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# Foreword

This RECORD brings together a group of reports of interest to all proponents of soil-cement, soil-lime and soil-bituminous stabilization. Ranging in context from the practical to the basic aspects of soil stabilization, these papers should be of specific interest to personnel of materials, design and construction.

Under freeze-thaw exposure, the strength loss of a lime-stabilized clay was examined by Walker, Esmer, and Krebs. Most strength loss occurred during the first freeze-thaw cycle with those specimens having a "moisture treatment" prior to F-T testing showing the greatest loss. Strength of all lime-stabilized specimens following freeze-thaw was greater than that of the nonstabilized specimens, however. In addition, freezing and thawing had little effect on the porosity of the lime-treated specimens.

Swanson and Thompson evaluated the general flexural fatigue response of four soil-lime mixtures to determine if the response to fatigue would limit the use of these materials in subbase and base course applications. The fatigue response, represented by a linear regression line on a scatter diagram of stress level versus log of the number of cycles to failure, was typical of general fatigue and of similar materials. However, there were significant differences between each soil-lime mixture, and it was concluded that the fatigue responses of all such mixtures are not equivalent. Further studies were discussed and recommended.

A quick laboratory procedure for reduction of testing time in determination of minimum cement content for use in soil-cement pavements is presented by Kemahlioglu, Higgins and Adam. Termed the "Louisiana Slope Value Method," it makes maximum use of AASHO procedures for wet-dry testing and PCA weight loss criteria. Minimum cement requirement is determined from a relationship between durability and unconfined compressive strength of a soil-cement mixture at several selected cement contents. The procedure has been extensively tested and compared with the wet-dry test. Correlation curves and tables are presented for simplified use of the method.

Arman and Saifan present data, based on more than 2000 specimentests, that compressive strength, density and durability of soil-cement mixtures are decreased considerably by delay of compaction. Compaction delay of four hours, following mixing, resulted in a 62 percent reduction of compressive strength. The major loss of strength, density and durability occurred after a delay of only two hours, however. Fluctuation in cement and moisture content of the mixes, both below and above optimum, did not affect the losses. The authors present recommendations for compensation of these losses.

Noble presents information relative to a better understanding of the reactions, and rates of reactions, in mixtures of Types I and II portland cement and relatively pure kaolinite, illite and montmorillonite clays. Based on the amount of  $Ca(OH)_a$  produced, the cement-kaolinite mixtures experienced the most rapid hydration, with the cement-illite mixes hydrating one-half to three-fourths, and the cement-montmorillonite mixes hydrating one-tenth to one-fifth as rapidly. A concept of the mechanism of strength gain of the cement-clay mixtures, as based on early flocculation of the clay particles, hydration of the cement, possible chemical combination with elements of the clays, and crystallization of  $Ca(OH)_a$ , is presented. The restriction of hydration of cement grains due to fine-grained clay

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encapsulation of the cement and crystallized gels of calcium silicate hydrate is discussed.

Uppal reports on the effect of three cationic surface active (antistripping) agents on the waterproofing properties of cutback asphalt used in soil stabilization. Paraffin wax and a rubber solution were also used in the study for comparative purposes. Each of the cationics were found to enhance the waterproofing properties of the bitumen, though an optimum quantity of chemical and optimum proportion of cutback to soil produced the most effective results. Based on observations of water absorption and wet compressive strength, treatment with 3 percent cutback and 0.015 to 0.03 percent cationics, by dry soil weight, compared favorably with 4 to 5 percent cutback only. It was observed that paraffin wax was the most effective of all additives.

A discussion of Uppal's paper is presented by Winterkorn, with added comments on the theoretical knowledge of bituminous soil stabilization developed within the past forty years.

-James M. Hoover

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# Strength Loss in Lime-Stabilized Clay Soils When Moistened and Exposed to Freezing and Thawing

RICHARD D. WALKER, Virginia Polytechnic Institute, ERKAN ESMER, Virginia Highway Research Council, and ROBERT D. KREBS, Virginia Polytechnic Institute

> A total of 216 specimens of an A-7-5 silty clay soil were compacted with Harvard miniature apparatus with specimens divided between two test procedures (open and closed) and 2 levels of lime addition (0 to 10 percent). Curing was at 120 F for 2 days while sealed in aluminum foil.

> Half of the 108 open-system specimens were placed in a 70 F, 100 percent humidity environment for 5 days before testing. All open-system specimens were allowed to absorb moisture while freezing at 0 F for 12 hr and thawing in the 70 F, 100 percent humidity environment for 12 hr. Unconfined compressive strength, moisture absorption, length changes, degrees of saturation, and porosities were determined at zero, 1, 3, or 5 cycles of freezing and thawing.

Half of the closed-system specimens were soaked in water for 24 hr before testing. All specimens were frozen and thawed 12 hr each under the same conditions as the opensystem specimens except for being sealed in aluminum foil. Results were obtained at zero, 1, 5, or 20 cycles. Results of the study indicated that (a) the strengths of all lime-stabilized specimens, whether or not exposed to freezing and thawing, are several times greater than the strengths of nonstabilized specimens; (b) moisture treatments prior to freezing and thawing greatly increased strength losses after freezing and thawing; (c) most strength loss occurred during the first cycle of freezing and thawing; and (d) freezing and thawing seemed to have little, if any, effect on porosity.

•IN Virginia, hydrated lime is being used extensively to improve plastic clay subgrade materials under primary highways. These improvements may permit reduction in base thicknesses of highways, resulting in large monetary savings. However, even though definite improvements result from adding lime to the clay soils, one hesitates to actually decrease base thicknesses from normal design values because of a lack of knowledge as to possible loss of improvement when the stabilized soils are exposed to field moisture conditions and such weathering agencies as freezing and thawing.

The effect of freezing and thawing on unconfined compressive strength of lime-stabilized soils was discussed by Walker and Karabulut (1) in 1964. It was shown that stabilized clay soils may lose strength on exposure to freezing and thawing, perhaps due to hydraulic pressures as described by Powers' hypothesis (2) for concrete.

The purpose of this discussion is to present results of additional freeze-thaw tests conducted with and without pretreatment of samples with moisture, extending the 1964

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study previously mentioned, and to describe briefly the current work being conducted at Virginia Polytechnic Institute aimed at measuring changes in pore characteristics in lime-stabilized soils.

# FREEZING AND THAWING TESTS

Since the work reported in 1964 dealt only with closed-system testing, it was decided to use both open and closed-system tests in order to determine how the soil actually behaves. In an open system, the specimen is free to take on or lose water; in a closed system it is prevented from gaining or losing moisture. The advantage of using a closed-system test is its ease of operation. If a relatively impermeable soil is exposed to an open-system test, the results should be similar to those obtained from a closed test, since little water would be gained or lost in either case.

# **Open-System Test**

A total of 108 specimens were subjected to the open-system test, 54 having zero percent lime and 54 with 10 percent lime by dry weight of soil. Half of each group of specimens were placed in a 70 F, 100 percent humidity environment for 5 days before being subjected to freezing and thawing (hereafter referred to as the 5-day moist-room treatment). The rest were subjected to freezing and thawing immediately after curing. All specimens were set on a porous stone in a water bath while freezing in a home-type deep freeze unit (zero F environment) and while thawing in the 70 F, 100 percent humidity environment. Both the freezing and thawing periods were 12 hr. Strength determinations were made after zero, 1, 3, or 5 cycles of freezing and thawing. Moisture and height measurements were determined for each specimen prior to strength testing.

# **Closed-System Test**

A like number of specimens were prepared for the closed-system tests having zero and 10 percent lime content by dry weight of soil. Half of the specimens were soaked in water for 24 hr and then wrapped in aluminum foil and sealed with paraffin prior to exposure to freezing and thawing. The remaining specimens, also wrapped in foil and sealed with paraffin, were exposed to freezing and thawing immediately after curing. The same 12-hr freeze and 12-hr thaw cycle was used as with the open-system testonly the porous stone and water bath were omitted. The closed-system specimens were tested for unconfined compressive strength after zero, 1, 5, and 20 cycles of freezing and thawing. The same measurements were made as for the open-system specimens.

### MATERIALS

# Soil

The clay soil used for this study was obtained from an excavation near the coliseum on the Virginia Polytechnic Institute campus, Blacksburg, Virginia. The reddishbrown soil had a liquid limit (LL) of 61 percent, a plastic limit (PL) of 30 percent, and a plasticity index (PI) of 31 percent. The soil was classified according to the AASHO classification system as A-7-5. Standard AASHO density was 96 pcf at an optimum moisture content of 27 percent.

# Lime

The hydrated lime used was manufactured by the Gibsonburg Lime Products Company, Gibson, Ohio. It contained 5 percent calcium cabronate, as determined by drying a sample in an oven at 900 C for 3 hr.

# Soil-Lime Mixture

When 10 percent lime by dry weight of soil was added, the LL dropped from 61 to 59 percent, the PL increased from 30 to 48 percent, the PI dropped from 31 to 11 per-

cent, standard AASHO density dropped from 96 to 84 pcf and the optimum moisture increased from 27 to 29 percent.

# PREPARATION OF SAMPLES

The air-dried soil was moist cured in a plastic bag for 24 hr and then compacted with a Harvard miniature apparatus at approximately optimum moisture content. Compaction was in 3 layers using the Harvard plunger apparatus loaded with a 40-lb spring, applying 25 blows to each layer. For the soil used, this compaction procedure produces densities closely approximating those obtained with the standard AASHO procedure.

Three specimens were produced per batch. After weight and volume determinations, the specimens were immediately wrapped in aluminum foil and sealed with wax to preserve their moisture content while curing. One specimen from each batch was not subjected to freezing and thawing, permitting the establishment of zero-cycle strength values.

Curing was accomplished by placing the sealed specimens in an oven at 120 F for 48 hr. Previous investigations by Anday (3) showed that this is approximately equivalent to 45 days of simulated field curing near Charlottesville, Virginia.

# RESULTS AND DISCUSSION

# Effect on Unconfined Compressive Strength

Tables 1 and 2 summarize the unconfined compressive strength results for the closed-system and open-system tests. Several points may be noted:

1. Strengths of all lime-stabilized specimens, whether or not exposed to freezing and thawing, are several times greater than the strengths of nonstabilized specimens.

2. The test employed was highly destructive to un-limed soil, with only that soil protected from moisture before and during freezing surviving with a significant amount of its original strength.

3. Resistance of lime-stabilized specimens to the test conditions depended to a greater extent on whether or not they were exposed to moisture prior to freezing than whether or not the test was open system or closed system in its design.

4. For the closed-system test, one cycle of freezing and thawing appeared to produce most, if not all, of the damage observed.

Test Condition	No. of Specimens	Cycles of Freezing and Thawing	Unconfined Compressive Strength (psi)	Strength Loss (percent) <sup>2</sup>
0\$ lime,	9	0	58	_
no soaking	6	1	41	36
	6	5	34	45
	6	20	25	39
10\$ lime,	9	0	323	-
no soaking	6	1	232	29
-	6	5	276	18
	6	20	223	25
10\$ lime.	9	0	340	
1-day soaking	6	1	119	61
	6	5	183	50
	6	20	131	61
0\$ lime, 1-day soaking	All specime	ns disintegrated	upon immersion	in water

TABLE 1 EFFECT OF CLOSED-SYSTEM FREEZING AND THAWING ON UNCONFINED COMPRESSIVE STRENGTH

<sup>2</sup>Percent strength losses were calculated on a batch basis and then averaged.

1	L		
7	z		
-	-		
	9	4	4

TABLE 2 EFFECT OF OPEN-SYSTEM FREEZING AND THAWING ON UNCONFINED COMPRESSIVE STRENGTH

Test Condition	No. of Specimens	Cycles of Freezing and Thawing	Unconfined Compressive Strength	Strength Loss (percent) <sup>2</sup>
			tpst/	
0 <sup>\$</sup> lime,	9	0	63	
no moist-	6	1	14	79 <sup>b</sup>
room treatment	6	3	2	97
	6	5	1	98
10≸ lime.	9	0	305	_
no moist-	6	1	209	24
	6	3	129	52
	6	5	225	37
0≸ lime.	9	0	54	-
5-dav	6	1	5	92
moist-room	6	3	- 1	99
treatment	6	5	1	98
10\$ lime.	9	0	264	_
5-day	6	1	177	45
moist-room	6	3	44	81
treatment	6	5	60	76

<sup>a</sup>Percent strength losses were calculated on a batch basis and then averaged.

bOf four samples compacted at 24 percent moisture, three lost 64 percent and one lost 91 percent; and of two samples compacted at 26 percent moisture, both lost 94 to 95 percent.

