of Various Waterproofing Agents

Due to the unusual disintegration observed, as described previously in the section on the effect of sodium content in VBC, and in view of the high humid cure strengths developed by some formulations (310 ± 10 psi, as compared to approximately 200 for VBC alone), it was thought that perhaps a stabilizing gel is being formed in the soil, but that the swelling of sodium montmorillonite when the samples are immersed causes the rupture of this gel. If this were true, all that would be necessary to successfully stabilize VBC with a silicate system would be to find a suitable waterproofing agent. Such a waterproofer should be cationic so that it would undergo cation exchange with the calcium and magnesium montmorillonite in the soil.

A number of these were tried in a vain attempt to produce a combination which would stabilize the clay. Results of each of these efforts are shown in the table below.

Waterproofer Added and Amount	24 Hour Humid Cure Strength
None	310
Rosinamine D-acetate (0.1%)	215
Arquad 16 (0.1%)	120

All samples disintegrated upon immersion.

It will be noted that not only was the VBC not stabilized to immersion, but also the humid cure strengths were reduced when these waterproofers were used. When the samples disintegrated under water it was obvious visually that swelling had not been halted.

A possible reason for both the failure to halt swelling of the soil and the lessening of humid cure strengths is that the cationic waterproofer exchanges with the sodium silicates rather than with the montmorillonite. This would inhibit the magnesium cross-linkage of the silica chains, as well as failing to prevent swelling.

There are still a number of waterproofing agents (ferrous and ferric salts, melamine, and others), which have not been tried, but the outlook for developing a sodium silicate system which will stabilize VBC is not very bright.

5. Investigation of Precipitating Agents Other than Basic Magnesium Carbonate

a. Gel Formation in the Absence of Soil

Screening tests in the absence of soil performed during the period September 1958-June 1959 had investigated the gel forming characteristics of calcium chloride, calcium dihydrogen phosphate, magnesium sulfate, calcium carbonate, calcium hydroxide, and calcium sulfate, besides basic magnesium carbonate. However, a number of other salts warranted investigation. A few of them were investigated during the period September 1959-June 1960.

Results obtained upon reacting zinc carbonate, aluminum hydroxide, and three types of reactive magnesium oxide with sodium silicates are given in Table V-6, where they are compared with identical experiments using BMC.

The only test, other than the one with BMC, which resulted in formation of a gel was that made with MgO No. 213 with the most siliceous silicate. Apparently MgO is very insoluble in the most basic silicate solution and cannot react. The gel formed with MgO was very dense, forming only at the very bottom of the beaker. A large volume of supernatant liquid was present.

These results indicated that the most reactive MgO (No. 213) might possibly be an alternate to $MgCO_3$ in silicate stabilization. The other substances investigated do not appear promising.

b. Stabilization of Massachusetts Clayey Silt with Magnesium Oxide Number 213 and Sodium Silicates

A comparison of the results obtained upon stabilization of M-21 with sodium silicates with magnesium oxide and basic magnesium carbonate as precipitating agent is presented in Table V-7.

The table shows that for cures up to seven days the samples compacted with MgO are considerably weaker, but that the strength developed by these samples continues to increase with time, until, after twenty-eight days, the strength of the MgO samples is equal to that of BMC samples with the same number of equivalents of magnesium. Thus the gel-forming abilities of the two magnesium salts are about equal, but the magnesium oxide is considerably slower to react. The reaction can be speeded up by increasing the amount of MgO added as is evidenced by the increase in strength with magnesium content for the MgO samples.

Upon testing the BMC samples at various cure periods, it was found that the strength developed decreases with time to a minimum at about two weeks cure and then begins to increase again.(Table V-3). The original decrease is probably due to shrinkage of the gel formed with resultant weakening of the soil. As the gel shrinks, however, it can be postulated that the cross-linking bonds now placed under considerable stress slowly rearrange themselves to form a more stable configuration. As the bonds are rearranging the strength increases up to its ultimate value, which is reached when the final stable configuration has been attained.

Since the reaction of the MgO is very slow, the strength would be expected to increase steadily with time until the ultimate value is obtained.

MgO has three advantages over BMC as far as use in silicate stabilization is concerned: (1) a smaller weight must be added to the soil per equivalent of magnesium; (2) MgO is more dense than BMC and therefore less bulky for a given weight; and (3) the carbonate ion is not present in MgO and the problem of possible sodium carbonate crystallization is eliminated. Its one disadvantage is that the gel-forming reaction is so slow that a considerable amount of time is necessary to build up good strengths. A good stabilizing system might then be the sodium silicates with a combination of BMC and MgO.
Enough basic magnesium carbonate should be added to develop barely satisfactory strengths within a reasonable time and the remaining magnesium to give the desired ultimate strength should be added in the form of the oxide. One such experiment was carried out and the

results are shown in Table V-7. From these results such a procedure appears favorable. Further work should be done to investigate the best relative amounts of the oxide and carbonate which should be used.

c. Stabilization of Massachusetts Clayey Silt with Sodium Silicate and Calcium Hydroxide

Previous screening tests had suggested that calcium hydroxide might be a successful precipitating agent with sodium silicates in the stabilization of soil. However, as is shown in Table V-8, significant strength after immersion is not obtained until the lime content reaches about five per cent of dry soil weight. At this point, it is believed that the stabilization is primarily due to the lime rather than the silicate, since the same amount of lime with much smaller quantities of silicate stabilized M-21 almost as effectively. In any event, the strengths obtained with five per cent lime were far less than those obtained using only 1.6 per cent basic magnesium carbonate. This may be caused by premature reaction of the lime with the silicate, as is evidenced by the low densities obtained.

6. Stabilization of Massachusetts Clayey Silt with Sodium Silicate and Basic Magnesium Carbonate

a. Effect of Silica Content

The results of this study are presented in Table V-9. This investigation was begun during the previous research period and some of the results presented in the table are taken from the report of that period. These results obtained previously show that in the low range of silica contents increasing silica content increases strength development for one day humid cure and one day humid cure-one day immersion and two week humid cure. It would be expected that the same results would be obtained from testing at seven day humid cure followed by one day immersion.

The table shows, however, that in the intermediate to high range of silica contents, strength decreases with increasing silica content. This can be explained by the less dense cross-linkage which must result when the high silica silicates are used. There is less cross-linking per unit length of silica chain. In NHS and VL, it was found that the high silica samples gave higher wet strengths after seven days humid cure, possibly due to slow formation of additional cross-linkages by gel forming cations in the soil. In M-21, no significant wet strength improvement upon longer cure is noted. It may be supposed that the more easily liberated exchangeable cations of M-21 react without great delay with the silicate.

It was observed that, in M-21, an optimum silica content exists, and that this optimum is lower than the silica content which gives the best strengths in NHS and VL. Since metal cations are more reactive in an acid medium, and since silica is more reactive in a basic medium, it is likely that an optimum pH for the formation of a gel with silica and metal cations exists.

Other things being equal, a higher optimum pH for gel-formation would result from the presence of more reactive metal ions. If the cations in M-21 are indeed more reactive than those in NHS and VL, it would be expected that the pH for stabilization of M-21 would be higher than for the other two soils. At a constant sodium content, the pH in the pore fluid will increase with decreasing silica. Then the greater reactivity of cations in M-21 is a possible explanation of the lower optimum silica content in that soil.

Other factors which probably affect the pH of the pore fluid are the differences in buffer action and water content among the soils.

This last study of the effect of additives in M-21 stabilized with BMC and sodium silicates fixes the optimum ratios of additives for stabilization of M-21. This optimum is a soda to silica weight ratio of 1:2.4, and a magnesium to sodium equivalent ratio of 0.75. At a total additive content of 7.2% of dry soil weight, wet strengths of 650 and 610 psi are obtained after one and seven days humid cure respectively.

b. Effect of Total Additive Content at Constant Silica: Soda: Magnesium Ratios

Once the optimum silica:soda:magnesium ratios have been determined, it was felt that a study of the effect of total additive content in the optimum formulation should be studied. This was accomplished by varying the silica content holding the ratio of sodium to silica, and the ratio of magnesium to sodium constant. The results of the study are presented in table form in Table V-10 and in graphical form in Figure V-4. From the figure, the necessary amounts of silica, magnesium, and sodium necessary for development of any given strength can be calculated (using the constant ratio).

As was expected, the strength developed increased with increasing amount of additive. However, at some point above the range investigated the curve of strength versus additive content may be expected to level off.

D. Conclusions

1. The optimum formulation for the stabilization of NHS with sodium silicates and BMC is a 1:3.2 soda to silica weight ratio and a 0.75 magnesium to sodium equivalent ratio. Soaked strengths of 300 and 365 psi are obtained after one and seven days humid cure, respectively, at a total additive level of 8.5% of dry soil weight. This optimum was determined to give the best results after seven day cure and one day immersion.

2. The optimum formulation for the stabilization of VL with sodium silicates and BMC is a 1:3.2 soda to silica weight ratio and a 1.0 magnesium to sodium equivalent ratio. Soaked strengths of 90 and 235 psi are obtained after 1 and 7 days humid cure, respectively, at a total additive level of 9.1% of dry soil weight. This optimum was determined to give the best results after seven day cure and one day immersion.

3. The optimum formulation for the stabilization of M-21 with sodium silicates and BMC is a 1:2.4 soda to silica weight ratio and a 0.75 magnesium to sodium equivalent ratio. Soaked strengths of 650 and 610 psi are obtained after one and seven days humid cure, respectively, at a total additive level of 7.2% of dry soil weight.

4. The optimum soda to silica weight ratio in NHS and VL is lower than that for M-21.

5. In order to increase the humid cure strength of VBC, dense cross-linkage between silica chains is required; hence, high sodium to silica ratios and about a unit equivalent ratio of magnesium to sodium are necessary. At these ratios, increased silica content gives increased humid cure strengths. Strengths up to 480 psi were recorded at high silica contents.

6. In the ranges studied, sodium silicate and magnesium carbonate alone will not stabilize VBC to immersion, evidently due to the formation of highly swelling sodium montmorillonite. All waterproofing agents tried thus far do not help to stabilize the soil. Consequently, it is felt that stabilization of montmorillonoid clays with silicate systems is very unlikely.

7. Calcium hydroxide and sodium silicate are not as successful in stabilizing M-21 as basic magnesium carbonate and sodium silicate (223 psi as compared to 610 psi after 7 day cure and 1 day immersion).

A fast setting, reactive magnesium oxide with sodium silicate will stabilize M-21 as well as an equivalent amoung of $MgCO_3$. However, the strength development is so slow that at least some basic magnesium carbonate should be used to realize good strengths within a reasonable length of time.

E. Recommendations

1. Salts of other di- and trivalent ions should be investigated as possible substitutes for basic magnesium carbonate as a precipitating agent. Ferrous, zinc, barium, and ferric salts have been suggested.

2. A study to find the optimum formulations more accurately in NHS and VL should be made.

3. The variation of strength developed with additive content at the optimum formulations of NHS and VL should be made so that the necessary additive contents to obtain a desired strength can be determined.

4. Use of a mixture of magnesium oxide and basic magnesium carbonate as a precipitant for sodium silicate should be investigated. The optimum relative amounts of each should be determined.

5. Stabilization of NHS, VL, and M-21 without a precipitating agent should be tried to study the action of cations which may be present in the soil.

TABLE V-1

SILICATE STABILIZATION OF NEW HAMPSHIRE SILT

						1 Day Humid Cure			1 Day Humid Cure 1 Day Immersion			7 Day Humid Cure 1 Day Immersion		
Wt. %[1] SiO₂	Wt. %[1] Na₂O	Wt. %[1] BMC[3]	Per cent Water As Molded on Dry Solids	Wt. Ratio Na₂O:SiO₂	Equiv. Ratio Mg++/Na+	Compressive Strength, psi	Dry Density	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test
a. Effect of Silica Content														
2.51	1.59	1.80	21.0	1:1.58	0.75	115 ± 10	106.5	19.7	90 ± 10	104.0	22.5	100 ± 5	106.0	22.3
3.82	1.59	1.80	22.0	1:2.4	0.75	215 ± 5	107.0	21.1	175 ± 5	105.0	22.7	200 ± 15	104.0	24.0
5.12	1.59	1.80	21.0	1:3.22	0.75	125 ± 5	102.0	20.5	180 ± 10	101.5	25.4	335 ± 40	103.5	23.8
b. Effect of Magnesium Content														
3.82	1.59	1.80	22.0	1:2.4	0.75	215 ± 5	107.0	21.1	175 ± 5	105.0	22.7	200 ± 15	104.0	24.0
3.82	1.59	2.40	21.0	1:2.4	1.00	255 ± 5	106.0	20.4	205 ± 5	105.0	23.2	200 ± 5	104.5	23.9
5.12	1.59	1.20	21.0	1:3.22	0.50	110 ± 0	106.5	19.9	Disintegrated			305 ± 5	103.5	23.7
5.12	1.59	1.80	21.0	1:3.22	0.75	125 ± 5	102.0	20.5	180 ± 10	101.5	25.4	335 ± 40	103.5	23.8
5.12	1.59	2.40	21.0	1:3.22	1.00	180 ± 20	103.5	20.4	235 ± 5	102.5	25.0	270 ± 25	102.5	25.1
c. Effects of Sodium Content														
5.12	1.59	1.80	21.0	1:3.22	0.75	125 ± 5	102.0	20.5	180 ± 10	101.5	25.4	335 ± 40	103.5	23.8
5.12	2.14	1.80	21.0	1:2.4	0.56	425 ± 0	110.0	19.1	350 ± 25	108.0	21.5	315 ± 20	108.5	22.1
5.12	3.24	1.80	21.0	1:1.58	0.37	130 ± 10	115.0	18.0	85 ± 5	111.5	21.2	125 ± 5	110.5	20.6
d. Effect of Water Content														
5.12	1.59	1.80	21.0	1:3.22	0.75	125 ± 5	102.0	20.5	180 ± 10	101.5	25.4	335 ± 40	103.5	23.8
5.12	1.59	1.80	19.0	1:3.22	0.75	290 ± 10	105.0	18.1	300 ± 10	102.5	24.3	365 ± 40	103.0	--

[1] - All percentages based on dry solids
[2] - Pounds Dry Solids per Cubic Foot [illegible]
[3] - Basic Magnesium Carbonate ($4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$)

TABLE V-2

SILICATE STABILIZATION OF VICKSBURG LOESS

						1 Day Humid Cure			1 Day Humid Cure 1 Day Immersion			7 Day Humid Cure 1 Day Immersion		
Wt. %[1] SiO_2	Wt. %[1] Na_2O	Wt. %[1] BMC[3]	Per cent[1] Water As Molded	Wt. Ratio $Na_2O:SiO_2$	Equiv. Ratio Mg^{++}/Na^+	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test
a. Effect of Silica Content														
2.51	1.59	1.80	20.0	1:1.58	0.75	150 ± 10	107.0	18.5	80 ± 10	104.0	22.8	95 ± 5	105.0	22.6
3.82	1.59	1.80	20.0	1:2.4	0.75	140 ± 5	103.0	18.7	140 ± 5	102.0	24.1	145 ± 10	102.5	24.4
5.12	1.59	1.80	20.0	1:3.22	0.75	140 ± 5	101.0	18.3	80 ± 5	99.0	26.7	190 ± 5	101.0	25.8
b. Effect of Magnesium Content														
5.12	1.59	1.20	20.0	1:3.22	0.50	95 ± 10	102.0	19.4	55 ± 5	101.0	25.8	110 ± 5	101.0	25.1
5.12	1.59	1.80	20.0	1:3.22	0.75	140 ± 5	101.0	18.3	80 ± 5	99.0	26.7	190 ± 5	101.0	25.8
5.12	1.59	2.40	20.0	1:3.22	1.00	150 ± 20	102.0	19.0	90 ± 10	99.0	27.1	235 ± 10	100.0	26.8
5.12	1.59	3.00	20.0	1:3.22	1.25	150 ± 20	100.0	19.3	105 ± 10	98.5	27.4	195 ± 5	98.5	27.2

[1] - See Note 1, Table V-1

[2] - See Note 2, Table V-1

[3] - Basic Magnesium Carbonate

Table V-3

EFFECT OF WATER CONTENT ON VBC STABILIZED WITH SODIUM SILICATES AND $MgCO_3$

1.6% Na_2O, 3.8% SiO_2, 2.2% BMC[3]

% Water[1] at Molding	% Volatiles[1] at Test	Compressive Strength, psi after 24-Hour Humid Cure	Dry Density[2]
23.5	22.5	260 ± 10	98.5
25.0	22.8	200 ± 10	99.0
27.0	27.6	205 ± 5	96.0
28.0	26.7	225 ± 15	98.5
30.0	31.0	200 ± 10	94.5
32.0	31.3	185 ± 5	93.5

[1] See Note 1, Table V-1

[2] See Note 2, Table V-1

[3] Basic Magnesium Carbonate

Table V-4

VBC STABILIZATION WITH SODIUM SILICATES & $MgCO_3$

EFFECT OF SILICA CONTENT

Wt. %[1] Na_2O	Wt. %[1] SiO_2	Wt. %[1] BMC[6]	Wt. Ratio Na_2O:SiO_2	$\frac{eq\ Mg^{++}}{eq\ Na^{+}}$	Wt. %[1] Volatiles at Mold	Wt. %[1] Volatiles at Test	Dry Density[2]	24-hr Humid Cure Strength psi
1.6	5.1	2.2	1:3.22	0.92	23.5	22.4	95.0	180
1.6	3.8	2.2	1:2.4	0.92	23.5	22.5	98.5	260
1.6	2.5	2.2	1:1.58	0.92	23.5	21.0	102.0	310
1.6[5]	0	2.2	--	0.92	23.5	23.0	103.5	230
3.2	7.6	4.4	1:2.4	0.92	23.5	23.1	99.0	300
3.2	7.6	4.4	1:2.4	0.92	30.0	33.8	91.0	---[3]
3.2	5.0	4.4	1:1.58	0.92	23.5	23.0	104.0	480[4]

[1] See Note 1, Table V-1

[2] See Note 2, Table V-1

[3] After 2-week humid cure and 24 hour immersion, one of two samples showed a strength of 155 psi. During immersion it decreased 5% by volume, and 10% by weight. Other sample, and those with shorter cure before soaking disintegrated when immersed.

[4] After 1 week humid cure and 24-hour immersion, samples had decreased 30-50% in volume, having a paraboloidal shape. One showed a strength of 155 psi at this time; the other was allowed to remain immersed and completely disintegrated in a total of 48 hours.

[5] Made with 2 per cent NaOH, equivalent to 1.6 per cent Na_2O

[6] Basic Magnesium Carbonate

TABLE V-5

EFFECT OF SODIUM AND MAGNESIUM CONTENTS ON VBC STABILIZATION

%[1] Na_2O	%[1] SiO_2	%[1] BMC[5]	Wt. Ratio $Na_2O:SiO_2$	$\frac{\text{eq Mg++}}{\text{eq base}}$	% Volatiles at Mold[1]	% Volatiles at Test[1]	Dry Density[2]	24-hr Humid Cure Strength psi
A. Effect of Sodium								
1.6	2.5	2.2	1:1.58	0.92	23.5	21.0	102.0	310
0.75	2.4	2.2	1:3.22	1.97	23.6	22.4	98.2	175[3]
0.78% Na_2O)[4] 1.47% KOH)	2.5	2.2	1:3.22	0.92	23.5	23.4	99.5	190
B. Effect of Magnesium								
1.6	3.8	1.5	1:2.4	0.63	23.5	23.8	97.0	190
1.6	3.8	2.2	1:2.4	0.92	23.5	22.5	98.5	260
1.6	3.8	2.9	1:2.4	1.21	23.5	24.2	97.0	250
1.6	3.8	1.5	1:2.4	0.63	28.0	26.7	97.5	220
1.6	3.8	2.2	1:2.4	0.92	28.0	26.7	98.5	225
1.6	5.1	1.5	1:3.22	0.63	23.5	23.9	94.0	160
1.6	5.1	2.2	1:3.22	0.92	23.5	22.4	95.0	180
1.6	5.1	2.9	1:3.22	1.21	23.5	23.8	95.0	195
1.6	2.5	2.2	1:1.58	0.92	23.5	21.0	102.0	310
1.6	2.5	4.8	1:1.58	2.00	23.5	23.2	96.5	190

[1] - See Note 1, Table V-1

[2] - See Note 2, Table V-1

[3] - Other sample, 71 psi

[4] - 1.47 per cent KOH + 0.78 per cent Na_2O is the same amount of base as 1.6% Na_2O.

[5] - Basic Magnesium Carbonate

TABLE V-6

SUMMARY OF GEL TESTS IN ABSENCE OF SOIL

No.	Sample	Gelation Time	Gel Characteristics
1.	1 Na_2O:1.58 SiO_2 BMC[1]	1 hour	Firm, moist gel becoming more rigid as it dries out. No supernatant liquid.
2.	1 Na_2O:1.58 SiO_2 MgO No. 213		Powdery ppt.
3.	1 Na_2O:1.58 SiO_2 MgO No. 210		Powdery ppt.
4.	1 Na_2O:1.58 SiO_2 MgO No. 211		Powdery ppt.
5.	1 Na_2O:1.58 SiO_2 $ZnCO_3$		Powdery ppt.
6.	1 Na_2O:1.58 SiO_2 $Al(OH)_3$		Powdery ppt.
7.	1 Na_2O:3.22 SiO_2 BMC[1]	4 1/2 hours	Firm, moist gel very similar to No. 1, but perhaps a little weaker. No supernatant liquid.
8.	1 Na_2O:3.22 SiO_2 MgO No. 213	4 1/2 hours	Hard, amorphous, small-volumed gel. Essentially all of the liquid remains supernatant.
9.	1 Na_2O:3.22 SiO_2 MgO No. 210		Powdery ppt.
10.	1 Na_2O:3.22 SiO_2 MgO No. 211		Powdery ppt.
11.	1 Na_2O:3.22 SiO_2 $ZnCO_3$		Powdery ppt.
12.	1 Na_2O:3.22 SiO_2 $Al(OH)_3$		Powdery ppt.

[1] - Basic Magnesium Carbonate

TABLE V-9

COMPARISON OF MgO AND $MgCO_3$ AS PRECIPITATING AGENTS IN SILICATE STABILIZATION OF MASSACHUSETTS CLAYEY SILT

All Samples Compacted with 1.59% Na_2O, 3.82% SiO_2, 12.5% H_2O[1]

Precipitating Agent and Weight Per cent[1]	1.8% BMC[3]	0.77% MgO	1.03% MgO	1.54% MgO	1.2% BMC[3] + 0.26% MgO	1.2% BMC[3]
Equivalent Ratio Mg++ / Na+	0.75	0.75	1.00	1.50	0.75	0.50
1 Day Humid Cure						
Compressive Strength, psi	705 ± 150	200 ± 25	295 ± 5	330 ± 10	490 ± 20	465 ± 50
Dry Density[2]	128.0	123.5	125.0	126.5	125.0	128.0
Per cent Volatiles at Test[1]	11.5	11.9	11.5	11.1	11.1	11.8
1 Day Humid Cure + 1 Day Immersion						
Compressive Strength, psi	650 ± 35	165 ± 15	250 ± 5	225 ± 15	565 ± 60	595 ± 25
Dry Density[2]	127.0	121.5	123.0	123.5	123.5	126.0
Per cent Volatiles at Test[1]	13.0	14.7	14.0	12.1	14.1	12.7
7 Day Humid Cure + 1 Day Immersion						
Compressive Strength, psi	610 ± 85	230 ± 10	350 ± 10	390 ± 30	625 ± 10	---
Dry Density[2]	124.5	122.0	123.0	124.0	122.0	---
Per cent Volatiles at Test[1]	14.0	14.1	13.2	12.1	13.9	---
14 Day Humid Cure + 1 Day Immersion						
Compressive Strength, psi	450 ± 30	300 ± 60	---	---	---	---
Dry Density[2]	120.0	124.0	---	---	---	---
Per cent Volatiles at Test[1]	15.1	12.9	---	---	---	---
28 Day Humid Cure + 1 Day Immersion						
Compressive Strength, psi	560 ± 50	585 ± 90	---	---	---	---
Dry Density[2]	120.0	124.0	---	---	---	---
Per cent Volatiles at Test[1]	15.5	12.5	---	---	---	---

[1] - See Note 1, Table V-1

[2] - See Note 2, Table V-1

[3] - Basic Magnesium Carbonate

TABLE V-8

STABILIZATION OF M-21 WITH SODIUM SILICATES AND $Ca(OH)_2$

Wt. %[1] $Ca(OH)_2$	Wt. %[1] Na_2O	Wt. %[1] SiO_2	Wt. Ratio $Na_2O:SiO_2$	eq Ratio $\frac{Ca{++}}{Na{+}}$	Dry[2] Density	Strength, psi upon cure followed by 24-hour soak (days)				24 Hour Humid Cure
						1	7	14	28	
0.95	1.6	3.8	1:2.4	0.5	116	Disintegrated	---	34	---	39
1.4	1.6	3.8	1:2.4	0.75	111	"	---	50	---	73
1.9	1.6	3.8	1:2.4	1.00	110	"	---	52	---	115
5.7	1.6	3.8	1:2.4	3.00	108.5	173	223	---	---	235
5.0[3]	0.39	0.37	1:0.97	10.8	---	---	175	---	610	---
5.0[3]	0.20	0.19	1:0.97	21.6	---	---	100	---	160	---
5.0[3]	0.097	0.093	1:0.97	43.1	---	---	80	---	125	---
1.8% BMC[4]	1.6	3.8	1:2.4	0.75	127.5	650	610	---	---	680

[1] - All percentages on dry soil

[2] - lb dry solids/ cu. ft.

[3] - Commercial lime used (Personal communication from Charles C. Ladd)

[4] - Basic Magnesium Carbonate

TABLE V-9

EFFECT OF SiO_2 CONTENT ON STABILIZATION OF M-21
WITH SODIUM SILICATE

						1 Day Humid Cure			1 Day Humid Cure 1 Day Immersion			7 Day Humid Cure 1 Day Immersion		
Wt. %[1] SiO_2	Wt. % Na_2O	Wt. %[1] BMC[3]	Per cent[1] Water As Molded	Wt. Ratio $Na_2O:SiO_2$	Equiv. Ratio Mg^{++}/Na^+	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test
3.04[4]	1.92	2.17	12.5	1:1.58	0.75	490 ± 60	127.5	9.8	380 ± 10	126.0	12.7	--	--	--
3.82[4]	1.91	2.16	12.5	1:2.0	0.75	750 ± 10	129.0	11.8	665 ± 5	129.0	12.8	--	--	--
3.82	1.59	1.80	12.5	1:2.4	0.75	705 ± 150	128.0	11.5	650 ± 35	127.0	13.0	610 ± 85	124.5	14.0
5.12	1.59	1.80	12.5	1:3.22	0.75	395 ± 35	116.0	11.8	410 ± 30	118.5	13.5	460 ± 50	118.5	13.7

[1] - See note 1, Table V-1

[2] - See Note 2, Table V-1

[3] - Basic Magnesium Carbonate

[4] - From Report on Soil Stabilization Research for the Period February 1959 - June 1959, Soil Stabilization Lab, MIT, June, 1959