5. For open-system testing, samples with 10 percent lime showed higher strengths after 5 cycles of freezing and thawing than after 3 cycles. The data presented here are preliminary and not conclusive on this point, but this anomaly has since been observed to appear consistently in similar tests with other heavily limed clay soils in this laboratory.

Table 3 indicates other interesting comparisons. In all cases, percent strength losses are considerably lower for the stabilized specimens as compared to the nonstabilized specimens. Perhaps of more importance is that with but one exception, the strength of stabilized specimens, as a factor of nonstabilized specimen strength, did not significantly change with increasing cycles of freezing and thawing. The stabilized open-system specimens that received no moisture treatment apparently were able to withstand the additional cycles of freezing better than the moisture-treated open-system specimens.

STABILIZED AND NONSTABILIZED SPECIMENS					
Test	Cycles of	Percent Strength Loss		Strength <sup>2</sup> as Factor of Non-	
Condition	Thawing	Nonstabilized	Stabilized	stabilized Strength	
Closed system.	1	36	29	5.7	
no soaking	5	45	18	8.3	
	20	39	25	9.1	
Open system,	1	79	24	15.0	
no moist-room	3	97	52	75.0	
treatment	5	98	37	204.0	
Open system,	1	92	45	39.4	
5-day moist-	3	99	81	68.0	
room treatment	5	98	76	50.0	

TABLE 3 COMPARISON OF UNCONFINED COMPRESSIVE STRENGTHS FOR

<sup>2</sup>Calculated on a batch basis and then averaged.

Test Condition	Cycles of Freezing and Thawing	Average Porosity (percent) <sup>2</sup>	Degree of Saturation (percent) <sup>b</sup>
Closed system,	0	49.0	88.7
no soaking	1	48.2	88.3
•	5	49.7	92.2
	20b	-	-
Closed system.	0	48.6	81.0
1-day soaking	1	49.1	91.9
	5	47.7	87.9
•	20	-	-
Open system.	0	49.5	87.8
no moist-	1	48.9	87.1
room	3	50.1	90.5
treatment	5	49.8	94.0
Open system.	0	49.0	91.5
5-day moist-	1	48.7	96.3
room	3	51.6	98.6
treatment	5	50.5	96.8

TABLE 4

<sup>a</sup>Measured after designated number of freeze-thaw cycles. b20-cycle specimens were too mutilated for accurate measurement.

# Effect on Porosity and Degree of Saturation

As shown in Table 4, there is no apparent effect of freezing and thawing on porosity, with the possible exception of the opensystem moisture-treated specimens. Townsend and Klym (4) discussed the possibility of sufficiently high hydraulic pressures being generated to exceed the tensile strength of the pore structure of the stabilized soil. They suggested that subsequent rearrangement of pore geometry and loss of pore structure confinement can result in strength loss and porosity increase. Because of this, work currently being done at Virginia Polytechnic Institute is aimed at more closely examining possible changes in porosity occurring on exposure to freezing and thawing.

The degree of saturation data given in Table 4 seem to indicate the expected moisture increases with time (if not cycles of freezing and thawing) in the open system, but not the closed.

# Destructive Mechanism of Freezing and Thawing

The destructive mechanism of freezing and thawing on the strength of clay soils may be considered to be a process whereby:

1. Ice lenses are built up in the soil with consequent expansion and loss in strength upon thawing;

2. The pore structure is sufficiently strong to generate hydraulic pressures by the movement of unfrozen water in advance of a moving ice front; or

3. Perhaps a combination of 1 and 2.

For the unstabilized soil specimens, the open-system test was truly open system in its effect; the development of ice lenses was apparently a dominant factor in their destruction. Figure 1b shows a typical example of this. Figure 1a shows the specimens prior to freeze-thaw testing. Closed-system specimens, with or without lime stabilization, showed no such damage. Figure 1d shows typical closed-system specimens, these having been exposed to 5 cycles of freezing and thawing. From the appearance of lime-stabilized samples subjected to open-system freezing, Figure 1c, one would question whether the open-system freezing of lime-stabilized clay was open system in its effect. No bulging or similar signs of ice-lens development are evident. A review of data on change in length and water content during treatment for the lime-stabilized specimens, some of which are summarized in Table 5, suggests that the action of opensystem freezing varied little from that of closed-system freezing. These results are in line with the hypothesis of Townsend and Klym (4) which suggests that lime-stabilized clays having more than 200-psi compressive strength together with less than 95 percent saturation may effectively resist the heave and ice-lens development associated with open-system freezing. Only in the case of samples held in the moist room for 5 days did the average degree of saturation exceed the 95 percent level.

The fact that the severe effects of open-system freezing may, as suggested by Townsend and Klym, be overcome with lime stabilization and the prevention of a high degree of saturation in the stabilized soil is gratifying. The problem remains, however, of understanding and dealing with the destructive effects of closed-system freezing. Of particular importance seems to be the condition of the soil just prior to freezing and the effects of the initial freezing cycle. It is apparent at this time that the soil responds



Figure 1a. Open-system specimens with zero percent lime after 5 days in moist room.



Figure 1b. Specimens after 3 cycles of opensystem freezing and thawing, 5 days in moist room, lime content-zero percent.







Figure 1d. Typical closed-system specimens, zero percent lime, 5 closed cycles.

	ST	ABILIZED	SPECIME	INS		
			No. Freez	ing Cycles		
Test	1			2		
Condition	\$ Strength Loss	Water Content Change	\$ Length Change	\$ Strength Loss	Water Content Change	≸ Length Change
Closed system, no soaking	20	-0.3	<1	18	1.4	<1
Open system, no moist- room treatment	24	0	<1	37	2.0	<1
Closed system, 1-day soaking	61	1.4	<1	50	3.42	<1
Open system, 5-day moist- room treatment	45	1.0	<1	76	2.8	2

TABLE 5 CHANGES IN LENGTH AND WATER CONTENT FOR STABILIZED SPECIMENS

Samples compacted appreciably drier than other samples employed in study.

in some way to adjust to the hydraulic pressures induced by freezing. Also apparent is that pretreatment of the compacted soil with moisture emphasizes its damaging influence.

#### CURRENT WORK

It seems likely that the damage caused by closed-system freezing is related to a remolding involving adjustment of pore-size distribution early in the freezing process, possibly with little change in total porosity, as observed in these experiments. Current work at Virginia Polytechnic Institute is aimed at studying pore-size distribution in lime-stabilized soils in relation to frost-thaw treatments with a view toward characterizing this change and studying its progression, if such exists, with repeated cycles of freezing and thawing.

Perhaps of equal merit of investigation is the effect of freezing and thawing on permeability. Townsend (9) noted that the change in permeability was negligible in specimens which had attained adequate resistance to frost deterioration. However, if a sample is subjected to freezing and thawing, perhaps some of the reaction products of the lime-clay combination are present in the voids. As hydraulic pressures begin to move water through the soil, some of these products may be moved out and thus initially increase the permeability while the soil generally retains its strength. If at this time the strength of the soil is overcome by excessive pressures, a collapse of the soil structure might take place causing a decrease in permeability. If not, the increase in permeability may be retained and the front susceptibility of the clay increased. Since this is speculation, permeability studies are also under way at Virginia Polytechnic Institute in order to present a clearer picture of the situation.

Although it is felt that conclusions presented in this study are valid for the data obtained, a method of freezing and thawing closer to that occurring in nature would be preferable. A new procedure has been developed and is being used in an extensive series of tests in connection with porosity and permeability studies covering a wider variety of soils.

#### CONCLUSIONS

It is hoped that the results of this study help shed a little light on what happens to lime-stabilized clays exposed to freezing and thawing. Unfortunately, the results are not sufficiently definitive to recommend specific reductions in pavement or base thicknesses. However, it should be noted that the largest strength loss suffered by the stabilized specimens (60 psi for 10 percent lime, 5 cycles, 5-day moist-room treatment) resulted in a compressive strength roughly equivalent to nonstabilized specimens not exposed to freezing and thawing. This might suggest that designs for pavements built on stabilized soils could be based on strengths of nonstabilized soils tested without moisture treatment (such as is used in the CBR test) until more definitive information can be obtained.

Where a significant lime-soil reaction takes place, there is no doubt that important strength benefits are derived. Work should continue in evaluating exactly what these benefits are.

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# **Flexural Fatigue Strength of Lime-Soil Mixtures**

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Flexural fatigue tests were conducted with four selected limesoil mixtures. The test results presented show that the fatigue response of cured lime-soil mixtures is similar to such materials as lime-fly ash aggregate mixtures and concrete.

Statistical techniques were employed to establish relations between S (fraction of ultimate flexural strength) and log N (number of applications to failure). The S log N plots displayed the same general form, but the same fatigue response curve was not obtained for all of the four mixtures studied.

Flexural fatigue should be considered in pavement design when lime-soil mixtures are utilized, but this does not limit the effective utilization of lime-soil mixtures for subbases and base courses.

•THE addition of small amounts of lime (3 to 7 percent by weight) to fine-grained soils produces a marked improvement in engineering properties. Plasticity is reduced, workability improved, and with reactive soils substantial strength increases are obtained.

A strength-producing pozzolanic reaction occurs between the lime and soil silica and/or alumina. This reaction forms various cementing agents, primarily calcium silicates and aluminates, which greatly increase the strength of the lime-soil mixtures after curing. The pozzolanic reaction occurs to varying degrees and those soils which develop a substantial strength increase are termed "lime reactive" (1).

Thompson (2) has shown that the shear and compressive strength of reactive limesoil mixtures is not the limiting factor in their application as subbase and base course material. In pavement applications, these materials will be subjected to repeated flexural stresses and therefore their flexural strength and fatigue response are important considerations. McDowell (3) has indicated that fatigue should be considered when evaluating tensile strength properties, and Ahlvin and Brown (4) have recently emphasized the importance of stress repetitions in pavement design.

Fatigue is the progressive failure of a material produced by repeated stresses which are less than the ultimate strength of the material. Fatigue response, generally defined as the reaction of a material to repeated loading at various stress levels as indicated by the number of load applications required to cause failure, is usually shown on scatter diagrams of stress level vs number of cycles to failure. The plot of the best fit line through the points on the scatter diagram can be linearized by plotting the stress level vs the log of the number of cycles to failure. The slope of this line is a measure of the fatigue response of the material. The susceptibility of the material to fatigue failure increases as the slope of the line becomes steeper.

Fatigue strength is defined as the greatest stress which can be sustained for a given number of stress cycles without fracture (5). Because of the scatter inherent in fatigue testing, the fatigue strength of a material at a given number of load applications is best represented as a range of stress levels. This range can be represented by different fatigue response curves for selected probabilities of failure.

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# OBJECTIVES

The primary objectives of this investigation were:

 Evaluate the general flexural fatigue response of selected lime-soil mixtures.
Determine if the fatigue response will limit the use of these materials in subbase and base course applications.

# METHODS AND PROCEDURES

# Materials

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The study was limited to four soils typical of the reactive soils in Illinois. The test soils were: Champaign County till; Bryce B; Illinoian till, Sangamon County; and Sable B. A hydrated high-calcium lime containing 96 percent available  $Ca(OH)_2$  with 95 percent passing the No. 325 sieve was used in all test mixtures.

Table 1 summarizes the natural soil properties and the properties of the lime-soil mixtures used. As indicated in Table 1, the natural soil properties showed substantial variation.