TABLE V-10

EFFECT OF TOTAL ADDITIVE CONTENT ON SILICATE STABILIZATION OF M-21

1:2.4 $Na_2O:SiO_2$, 0.75 equiv. Mg^{++}/equiv. Na^+

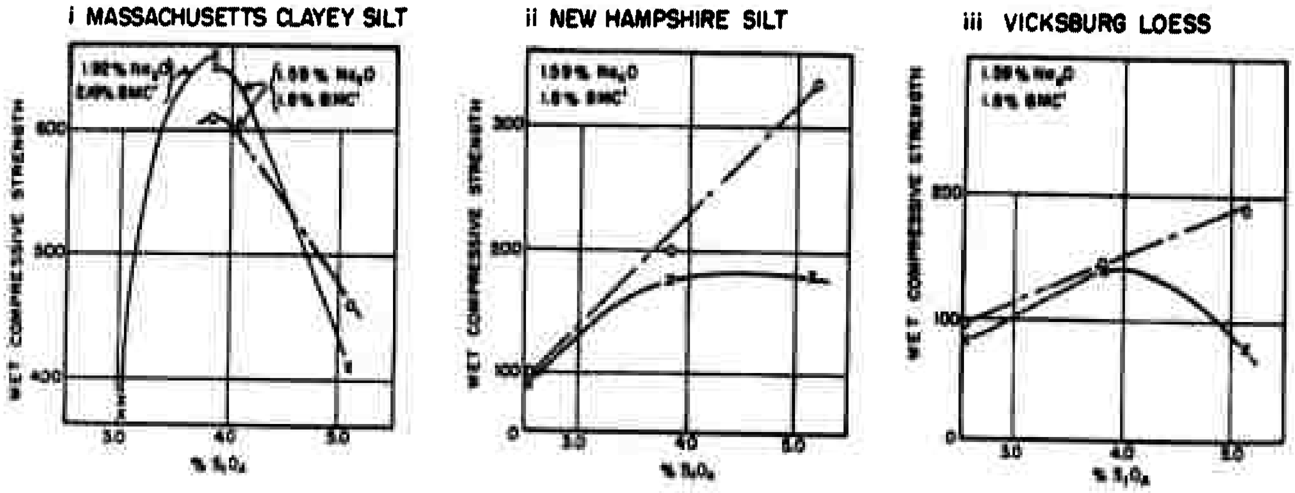
Wt. %[1] Total Additives	Wt. %[1] SiO_2	Wt. %[1] Na_2O	Wt. %[1] BMC[3]	% Water As Molded	1 Day Humid Cure			1 Day Humid Cure 1 Day Immersion			7 Day Humid Cure 1 Day Immersion		
					Compressive Strength psi	Dry Density[2]	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test	Compressive Strength, psi	Dry Density[2]	Per cent[1] Volatiles at Test
1.89	1.00	0.42	0.47	12.5	--	--	--		Disintegrated			Disintegrated	
3.78	2.00	0.83	0.94	12.5	170 ± 5	123.0	12.1	125 ± 5	122.0	14.1	135 ± 5	122.0	14.1
5.66	3.00	1.25	1.41	12.5	590 ± 35	126.0	11.8	505 ± 55	125.5	13.2	465 ± 65	123.0	13.6
7.21	3.82	1.59	1.80	12.5	705 ± 150	128.0	11.5	550 ± 35	127.0	13.0	610 ± 85	124.5	14.0

[1] - See Note 1, Table V-1

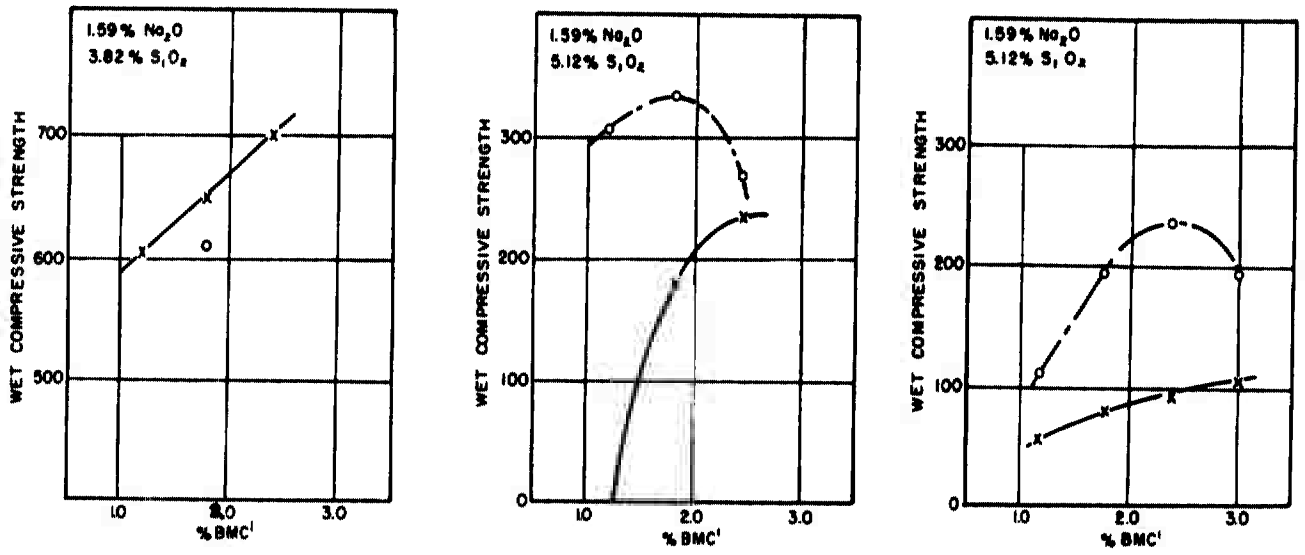
[2] - See Note 2, Table V-1

[3] - Basic Magnesium Carbonate

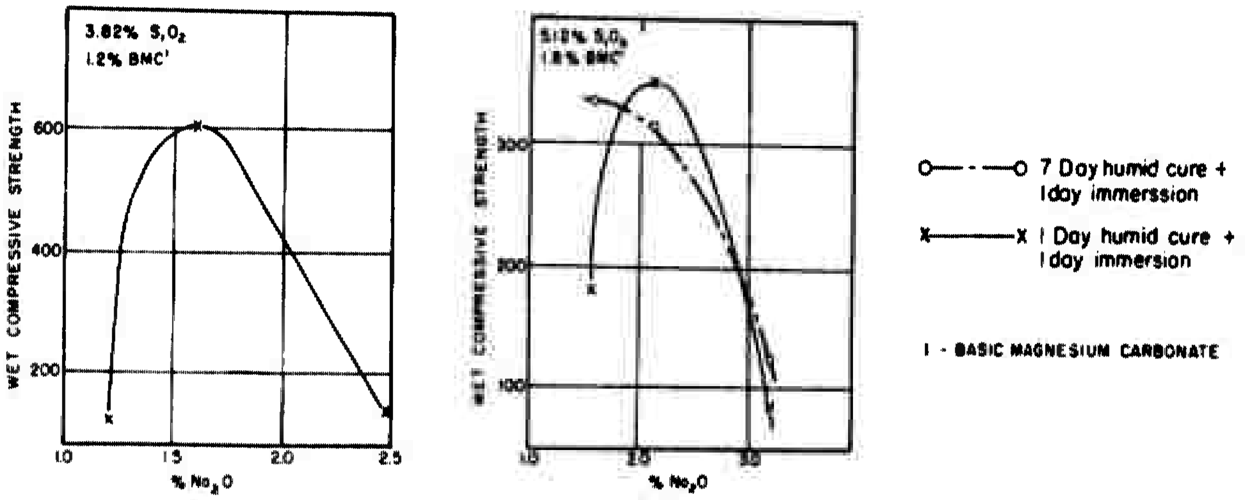
FIGURE V-1 EFFECT OF SILICA, SODIUM, AND MAGNESIUM CONTENTS ON WET STRENGTH OF M-21, N.H.S AND VL



A. EFFECT OF SILICA

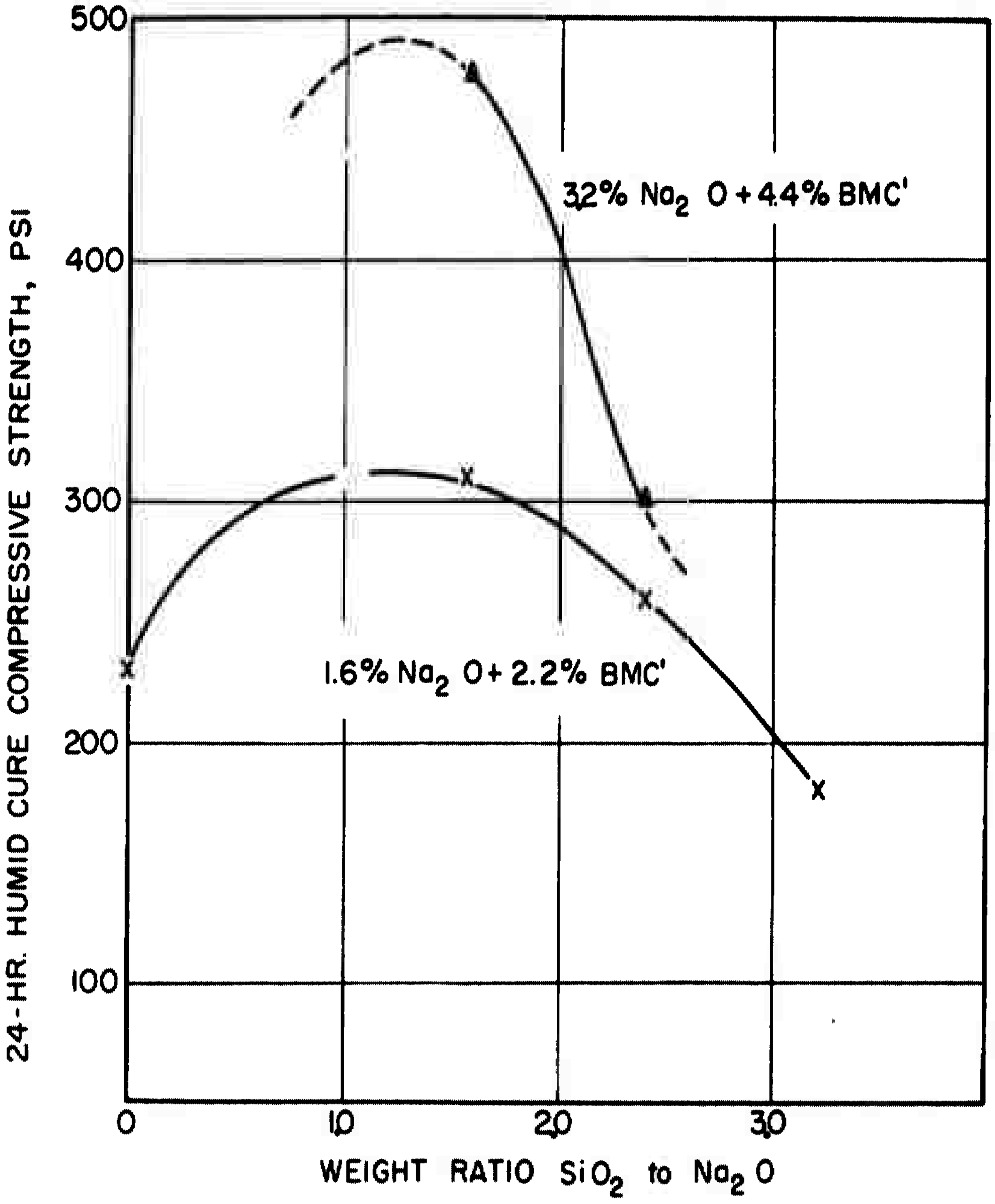


B. EFFECT OF MAGNESIUM



C EFFECT OF SODIUM

FIGURE V-2 EFFECT OF SILICATE BASICITY ON STABILIZATION OF VBC



Note:
1 - Basic Magnesium Carbonate

FIGURE V-3 COMPARISON OF WET STRENGTH DEVELOPED vs CURE PERIOD FOR EQUIVALENT AMOUNTS OF BMC AND MgO IN M-21

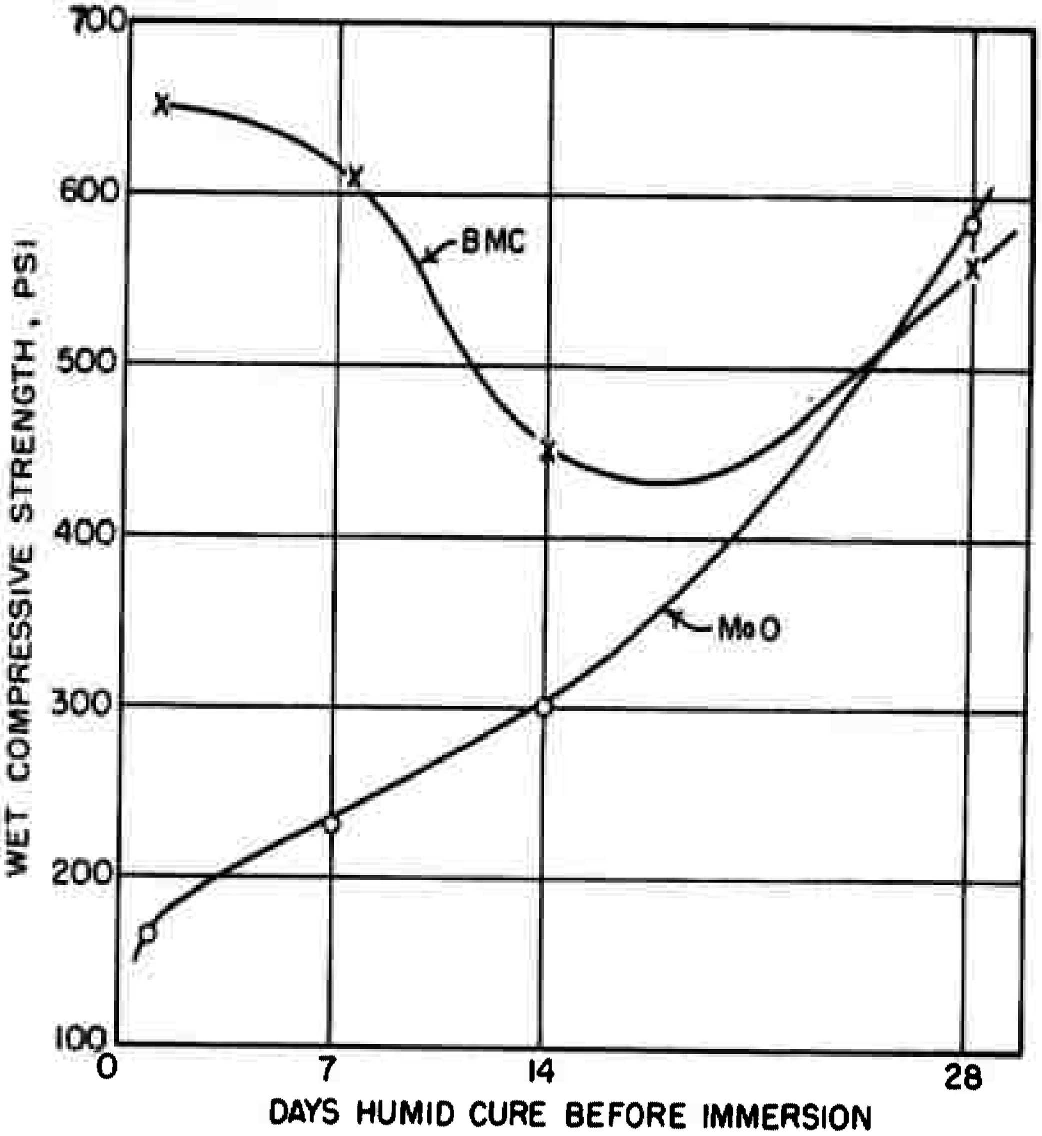
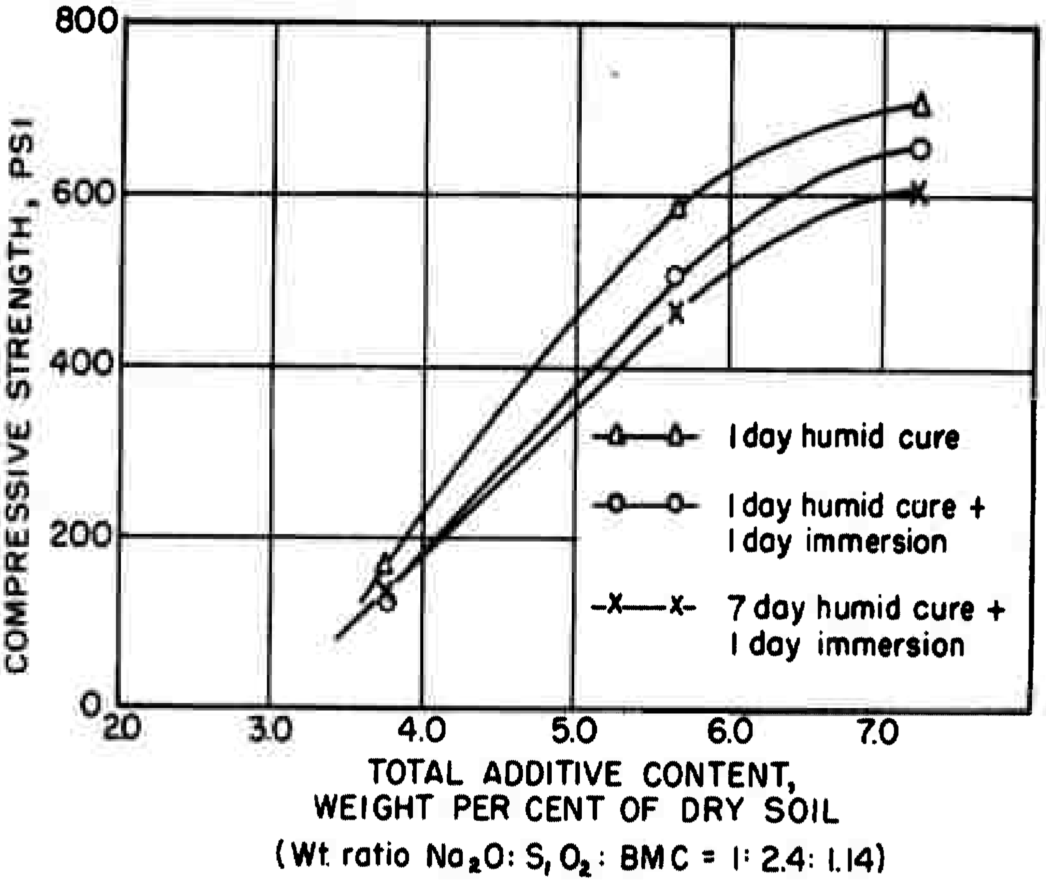


FIGURE V-4 VARIATION OF STRENGTH DEVELOPED WITH TOTAL ADDITIVE CONTENT IN M-21



VI. SOIL STABILIZATION WITH ASPHALT EMULSIONS

A. Introduction

Asphalt is an inexpensive and convenient means for the stabilization of fine-grained soils, but it must be reduced in viscosity for adequate admixture with or penetration of the surfaces of these soils. Two means of accomplishing this are: (a) solution of the asphalt in an organic solvent, the mixture being known as a "cutback", and (b) emulsification of either the asphalt or a cutback in an aqueous solution. The use of asphalt emulsions in preference to cutbacks permits the incorporation of a water-soluble soil-stabilizing additive, phosphoric acid, in one step and reduces the danger of flammability. Thus, the object of this year's research with asphalt as a soil stabilizing and waterproofing agent has been the preparation of asphalt-in-water emulsions containing phosphoric acid for the purposes of (a) strengthening and improving the erosion resistance characteristics of soils by topical application of emulsions to the surface, and of (b) increasing the load bearing capacity of soils by incorporation of emulsions in a compacted soil mass by mechanical mixing.

The topical application of soil-stabilizing materials in liquid form would provide a highly satisfactory method for the surface treatment of soils. In those instances in which mechanical incorporation of these materials is not feasible from economical or practical considerations, the application of asphalt in emulsified or cutback form shows excellent promise as a means of increasing the water and abrasion resistance and of improving the load bearing characteristics of a thin layer of soil surface. Topical treatment of soils is of importance in controlling the erosion of earth slopes, the seepage in reservoirs and ponds, and in increasing the resistance of surfaces to mechanical deterioration.

Previous work performed in this laboratory (Ref. 1) demonstrated the possibility of preparing emulsions stabilized with a nonionic emulsifying agent, and containing phosphoric acid which are capable of penetrating the surface of a fine sand to depths of up to two inches. Thus, after ascertaining what variables of emulsion composition and method of preparation were of importance in promoting soil penetrability, several promising emulsions were prepared and applied under specified conditions to the surfaces of several soils. The permeability of these treated surfaces to a constant head of water, the resistance of the surfaces to penetration by a given weight load, and the stability and erosion resistance of the surfaces when subjected to a high-speed water spray directed perpendicularly to the surface were employed as tests to evaluate the performance of the stabilized surfaces under anticipated natural weathering conditions.

For the corporative stabilization of soils with asphalt, emulsions prepared with the use of cationic emulsifying agents are believed to "plate out" on the soil particles, producing a more effective adhesion between asphalt and soil than that obtained with the use of anionic and nonionic emulsifiers, and providing more effective waterproofing of the compacted soil samples. Stabilization of Massachusetts Clayey Silt (M-21) and Vicksburg Buckshot Clay (VBC) with cationic emulsions containing phosphoric acid have in the past given compressive strengths much less than those which can be obtained using phosphoric acid alone. The observable inhomogeneity of the mixtures of emulsion and soil, due to rapid breaking of the emulsion and deposition of the asphalt particles before adequate mixing had been obtained, is undoubtedly the reason for these low strengths. Since the depths of penetration achieved with emulsions prepared for topical treatment indicated that these emulsions were more stable and less likely to deposit asphalt at a rapid rate, the stabilization of the aforementioned soils was investigated with the use of these emulsions.

B. Experimental Procedure

1. Materials for Emulsification

A moderately hard (40-50 penetration) and a moderately soft (100-120 penetration) straight run, vacuum distilled asphalt were emulsified in tap water using several promising cationic and nonionic emulsifiers, including:

a. Duomeen S -- a diamine of the form $R\text{-}NH\text{-}C_3H_6\text{-}NH_2$, where R is derived from soya oil,

b. Duomeen T -- a diamine of the same form, derived from tallow,

c. Rosin Amine D -- a diamine of abietic acid $(C_{20}H_{30})_2)$, and

d. Nonic 218 -- a nonionic condensation product of dodecyl mercaptan and ethylene oxide.

Technical grade samples of the Duomeens were supplied by the Armour Industrial Chemical Company, of the Rosin Amine D by the Hercules Powder Company, and of the Nonic 218 by Sharples Chemicals. Reagent grade meta- and ortho-phosphoric, acetic, hydrochloric and sulfuric acids constituted the water soluble portions of the diamine emulsifiers. The chlorides of transition series metals ($CrCl_3 \cdot 6H_2O$, $FeCl_3 \cdot 6H_2O$, $CoCl_2 \cdot 6H_2O$, $NiCl_2 \cdot 2H_2O$, $CuCl_2 \cdot 2H_2O$, and $CdCl_2 \cdot 2.5H_2O$) and calcium chloride-dihydrate and aluminum chloride-hexahydrate, added in trace amounts to specified Duomeen T-hydrochloride emulsions, were also reagent grade. Methyl cellulose and cetyl alcohol, which were employed as emulsion stabilizers, were of technical grade. The four volatile, asphalt-miscible organic solvents used to reduce the viscosity of the asphalt, gasoline, kerosene, xylene and carbon disulfide, were commercial grade reagents.

2. Emulsion Preparation and Preliminary Evaluation Techniques

Asphalt of the desired penetration range was heated to its melting point and, when desired, was diluted with a measured volume of gasoline, kerosene, xylene or carbon disulfide. When aliphatic diamine emulsifiers were employed, a measured amount of the diamine was added to the asphalt. To an amount of water approximately equal in weight to that of the asphalt phase, one of five acids (meta-phosphoric, orthophosphoric, acetic, hydrochloric and sulfuric) was added in an amount slightly in excess of the quantity required for the neutralization of the diamine. One-tenth to one-half of one per cent of a metallic chloride, such as those of copper, calcium, cadmium, chromium, iron, nickel, cobalt, zinc, and aluminum, was incorporated into the water phase of diamine-hydrochloric acid emulsions, when desired. When Nonic 218 was employed, it was added to that phase in which the emulsification was to be carried out. Secondary emulsifying agents, which included cetyl alcohol and methyl cellulose, were dissolved in the water phase.

Emulsification was carried out in most cases either by (a) adding the asphalt slowly to the aqueous phase with agitation, forming an asphalt-in-water emulsion directly (mayonnaise method), or (b) adding the aqueous solution to the asphalt, to produce a water-in-oil emulsion, which usually inverted to an asphalt-in-water emulsion just before the addition of the aqueous solution was complete (inversion method). A third technique, the alternate addition of the asphalt and aqueous solution resulting in an inversion with each addition, was employed less frequently.

A Premier Dispersator, the speed of which could be adjusted by a Variac, has been the most effective laboratory-scale emulsification device employed to date. A volume of foam usually ranging from 100 to 300 per cent of the liquid emulsion volume is obtained at high mixing rates.

An optical microscopic examination of the stable emulsions was undertaken to obtain an estimate of the particle size distribution. Previous investigation (Ref. 1) has indicated that the more finely-divided emulsions penetrate soil surfaces to greater depths than coarse emulsions, so that droplet size provided a convenient means of selecting promising emulsions for further study.

The promising emulsions prepared as described above were applied directly (without filtration) to the uncompacted surface of 80-150 mesh Ottawa sand or dry M-21 confined in 3-inch diameter Petri dishes or in 4.5-inch Buechner funnels. The weight of emulsion applied, the area over which the treatment was applied, and the average depth of penetration were measured. The average asphalt concentration in the penetrated soil layer, the fraction of the void volume of the soil filled with asphalt, and the maximum asphalt dosage per unit area of application which would penetrate the soil, could be calculated from these measurements. The density of the uncompacted Ottawa sand measured 1.51 g/cc, while that of uncompacted M-21 was 1.47 g/cc; the void volumes of these materials were calculated to be 33 and 36.5 per cent, respectively, as obtained from the densities of the mineral components which make up these soils. Those emulsions with an adequate penetration range which could be applied in sufficient quantities for stabilization of the surface of these soils were then subjected to water permeability, penetration resistance and erosion tests using several types of soils.

3. Topical Treatment of Soil Surfaces

An asphalt cutback or freshly prepared mixture of emulsions and ortho-phosphoric acid (if the addition of the latter was desirable for improved stabilization of the soil) was applied in one or several dosages to the surface of an uncompacted soil contained in a 4.5-inch diameter Buechner funnel. The application in a funnel permitted the escape of air from the voids of the soil and prevented channeling in the soil during the uptake of emulsion. The emulsion was diluted in most cases with an equal weight of water. The soils employed in this investigation included Massachusetts Clayey Silt (M-21), New Hampshire Silt (NHS) and Wisconsin sand (1056); the properties of these soils are presented in Table II-1.

4. Evaluation of Stabilized Surfaces

a. Permeability to Water

The level of the stabilized soil surface in the Buechner funnel was placed one inch below the top so that a one-inch head of water was readily maintained by keeping the funnel full. The amount of water passing through the filter was recorded as a function of time.

In future work, it will be necessary to compact the soil before treatment with emulsion or cutback to prevent a separation between the stabilized surface and the underlying non-treated soil during the process of wetting. In order that the permeability of the surface layer may be computed from these experiments, a continuous column of water should be maintained through the Buechner funnel and should exit from the funnel below the surface of a reservoir of water. The flow rate through the surface is obtained by measuring the amount of water needed to maintain a constant head of water in the column.

b. Penetration Resistance

A one-fourth inch diameter brass piston was forced perpendicularly into the stabilized soil surface at a constant rate of 2.5 mm/min to provide a measure of the penetration resistance of the soil. The maximum resistance of the surface and the resistance obtained when the piston had penetrated the surface to a depth of one-sixteenth inch were noted; these values were generally found to be the same for the soils investigated. As the concentration of stabilizing materials--asphalt and phosphoric acid--in the surface decreases with increasing depth, the penetration resistance falls to the value obtained for a wet, unstabilized soil.

c. Water Erosion Resistance

By placing the level of the treated soil surface as close to the top of the Buechner funnel as possible, a spray of water can be directed at the surface, eliminating interference to erosion which would be offered by a continuous film of water on the surface. The interface between the stabilized surface and the porcelain wall was coated with asphalt to prevent erosion at the wall and to confine erosion to the bulk surface of the sample.