Soil	Champaign County Till	Bryce B	Sable B	Illinoian Till Sangamon County
General Description:	A Wisconsian loam till. Very typical through- out the Midwest.	A Humic-Gley (B horizon) derived from thin loess over Wisconsinan drift.	A Humic-Gley (B horizon) derived from loess.	Typical Illinoian Till
Soil Properties				
AASHO Classification	A-4 (6)	A-7-6(18)	A-7-6(16)	A-6(6)
< 2 µ Clay, %	16	52	36	14
Liquid Limit, 🖇	22.5	53.1	50.7	25.5
Plasticity Index, 🖇	7	28.8	23.5	11.0
Carbonates	Calcareous	Noncalcareous	Noncalcareous	18.6%
pH	8.3	7.4	7.8	8.3
Predominant Clay Mineral	Illite-Chlorite	Illite	Mixed Layer	Illite-Chlorite
Lime Soil Mixture Properts				
Lime Treatment, 🖇	3	5	3	3
Optimum Water Content, 🖇	11.5	25.8	20.1	13.0
Maximum Dry Density, pcf	120	97-3	100	121
Compaction, Elows/Layer (Top-Middle-Bottom)	40-25-10	50-35-20	65-40-15	35-23-8
Initial Cure	48 hrs @ 120°F	24 hrs @ 120°F	24 hrs @ 120 <sup>0</sup> F	24 hrs @ 120°F
Fatigue Specimens Tested	32	52	43	34

#### TABLE 1

# Mixtures Design and Preparation

Only that portion of the soil passing the No. 4 sieve was used in the test mixtures. The optimum percentage (dry weight basis) of lime, as determined from previous studies (1), was added to the soil and then dry-mixed until the lime was evenly distributed. Water was added to bring the mix to optimum moisture content and the materials were mixed mechanically for approximately 3 min. After mixing, the lime-soil mixture was tightly covered to prevent moisture loss and allowed to mellow 1 hr before compaction.

# **Compaction of Test Specimens**

Test specimens were prepared by compacting the lime-soil mixtures into  $2 \times 2 \times 7$ in. steel molds in three equal layers. The surface of each layer was scarified before placing the next layer to insure bonding. Compaction was accomplished through a hammer with base dimensions of  $2 \times 7$  in. The compactive effort was applied by a 7-lb weight free falling 12 in. The water content and dry density of each specimen were carefully controlled to produce as nearly identical specimens as possible.

The specimens were rotated before testing so that the load was applied perpendicular to the direction of compaction, thus minimizing the effects of bonding between layers. Density studies revealed that with an equal compactive effort on each layer unequal densities were produced. The difference in strength of the layers as indicated by the unequal densities was undesirable as load eccentricities and a tendency to crack first on the weak side would result. This problem was minimized by using a different compactive effort, as indicated in Table 1, on each layer to achieve a uniform density throughout the specimen. The compactive effort required was greatest on the top layer and least on the bottom layer. Average densities of the specimens were equivalent to those obtained from AASHO T-99.

# Curing Procedures

Immediately after compaction the specimens were wrapped in plastic food wrap and sealed with cellophane tape. This proved to be an effective way to maintain the optimum moisture content of the lime-soil mixture for the duration of the curing and testing periods.

The specimens were fully supported on rigid boards to prevent cracking or warping and then placed in an oven at a constant temperature of 120 F. The high temperature was used to accelerate the curing of the lime-soil mixtures. Curing the Champaign County till for 48 hr and the other soils for 24 hr at 120 F produced strengths approximately equivalent to a 30-day cure at 70 F. This length of cure was assumed to be representative of field conditions.

After removal from the oven, the specimens were stored in a constant temperature room at 70 F. The specimens were tested at ambient room temperature and no attempt was made to control the temperature during the fatigue testing period.

# Static Flexural Strength Studies

The stress level in a fatigue test is commonly defined as the ratio of the applied stress to the static ultimate strength of the material. It is thus very important to be able to predict the static strength of the material during the fatigue test. A strength study was statistically designed to determine the mean static flexural strength of each lime-soil mixture immediately after the accelerated cure and for curing periods of 1, 3, 5, and 9 days after the accelerated oven cure.

The flexural test specimens were tested under third-point loading using a system of balls and rollers to prevent eccentricities. All test specimens were loaded at a constant rate of 0.05 in./min. Stress in the specimen was calculated assuming a constant cross section and a linear stress distribution.

The static flexural tests showed that the lime-soil mixtures continued to gain strength at a decreasing rate after the accelerated oven cure. The test results also indicated variations in static strength for "identically prepared" specimens with the coefficients



Figure 1. Flexural strength—curing study.

of variation ranging from 8.4 to 13.3 percent. This amount of variation can be expected even though the preparation, curing, and testing of the specimens are carefully controlled. Figure 1 shows the average static flexural strength vs curing time curves for each lime-soil mixture.

# **Fatigue Testing Machines**

Two testing machines of the constant displacement type were used to apply repeated flexural stresses to the specimens. The displacement was produced by an adjustable motor-driven eccentric operating a series of levers. Figure 2 is a schematic drawing of the testing machine.

The specimen was placed on the test cradle which was oscillated vertically by the testing machine. A loading head for applying load at the specimen third points was



Figure 2. Schematic drawing of fatigue testing machine.

placed between the specimen and the calibrated steel bar dynamometer. The loads applied to the specimen were equivalent to the force required to cause a deflection in the steel bar equal to the vertical movement of the oscillating shaft on the test machines.

Two SR-4 strain gages were attached to the top and bottom of the steel bar dynamometer and the gages were connected to add both the tensile and compressive strains of the bar as it was bent. The bar was then calibrated so the load applied to the specimen could be determined by reading the difference in strain as the bar was deflected. Using these calibrated bars, the stress in the sample could be adjusted to  $\pm 0.5$  psi.

The loads were applied at rates of 730 cpm and 690 cpm by the two machines. Based on Murdock and Kesler's work (6) on fatigue of concrete, it was assumed that this small difference in loading rate would have no effect on the fatigue response of the lime-soil mixtures.

Mechanical counters were used to record the total number of load applications on each specimen. When a specimen failed it fell on a microswitch which turned off the machine. The counters stopped with the machines and indicated the number of cycles to failure.

The testing machines were set to apply a given load under static conditions. Tests revealed that the loads applied under dynamic loading were about 6 percent greater than those indicated by the static adjustment. The static loads were adjusted to correct for the dynamic loading effects.

Figures 3 and 4 show one of the testing machines and a close-up of the loading apparatus and test specimen.

# **Fatigue Testing Procedures**

The specimens were tested at stress levels ranging from 95 to 40 percent of their ultimate strength. Several specimens were tested at no less than three different stress levels for each lime-soil mixture.

A stress level was chosen for a given test specimen. The test specimen was placed in the machine and the load adjusted using a strain indicator. Load adjustments were made carefully so the required load was approached from the low side. A minimum stress of 10 percent of the ultimate strength was maintained on the specimens during all tests in order to avoid the dynamic effects caused by complete stress removal and subsequent reloading. Since range of stress has an effect on the fatigue response of the materials (7), the minimum stress level was held constant for all tests so comparison between tests could be more easily made. Figure 5 presents the typical sine wave loading cycle used in the tests.

After the load had been adjusted, the automatic counter was set on zero and the test machine activated. The load was adjusted at least every 24 hr to correct for changes



Figure 3. Fatigue testing machine.



Figure 4. Test specimen and loading apparatus.



Figure 5. Typical loading pattern.

in stress level due to the effects of creep and curing of the specimen. As the load was applied, the test specimens sagged slightly due to creep in the material. The effect of this sagging was a reduction in the stress level due to reduced deflection of the steel bar dynamometer. Each day of testing was assumed to be equal to one day of curing at 70 F, and as the test specimen became stronger the stress level decreased. By frequent readjustment of the load on the specimen the stress level was held nearly uniform. The maximum reduction in the stress level, due to the effects of creep and curing of the specimen, was 4 percent; however, the average reduction over the 24 hr between adjustments was less than 1 percent.

The results of the flexural fatigue tests are presented on scatter diagrams, Figures 6-9, of stress level, S, vs the log of the number of cycles to failure N.

Runouts, specimens which did not fail during the test, are shown on the scatter diagrams as a point with an arrow. It was necessary to terminate a test when the curing period of the specimen was longer then the curing periods used in the static strength study. If the test specimen did not break after five million stress cycles (5 days of load repetitions), the test was stopped if other specimens were ready for testing.

For stress levels above 90 percent about one-half of the specimens failed on the first load application. This is to be expected because of the difference between the



Figure 6. Fatigue test results.



Cycles To Failure, N



actual specimen strength and the estimated average strength of the material. Specimens which failed on the first load application were plotted at N equal to 1 and S equal to the estimated value even though it was apparent that the actual strength was less than estimated. These specimens were as significant statistically as those which withstood several stress applications.

Similar reasoning was used to justify the use of runouts in the analysis of the test data. It is clear that runouts cannot be ignored in the analysis of the test data, but a question arose as to what value of N should be assigned to them. It was decided that



Figure 8. Fatigue test results.



Cycles To Follure, N

Figure 9. Fatigue test results.

the runouts should be treated as failures at N values equal to those recorded when the test was terminated. This method of interpretation will give a conservative estimate of the fatigue response of the material.

The test data were analyzed statistically using linear regression techniques. Linear regression lines were plotted on the scatter diagrams and were used to represent the general flexural fatigue response of the lime-soil mixtures tested. The regression lines were analyzed statistically and all were found to be significant ( $\alpha = 0.01$ ). Correlation coefficients, R, for the linear regression lines are recorded on the scatter diagrams. Figure 10 shows the fatigue response of the four lime-soil mixtures tested. The average fatigue strength at five million stress applications was approximately 54 percent with the values ranging from 66 to 41 percent of the ultimate flexural strength. The slopes of the linear regression lines of the different lime-soil mixtures were statistically compared to determine if they were significantly different. The comparisons indicated that the slope for the Sable B lime-soil mixture was significantly different from all the others,  $\alpha = 0.05$ , but there was no significant difference ( $\alpha = 0.05$ ) in the slopes for the other three soils.

# DISCUSSION

The cementitious materials in portland cement concrete, lime-fly ash-aggregate mixtures and lime-soil mixtures are similar. Because of this similarity, it was expected that the fatigue response of the lime-soil mixtures would compare favorably with the other materials. Figure 10, a plot of the general fatigue response for the three materials, indicates the close resemblance of the S-log N plots.

The length of cure of the materials tested was limited to the equivalent of 30 to 40 days at 70 F. Lime-soil mixtures continue to gain strength for long periods of time and the ultimate strength is a function of the curing period and temperature. Under actual field conditions lime-soil mixtures may continue to gain strength for several years. The magnitudes of the stress repetitions applied to a lime-soil mixture used as a paving material are relatively constant throughout its design life. Therefore, as the ultimate strength of the material increases due to curing, the stress level (as a percent of ultimate strength) will decrease and the fatigue life of the lime-soil mixtures will be increased. This would suggest the possibility of increasing the fatigue design stress level to take advantage of the increase in ultimate strength over long curing periods.



Figure 10. Flexural fatigue response curves.

# SUMMARY AND CONCLUSIONS

The general flexural fatigue responses of four typical reactive Illinois soils mixed with lime were determined. The fatigue response was represented by a linear regression line on a scatter diagram of stress level, S, vs the log of the number of cycles to failure, N. The S-N plots were typical of fatigue in general and of similar materials, specifically portland cement concrete and lime-fly ash-aggregate mixtures.

The slopes of the regression lines representing the fatigue response of the four limesoil mixtures were statistically compared. There were significant differences between the lime-soil mixtures and, therefore, it cannot be concluded, based on this limited study, that the fatigue response of all lime-soil mixtures are equivalent.

If lime-soil mixtures are to be used in subbase and base course construction, the flexural fatigue response of these materials should be considered in selecting a design stress. The stress used for design must be low enough to allow the design number of load applications before fatigue failure. Consideration of the flexural fatigue response will limit the design stress, but the limitation does not make the use of these materials impractical for subbase and base course construction. Flexural fatigue response tests to be used for design purposes should include provisions for determining a range in fatigue response for various probabilities of failure.

The loading conditions in the field are different from those used in this study in the following respects: First, the random loads applied in the field by heavy trucks interspersed with passenger vehicles are not equivalent to the uniform loads applied in the laboratory. The second difference is in the range of stress applied to the materials. In the field the minimum stress level will be zero, whereas in the laboratory, the minimum stress was 10 percent of the ultimate strength. A decrease in the range of stress increases the number of load cycles required to cause failure. The third difference in applied stress between field and laboratory conditions is the constant loading in the laboratory as compared to the rest periods which can be expected between loads in the field. Hilsdorf (8) has shown that rest periods have beneficial effects on fatigue strength

of concrete. Under actual service conditions, paving materials are subjected to stress reversals (compression-tension), but this feature was not present in the laboratory testing program. Further studies to determine the effects of these factors on the flexural fatigue response of lime-soil mixtures may be desirable.

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The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the State of Illinois, Division of Highways, or the Bureau of Public Roads.

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# A Rapid Method for Soil-Cement Design: Louisiana Slope Value Method

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A laboratory design method is developed to reduce the testing time for determination of minimum cement content for use in soil-cement pavement construction. This method makes maximum use of AASHO procedures for wet-dry testing and the PCA weight loss criteria and is based on an apparent relationship observed between the durability of soil-cement mixtures at selected cement contents and the slope of the unconfined compressive strength line at identical cement contents. The "Louisiana Slope Value Method" has been extensively tested and compared against the wet-dry test method; 92 percent of all cement contents recommended were within ±2 percent of those cement contents called for by the latter method.

Normal testing time for the Louisiana Slope Value Method is about 8 days. Actual testing is easy to perform and use of the developed correlation curves and tables is simple and rapid.

•THE testing procedure used until very recently by the Louisiana Department of Highways for laboratory design of soil-cement mixtures for base and subbase courses was taken from standard AASHO (1) procedures for wet-dry testing and PCA (2) weight loss criteria. These methods have been used by the Department for more than a decade with the results applied to approximately 7000 lane-miles of soil-cement construction producing very satisfactory performance without any significant failures. However, they had the disadvantage of involving extensive laboratory work covering a rather long period of testing time which has not been always desirable. The need for a faster test method became apparent under an accelerated construction program when the Department required contractors to furnish all the materials and labor for soil-cement construction where the final approval of the soil and the determination of amount of cement required to produce soil-cement would be made after the soil material was placed on the embankment. In an attempt to eliminate some of the lengthy laboratory test procedures, the Department initiated a study in July 1961, in cooperation with the U.S. Bureau of Public Roads. The intent of this work was not to compare the relative merits of various soil-cement test methods, but to establish a correlation between the "wetting and drying test of compacted soil-cement mixtures" (ASTM D 559-57 or AASHO T 135-57) and a 7-day compressive strength requirement in order to make full use of the proven qualities of the accepted standard method and PCA criteria.

# SCOPE AND OBJECTIVE

When this research program was first conceived, it was decided that the best solution to the problem would be to establish a minimum compressive strength requirement for satisfactory soil-cement stabilization.

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With this in mind, the investigation was to be conducted in the following phases:

1. Determination of in-place strengths of existing soil-cement projects to cover predominant Louisiana soil types, climatic conditions, and laboratory behavior of similar material.

2. Correlation of satisfactory pavement behavior to soil-cement strength data observed in place.

3. Determination of in-place 7-day strengths of soil-cement projects under construction and laboratory behavior of similar material.

4. Establishment of a laboratory correlation of unconfined compressive strength vs wet-dry test losses of samples molded at identical cement contents and moisture conditions.

Soon after the work was started, it was obvious that the compressive strength vs the wet-dry test relationship of soil-cement mixtures is much too complex to be defined accurately by some minimum or maximum strength value especially when soils of different types and geologic origin are encountered. Also, the in-place strength data obtained from existing projects as compared to the laboratory behavior of what was presumed to be similar untreated material was too erratic to offer any conclusions. However, the results obtained from phase 4 looked promising and it was felt that all efforts should be concentrated in this area.

# MATERIALS TESTED

All soils which were submitted from routine Department of Highways construction projects during the duration of the research project were tested without any discrimination. Since soil-cement projects are fairly common and widespread throughout the state, a good representation of soil types suitable for cement stabilization in Louisiana was achieved.

A total of 637 different soil samples were tested. The classifications tested included A-2-4, A-3, A-4, and A-6 soils with plasticity indices of 15 or less. Also tested were A-6 or A-7-6 soils with plasticity indices of 15 or more which had been modified by the addition of lime to have plasticity indices of 15 or less. Approximately 22 percent of the samples represented A-2-4 and A-3 soils, 15 percent represented A-4 soils with low plasticity, 24 percent represented plastic A-4 soils, and 39 percent represented A-6 soils and those A-6 and A-7-6 soils modified with lime.

The cement used in this investigation was commercially available Type I, portland cement, meeting requirements of AASHO designation M 85-60.

# METHOD OF PROCEDURE

Standard laboratory procedures were used in the investigation with a very few minor variations such as capping ends of molded samples with a commercially available capping compound before testing for unconfined compressive strength. Further details are given in the Appendix.

Once the work was under way, it became apparent that by plotting the unconfined compressive strength and the corresponding wet-dry loss at each percentage of cement content on a combined graph as shown in Figure 1, one could predict the minimum compressive strength required to fulfill the wet-dry loss criteria provided this relationship were established for each soil type encountered. The minimum cement content required for soil-cement mix design is determined by point (a) which is the intersection of the wet-dry loss line with the PCA criteria allowable loss line. It follows that this particular soil specimen would develop a strength of 245 psi to meet the wet-dry loss criteria as indicated by point (b). In other words, 245 psi would be the minimum strength requirement for this soil if stabilization criteria were applied through a measure of strength instead of wet-dry losses.

The next step was to establish generalized relationships in the form of a family of curves for major soil types encountered. It was also seen that there were quite a few soil specimens which did not present a neatly defined relationship as described above. In about a third of the total observations made, the wet-dry loss line turned out to be



Figure 1. Strength vs wetting-drying loss relationship for a typical soil.

too flat to intersect the allowable loss line at the percentages of cement used in the laboratory or in the field. It was decided to attack this problem through a statistical analysis of a large amount of data. An arrangement was made with the Soils Unit of the Materials and Testing Section, whereby every soil sample being submitted to the unit to be tested for suitability for stabilization with cement and cement content requirement would be tested by each of two different methods.

In addition to standard Louisiana testing, two additional cylinders of the same type used for the wet-dry test were molded at each of the cement contents for which the wet-dry test would be performed. These cylinders were cured in a moisture-controlled room where the relative humidity was maintained near 100 percent for 7 days. The cylinders were then removed from the moisture room, measured, capped with a commerical capping compound, immersed in water for 4 hr, and then tested for unconfined compressive strength. The results obtained for the two cylinders at each cement content were averaged to obtain a compressive strength value at that cement content. Since the wet-dry test is normally run at three cement contents, a total of six compressive strength cylinders were usually tested to obtain average strength results.

This process, over a period of 5 years, made available an extensive amount of data regarding 7-day strength vs wet-dry loss curves with associated percentages of cement for soil-cement mix design as recommended by the Soils Unit using standard Louisiana procedures. It appeared that there was a relationship, at least empirical, between the rate of strength increase of a soil with additional cement and its expected strength at the cement content necessary to stabilize it. A trial-and-error approach was used to arrive at a set of curves where the general relationship in Figure 2 was assumed to represent the idealized correlation between the wet-dry loss and strength for the corresponding soil group. Figure 2 represents the family of curves developed for A-2-4 and A-3 soils using the test results of some 140 different soil samples. This chart actually represents the superimposition of a large number of strength and wet-dry loss curves for individual samples comparable to that shown in Figure 1. Lines numbered 1, 2, and 3 represent strength curves and lines 1A, 2A, and 3A represent the matching wet-dry loss curves. The lines are matched in pairs such as 1 and 1A, etc. It was found out that if a set of wet-dry loss tests were run and the results plotted to define line 1A, a plot of the results of a set of strength tests on the same soil would define line 1. Consequently, if the location of either line of a matched pair were determined through laboratory testing, the location of the other line could be predicted without the necessity of actual testing. For those test results which did not determine lines coinciding with the three matched pair of lines shown in Figure 2, a suitable interpolation could be made as indicated by the dashed lines.

Predictions of recommended cement content using this approach were compared by those made by the Soils Unit using standard methods and boundary lines in the family



Figure 2. Determination of minimum cement content using family of curves.

of curves shifted back and forth on a trial-and-error basis until the greatest percentage of agreement was reached between the two methods.

Figures 3a through 3d represent the family of curves for each of the following four general soil groups determined in the same manner as discussed previously: (a) A-2-4 and A-3 soils, (b) A-4 soils with plasticity indices less than 3, (c) A-4 soils with plasticity indices greater than 3, and (d) A-6 soils with plasticity indices less than 15 and those A-6 and A-7 soils originally with higher plasticity indices but later modified to less than 15 with the addition of lime.

It will be noted that a further refinement has been added to these figures by assigning a slope value to the strength lines and hence the name "Louisiana Slope Value Method." The slope value is determined by dividing the difference in unconfined compressive strength in pounds per square inch by the difference in the corresponding cement contents in percentage by weight.

Since only the placement or the slope value of the strength lines and the cement content at the intersection of the wet-dry loss lines and the allowable loss line is of significance in practical applications, the family of curves can be very simply expressed in tabular form. A detailed procedure for using such a method is given in the Appendix for routine laboratory use.

#### DISCUSSION OF RESULTS

As mentioned earlier, an initial attempt was made to establish a minimum compressive strength to be required for satisfactory soil-cement stabilization. This approach had to be abandoned as the work progressed, but nevertheless, some interesting observations were made as indicated in Figures 4 and 5.

1. Within any AASHO soil group tested, as the soil specimens require more cement to meet the PCA criteria, the strength developed at these cement contents also increases. This is indicated by the ascending slopes of the lines which represent an approximate average of the scatter pattern.

2. The rate of increase in strength with corresponding increases in the cement content to meet the maximum allowable loss is the highest in A-2-4 and A-3 groups, intermediate in the A-4 group, and lowest in the A-6 group. Actually, the slope of the strength line is almost flat in the A-6 group. In other words, no matter how much cement it takes to stabilize adequately an A-6 soil, the average strength gain at that cement content is not appreciably higher than the average strength of another soil which will require a much lower cement content. The same point can further be discussed and elaborated on through the use of Figure 5a, b, c, d. These charts illustrate the







Figure 4. Strengths developed at the required minimum cement contents.







Figure 5. Strength vs wetting-drying loss of specimens molded at various cement contents.









 $\begin{array}{c} e^{i \frac{\pi}{2}} &= e^{-i \frac{\pi}{2}} e^{-i \frac{\pi}{$ 

compressive strength-wet-dry test loss for A-2-4 and A-3, plastic and nonplastic A-4 and A-6 soils.

If an arbitrary minimum compressive strength requirement of 350 psi were selected for A-2-4 and A-3 groups shown in Figure 5a, 24 percent of the soils tested at that time that would normally stabilize at the indicated cement contents would either completely fail or require a higher cement content. The soils that would be suitable using the wetdry test and would fail this minimum compressive strength are shown in the shaded area.

If an arbitrary minimum compressive strength of 300 psi is specified for A-4 and A-6 soils (Fig. 5b, c, and d), the percentage of soil specimens that would pass the wetting-drying test and yet fail this minimum compressive strength requirement would be 33 percent for nonplastic A-4 soils, 49 percent for plastic A-4 soils, and 44 percent for A-6 soils. Here again, the areas where the soils would pass the wet-dry test and fail the arbitrarily specified compressive strength test are shaded. In other words, in soil stabilization, the use of a minimum compressive strength value as a criterion for the selection of the minimum adequate cement content will result in the elimination of a large percentage of soils that would perform satisfactorily.

It was at this point in the research study that it was decided to concentrate all work on developing some other type of approach where individual characteristics of different soil types were recognized and treated accordingly.

3. Minimum compressive strengths required for various AASHO soils groups to meet the PCA criteria can be established to insure that all the specimens tested would meet the PCA criteria. However, this method would not be economically desirable leading to the use of excessive cement on a large portion of the samples tested.

A detailed procedure for the use of the Louisiana Slope Value Method is given in the Appendix. This method has been used by the Department as a tentative procedure for the past two years and has finally been approved as a standard method. The wet-dry test (AASHO T 135-57) is also retained for reference in those cases where soils are encountered which are obviously outside the boundaries covered by the slope value test.