Samples were subjected to linear water velocities ranging from 300 to 1500 cm/sec. with the use of a modified flat-face shower head fixture with 34 streams of water emanating from the shower with radii of one-sixteenth inch and directed uniformly at all parts of the surface at a distance of nine inches above the surface. The linear terminal velocity of spherical rain droplets having the same radius was calculated to be 165 cm/sec. The tests extended from immediate failure to forty hours without gross disruption of the stabilized surface. In general, at high linear water velocities, one hour of erosion was an adequate period of testing, since either gross failure occurred during that time or the weight of material eroded and collected by sedimentation in a large overflow reservoir of water was almost negligible for all samples which did not fail.

The flow rate of water striking the surface was obtained volumetrically before and after each erosion test, and the linear water velocity of the impinging streams was calculated from this figure on the basis of the assumption that the radii of these streams did not increase substantially in the distance between the shower head and the surface. The variations in water temperature during the course of a test, which are believed to influence the rate of erosion significantly, were negligible. Since the volume of water required for a test was great, controlling the water temperature was impractical.

The penetration resistance of the surface was measured just prior to, immediately after, and two days after an erosion test. Compaction of the soil before treatment with a cutback or an emulsion is again necessary to prevent separation of the stabilized from the untreated layer of soil, a phenomenon which markedly accelerates gross disruption of the surface.

5. Corporative Stabilization of Soils

To a given weight of soil placed in the bowl of a finger-prong mixer, a cutback or an emulsion, containing phosphoric acid as a secondary stabilizer if desired, was added rapidly and the soil mixed initially for a period of from five to ten minutes. After scraping the bowl clean, the soil was mixed again until its appearance was homogeneous (which usually required ten minutes) or until twenty minutes had elapsed. Samples were molded at a constant pressure of 1000 psi by two-end static compaction in a Harvard miniature size mold. These were cured at room temperature for 1 day at 100 per cent relative humidity, for 1 day at 100 per cent relative humidity followed by 1 day immersion, and for 7 days at 100 per cent relative humidity followed by 1 day immersion, and then tested to failure in unconfined compression at a constant, measured rate of strain.

C. Results and Discussion

1. Emulsion Preparation

A study of the properties of asphalt emulsions as a function of the methods of preparation and the emulsifying materials was warranted by the successful but accidental and non-reproducible formation of one such nonionic emulsion in a previous investigation (Ref. 1).

a. Effects of Variations in Emulsification Method

From an analysis of the particle size distributions for the emulsions described in Table VI-1, the following conclusions can be drawn:

i.) The inversion technique gives emulsions of comparable particle size but having less particle aggregation than those produced by other emulsification methods. Since mixing of the emulsion ceases immediately after inversion takes place for the relative amounts of asphalt and water employed, a minimum of aggregation (clusters of small particles about larger particles formed by coalescence) will take place with this technique.

ii.) The rate of mixing during emulsification has no effect on the particle size distribution or the extent of particle aggregation using the Premier Dispersator. The expected minimum in the mean particle size, which is a function of the shear rate in particle formation and of the translational energy of the newly-formed particles, is not observed for the range of mixing rates studied (Ref. 2). It was observed that those emulsions with high particle aggregation foamed excessively during preparation.

iii.) An increase in the emulsifier concentration from 2.5 to 5 per cent (based on total weight of emulsion) results in an increased tendency for the particles to aggregate and eventually to coalesce. The results presented in Table VI-2 indicate that as little as one per cent emulsifier can be employed to form an emulsion using asphalt diluted with up to as much gasoline by weight as asphalt.

iv.) The free mineral acid concentration does not affect particle size distribution or extent of aggregation for small excesses of the acid in this system.

v.) Furthermore, it was observed experimentally that the temperatures of the materials to be emulsified and the temperature of the resultant emulsions have no effect on the particle size distribution for the emulsions prepared with cut-back asphalt and cationic or nonionic systems. Of course, the asphalt phase must be maintained at a temperature sufficient for it to remain in the fluid state necessary for emulsification (70-95°C).

b. Effect of Variations in Emulsifying Materials

In addition to a microscopic determination of particle size distributions, the application of given amounts of the emulsions prepared with various emulsifiers and additives to different types of soil surfaces provided a means of comparison of these emulsions on the basis of their penetrating characteristics. A summary of the experiments designed to determine the effect of variations in the composition and fluidity of the asphalt phase, of variations in the emulsifier systems, and of variations in the asphalt to water phase ratios in these emulsions is presented in Table VI-2.

(i.) Asphalt Phase Fluidity and Composition

It was established that the use of a volatile organic solvent to reduce the viscosity of the asphalt is a prerequisite for successful penetration of sandy or silty soils by emulsions prepared with any of the emulsifying agents studied in this period, as can be observed from the results presented in Table VI-2. The penetration number of the original asphalt, the type of organic solvent used to dilute the asphalt, and the relative amounts of asphalt and organic solvent are the variables which alter asphalt phase composition and fluidity and whose effects were studied for typical cationic (Duomeen T-acid) and nonionic (Nonic 218) emulsifier systems.

For two organic cutback solvents, gasoline and kerosene, mixed in the same ratios with the moderately soft (100-120 penetration) and the moderately hard (40-50 penetration) asphalt, the maximum depth of penetration in a fine sand was significantly greater for the softer asphalt. The maximum asphalt density in the treated layer varied inversely, however, as the penetration number of the original asphalt. The more volatile and more fluid cutback solvent of the two, gasoline, is more effective in reducing the asphalt viscosity and in increasing the maximum depth of penetration. Emulsions prepared with xylene and carbon disulfide in weight ratios to the asphalt that would make these solvents economically competitive with a satisfactory two parts asphalt-one part gasoline emulsion were much less effective in the penetration of the sand surface than the emulsified gasoline cutback.

A minimum mean particle size and a maximum depth of penetration has been observed at a two-to-one ratio of asphalt to solvent for the Nonic 218 system. This optimum ratio has been established for the use of gasoline and 100-120 penetration asphalt. The decrease in asphalt phase viscosity is evidently necessary to make the emulsified droplets sufficiently deformable to pass through the pores of a fine-grained soil; an excess of the cut-back solvent in the asphalt phase, on the other hand, is responsible for an increased rate of coalescence of the emulsified particles, which are no longer stable enough to maintain their identity on collision.

For the Duomeen T-hydrochloric acid system, maintaining a one-to-one weight ratio of asphalt phase to water phase, the reduction of the asphalt phase viscosity initially improved the penetration characteristics of the emulsion, but the formation of a water-in-asphalt emulsion was observed when a ratio of one part asphalt to two of gasoline was reached. This is caused by an increase in the phase volume of the asphalt particles resulting from the replacement of a weight of asphalt with an equal weight of the less dense solvent. By operating with a lower asphalt phase to water phase volume ratio, the formation of an asphalt-in-water emulsion is assured and a better determination of the optimum asphalt-to-solvent ratio for Duomeen T-hydrochloric acid systems can be made. This optimum ratio is approximately one part asphalt to one part solvent. The formation of a water-in-asphalt emulsion was not encountered with the Nonic 218 system, since in this case the emulsifying agent is much more soluble in the water phase, and the external phase is generally that in which the emulsifying agent is more soluble (Ref. 2).

ii.) Emulsifying Agents

Duomeen T

The most extensive investigation pertained to emulsifier systems consisting of the N-alkyl propylene diamine Duomeen T, and one of five acids: meta-phosphoric, ortho-phosphoric, hydrochloric, acetic and sulfuric. The following observations were made:

With meta-phosphoric acid both the inversion method and the mayonnaise method produced water-in-asphalt rather than asphalt-in-water emulsions. The use of four times as much water as asphalt phase did not alter this result.

Duomeen T-ortho-phosphoric acid emulsions penetrate 80-150 mesh Ottawa sand to a depth of one-fourth inch, but fail to penetrate a clayey silt, M-21, to any measurable depth. These emulsions contain particles somewhat larger in size than those obtained with Nonic 218 and Duomeen T-hydrochloric acid systems.

Of the acids studied, hydrochloric acid is the best water-soluble portion of this cationic emulsifying agent. The emulsions are extremely fluid and the particles are not aggregated; ten per cent by weight of ortho-phosphoric acid may be added subsequent to emulsification without any alteration in the particle size distribution, the extent of aggregation, or the depth of penetration in soils. The treated layer of 80-150 mesh Ottawa sand is remarkably flexible and strong. Twenty-four hour immersion of this uniformly thick layer in water weakens the cohesive binding between sand particles, but strength is regained on drying. The degree of penetration of M-21 is insufficient to give any surface strength.

Emulsions prepared with acetic acid and Duomeen T show little promise. Excessive foaming is encountered during emulsification, the foam reaching a volume three times that of the liquid emulsion. Particles are nearly five-fold greater in size than those found in the satisfactory nonionic and Duomeen T-hydrochloric acid emulsions.

Water-in-asphalt emulsions were formed when sulfuric acid was employed as the water soluble portion of the Duomeen T emulsifier; the addition of excess water did not invert this emulsion.

Chromic and ferric chlorides, added in trace amounts to Duomeen T-hydrochloric acid emulsions, are both equally effective in reducing the particle size and in improving the penetration characteristics of these emulsions. A maximum depth of 3/4 to one inch of penetration was obtained in the Ottawa sand. One tenth of one per cent chromic or ferric chloride on the weight of the emulsifying agent is sufficient, and increased quantities are not as effective. Other metal chlorides fail to affect the quality of the emulsion when added in comparable amounts; these include the chlorides of aluminum, copper (II), cadmium, calcium, cobalt, nickel and zinc, most of which belong to the transition series metals of the periodic table. Chromium (III) forms extremely stable coordinate complexes with amino compounds (Ref. 3); it is postulated that the emulsified asphalt droplets are prevented from coalescing on collision with one another (or from depositing too rapidly on soil particles) by the formation of solid chrome-amine complexes around the droplets. The same mechanism is believed to be valid for the effect of ferric ion. The failure of other cations, which are chemically similar to these two, to achieve the same results is not fully understood, since the amine complexes of cobalt, nickel, etc. are also known to be stable.

Other Surface-Active Agents

Duomeen S, with hydrochloric acid as the water-soluble portion of the emulsifier, and using $CrCl_3 \cdot 6H_2O$ as a stabilizing agent, did not yield as satisfactory an emulsion as Duomeen T, confirming the results of previous work with this material. Rosin amine D failed to produce a stable emulsion when employed as a substitute for Duomeen T.

No combination of variables has served to produce as fine-grained an emulsion as had been previously obtained with Nonic 218. The present reproducible Nonic 218 system will penetrate 80-150 mesh sand to a depth of 2/3 inch, as compared to a depth of two inches for the accidental formulation. Furthermore, the maximum application which will leave no surface residue of unpenetrated asphalt is half that of the Duomeen T-hydrochloric acid emulsion containing chromium (III) or ferric chloride and much less than that of the accidental preparation. At least ten per cent on the weight of the nonionic emulsion of ortho phosphoric acid may be added without altering the penetration characteristics of these Nonic 218 emulsions.

Secondary emulsifiers, such as cetyl alcohol and methyl cellulose, have failed to decrease the amount of particle aggregation and improve the penetration levels for both cationic and nonionic emulsions.

iii.) Asphalt to Water Phase Ratio

Maintaining a two-to-one weight ratio of asphalt to gasoline throughout, emulsions containing 60 and 65 per cent asphalt phase by weight have been successfully prepared and appear to be stable for an indefinite length of time. Several attempts to produce a 70 per cent emulsion of the asphalt phase in water resulted in failure. Duomeen T and hydrochloric acid served as the emulsifying agents and constituted 2.5 per cent of the weight of the emulsion in these preparations. The depth of penetration obtained in 80-150 mesh Ottawa sand using the 65 per cent asphalt phase emulsion was 2/3 that of a 50-50 asphalt-in-water emulsion, or 1/2 to 2/3 inch.

iv.) Reproducibility

Repeated microscopic examination and penetration tests have demonstrated that the satisfactory emulsions, which include the Duomeen T-hydrochloric acid-chromic or ferric chloride and the Nonic 218 preparations, are readily reproducible.

2. Characteristics of Surfaces Treated with Emulsions and Cutbacks

a. Generalities

Table VI-3 lists the emulsions used in topical treatment studies and gives each a "coded name" to facilitate further discussion.

Table VI-4 is a summary of the depths of penetration achieved by application of cutbacks and some of the more promising emulsions to the surface of four soils: 80-150 mesh Ottawa Sand, M-21, New Hampshire Silt, and a Wisconsin sand (1956).

For limited applications to the surface, emulsions will distribute asphalt over a thicker layer than straight cutbacks containing the same ratio of asphalt to gasoline. These limited dosages of cutbacks and emulsions initially fill all the void volume in each of the soils considered. Therefore, the concentration of asphalt in the penetrated soil will be greater when cutbacks rather than emulsions of these cutbacks are employed, since the volatiles contents of the emulsions are much greater than those of the cutbacks.

As the amount of applied cutback is increased, the depth of penetration will be directly proportional to the amount applied, so that the average asphalt concentration (or the percentage of the void volume filled with asphalt) will remain the same as for limited applications. Up to 0.40 g. asphalt/ sq. cm. of surface may be applied to the surface of the Ottawa Sand, for example, without leaving a residue of asphalt on the surface; the penetration obtained is 36 mm., as compared with 8 and 18 mm. for applications of 0.10 and 0.20 g. asphalt/ sq. cm., respectively.

For emulsions, however, doubling the dosages produces less than half again as much of an increase in the depth of penetration. Premature deposition of asphalt droplets from the emulsion and rapid blocking of the soil pores not only results in a separation of the asphalt and water phases during penetration, giving a surface layer containing a greater average asphalt concentration, but limits the asphalt dosage per unit soil surface which will penetrate the soil. This maximum dosage is approximately 0.05, 0.08, 0.15 and 0.20 g. of asphalt/ sq. cm. of surface for the soils Massachusetts Clayey Silt, New Hampshire Silt, Wisconsin Sand (1056), and Ottawa Sand, respectively.