The slope value method is simply a formalized expression of available statistical data. It is based on a tangible relationship between the susceptibility of different types of soil-cement mixtures to additional cement, as expressed by the slope of the 7-day unconfined compressive strengths developed at respective cement contents, and the wet-dry test losses realized at identical cement contents. At this point, it should be recognized that very low cement contents such as from 1 percent to 4 percent by weight are mainly of academic significance in the laboratory and not feasible for use in soil-cement construction with the predominant soil types in Louisiana. It was, therefore, decided to use an arbitrary minimum cement content of 5 percent by weight to analyze and evaluate the laboratory data which were not well defined. This minimum figure of 5 percent by weight was arrived at after reviewing all the available soil-cement test data in the Department's laboratory. Since the objective of the testing program was to

TABLE 1

ACCURACY OF CEMENT REQUIREMENT DETERMINATIONS THROUGH USE OF CORRELATION CHARTS AS COMPARED WITH THOSE OBTAINED FROM WETTING-DRYING TEST LOSSES

	Percent of Samples for Soils:				
Cement Content Correlation With Wetting-Drying Test	A-2-4 and A-3 (139 Observations)	Nonplastic A-4 <sup>a</sup> (98 Observations)	Plastic A-4 <sup>b</sup> (155 Observations)	A-6 and Mod. A-7-6 (245 Observations)	
No deviation	32	35	32	29	
Within ±1% cement	73	71	73	75	
Within -1 and +2% cement	97	81	97	86	
Within ±2% cement	98	92	98	94	
More than 2% cement	2	8	2	5	
Less than 2% cement	ō	ō	ō	1	

PI less than 3.

bpl greater than 3.

arrive at a practically feasible and workable method from a construction standpoint, it was felt that imposing such an arbitrary boundary condition would be in order.

The accuracy of cement requirement determinations through use of the slope value method as compared with those obtained from wet-dry test method is indicated in Table 1 for major soil groups. It will be seen that better than 92 percent of the results are within  $\pm 2$  percent content as determined by the wet-dry test and better than 78 percent are within  $\pm 2$  or -1 percent of cement content by weight.

#### CONCLUSIONS

1. A minimum compressive strength requirement would not necessarily result in the most economical cement requirement due to the fact that different soil-cement mixtures exhibit different strengths at similar degrees of durability.

2. The minimum compressive strength required for various AASHO soil groups to meet PCA criteria applied through the wet-dry test is not a constant but probably varies as a function of other parameters (i.e., physical, chemical properties, etc.).

3. A method, using correlation curves or a numerical chart or both, has been devised for determining minimum cement content requirements for soil-cement stabilization of soils with a resulting accuracy of 92 percent of predictions within  $\pm 2$  percent of those determined by the wet-dry test method.

4. The use of the slope value method results in a decrease in testing time from a minimum of 32 days to 8 days.

5. The accuracy of the slope value method based on compressive strength is not dependent upon a great deal of procedural control. The test method is simple to perform and the reproducibility is satisfactory.

Since the PCA wet-dry weight loss criteria are incorporated into the slope value method, it includes most of the relationships to field performance that have already been established in Louisiana.

# ACKNOWLEDGMENTS

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# Appendix

# LABORATORY PROCEDURE

# Scope

1. This method is intended for determining the minimum cement requirement for design use in the construction of soil-cement base and subbase courses.

**Test Methods** 

2. (a) Soil samples shall be prepared in accordance with AASHO designation T 87-49 (LDH designation 411-58)—standard method of dry preparation of disturbed soil samples for test.

(b) Soils shall be classified in accordance with AASHO designation M145-49the classification of soils and soil aggregate mixtures for highway construction purposes.

(c) The moisture-density relations of the soil-cement mixture shall be determined by adhering to AASHO designation T 134-57-standard methods of test for moisture-density relations of soil-cement mixtures.

(d) Specimens for unconfined compressive strength determinations shall be molded in accordance with paragraph 4, ASTM designation D 559-57—wetting and drying tests of compacted soil-cement mixtures.

(e) The compressive strength specimen shall be tested in accordance with ASTM designation D1633-59T with the following exceptions:

- (1) Test specimens shall have a diameter of 4.0 in. and a height of 4.6 in.
- (2) Specimens shall be moist-room cured at approximately 100 percent relative humidity for 7 days.
- (3) Immediately upon removal from the moist room, the specimens shall be measured for height and diameter, capped with a commercial capping compound (trade name: Vitrobond or gypsum plaster), and immersed in clean water for 4 hr prior to testing.

# Procedure

After the soil is classified, a range of cement contents is selected according to the following: A-2-4, A-3 and A-4 should be molded at cement contents ranging from 5 to 9 percent by weight, and the range for A-6 soils should be from 6 to 10 percent by weight.

A minimum of two (preferably three) cylinders are molded at each of the three cement contents selected, tagged and cured in the moist room for the required 7 days, after which the samples are measured, capped and immersed in water for 4 hr prior to testing for unconfined compressive strength. Upon completion of the compressive strength, the appropriate "slope values" are determined by the following formula:

Slope value = 
$$\frac{B-A}{Y-X} \times \frac{1}{100}$$
 or  $\frac{C-B}{Z-Y} \times \frac{1}{100}$ 

where:

A = unconfined compressive strength at the lowest cement content,

B = unconfined compressive strength at the median cement content,

C = unconfined compressive strength at the highest cement content,

X = lowest cement content by weight,

Y = median cement content by weight, and

Z = highest cement content by weight.

"Maximum slope value" represents the highest value obtained from the above expression and is used for A-2-4, A-3, and all A-4 soils with plasticity indices of 10 or less. "Minimum slope value" would be the lowest value derived from the above formula and is used for the A-6 and A-7-6 groups of soils. For example:

Point	Cement Content (% by wt)	Failure Stress (psi)
Α	5.08	342
B	6.89	455
С	8.77	603
Maximum slope value =  $\frac{603 - 455}{8.77 - 6.89} \times \frac{1}{100} = 0.79$ 

Minimum slope value = 
$$\frac{455 - 342}{6.89 - 5.08} \times \frac{1}{100} = 0.62$$

The appropriate slope value is then located on the proper chart or on the following table for the minimum cement content requirement.

MINIMUM CEMENT REQUIREMENT USING THE

Soil Classification	Slope Value Type	Slope Value	Min. Cement Requirement by Weight (\$)	
A-2-4, A-3	Maximum	0.46-0.60 0.61-0.85	6 7	
Nonplastic A-4 (PI 0.0-3.0)	Maximum	0.24-0.36 0.37-0.56 0.57-0.75 0.76-0.90 0.91-0.94	5 6 7 8 9	
Plastic <b>A-4</b> (PI 3. 0-10. 0)	Maximum	0.18-0.20 0.21-0.30 0.31-0.67 0.68-1.25	5 6 7 8	
A-6 and A-7-6	Minimum	0.17-0.27 0.28-0.34 0.35-0.36	8 7 6	

Note: Slope values which vary greatly from the limiting values should be verified by the complete wetting-drying test (AASHO designation T 135-57).





Figure 6. Cylinder capping mold.

# The Effect of Delayed Compaction on Stabilized Soil-Cement

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The reaction of cement with soil occurring in the soil-cement stabilized base material is of a chemical nature. Such a reaction is not only a function of the reacting chemical agents, but is also a function of time. Such is the case with a soilcement stabilized material.

The construction of a cement-stabilized soil foundation, be it for roads or other structures, is a time-consuming procedure. The most time-consuming phase of the construction is pulverizing the soil, the initial part of mixing the cement and soil uniformly, and obtaining the optimum moisture content for the final mixture. This procedure in the field, under normal conditions, takes anywhere from 2 to 4 hr, which means that the compaction of a soil-cement mixture cannot be started in most cases before 2 to 4 hr after mixing the soil with the cement. In the meantime a very vigorous reaction starts between the soil and the cement, and as the reacting particles are left loose for different periods of time, the final compacted and finished product attains physical characteristics that are different from each other.

This study attempts to establish the working range of some of the physical variables produced by delayed compaction after mixing of soil-cement stabilized material. The effects of the delayed compaction and fluctuations in the optimum cement content, and optimum moisture content of the mix on the density, unconfined compression strength, and durability of the final product are investigated in this report. Compaction has been delayed for periods from zero to 6 hr after mixing to determine its effect on compressive strength, durability, and the density of the final product. The results of the study show that the intensity of the effect of the delayed compaction depends to a large extent on the type of soil. Most of the silty and clayey soils undergo serious losses of strength, durability, and density. Results indicate that the reduced density is one of the causes of loss in strength.

A study of the initial and final setting times of the portland cement used in this study has indicated that serious losses of strength density and durability occur at a time coinciding with the initial setting time of cement paste.

•CONSTRUCTION with soil-cement stabilized material requires the pulverization of the soil, uniform application and mixing of portland cement, moisture control at the time of compaction, density control, curing, and time restrictions to prevent partial

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hardening of the mix before compaction. The main purpose of this treatise is to study the behavior of soil-cement mixes when a delay between the time of mixing and compaction is introduced, and to investigate the effect of delayed compaction. The two main factors affecting the compressive strength and durability of soil-cement are dry density and cement content. Variations in these factors are investigated in connection with delayed compaction and variations in the optimum moisture content.

#### TESTING PROCEDURE

Soil samples from different locations in Louisiana representing predominant soils covering a given geographical area which are suitable for stabilization with portland cement were selected for this study. These soils were tested for the following variables:

1. <u>Portland cement content</u>: each soil was tested by varying the cement content 2 percent above and below the optimum cement content. They were also tested at optimum cement content, according to criteria established by the Portland Cement Association.

2. <u>Moisture content</u>: each soil was tested at 2 and 4 percent below the optimum moisture content, at optimum moisture content, and at 2 and 4 percent above the optimum moisture content.

3. Detention period before compaction: one specimen was made for each of the following detention periods: 0,  $\frac{1}{2}$ , 1, 2, 4, and 6 hr after mixing.

A total of approximately 1800 samples were tested. In addition, 300 samples were retested because of deviations from certain patterns of behavior detected during testing. The disturbed soil samples were prepared according to ASTM designation D 421-58; then grain size analysis was performed in accordance with ASTM designation D 422-63 and Atterberg limits were determined by standard tests, ASTM designation D 423-61T and ASTM designation D 424-59. The AASHO soil classification method was used to identify the soil types. Periodic tests were performed to determine the initial setting time of the cement according to ASTM designation C 266-64. The average, final setting time for the cement paste proved to be 2 hr and 5 min.

After the soils were classified, the estimated cement content required for soil stabilization was determined using the criteria developed by the Portland Cement Association. With this cement content, the moisture-density relations of the soil-cement mix and of the soil were determined by using the standard proctor test ASTM designation D 558-57. The results of the moisture-density test and the classification tests were used to determine a more accurate optimum cement content as in the data provided by the Portland Cement Association. Specimens were then prepared according to the study schedule for the unconfined compressive strength test and wet-dry test. Soil and cement were mixed for 2 min by a counter current batch mixer, then the water required was added and the mixing was continued for 8 min more. This mixing procedure was standardized throughout the test. Some of the soil-cement specimens required remixing after a period of 4 to 6 hr of delayed time. Some soil-cement mixtures after this period of time formed into clusters and lumps that had to be broken up before compaction in order to obtain a uniform end product. Therefore, they were remixed by hand tools for on the average period of 6 min; the results of specimens where the soil has been re-mixed are shown as such in the curves and tables. However, there was not a special effort made to study the effect of re-mixing by preparing special samples and re-mixing the mixture after a given period of time. Unconfined compression test specimens and wet-dry test specimens were molded according to ASTM designation D1632-63. Cylinders 6 in. long and 4 in. across were used throughout. Initial compaction of zero hr was performed about 12 min after mixing was started. Specimens were molded at  $\frac{1}{2}$ , 1, 2, 4, and 6 hr from initial compaction. They were then weighed before being placed in a moist room for 7 days. At the end of moist curing, specimens were submerged in potable water at room temperature for a period of 4 hr before testing for compressive strength. The failure of the specimens was consistent cup and cone with a failure angle of approximately 60°. The remaining

samples were used to run the wet-dry test according to ASTM designation D 559-57. At the end of the moist curing period the specimens were submerged in potable water for a period of 5 hr, after which they were placed in an oven at 160 F for 48 hr. The specimens were then removed, weighed and given two firm strokes of approximately 3-lb force on all areas with a standard wire scratch brush. Eighteen to 20 strokes were required on the sides and 4 strokes were required at each end. This procedure constituted 1 cycle of the wet-dry test and it was repeated for 12 cycles, after which the specimens were dried to constant weight at 230 F.

The following deviations from design factors were used for laboratory practice: moisture content  $\pm 2$  and 4 percentage point; density  $\pm 3$  pcf. All specimens within these deviations were accepted and tested.

The testing procedures are summarized by the flow diagram shown in Figure 1.

#### DISCUSSION OF RESULTS

The results of the tests performed on each of the ten soils suitable for stabilization with portland cement in the State of Louisiana show different patterns of behavior when tested for compressive strength and durability. The results varied in accordance with the physical characteristics of each soil.

The soils identified as W-1, W-2, and W-6 are classified as silty loam. The effects of delayed compaction, after mixing the soil at optimum cement content and optimum moisture content, on the compressive strength, dry weight density, and percentage weight loss are shown in Figures 2, 3, and 4, respectively. The data on the three silty loam soils are given in Table 1a.

A time delay of 6 hr reduced the compressive strength by 75 percent and the dry weight density by 18 percent. At or below optimum water contents both the compressive strength and the dry weight density decreased as the time between mixing and compaction increased. At water contents 2 and 4 percent above the optimum, both the compressive strength and density increased to a certain value before they again decreased with time delay. When either the water content, cement content, or both ex-



Figure 1. Flow diagram of testing procedure.

ceeded the optimum by 2 percent, the durability showed tangible improvement.

Soils identified as W-3, W-4, and W-9 are classified as silty clay loam. The typical effects on these soils of delayed compaction at optimum moisture content and optimum cement content on compressive strength, dry weight density, and percent weight loss are shown in Figures 5, 6, and 7, respectively. Typical data on the silty clay loam soils are given in Table 1b. A 4-hr delay in compaction



Figure 2. Effect of delayed compaction after mixing on compressive strength.





Figure 3. Effect of delayed compaction after mixing on density.

Figure 4. Effect of delayed compaction after mixing on percent weight loss.

reduced the compressive strength by 55 percent and the density by 13 percent. The behavior of the silty clay loam differed from the silty loam in that larger compressive strengths were developed in the silty clay loam when the mix was compacted  $\frac{1}{2}$  hr after mixing with moisture contents at or below optimum than specimens compacted immediately after mixing, and attained lower durability than the silty loam. The dry weight density decreased as time delay increased for all moisture contents.

The sandy loam soils W-7 and W-8 are comparatively better materials for soilcement stabilization. With less cement content than had been used for the other soils, sandy loam attained larger compressive strengths and proved to be more durable under the wet-dry test. The effects of time delay on the compressive strength, density, and durability of the sandy loam soils with optimum cement and moisture content are shown, respectively, in Figures 8, 9, and 10. Table 1c gives the data for these soils.

The compressive strength and the dry weight density for moisture contents at or below optimum both decreased as the time delay before compaction was extended. But at moisture contents above the optimum, the compressive strength and density both increased to a certain level before starting to decrease again.

TA	BL	E	1	
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			5011	DATA				
Call	AASHO	Cement	Water Content (\$)	Liquid	Plasticity	Composition (%)		
301	Group	(%)		Limit	Index	Sand	Silt	Clay
			(a) Silty	Loam So	ils			
W-1	A-4	10	17.0	26.5	8.0	28.0	60.0	12.0
W-2	A-4	10	17.0	28.2	8.4	6.5	79.0	14.5
W-6	A-6	12	15.5	30.6	11.1	31.6	51.4	17.0
			(b) Silty Cl	ay Loam	Soils			
W-3	A-4	10	17.5	28.0	9.3	26.0	53.0	21.0
W-4	A-6	12	17.0	37.0	19.0	19.0	62.0	19.0
W-9	A-6	12	20.0	37.5	14.0	28.0	51.0	21.0
			(c) Sandy	Loam So	oils			
w-7	A-2-4	7	15.0	25.0	6.6	71.0	16.0	13.0
W-8	A-2-4	7	10.5	18.4	1.0	66.6	21.4	12.0
			(d) Si	lty Soils				
W-5	A-4	10	17.5	28.2	5.8	4.4	80.3	15.3
W-10	A-6	12	19.0	36.0	11.0	0	86.5	13.5
_				_				



Figure 5. Effect of delayed compaction after mixing on compressive strength.



Figure 6. Effect of delayed compaction after mixing on density.



Figure 7. Effect of delayed compaction after mixing on percent weight loss.



Figure 8. Effect of delayed compaction after mixing on compressive strength.





Figure 9. Effect of delayed compaction after mixing on density.

Figure 10. Effect of delayed compaction after mixing on percent weight loss.



Figure 11. Effect of delayed compaction after mixing on compressive strength.



Figure 12. Effect of delayed compaction after mixing on density.



Figure 13. Effect of delayed compaction after mixing on percent weight loss.

moisture-density test, respectively, when the compaction of the mix is delayed.

Throughout each test, the moisture content of the mix remained almost constant. The durability of the compacted soil and portland cement mixtures, in most cases, proved to be the limiting factor on the working range of the soils tested. There was no direct relationship between the compressive strength and durability which could be generalized so that it might apply to all soils or even to soils of the same type. In

Soil W-8 behaved differently from soil W-7 in that: (a) re-mixing the soil did not seem to affect the loss of compressive strength at all; and (b) at moisture contents 2 percent above optimum, though the compressive strength increased with time to a certain value before starting to decrease again, the dry weight density consistently decreased with time delay. A 4-hr delay in compaction reduced the compressive strength by 62 percent and the density by 16 percent.

The data on silts, W-5 and W-10, are given in Table 1d. The effect of delayed compaction on the compressive strength, dry weight density, and durability are shown in Figures 11, 12, and 13, respectively.

Soil W-5 attained rather low compressive strengths, yet it proved to be a very durable material for soil-cement stabilization. The dry weight densities and compressive strengths at and 2 percent below optimum moisture contents decreased as the time between mixing and compaction were increased, while at water contents above the optimum they both increased to certain values before they decreased again. Re-mixing this soil before the 6-hr compaction did not show any appreciable increase in strength. An increase in cement content improved the strength but had little effect on the durability.

Soil W-10 had a higher compressive strength and lower durability than W-5. An increase of 2 percent in the cement content increased the strength but did not have much effect on the durability. There was a consistent decrease in density at all different moisture contents when the time delay before compaction was increased. The compressive strength at above optimum moisture contents increased with the time by approximately 10 percent before starting to decrease again.

A study of the results shows that soils do not produce expected durability and compressive strengths at optimum moisture and cement content as determined by the criteria established by the Portland Cement Association and by the standard



Figure 14. Hypothesis of cementation in fine (left) and coarse (right) grained soils when compaction is delayed.

some cases, even though there were large losses in weight as a result of the wet-dry test, the same soil showed very high compressive strength, while in other instances there were lower losses in weight for soils with smaller compressive strengths.

To analyze the results of the tests performed, some assumptions based on observations made during testing need to be formalized.

A most important occurrence detected during the wet-dry test was that during the brushing cycles the particles loosened

from specimens exhibited significant characteristics in relation to delayed compaction and to the grain size of the soil itself.

It was observed that when the specimen was compacted immediately after mixing and when the soil was composed of fine-grained material, the grains loosened by brushing had approximately the size and shape of the original soil particles. This shows that the test specimen en masse was the product of the cementation among the individual grains themselves (Fig. 14(left)). In specimens containing coarse-grained soils and specimens compacted after a time delay after mixing, the grains loosened were considerably larger in size, indicating that groups of individual particles were cemented together before compaction was performed. This conglomeration effect is represented in Figure 14 (right).

According to Lilley, the optimum moisture content of the cohesive soils does not change significantly as a result of time delay before compaction, while in sand it increases from 12 to 17 percent after a 24-hr delay. This fact explains the behavior of the soils tested when the moisture content at mixing was above optimum. The silty clay soils, the most cohesive soils tested, lost compressive strength and density consistently with time delay, while the other less cohesive soils attained higher strength for a certain delay in compaction before they started to lose strength again. The silt, silty loam, and sandy loam soils all showed higher strengths after some delay when the moisture contents were above optimum. This behavior obviously has been caused by the increase in optimum moisture content requirement due to time delay to where the excess water present during mixing was enough to have the mix reach the optimum moisture content some time after mixing. This phenomenon was not detected in the silty clay loam.

At or below optimum moisture and cement contents the silty clay loam acquired higher compressive strength when compacted  $\frac{1}{2}$  hr after mixing than when compaction was performed immediately after mixing.<sup>1</sup>

Although some of the soils in Louisiana classified as silts might be suitable for soilcement stabilization from the standpoint of meeting minimum requirements for compressive strength and durability, their use is undesirable because of their inconsistent characteristics. The two silts tested showed contradictory results in that while one, W-5, proved to be a very durable material though it attained low compressive strengths, the other soil, W-10, acquiring larger compressive strength proved to be much less durable (see footnote 1). An increase in cement content increased the compressive strength of the silts but did not have much effect on their durability.

It has been observed that in connection with the use of these soils in soil-cement stabilization, bituminous hot-mix concrete, and portland cement concrete, the grains of the river-deposited silts have rounded shapes with smooth surfaces quite often referred to as the "ball bearing" silts. This shape factor results in low durability and low compressive strength.

<sup>&</sup>lt;sup>1</sup>Detailed tabular matter not reproduced here is available from the Highway Research Board at cost of handling and reproduction. Refer to XS-11, Highway Research Record 198.

The compressive strength of soil-cement is a function of the size and shape of the soil particles and of the bond created by the cementation process. Durability is mainly a function of the bond created among the particles by cementation. It should be noted that in both cases the bond created still remains dependent on the size and shape factor; however, in the case of compressive strength, the shape and size may contribute additional strength by proper load distribution and interlocking of particles.

In fine-grained soils, the particles have larger areas of contact which result in a stronger bond due to cementation. More cement is needed in this case because there are larger surface areas to cover. The structure of the soil, on which the strength of the mixture depends, is of the honeycomb type.

In granular soils, the particles are cemented together only at points of contact. The area of contact is small, thus requiring less cement.

An increase in cement content above the optimum does not necessarily improve the quality of soil-cement (see footnote 1). If an adequate amount of cement is mixed with the soil, an optimum coverage of the surface area of the aggregate is obtained. An additional application of portland cement does not insure a better soil-cement product.

During testing, the batch of soil-cement mix, in some cases, had to be re-mixed either immediately before the 4-hr compaction or the 6-hr compaction. Re-mixing did not reduce the loss in compressive strength and dry weight density of all the soils. It is the opinion of the author that the reason for such inconsistency was mainly due to the presence of free water in the mix at the time of re-mixing plus some free portland cement which had not already gone through the process of hydration. Re-mixing, in this case, brings these two agents together, thus producing an improved product.

A review of the results of this study indicates that changes in compressive strength, density and durability of compacted soil-cement mixes occur at different periods of delayed compaction time. However, the only typical trend established by all soils for all variables is the trend established by the considerable loss of compressive strength, durability and density as a result of 2 hr of delay in compaction. It is very significant that the initial setting time of the cement paste was found to be 2 hr and 5 min. This indicates that the behavior of the variables being investigated is directly related to the initial setting time of the cement paste. When a mixture of soil-cement has been left relatively loose for a period of time, the cementation reaction takes place during this period. Although there is no intimate contact between all soil particles and the cement gel present in the mixture, there is enough cementation taking place to create large cemented lumps of soil-cement mixtures.

At the end of 2 hr or longer periods of time, if this cemented mixture is disturbed and bond between the ingredients of the mix is broken during the compaction operations, the improvement obtained by the presence of cement is minimized or nullified. Unless additional cementing agents are added, or unless unreacted cement and free moisture are available in the mixture, there will be very minor amounts of cementation taking place, after the compaction is completed. This explains the loss of strength and durability occurring after a period of time delay exceeding the final setting time of the cement gel. The loss in density is a result of the changes in the physical characteristics of the soil-cement mix after a period of time. For different types of cement with different setting time characteristics the losses and the fluctuations appearing in the mix due to delay in compaction shall also differ.

A parallel study performed at the Louisiana State University using dispersant admixtures in soil-cement mixes indicated that the cementation reaction can be delayed without ill effects, and in most cases resulting in considerable improvements in the final product, by the use of these agents. Since most construction procedures require a time delay between mixing and compaction of soil-cement mixes in the field, the use of dispersant admixtures will be advantageous in delaying the setting of the cement gel in the mix.

#### CONCLUSIONS

Based on the results of this study, the following conclusions are derived:

1. Each of the soils tested showed different patterns of behavior when tested for compressive strength, durability, and density within the first 2 hr of the compaction

period. One given pattern of behavior of all variables for all soil-cement mixes de not exist.