The absorption of water by layers of Ottawa Sand, stabilized with several cutbacks and promising emulsions, is presented in Table VI-5. Surfaces stabilized with emulsions absorb, on the average, 25 per cent of their weight of water on immersion, while those stabilized with cutbacks absorb five per cent of their weight of water.

Permeation of Stabilized Surfaces to Water Seepage

In Table VI-6 the permeability studies are summarized.

The per cent of the stabilized surface covered with asphalt that did not penetrate the soil is the primary factor governing the rate of seepage of water through the stabilized surface of New Hampshire Silt. While a residual layer of asphalt makes the surface impermeable to water, it prevents the escape of volatiles from the treated soil layer and markedly decreases the rate of strength development in this layer. For those surfaces not entirely coated with residual asphalt, the flow rate of water through the surface remained constant after decreasing from a very high initial rate of seepage. Since the stabilized surface layer of New Hampshire Silt is very thin, channelling of water through fissures which developed in the surface of the soil occurred, but the rates of seepage were still much less than those observed for unstabilized soil surfaces.

Improvement of the testing procedure should yield more reproducible results than those obtained in this study to date. The permeability of the stabilized surfaces of M-21, Ottawa Sand, and 1056 to water seepage may then be investigated.

c. Penetration Resistance

Table VI-7 presents the maximum penetration resistances of topically treated soils cured at room temperature and humidity for specified periods of time.

A comparison of the emulsified cutbacks prepared with Duomeen T, hydrochloric acid, ferric and chromic chlorides and those prepared with Nonic 218 when applied to the surface of Wisconsin Sand (1056) indicates that the latter develop significantly greater strength for long periods of cure. The penetration resistance reaches a value of 465 psi when 0.20 g. asphalt/ sq. cm. of the Nonic emulsion containing ten per cent ortho-phosphoric acid is applied and the surface cured for a period of thirty days. The addition of phosphoric acid is essential for the eventual development of strengths greater than 50 psi. The incorporation of phosphoric acid in the surface layer provides strength while the primary function of the asphalt appears to be that of waterproofing. Dilution of these fifty-fifty cutback-in-water emulsions with another part by weight of water significantly improves the initial strength development of the surface, and increases the depth of penetration of the emulsion into the soil. It is presumed that the lower asphalt concentration in these soil surfaces permits a more rapid escape of the volatiles from the soil, and hence, an accelerated rate of cure. Dilution of the emulsion with more than one additional part of water does not give a further improvement in strength. Increasing the temperature of the applied emulsion does not result in a more rapid curing rate; apparently, the heat capacity of the soil is sufficient to cool the emulsion quickly.

M-21 and NHS absorb only a small amount of emulsion and the resulting treated layer is extremely thin; on the other hand, if a larger amount of emulsion is added to cover the surface with a residue of asphalt, the rate of strength development is rather slow. In either case, the penetration resistances obtained after treatment of these soils with Duomeen T emulsions containing phosphoric acid are much lower than those obtained for Wisconsin Sand.

d. Water Erosion Resistance

Comparative conditions for the preparation, curing, and erosion of stabilized surfaces of M-21, New Hampshire Silt and Wisconsin Sand (1056) are given in Table VI-8. The types of erosion fell into two categories: complete disruption of the stabilized surface and washing out of the underlying soil, and gradual, almost negligible erosion of individual soil particles from the surface. The first type was more commonly observed at high water velocities, while the second type occurred very slowly at lower flow rates. Increasing the velocity of the impinging water spray generally resulted in surface failure rather than increasing the amount of material eroded.

Penetration resistance tests indicate that samples subjected to erosion and which did not fail lost most of their original strength when tested just after erosion but regained most of the strength after drying for a period of two days. Surfaces covered with a residue of asphalt did not erode gradually but failed completely when not cured for a sufficient length of time.

The treated surfaces of M-21 and New Hampshire Silt either failed completely earlier or eroded gradually at a more rapid rate than those of Wisconsin Sand, depending on whether the surface was or was not covered with a residue of asphalt. Dilution of the emulsion with increasing amounts of water before application resulted in increasing penetration resistance but decreasing erosion resistance for stabilized surfaces of Wisconsin Sand. When the surface of this soil is treated with cutbacks or emulsions not containing phosphoric acid, disruption of the surface occurs in very short order at high water velocities. Erosion resistance is directly related to length of curing of the stabilized layer if none of the surface is covered with a residue of asphalt. When the surface of the treated soil is partially covered with asphalt, so that the volatiles can escape from the surface, the erosion resistance of the soil is the greatest for any given phosphoric acid content and curing period.

3. Characteristics of Soils Stabilized by Mechanical Incorporation of Emulsions and Cutbacks

a. Stabilization of Massachusetts Clayey Silt (M-21)

The corporative stabilization of M-21 using Duomeen T-orthophosphoric acid emulsions of 100-120 penetration asphalt had been investigated prior to this year's research (Ref. 1). The best results obtained were compressive strengths of 105 ± 5 psi for 24 hours cure at 100 per cent RH and 1 day immersion, and of 305 psi for 6 days cure at 43 per cent RH and 1 day immersion, using 10 per cent asphalt and two per cent free ortho-phosphoric acid on the weight of the soil. Stabilization of M-21 with a two-to-one asphalt to gasoline cutback and addition of 1.5 per cent P_2O_5 to the soil, however, gave a strength of 325 psi for a 14 day 43 per cent RH cure and 7 days immersion.

Table VI-9 presents the results of M-21 stabilization using some of the emulsion preparations developed during the course of this year's work which proved effective in the topical treatment of soils. More stringent curing conditions were employed to test these samples than the 43 per cent relative humidity cure employed in previous years. One set of samples, stabilized by an emulsified cutback prepared with Duomeen T, hydrochloric acid and chromic chloride and containing 7.5 per cent asphalt and 1.5 per cent phosphoric acid on the weight of the soil (Series N) compared closely with the best results obtained in the past; this was by no means the best set of results obtained this year, however.

Comparison of the results presented in Table VI-9 demonstrates that: (1) the addition of phosphoric acid to these emulsions is necessary for adequate soil stabilization; asphalt alone is incapable of givine one day 100 per cent relative humidity, one day immersion strengths of better than 100 psi; (2) 100-120 penetration asphalt is a superior waterproofing agent to 40-50 penetration asphalt; (3) the asphalt to be emulsified must be diluted with an organic solvent (preferably gasoline) in the ratio of two parts of asphalt to one of solvent, to prevent the asphalt droplets from plating out too rapidly on the surface of the soil; (4) the emulsions prepared using Duomeen T-hydrochloric acid with chromic or ferric chloride as trace additives give superior ultimate strengths and result in less of a loss of strength on immerson than emulsions prepared using Nonic 218 or Duomeen T-phosphoric acid, or than emulsions lacking the trace additives; (5) the use of 7.5 and 10 per cent asphalt on the weight of the soil results in significantly lower strengths than obtained with 5 per cent asphalt; even though 60 and 65 per cent asphalt emulsions were employed to stabilize dry M-21, the water content obtained at these increased asphalt dosages was in excess of optimum and was partly responsible for the decrease in strength.

The best stabilization obtained for 2 per cent phosphoric acid and 4 per cent asphalt on the weight of the soil was 265 psi for a one day 100 per cent relative humidity cure and one day immersion, representing a loss of only 40 psi on immersion, and 455 psi for a 7 day 100 per cent relative humidity cure and one day immersion (Series K). For the extended curing period these are significantly better than the strengths obtained using a solution of phosphoric acid in water and a two-to-one asphalt to gasoline cutback, which were 255 psi and 305 psi for the same cure periods respectively (Series Q). Extreme difficulty was encountered in mixing the cutback with the soil; thirty minutes of mixing were required to obtain a soil of homogeneous appearance. This difficulty was encountered with none of the emulsified cutbacks. From a comparison of Series P and Series Q, the waterproofing effect of the Duomeen T can be observed in the relative losses of strength on immersion with and without this additive in the cutback.

b. Stabilization of Vicksburg Buckshot Clay (VBC)

In previous work (Ref. 1) the corporative stabilization of VBC using Duomeen T-ortho-phosphoric acid emulsions of 100-120 asphalt had given a 1 day 100 per cent relative humidity and 1 day immersion strength of 65 psi using 2 per cent phosphoric acid and 10 per cent asphalt on the weight of soil.

Table VI-10 presents the results of VBC stabilization using a Duomeen T-hydrochloric acid emulsion containing trace amounts of ferric chloride. Using these emulsified cutbacks, a compressive strength of 85 psi had been obtained with 10 per cent asphalt and 2 per cent phosphoric acid on the weight of the soil for a cure of 1 day at 100 per cent relative humidity and 1 day immersion, a result just slightly better than that obtained last year. Furthermore, it can be concluded from these results that (1) ten per cent asphalt on the weight of soil gives the best humid cure strength, while 12.5 per cent gives the best one day humid cure and one day immersion strength; and (2) both the humid cure and immersion strengths obtained with these emulsions are significantly better than those achieved with cutbacks and the same amount of phosphoric acid added separately, and at approximately the same soil water content.

D. Conclusions

1. The use of N-alkyl propylene diamines in conjunction with hydrochloric acid as emulsifying agent and including trace amounts of chromium (III) chloride or ferric chloride as complexing agents leads to the preparation of satisfactory cationic asphalt emulsions for topical treatment of soils. Nonic 218 serves as the emulsifying agent for successful nonionic formulations. In all cases it is necessary to dilute the asphalt to be emulsified with approximately half

as much gasoline or kerosene in order to make the emulsified droplets sufficiently deformable to pass through the pores of a fine sand.

2. These emulsions will penetrate the uncompacted surfaces of a fine sand and clayey silt to depths of one inch and 1/4 inch, respectively. The treated surface layer of the fine sand is remarkably flexible and cohesive; the asphalt is rather uniformly distributed throughout this layer. The best Nonic 218 emulsions will fill 18 per cent of the void volume of the soil, while the best Duomeen T-hydrochloric acid emulsions will fill 30 per cent of this volume.

3. If equal amounts are applied per unit area of exposed soil surface, these emulsions will penetrate soils to greater depths than cutbacks containing the same ratio of asphalt to gasoline, but, at low dosages, the average asphalt concentration in the penetrated layer will be greater for cutbacks. Increasing the dosage of emulsion per unit surface will result in a greater average asphalt concentration in the penetrated layer, even higher than that obtained with cutbacks. Blocking of soil pores by rapid deposition of asphalt droplets from the emulsions, however, limits the dosage of emulsion which will penetrate the soil. For cutbacks, the depth of penetration is directly proportional to the amount applied, the asphalt concentration in the treated layer remaining invariant.

4. As much as 10 per cent of ortho-phosphoric acid by weight of the emulsion may be incorporated into either of the aforementioned cationic or nonionic emulsion types without impairing their soil penetrating characteristics.

5. Satisfactory emulsions containing as much as 65 per cent asphalt phase by weight can be prepared using Duomeen T, hydrochloric acid and ferric chloride as emulsifying agents, the depths of penetration obtained in a fine sand being two-thirds that obtained for 50-50 asphalt-in-water emulsions prepared with the same materials.

6. Topically treated surfaces of fine sands stabilized with emulsions absorb much more water on immersion (25%) than those stabilized with cutbacks (5%). The bulk of this water uptake is probably contained in the untreated sublayer of the soil.

7. The per cent of the stabilized surface of a soil covered with residual asphalt that does not penetrate the soil governs the rate of water seepage through the surface.

8. Dilution with an equal weight of water of a cutback emulsion prepared with Nonic 218 and containing ten per cent phosphoric acid and application of 0.20 g. asphalt/sq. cm. with this emulsion to the surface of a silty sand results in the development of a surface

penetration resistance of 465 psi when cured at ambient conditions for thirty days.

9. Erosion of two kinds has been obtained: complete disruption of the stabilized surface and washing out of the underlying soil, and gradual but slow erosion of individual soil particles from the surface. When the surface of the treated soil is only partially covered with residual asphalt, permitting the volatiles to escape from the surface and allowing the surface to cure, the erosion resistance of the soil is greatest for any given phosphoric acid content and curing period.

10. Compressive strengths of 265 ± 10 psi for a one day 100% RH cure and one day immersion and of 455 psi for a seven day 100% RH cure and one day immersion were obtained by stabilizing M-21 with 2 per cent phosphoric acid and 4 per cent asphalt in the form of a cutback emulsion prepared with Duomeen T, hydrochloric acid and ferric chloride. For the same curing conditions, a solution of phosphoric acid in water and an asphalt cutback gave compressive strengths of 255 psi and 305 psi, respectively, significantly less than those for the best emulsion for the extended curing period.

11. A compressive strength of 85 psi has been obtained in the corporative stabilization of VBC with the aforementioned emulsified cutback; the asphalt and phosphoric acid concentrations were 10 and 2 per cent of the weight of the soil, respectively, and the samples referred to were cured at 100% RH for one day and immersed for another day. Both humid cure and immersion strengths using this emulsion exceed those obtained with a cutback and a solution of the same amount of phosphoric acid.

E. Recommendations

1. The study of the permeability of soils to water seepage should be extended to stabilized surfaces of Massachusetts Clayey Silt and Wisconsin Sand (1056) following improvement of the testing procedure. The permeability of the surface layer should be computed from the measured pressure heads and flow rates through the soil layer by maintaining a constant head of water on the soil and a continuous column of water through the soil undergoing the permeability test.

2. A systematic determination of the penetration resistance of the stabilized surfaces of a variety of soils for extended curing periods should be taken.

3. Modification of the present erosion apparatus is desirable so that the gradual erosion of stabilized soil surfaces can be determined by weighing a significant quantity of particles eroded from the surface.