2. The durability, compressive strength, and density of soil-cement mixes con pacted at, below, or above their optimum cement and moisture contents decrease ( siderably and uniformly after a delay of 2 hr or more in compaction of the soil-cer after mixing. Some of the above variables show periodic increases before they tak turn to a sharp drop. These, from an engineering standpoint, are insignificant fluctuations.

3. The loss of compressive strength, durability, and density due to delayed cor paction can become so great in intensity that any physical improvements to be derifrom the addition of portland cement to the mix are reduced.

4. The effect of delayed compaction is directly dependent on the final setting tin of the cement used. Compaction in soil-cement mixes should not be delayed beyon the initial setting time of the cement gel. It is also recommended that a factor of ( be used as a multiplier of the initial setting time to determine the maximum allowa period of delay in compaction.

5. Since a delay between mixing and compaction cannot be avoided, steps should taken to counteract the detrimental effect of delayed compaction. When the delay e ceeds the specified limits already established, one of the three following methods n be employed to compensate for losses: (a) Increase the thickness of the base to ena it to carry a specified load. (b) Use set-retarding agents such as calcium lignosulfe nate and hydroxylated carboxylic acid in trace amounts to slow down the cementatio process. (This is the most economical method.) The use of these additives should be further studied before any conclusive statements can be made. (c) If economical feasible, allow the use of plant-mixed soil-cement, which can be delivered and com pacted in a period of time not exceeding 0.80 times the initial setting time of the cement as determined by the ASTM designation C 266-64.

6. The optimum moisture content as obtained by standard compaction test of so cement mixes changes due to delay between mixing and compaction.

7. When the compaction of a soil-cement mixture is delayed, the optimum cemcontent determined using the criteria developed by the Portland Cement Associatio does not necessarily produce the best final product obtainable. It should be noted that the criteria established by the Association are valuable guidelines and their us results in a satisfactory final product when the compaction is not delayed beyond th initial setting time of the cement.

8. Considering the success of the soil-cement bases constructed to date and the results of this study, it is concluded that the criteria used presently for durability and strength should be revised and relaxed with provisions of counteracting the adv effect of delayed compaction. It is obvious that the initial product presently obtain where the compaction has been delayed is far inferior to the expected results, whic in most cases are established in the laboratory with a very short period of time del between mixing and compaction. Therefore, since material with lower strength an durability has produced excellent performance records, it will be more economical to design these mixtures for requirements lower than the ones presently in use.

# **Reactions and Strength Development in Portland Cement-Clay Mixtures**

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> The purpose of this study is to extend the knowledge and understanding of the reactions and rates of reactions in mixtures of cement and relatively pure clays. The investigative approach utilized was to examine the materials and reacted mixtures physically by optical microscopy and X-ray diffraction, and chemically by measuring the change in pH with time of dilute suspensions of mixtures, analyzing the leachates of distilled water and 1.5 N HCl obtained from mixtures cured for 1, 3, 7, 14, 56 and 112 days for Ca, Al, and Si, and by making chemical analyses of the untreated clay materials.

> Measurements of strength development with a Type II portland cement were also made and compared with similar measurements made on cement-clay mixtures containing a Type I portland cement.

> The following conclusions are presented: (a) the cement in the cement-Anna kaolinite mixtures experienced the most rapid hydration, with the cement-Fithian illite mixtures hydrating one-half to three-fourths, and the cement-Aberdeen montmorillonite mixtures hydrating one-tenth to one-fifth as rapidly as the cement-Anna kaolinite mixtures; (b) the strength gain appears to result from early flocculation of the clay particles (thus making their joint cementation easier), hydration of the cement and possible chemical combination of SiO2, Al2O3 and Ca(OH)<sub>2</sub> to create additional cementitious particles, and crystallization of Ca(OH)<sub>2</sub> which cements discrete particles as well as floccules to each other; (c) the clays suffered deterioration from the alkaline environment of the hydrating cement in the order Aberdeen montmorillonite, Anna kaolinite, and Fithian illite; (d) the hydration of cement grains may be restricted by encapsulation of the cement by very fine-grained clay and recently crystallized gels of calcium silicate hydrate and, perhaps, calcium aluminate hydrate; and (e) up to a curing period of 7 days, the Type I portland cement-clay mixtures developed somewhat greater strengths than the Type II mixtures because of the larger content of tricalcium aluminate and similar content of CaSO<sub>4</sub> in the Type I portland cement.

•IN THE first progress report (Sherwood and Noble, <u>11</u>) on this project, initial studies of the reactions and reaction products in portland cement-clay mixtures were presented. It was noted that:

1. There was a significant gain in strength in cement-treated clay samples relative to untreated clays.

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TABLE 1	
CHEMICAL AND PHYSICAL ANALYSES	OF
CEMENTS UTILIZED*	

Category	Type I	Type I
Oxide Analysia	ategory Type I alysis: 21.20 5.90 2.34 64.44 2.83 2.18 8 0.08 0.12	
SIO.	21 20	22 62
Al-O-	5 90	3 32
FerOn	2 34	3.36
CaO	64 44	63 63
MgO	2 83	2.58
80-	2 18	2 21
Ign loss	0.48	0.85
Na-O	0.12	0.00
K-0	0.73	0.60
Na-O emiy	0.60	0.48
Free CaO	1.00	0.67
Compounds (Calculated):		
C.S	52.0	53.5
CaS	21.6	24.5
CA	11.68	3,12
CAF	7.11	10.21
CaSO 4	3.71	3.76
Fineness:		1111
\$ passing No. 325 mesh	91.8	93,9
Blaine (cm <sup>2</sup> /gm)	3396	3396

\*Cements and analyses courtesy of Standard Lime and Cement Division of Martin-Marietta.

TA	BLE 2
PHYSICAL	PROPERTIES

Properties	Anna Kaolinite	Fithian Illite	Aberdeen Montmorillonite
Size Ranges:			
>16 µ	20.8	23.5	20.0
16-8µ	6.0	14.2	7.2
8-4µ	12,0	11.1	10.4
4-2µ	10.6	6.6	3.5
2-1µ	13.6	9.5	14.9
< 1µ	37.2	35.1	44.0
Cation exchange			
capacity (me/100gm)	18	28	64.7
Maximum density (pcf)	116.8	103.9	70.1
-			
Optimum moisture content (%)	13.82	20.84	46.48

2. The several clay species develop different strengths; the more hydrophil clays (montmorillonites, and attapulgit developed the least strength gain; the 1 hydrophilic clays (kaolinite, illite, grun developed the greatest strength gains.

3. The portland cement appeared to fully hydrated after curing for 43 days 79 C.

4. No crystalline lime was detected X-ray diffraction.

5. Based on the presence of the 3.0" 3.09 A peak, a calcium silicate hydrate seems to have formed.

All the specimens prepared for the str tests were cured for 7 days in a moisturoom at 21 C, and the specimens prepa for chemical and X-ray diffraction ana were mixed at a water to solids ratio o 1 with the exception of attapulgite and Wyoming montmorillonite which were mixed at water to solids ratios of 3 and respectively, and cured for periods of 4 and 100 days at 100 percent relative hu midity and 79 C.

The work performed in a later phase (Noble, 10) was designed to further the understanding of the nature of the reac tions and to establish rates of reaction cement-clay mixtures. Strength data f cement-clay mixtures using a Type II cement, because of its low tricalcium aluminate content, were obtained and copared with the strength data from the f phase for which a Type I cement was u The chemical and physical analyses of these two types of cement are given in Table 1.

# EXPERIMENTAL PROCEDURES

Eight clays were employed in the initial phase of the project (Sherwood and Noble. These 8 clays were also used in the strength studies with a Type II portland cement, the investigation of the change in pH with time of dilute suspensions of cement-clay mixtures, and in the total chemical analysis. However, to permit a more exhaustive study of individual specimen in the principal body of studies for this phase of the investigation, we decided to use only 3 clays, Anna kaolinite, Fithian illite, and Aberde montmorillonite. The physical properties of these clays are given in Table 2. The mixtures were prepared with 40 percent Type I portland cement and were cured at 100 percent relative humidity and 25 C for periods of 1, 3, 7, 14, 56, and 112 days. water to solids ratio of 1 was used for all the mixtures prepared for the second phas of this investigation. Twelve specimens per clay were prepared, 2 for each curing period, 1 for chemical and X-ray diffraction analyses and 1 for petrographic micros studies.

#### Physical Studies

Petrographic Investigation-Inasmuch as the petrographic microscope and X-ray diffraction techniques provide data that supplement each other, it was decided to limit

the number of specimens studied with the petrographic microscope. Therefore, powders of the untreated clays, and the clays mixed with 40 percent portland cement and cured for 1 and 43 days, were studied utilizing index oils. The material cured 43 days had been stored as a powder in a relatively airtight vial for approximately 1 yr. Thin sections were cut from 112-day and 2-yr 40 percent mixtures cured at 100 percent relative humidity and 25 C. Two  $\frac{1}{2}$ -in. thick disks were cut from cylinders of these mixtures which had been dried at 80 C. The disks were then impregnated under vacuum with "Permount," a synthetic resin cut with 40 percent of toluene. After saturation, which usually required 24 hr, the samples were put into an oven (80 C) for 24 to 48 hr to harden. The disks were then ground on a plate glass and mounted with an epoxy, Resiweld Adhesive 7004. After hardening, one of the samples was ground down to approximately  $30\mu$  and the other to approximately  $15\mu$ . Cover glasses were added using index oil so that removal and use of another oil could easily be accommodated.

X-Ray Analysis—Powder specimens were mixed in Nujol and spread in a shallow well holder. They were X-rayed through an arc from 2° two theta to 40° two theta. The X-ray diffractometer used was a G. E., model XRD-5 with Cu K  $\alpha$  radiation and a Ni filter.

## **Chemical and Physicochemical Studies**

pH of Dilute Suspensions of Mixtures—Dilute suspensions of cement-clay mixtures  $(0.3 \text{ gm clay} + 0.2 \text{ gm portland cement in 70 ml of } H_2O)$  were prepared and tightly capped. The variations with time of their pH's were measured. Nitrogen was played over the surface of the suspension to prevent carbonation when the pH measurements were made. The variation in pH of the several clays with variations in mix concentrations was also investigated for supplemental information.

Analyses of Leachates Made With Distilled Water and HCl-During the organization of this investigation, consideration was given to the importance and source of soluble Ca in the mixtures. Inasmuch as the hydration products of alumina, silica and iron are extremely insoluble in the liquid phase of hydrating cement (Copeland and Kantro, 3), it appears that the Ca(OH)<sub>2</sub> produced by the hydrating cement is the most soluble of the calcium compounds present. However, it was realized that Ca(OH)<sub>2</sub> is not especially soluble. Therefore, the quantity of distilled leach water used was more than needed to dissolve all the Ca(OH)<sub>2</sub> that could be produced by complete hydration of the cement present.

Two-gram aliquots of the various cement-clay mixtures were repeatedly leached with 200 ml of distilled water. The leachings were carried out in a nitrogen atmosphere to prevent carbonation. At least three leachings were run with distilled water prior to an acid leach, using 200 ml of 1.5 N HCl. The leachings were accomplished by placing cement-clay mixtures and distilled water in a stainless steel cup and rapidly agitating them with a Hamilton Beach mixer, model No. 33. An agitator blade of the standard type used to disaggregate soils was used. The pH was taken before and after removal of the suspended material, which was removed by centrifugation. Analyses of the leachate were made for Ca, Al, and Si, and are reported and discussed as oxides. The quantitative analyses for these selected cations were made using testing procedures currently used in the Council's geochemistry laboratory for analysis of soils and cement. Calcium was determined by titration with EDTA as described by Davis (4). Al and Si were determined by absorption spectrophotometric techniques also as detailed by Davis.

Total Chemical Analysis—A chemical analysis, which first required fusion of the specimen, was made of each of the untreated clays to provide supplementary information.

#### **RESULTS AND DISCUSSION**

#### **Physical Studies**

Observations With the Petrographic Microscope—These observations were divided into two parts: (1) work with powdered samples and index oils, and (2) work with thin sections and index oils.

Powders—Untreated Anna kaolinite occurs predominantly in well-developed flocs averaging about  $30 \mu$  in diameter. Quartz of comparable size is clear and relatively



Figure 1. Photomicrographs of powders; enlargement X70: (a) untreated Anna kaolinite, (b) Annc kaolinite plus 40 percent cement, (c) untreated Fithian illite, (d) Fithian illite plus 40 percent cement, (e) untreated Aberdeen montmorillonite, and (f) Aberdeen montmorillonite plus 40 percent cement.