F. References

1. Soil Stabilization by Chemical Methods, Final Report Phase XI, Soil Stabilization Laboratory, M.I.T., November, 1959

2. Becher, P., "Emulsions--Theory and Practice", Reinhold Publishing Corporation, New York, 1957

3. Moeller, T., "Inorganic Chemistry", John Wiley, New York, 1952

Table VI-1

EFFECT OF VARIATIONS IN EMULSIFICATION METHOD

	Emulsification Conditions				Particle Size Distribution (%)					
	Emulsifier Concentration (% Total Emulsion)	Excess Acid Concentration (% Total Emulsion	Rate of Mixing	Emulsification Technique*	Extent of Aggregation	< 1μ	1-2μ	2-4μ	4-10μ	>10μ
Duomeen T-orthophosphoric acid	2.5	1	Low	I	Low	9	29	42	17	3
				M	High	30	43	17	8	2
				A	Medium	30	47	15	7	1
	5	1	Low	I	Low	29	19	25	17	0
				M	Not fluid, completely aggregated					
				A	Not fluid, completely aggregated					
	2.5	2	Low	I	Low	20	41	17	15	7
				M	High	18	45	29	7	1
				A	High	18	33	31	18	0
	5	2	Low	I	Low	18	27	29	22	2 4
				M	Not fluid, completely aggregated					
				A	Semi-Fluid, very highly aggregated					
	2.5	1	High	I	Low	25	41	25	7	2
				M	Medium	62	24	10	4	0
				A	Medium	44	40	11	4	1
	5	1	High	I	Low	15	39	35	10	1
				M	Not fluid, completely aggregated					
				A	High	35	35	20	10	0
	2.5	2	High	I	Low	12	19	43	23	3
				M	High	67	19	6	7	1
				A	High	48	28	17	6	1
	5	2	High	I	Not fluid, completely aggregated					
				M	Not fluid, completely aggregated					
				A	Semi-fluid, highly aggregated					
	2.5	10	High	I	Low	27	26	29	15	3
	5			I	Semi-fluid, highly aggregated					
Duomeen T-orthorhombic P_2O_5	2.5	1	High	I	No emulsion					
		2		I	No emulsion					
		10		I		36	24	22	17	1
	5	1	High	I	No emulsion					
		2		I	Semi-fluid, highly aggregated					
		10		I		31	34	18	14	3

* I = Inversion
M = Mayonnaise
A = Alternate Addition

Ta

EFFECT OF VARIATION

AMOUNTS OF MATERIALS (in grams)	A	B	C	D	E	F	G	H	I	J	K	L	M
Water	119	119	115	115	115	115	115	115	119	119	119	119	119
Asphalt	83	83	83	83	83	83	125	42	125	83	100	115	125
Penetration No.**	100-120	40-50	100-120	40-50	100-120	40-50	100-120	100-120	100-120	100-120	100-120	100-120	100-120
Cutback Solvent													
Gasoline					42	42		83		42	25	10	
Kerosene	42	42	42	42									
Emulsifier													
Nonic 218	6.25	6.25							6.25	6.25	6.25	6.25	6.25
Duomeen T			5.00	5.00	5.00	5.00	5.00	500					
Duomeen S													
Rosin Amine D													
Acids													
M phosphoric (100%)													
O phosphoric (85%)													
Hydrochloric (35.6%)			4.70	4.70	4.70	4.70	4.70	4.70					
Acetic (100%)													
Additives			0.25 CrCl$_3$, 6H$_2$O	0.25 CrCl$_3$, 6H$_2$O	0.25 CrCl$_3$, 6H$_2$O	0.25 CrCl$_3$, 6H$_2$O	0.25 CrCl$_3$, 6H$_2$O						
EMULSIFICATION CONDITIONS													
Emulsification Technique**	M	M	I	I	I	I	I	I	M	M	I	M	I
Emulsifier Concentration (% of Total Emulsion)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Excess Acid Concentration (% of Total Emulsion)			0.5	0.5	0.5	0.5	0.5	0.5					
PENETRATION CHARACTERISTICS													
Type of Soil Treated	Sand	Sand	Sand	Sand	Sand/ M-21	Sand	Sand	Omitted	Sand	Sand	Sand	Sand	Sand
Max. Depth of Penetration (mm)	9	4	6	4	18/5	6	4		0.5	16	5	1.5	0.5
Max. Application Leaving No Surface Residue (g. asphalt/sq. cm. surface	0.15	0.18	0.10	0.10	0.20/ 0.10	0.10	0		0	0.10	0.05	0	0
% of Soil Void Vol. Filled with Asphalt	42	61	42	61	29/76	42	..	.	.	15	25	..	..
Wt. Asphalt on Wt. of Treated Soil Layer	0.11	0.16	0.11	0.16	0.076/ .152	0.11	.			.011	.065	..	..
PARTICLE SIZE DISTRIBUTION													
Less than 1 micron	Omitted	Omitted	Omitted	Omitted	50	17	41	Water in oil emulsion	Omitted	68	Highly aggregated	Omitted	3
1-2 microns					30	26	19			17			10
2-4 microns					11	34	26			13			15
4-10 microns					6	20	12			2			62
Greater than 10 microns					3	3	2			0			10

AMOUNTS OF MATERIALS (in grams)	N	O	P	Q	R	S	T	U	V	W	X	Y	Z
Water	119	112	119	119	119	115	115	115	115	115	115	115	115
Asphalt	125	125	83	42	83	83	83	83	83	83	83	83	83
Penetration No.**	100-120	100-120	100-120	100-120	100-120	100-120	100-120	100-120	100-120	100-120	100-120	100-120	100-
Cutback Solvent													
Gasoline			42	83	*42	42	42	42	42	42	42	42	42
Kerosene													
Emulsifier													
Nonic 218	6.25	12.5	6.25	6.25	6.25								
Duomeen T						5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Duomeen S													
Rosin Amine D													
Acids													
M phosphoric (100%)													
O phosphoric (85%)													
Hydrochloric (35.6%)						4.70	4.70	4.70	4.70	4.70	4.70	4.70	4.70
Acetic (100%)													
Additives					0.25 Methyl cellulose		0.25 AlCl$_3$, 6H$_2$O	0.25 CoCl$_2$, 6H$_2$O	0.25 NiCl$_2$, 6H$_2$O	0.25 ZnCl$_2$, 2H$_2$O	0.25 CaCl$_2$, 2H$_2$O		0.25 Cr 6H
EMULSIFICATION CONDITIONS													
Emulsification Technique**	M	I	M	I	M	I	I	I	I	I	I	M	M
Emulsifier Concentration (% of Total Emulsion)	2.5	5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Excess Acid Concentration (% of Total Emulsion)						0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
PENETRATION CHARACTERISTICS													
Type of Soil Treated	Sand	Omitted	Sand	Sand/ M-21	Sand	Sand/ M-21	Sand/ M-21	Sand	Sand	Sand	Sand	Sand	Sand
Max. Depth of Penetration (mm)	1		14	24/6	13	8/3	6/5	9	5	1	5	10	9
Max. Application Leaving No Surface Residue (g. asphalt/sq. cm. surface	0		0.18	0.20	0.10	0.18	0.10	0.10	0.10	0.05	0.10	0.10	0.10
% of Soil Void Vol. Filled with Asphalt	.		18	18/64	19	31/100	31/76	50	50	42	50	25	48
Wt. Asphalt on Wt. of Treated Soil Layer		.	.047	.058/ 0.11	.051	.063/ .22	.063/ .132	.132	.132	.11	.132	.066	.074
PARTICLE SIZE DISTRIBUTION													
Less than 1 micron	11	Highly aggregated	12	Max. ca. 2-4 microns	Omitted	12	51	17	16	Highly Aggregated	22	42	Omitted
1-2 microns	44		20			25	14	41	59		16	35	
2-4 microns	30		29			18	24	29	14		28	14	
4-10 microns	11		18			20	9	12	9		12	5	
Greater than 10 microns	4		1			5	2	1	2		2	4	

* All experiments were carried out at a high mixing rate.

** I refers to the inversion technique, M to the mayonnaise technique.

1

Table VI-2

EFFECT OF VARIATION IN EMULSIFYING MATERIALS[a]

	T	U	V	W	X	Y	Z	AA	BB	CC	DD	EE	FF	GG	HH	II	JJ	KK	LL	MM	NN	OO	PP	QQ	RR	SS	TT	UU	VV	WW	XX	YY	ZZ
-120	115 83 100-120	115 83 100-120	115 83 100-120	115 83 100-120	115 83 100-120	115 83 100-120	115 81 100-120	115 81 100-120	115 83 100-120	115 81 100-120	97 81 100-120	115 81 100-120	115 81 100-120	85 83 100-120	117 83 100-120	117 83 100-120	94 100 100-120	90 81 100-120	117 83 100-120	94 83 100-120	194 81 100-120	115 83 100-120	110 83 100-120	119 83 100-120	119 62 100-120	119 83 100-120	119 83 100-120	119 125 100-120	119 100 100-120	119 100 100-120	82 100 100-120	244 66 100-120	94 100 100-120
	42	42	42	42	42	42	42	42	42	42	42	42	42	42	42	42	25	42	42	42	42	42	42	62	62	42	42		25[1]	25[2]	54	33	50
0	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	20.0	5.00	5.00	5.00	5.40	5.40	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
																	26.25	30.25	2.88	26.0	26.0												
0	4.70	4.70	4.70	4.70	4.70	4.70	4.70	4.70	4.70	4.70	8.40	4.70	4.70	35.4	2.10	2.10						4.7	10.34	4.70	4.70	2.55[3]	4.70	4.70	4.70	4.70	4.70	4.70	4.70
	0.25 $AlCl_3$- $6H_2O$	0.25 $CuCl_2$- $2H_2O$	0.25 $NiCl_2$- $2H_2O$	0.25 $ZnCl_2$- $2H_2O$	0.25 $CaCl_2$- $2H_2O$		0.25 $CrCl_3$- $6H_2O$	0.25 $CoCl_2$- $6H_2O$	0.25 $CdCl_2$- $2.5H_2O$	1.25 $CrCl_3$- $6H_2O$	1.00 $CrCl_3$- $6H_2O$	0.25 $CrCl_3$- $6H_2O$ 0.25 Methyl Cellulose	0.25 $CrCl_3$- $6H_2O$ 0.25 Cetyl Alcohol									0.15 $CrCl_3$- $6H_2O$	0.15 $CrCl_3$- $6H_2O$	0.04 $CrCl_3$- $6H_2O$	0.1 $CrCl_3$- $6H_2O$		0.1 $FeCl_3$- $6H_2O$	0.1 $CrCl_3$- $6H_2O$	0.1 $CrCl_3$- $6H_2O$	0.1 $CrCl_3$- $6H_2O$	0.1 $CrCl_3$- $6H_2O$	0.1 $FeCl_3$- $6H_2O$	0.1 $CrCl_3$- $6H_2O$
	1	1	1	1	1	M	M	1	1	1	1	1	1	1	1	M	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	10	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	1.5	2.5	1.0	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	5	0.5	0.5	10	10	0.5	10	10	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
nd/ -21	Sand/ M-21	Sand	Sand	Sand	Sand	Sand	Sand	Sand	Sand	Sand	Sand	Sand	Sand	Omitted	Sand	Omitted	Sand	Sand	Sand	Omitted	Omitted	Sand	Omitted	Sand	Sand	Water-in-oil emulsion	Sand	Sand	Sand	Omitted	Sand	Omitted	Omitted
3	8/5	5	5	1	5	10	9	5	6	6	5	8	7		2		4	6	7			9		18	23		21	1	11		16		
10	0.10	0.10	0.10	0.05	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10		0.05		0.05	0.10	0.10			0.10		0.20	0.20		0.20	0.20	0.15		0.20		
/100	91/76	50	50	42	50	25	28	50	42	42	50	11	17		53		11	42	17			28		29	18		22	100	15		31		
3/ 2	.083/ .112	.132	.112	.11	.132	.066	.074	.112	.11	.11	.132	.081	.095		.167	- -	.083	.110	.095		.	.074		.076	.058		.066		.08		.088		
	81 14 24 9 2	17 41 29 12 1	16 39 34 9 2	Highly Aggregated	22 36 28 12 2	42 35 14 5 4	Omitted	Omitted	Omitted	32 45 16 7 2	Omitted	Omitted	Omitted	Water-in-oil emulsion	12 17 14 61 14	Highly aggregated excessive foaming	Omitted	Omitted	Omitted	Fine water-in-oil emulsion	Water in oil emulsion	Omitted	No emulsion										

[1] Xylene

[2] Carbon Disulfide

[3] Sulfuric (98%)

2

TABLE VI-3

SUMMARY AND CODING OF PROMISING EMULSIONS FOR TOPICAL APPLICATION TO SOILS

Emulsion No. (See Table VI-2)	Code Name	Emulsifying Agents	Asphalt Penetration No.	Cutback Solvent	Wt. Ratio Asphalt Solvent Water	Additives
E	Chromium-Conc.	Duomeen T-HCl	100-120	Gasoline	2-1-3	$CrCl_3 \cdot 6H_2O$
F	Low Penetration	Duomeen T-HCl	40-50	Gasoline	2-1-3	$CrCl_3 \cdot 6H_2O$
J	Nonic-Conc.	Nonic 218	100-120	Gasoline	2-1-3	--
Q	Nonic-Dilute	Nonic 218	100-120	Gasoline	1-2-3	--
S	HCl	Duomeen T-HCl	100-120	Gasoline	2-1-3	--
T	Aluminum	Duomeen T-HCl	100-120	Gasoline	2-1-3	$AlCl_3 \cdot 6H_2O$
RR	Chromium-Dilute	Duomeen T-HCl	100-120	Gasoline	1-1-2	$CrCl_3 \cdot 6H_2O$
TT	Ferric	Duomeen T-HCl	100-120	Gasoline	2-1-3	$FeCl_3 \cdot 6H_2O$
UU	No solvent	Duomeen T-HCl	100-120	None	3-0-3	$CrCl_3 \cdot 6H_2O$
VV	Xylene	Duomeen T-HCl	100-120	Xylene	2-0.5-2.5	$CrCl_3 \cdot 6H_2O$
XX	High Asphalt-1	Duomeen T-HCl	100-120	Gasoline	2-1-1.6	$CrCl_3 \cdot 6H_2O$
ZZ	High Asphalt-2	Duomeen T-HCl	100-120	Gasoline	2-1-2	$CrCl_3 \cdot 6H_2O$

TABLE VI-4

PENETRATION OF TOPICALLY APPLIED ASPHALT EMULSIONS AND CUTBACKS

Type of Soil	Emulsion or Cutback Applied	Amount Asphalt Applied (g/cm^2 surface) 0.10		o.20	
		(mm) Depth Penetration	% of Surface with asphalt residue	(mm) Depth Penetration	% of Surface with asphalt residue
80-150 Mesh Ottawa Sand	100-120 penetration asphalt:				
	Cutback: 2A/1G[1]	5	0	8	0
	1A/1G	8	0	18	0
	Emulsion: Chromium Conc.	14	0	18	20
	Nonic Conc.	16	0	--	--
	Nonic Dilute	--	-	24	5
	Chromium Dilute	--	-	23	10
	Ferric	--	-	21	5
	40-50 penetration asphalt:				
	Cutback: 1A/1G	10	0	15	10
	Emulsion:				
	Low Penetration	6	5	--	--
M-21	100-120 penetration asphalt:				
	Cutback: 2A/1G	1	100	--	--
	1A/1G	4	20	6	100
	Emulsion:				
	Chromium Conc.	4	100	--	--
	Nonic Dilute	6	80	--	--
	Ferric	2	100	--	--
NHS	100-120 penetration asphalt:				
	Emulsion:				
	Chromium Conc.	2	90	--	--
	Ferric	2	90	--	--
1056	100-120 penetration asphalt:				
	Cutback: 2A/1G	-	--	9	5
	Emulsion: Chromium Conc.[2]	7	5	--	--
	Ferric[2]	5	0	--	--
	Nonic Conc[2]	6	5	8	100
	Nonic Conc.[3]	10	10	--	--

[1] A refers to the weight of asphalt, G to that of gasoline

[2] Diluted with an equal weight of water

[3] Diluted with twice its weight of water

TABLE VI-5

WATER ABSORPTION OF STABILIZED SAND SURFACES

(80-150 mesh Ottawa sand layers treated with emulsion or cutback and immersed in water)

Emulsion or Cutback[1]		Application: g. Asphalt/ sq. cm. of Surface	Per cent Water Absorption on Weight of Sand	Hours to Reach Maximum Water Uptake
100-120 penetration asphalt:				
Cutback:	2A/ 1G[1]	0.10	5.3	8
	2A/ 1G	0.20	1.5	10
	1A/ 1G	0.10	6.8	6
	1A/ 1G	0.20	6.1	7
Emulsion:				
	Aluminum	0.10	23.5	6
	Chromium Conc.	0.10	21.9	6
	Nonic Dilute	0.20	24.5	4
	HCl	0.20	29.4	5

---

[1] A refers to the weight of asphalt, G to that of gasoline

TABLE VI-6

PERMEABILITY OF STABILIZED SURFACES TO WATER SEEPAGE (NHS)

Emulsion or Cutback[1]	Application g. Asphalt/ sq. cm. of Surface	Curing Period Before Test Hours	Permeability ml/min		Comment
			1st hr	Final	
Cutback[2]					
2A/1G	0.20	6	0	0	Surface 90% coated with asphalt
1A/1G	0.20	6	0.16	0	Surface 50% coated with asphalt
Emulsion					
Nonic Dilute	0.10	15	1.25	0.9	Channeling in soil; no surface residue of asphalt
Chromic Conc.	0.10	15	4.0	1.0	Channeling in soil; no surface residue of asphalt
Aluminum	0.10	15	0.16	0.11	Surface 80% coated with asphalt
HCl	0.10	15	13		Surface 50% coated with asphalt
No stabilizer	0	--	16	0.4	

---

[1] These emulsions were not mixed with H_3PO_4 before application

[2] A refers to the weight of asphalt, G to that of gasoline

TABLE VI-7

PENETRATION RESISTANCE OF STABILIZED SOIL SURFACES

Soil Type	Emulsion or Cutback 100-120 Penetration Asphalt	Application g. Asphalt/ sq. cm. Surface	Temp. of Application °C	% H_3PO_4 on Wt. of Emulsion	Dilution Ratio: Emulsion/ Water	Penetration Strength - psi			
						Curing Period - Days			
						1	2	10	30
1056	Chromium Conc.	0.10	23	0	--		22	38	
		0.10	23	10	--		16	69	
		0.10	23	10	1:1		82	124	
		0.10	57	10	1:1			54	
	Ferric	0.10	23	0	--		13	31	
		0.10	23	10	--		20	43	
		0.10	23	10	1:1	50	57	163	
	Nonic-Conc.	0.10	23	0	--		33+29	35	
		0.10	23	10	--		22	48	
		0.10	23	10	1:1		91[1]	247	[2]
		0.20	23	10	1:1	0		123	465
		0.10	54	10	1:1		[3]		
		0.10	23	10	1:2	54			
		0.10	23	20	1:1		94		
	Cutback 2A/ 1G	0.20	23	0	--		33		
	H_3PO_4 alone, equivalent to 20% on 0.10 g. asphalt/ cm^2+ 60g H_2O						80		
NHS	Chromium Conc	0.10	55	10	1:1			33	
	Ferric	0.10	48	10	1:1			51	
M-21	Ferric	0.10	49	10				[4]	

[1] 3 days - 87 psi

[2] 14 days - 145 psi; 21 days - 189 psi; 28 days - 225 psi

[3] 6 days - 175 psi

[4] 19 days - 62 psi

TABLE VI-8

EROSION RESISTANCE OF ASPHALT STABILIZED SOIL SURFACES

* Comparison based on variations in these quantities

	Application Data: (at 23°C)							Penetration Resistance, psi			Erosion Data			
Soil Type	Emulsion or Cutback 100-120 Penetration Asphalt Code Names	Amount-g Asphalt/ sq. cm. of Surface	% H_3PO_4 on Wt. of Emulsion	Dilution Ratio: Emulsion to Water	% of Surface Coated With Asphalt	Depth of Pene-tration mm.	Curing Time, Hours	Before Test	After Test	Two Days After Test	Water Flow Rate cc/sec.	Water Temp. °C	Erosion Time Min.	Comment
NHS*	Chromium Conc.	0.10	10	1:1	60	2	209	33	0	--	151	8	7h.	1.0 g. eroded
1056*	Chromium Conc.	0.10	10	1:1	5	7	229	54	47	--	159	8	23 h.	No erosion
NHS*	Ferric	0.10	10	1:1	80	2	211*	51	0	--	232	8	30.5 h.	0.1 g. eroded
M-21*	Ferric	0.10	10	1:1	100	2	461*	62	51	--	470	9	4.5 h.	No erosion
1056*	Ferric	0.10	10	1:1	5	5	24*	51	0	--	465	10	60	Gross failure
1056	Nonic-Conc.	0.10	10	1:0.5*	2	6	48	83	33	44	470	14	60	0 g, cracked surface
1056	Nonic-Conc.	0.10	10	1:1*	2	6	65	87	44	58	515	13	60	0.1 g. eroded
1056	Nonic-Conc.	0.10	10	1:2*	10	10	24	54	0	--	500	12	35	Gross failure
1056	Nonic-Conc.	0.20	5*	1:1	100	7	23	0	0	--	500	10	1	Gross failure
1056	Nonic-Conc.	0.20	10*	1:1	100	6	23	0	0	--	500	10	1	Gross failure
1056	Nonic-Conc.	0.10*	20*	1:1	5	8	48	94	40	75	470	14	60	0g cracked
1056	Nonic-Conc.	0.10*	10	1:1	2*	6	65	87	44	58	515	13	60	0.1 g. eroded
1056	Nonic-Conc.	0.15*	10	1:1	20*	7	68	65	36	51	480	13	60	0 g. eroded
1056	Nonic-Conc.	0.20*	10	1:1	100*	6	23	0	0	--	500	10	1	Gross failure
1056	Nonic-Conc.*	0.10*	20*	1:1	5	8	48	94	40	75	470	14	60	0 g cracked
1056	6g H_3PO_4*, equiv. to 20% on 0.1g asphalt/sq. cm.			60g H_2O	--	-	56	87	0	--	500	14	1	Gross failure
1056	Nonic-conc.	0.10	0*	1:1	10	9	56	29	0	--	500	14	1/2	Gross failure
1056	Nonic-Conc.	0.10*	0	1:1	10	9	56	29	0	--	500	14	1/2	Gross failure
1056	Cutback 2A/1G	0.20*	0	--	0	7	58	33	33	--	500	14	40 h.	Gross failure

TABLE VI-9

CORPORATIVE STABILIZATION OF MASSACHUSETTS

CLAYEY SILT WITH ASPHALT EMULSIONS AND CUTBACKS[1]

					CURING CONDITIONS									
					1 Day 100% RH			1 Day 100% RH, 1 Day Immersion			7 Days 100% RH, 1 Day Immersion			
Series No.	Emulsion No.[5]	Cutback or Emulsion Code Names	% Asphalt on Wt. of Soil	% H_3PO_4 on Wt. of Soil	Volatiles Content[4] %	Dry Density lb/ft³	Compressive Strength[2] psi	Volatiles Content[4] %	Dry Density lb/ft³	Compressive Strength psi	Volatiles Content[4] %	Dry Density lb/ft³	Compressive Strength psi
A	E	Chromium Conc.	5	0	6.7	127	95	8.0	127	50	7.6	127	75
B	E	Chromium Conc.	5	1.5	7.4	126	195	7.8	126	190	7.2	126	255
C	TT	Ferric	5	1.5	7.4	126	215 ± 20	7.8	125	195 ± 10	7.2	125	270 ± 10
D	J	Nonic-Conc.	5	1.5	7.2	128	155 ± 10	8.7	128	120	7.8	127	145 ± 10
E	F	Low Penetration	5	1.5	7.9	124	205	7.8	123	165	7.7	123	185 ± 10
F	UU	No Solvent	5	1.5	6.9	122	165	7.7	122	110	7.3	122	140
G	S	HCl	5	1.5	7.4	125	170	8.0	125	125	8.0	125	155
H	VV	Xylene	5	1.5	9.2	119	80	10.1	118	60	9.7	118	80
I	E	Chromium Conc.	5	2	8.1	126	205	8.3	126	190 ± 15	8.1	127	275 ± 10
J	TT	Ferric	4	5	7.8	128	275 ± 10	8.3	128	610 ± 20	8.0	129	870 ± 60
K	TT	Ferric	4	2	6.8	129	305 ± 30	7.0	130	265 ± 10	6.8	131	455
L	TT	Ferric	3	2	5.8	128	235 ± 25	6.4	128	155 ± 10	6.0	127	360 ± 50
M	VV	Xylene	7.5	1.5	9.2	119	80	10.1	118	60	9.7	118	80
N[3]	ZZ	High Asphalt-2	7.5	1.5	7.5	123	125	7.6	123	125	7.5	123	195
O	XX	High Asphalt-1	10	1.5	8.7	116	40	9.0	116	35	8.7	116	70
P		Cutbacks: 2-1-0 + 3 pts H_2O added separately	5	2	7.8	126	225	8.3	126	165	7.9	127	305
Q		2-1-0 + 3 pts H_2O added separately	5	2	9.1	124	260 ± 15	9.4	123	255 ± 10	9.6	123	305 ± 20

---

[1] Samples compacted at 1000 psi

[2] The error is ± 5 or less in these strengths, unless otherwise specified.

[3] A set of samples in this batch cured for 6 days at 43% RH and immersed for one day had a compressive strength of 105 psi.

[4] Weight per cent based on dry solids plus volatiles

[5] See Table VI-2

TABLE VI-10

CORPORATIVE STABILIZATION OF VICKSBURG

BUCKSHOT CLAY WITH ASPHALT EMULSIONS AND CUTBACKS[1]

EMULSION OR CUTBACK DESCRIPTION:
100-120 penetration asphalt

Emulsion No.	Emulsifier	Cutback Solvent	Water Content at Molding, % [4]	Wt. Ratio Emulsion, Asphalt - Solvent - Water	% Asphalt on Wt. of Soil	% H_3PO_4 on Wt. of Soil	CURING CONDITIONS: 1 Day 100%RH Volatiles Content [3] %	1 Day 100%RH Dry Density lb/ft³	1 Day 100%RH Compressive Strength psi	1 Day 100% RH 1 Day Immersion Volatiles Content [3] %	1 Day 100% RH 1 Day Immersion Dry Density lb/ft³	1 Day 100% RH 1 Day Immersion Compressive Strength psi	7 Days 100% RH 1 Day Immersion Volatiles Content [3] %	7 Days 100% RH 1 Day Immersion Dry Density lb/ft³	7 Days 100% RH 1 Day Immersion Compressive Strength psi
TT	Duomeen T, HCl-$FeCl_3$	Gasoline	17.5	2-1-3	5	2	22.3	89	55	23.3	87	25	22.4	90	35
TT	Duomeen T, HCl-$FeCl_3$	Gasoline	15.0	2-1-3	5	2	17.3	99.5	140	19.5	98	50	18.3	99.5	85
TT	Duomeen T, HCl-$FeCl_3$	Gasoline	12.5	2-1-3	5	2	15.6	104.5	220 ± 10	18.8	100.5	35	17.3	101	60
TT	Duomeen T, HCl-$FeCl_3$	Gasoline	10.0	2-1-3	5	2	14.1	107	205	21.3	91	15	17.0	101	60
TT	Duomeen T, HCl-$FeCl_3$	Gasoline	15.0	2-1-3	10	2	15.0	101	180 ± 10	17.0	98	85	--	101.5	135
TT	Duomeen T, HCl-$FeCl_3$	Gasoline	18.7	2-1-3	12.5	2	16.6	96	120 ± 10	17.8	94	95	17.5	95	115 ± 15
	Cutback	Gasoline	15.0	2-1-0 + 3 pts H_2O added separately	10	2	17.1	96	85	18.8	95	40	18.4	95	65

---

[1] Samples compacted at 1000 psi

[2] The error is ± 5 or less in these strengths, unless otherwise specified

[3] Weight per cent based on dry solids and volatiles

[4] Total water content at time of molding based on dry soil weight