Figure 2. Photomicrographs of thin sections of cured clay-cement mixtures: (a) Anna kaolinite X70, (b) Anna kaolinite X270, (c) Fithian illite X70, (d) Fithian illite X270, (e) Aberdeen montmorillonite X70, and (f) Aberdeen montmorillonite X270.

free of clay coating (see Fig. 1a). Accelerated curing results in larger  $(100 \mu)$  flocs and partially clay coated quartz (see arrows, Fig. 1b). Particles of lime, Ca(OH)<sub>2</sub>, occur in the mixture cured one day at 25 C.

The slightly coarser grained Fithian illite contains mica shards. Flocculation is ragged in the untreated clay (see Fig. 1c), but is much better developed in the mixture subjected to accelerated curing (see Fig. 1d), with flocs up to  $170 \,\mu$  common.

The untreated Aberdeen montmorillonite contained minor amounts of calcite, was very fine grained and only weakly flocculated (see Fig. 1e). After one day with 40 per cent cement the clay was strongly flocculated (see Fig. 1f).

Thin Sections—The Anna kaolinite mixtures were characterized by profuse, welldeveloped clay flocs. Profuse irregular patches of well-crystallized lime, Ca(OH)<sub>2</sub>, occur throughout the slide. This lime was deposited on clay flocs and quartz grains and has even cemented two discrete grains together (see Fig. 2a and b). The size of the flocs,  $60-80 \mu$ , is approximately the same as noted for the powders.

Flocculation in the Fithian illite mixtures was not as pronounced as it was in the kaolinite and montmorillonite mixtures. Mica shards are still present (see Fig. 2c and d). Irregular but fairly extensive patches of lime occur, but they are neither as profuse nor as well developed around the preexisting grains as in the kaolinite mixture

In the Aberdeen montmorillonite mixtures, flocculation is well developed with most flocs approximately  $40 \mu$  in diameter and with a few ranging up to  $250 \mu$  (see Fig. 2e and f). Lime is absent, but some high birefringent grains, probably calcite, are scattered throughout the mixture.

**Observations showed that:** 

1. Flocs or aggregations of the clay are much better developed and larger in the cement mixtures than in the untreated clays.

2. Where quartz is present, the cement-clay mixtures appear to coat the quartz grains to a much greater extent than the untreated clays.

3. Unhydrated cement could not be identified in 40 percent cement-clay mixtures cured for 43 days at 79 C.

4. Lime was common in the thin sections of cement-Anna kaolinite mixtures as a coating, or as cement on or between the clay flocs and quartz grains.

5. Lime was common in the cement-Fithian illite mixtures as very irregular patch

6. Lime was not observed in the cement-Aberdeen montmorillonite mixtures.

X-Ray Analyses of Mixtures-By using the naturally occurring quartz in the clays as an internal standard, it was possible to make semi-quantitative estimates of the relative changes in quantity of several of the hydration products of cement occurring in the mixtures. Inasmuch as Ca(OH)<sub>2</sub> is a product of the hydration of two of the principal anhydrous compounds of portland cement and is used in the formation of several compounds, it is interesting to note the variation with curing time in quantity of crystalline Ca(OH)<sub>2</sub> and calcium monosulfoaluminate in the several cement-clay mixtures. Observation of the variations in quantity of crystalline Ca(OH)<sub>2</sub> may permit speculation as to the extent of hydration of the cement and the extent of interaction between the Ca(OH)<sub>2</sub> and the clays. In considering the early hydration reactions of a typical portland cement, Greene (8) states that very early after the addition of water to form a paste, "The C<sub>3</sub>S is hydrating, and as evidence of this, crystalline calcium hydroxide begins to form. This compound can sometimes be detected by DTA after 2 or 3 hours. It continues to increase in amount with time, and is a rough indicator of the development of the calcium silicate hydrate phase." Greene's data also show a very steady, significant increase in crystalline Ca(OH)<sub>2</sub> up to 7 days, which is the longest curing period for which data were presented. If, as suggested, the extent of crystallization of  $Ca(OH)_2$  is commensurate with the extent of hydration in a portland cement paste, then the X-ray reflections should approach an apparent maximum intensity by a curing time of 7 to 10 days, when according to Copeland, et al (2), 72-80 percent of the C<sub>3</sub>S, 78-87 percent of the C3A, and 36-46 percent of the C2S should be hydrated.

A comparison of the diffractogram traces (see Figs. 3-5) shows that in the Anna kaolinite mixtures there was an increase in crystalline  $Ca(OH)_2$  up to a curing time of . 14 days and that from 14 to 112 days there was little or no apparent change. The 8.2 A





Figure 3. X-ray diffractogram patterns of cement-Anna kaolinite mixtures for the various curing periods.

Figure 4. X-ray diffractogram patterns of cement-Fithian illite mixtures for the various curing periods.

reflection of calcium monosulfoaluminate is quite distinct after one day of curing, and by the third day attains a maximum intensity which is maintained through 112 days. In the Fithian illite mixtures, there was a pronounced increase in crystalline  $Ca(OH)_2$  up to the 7-day curing period; followed by an apparent decrease at 14 days which continued to 56 days, when it reached the level it maintained through the 112-day curing period. The quantity of calcium monosulfoaluminate increased through 3 days, maintained that concentration to 14 days, and diminished through 56 and 112 days. After 1 day of curing, there were no  $Ca(OH)_2$  or calcium monosulfoaluminate reflections for the Aberdeen montmorillonite mixtures; 3-day material showed distinct reflections for both compounds; at 7 days the Ca(OH)<sub>2</sub> reflection was very weak and the calcium monosulfoaluminate reflection was absent. There was no reflection for either compound from 14 days through 112 days. The diffractogram traces shown in Figure 6 are of cementclay mixtures similar to those discussed above, except that they were cured for 2 yr. The prominence of the crystalline  $Ca(OH)_2$  in the kaolinite mixture seems to discount the possibility of any extensive  $clay-Ca(OH)_2$  interaction. The paucity of crystalline Ca(OH)<sub>2</sub> in the illite mixture, and its absence in the montmorillonite mixture, suggests that either  $clay-Ca(OH)_2$  interaction is extensive in these mixtures or little  $Ca(OH)_2$  is produced initially because of a lack of cement hydration. It will be shown later, during a discussion of the leachates, that those processes which use Ca(OH)<sub>2</sub> rapidly could not use enough  $Ca(OH)_2$  to account for the differences in  $Ca(OH)_2$  leached from the kaolinite mixtures, as compared to that leached from the illite and the montmorillonite mixtures; and that restricted hydration of the cement appears to be a plausible explanation of the paucity of crystalline  $Ca(OH)_2$ .

On the basis of the X-ray diffraction data, the relationship of the rate of hydration of the portland cement with the subsequent production of crystalline  $Ca(OH)_2$  to the rate of usage of crystalline  $Ca(OH)_2$ , as represented in Figure 7, can be observed. It is obvious that the accumulation of crystalline  $Ca(OH)_2$  in the kaolinite mixtures most





Figure 6. X-ray diffractogram patterns of cementclay mixtures cured for two years.

Figure 5. X-ray diffractogram patterns of cement-Aberdeen montmorillonite mixtures for the various curing periods.

closely approximates Greene's description of the buildup of crystalline Ca(OH in a cement paste. It also appears that the buildup of crystalline Ca(OH)<sub>2</sub> is slower in the illite mixtures and very slow in the montmorillonite mixtures.

As mentioned previously, this can be caused by usage or lack of production. Usat is certainly a factor when the crystalline  $Ca(OH)_2$  decreases. However, the importance of this usage depends on the degree of hydration of the cement.

#### **Chemical and Physicochemical Studies**

It might be beneficial to the following discourse to list some of the processes, whic are thought to occur in more isolated systems and which probably occur in the more complex system of cement-clay mixtures, in as near to the chronological order of their inceptions as is possible.

- 1. Hydration of cement compounds with production of Ca(OH)<sub>2</sub>.
- 2. Adsorption of Ca onto the cation exchange sites.

3. Adsorption of Ca(OH)<sub>2</sub> on clay surfaces (relatively easily leached by distilled water, Diamond and Kinter, 7).

- 4. Occurring concurrently.
  - (a) Encapsulation of Ca(OH)<sub>2</sub> crystallites by gels of calcium silicate hydrates and possibly calcium aluminate hydrates and clay particles.
  - (b) Possible encapsulation of cement grains by clay and gels.
  - (c) Chemical combination of Ca(OH)<sub>2</sub> and suggested SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>.

<u>pH of Dilute Suspensions of Mixtures</u>-Figures 8 and 9 show that 7 of the 8 pH plots from the dilute suspensions of mixtures belong to the same family of curves, with Wyoming montmorillonite being the exception. Despite the wide variation in the pH's of the untreated clays (see Table 3), the mixtures start at a high pH of 11.52 to 11.65 because of the rapid production of Ca(OH)<sub>2</sub> by the hydrating cement. The pH of 0.2-gn



Figure 7. Schematic representation of the rate of accumulation of crystalline Ca(OH)<sub>2</sub> in the cementclay mixtures.

Type I portland cement in 70 ml of distilled water with nitrogen played over the surface of the water was 12.00 at the start of hydration and 11.83 after 15 min of hydration. Thus the effect of the clay minerals in the mixtures is readily apparent. This instantaneous decrease in pH probably corresponds to the very rapid adsorption of  $Ca(OH)_2$ 





Figure 9. pH vs time.

as reported by Diamond and Kinter (7). The pH curves rise slowly from the start, and with the exception of the curve for the Wyoming montmorillonite, reach a peak at approximately 7 days with pH's which range from 12.10 to 12.25. Then the curves start to drop gradually, until at 28 days they start a rapid decline. From 28 t 274 days the curves within the family have their greatest variation. The steepest par of the curves occur between the following times:

Clay in Cement-Clay Mixture	Time Period in Days		
White Springs montmorillonite	28- 81		
Anna kaolinite	81-140		
Aberdeen montmorillonite	81-274		
Fithian illite	81-274		
Attapulgite	81-274		
Grundite	140-274		
Dry Branch kaolinite	140-274		

The steepest parts of the curves probably represent the times at which the  $Ca(OH)_2$ produced by hydration of the cement was depleted. Inasmuch as X-rays showed that all these mixtures had become carbonated, it is possible that the depletion of the dissolved  $Ca(OH)_2$  was due to carbonation.

Perhaps the most startling aspect of the pH curves is their similarity through 7 day of reacting. If the one variant, White Springs montmorillonite, is ignored, this similarity or tight bunching of the curves continues to 28 days at which time the pH's range from 12.0 to 12.2. It seems unlikely that if a Ca(OH)<sub>2</sub> consuming process were extremely active that the pH's could be maintained at the 12.0 and above level through 28 days. It should be kept in mind that these specimens were dilute suspensions rathe than pastes, and that the reactions may be quite different from those occurring in the pastes.

ph of clays				
Clay	рН			
Aberdeen montmorillonite	9.1			
White Springs montmorillonite	4.92			
Wyoming montmorillonite	9.4			
Fithian illite	4,18			
Grundite	7.05			
Anna kaolinite	6,48			
Dry Branch kaolinite	4.88			
Attapulgite	9.6			

2. 0.4-gm clay to 70-ml distilled water used.

All the mixtures, with the exception of the Wyoming montmorillonite, have pH's between 8.22 and 8.50 during the period of 274 to 529 days. The Wyoming montmorillonite reached a peak pH of 12.22 at a time of 1 day, maintained that pH until the 15-day reading, and then started a slow decline to a pH of 11.78 at 529 days.

X-ray analyses of the mixtures, after drying at 80 C, show reflections at 7.70 Å and 8.07-8.30 Å in all but the White Springs montmorillonite. These reflections may

be interpreted as indicating the presence of a tricalcium aluminate hydrate, similar to that type produced by the long-term interaction of  $Ca(OH)_2$  and  $Al(OH)_3$ .

Analyses of Leachates Made With Distilled Water and HCl—Data for the chemical analyses of the distilled water and acid leachates of the cement-clay mixtures are presented in Tables A-1 to A-9 in the Appendix.

Throughout the following discourse, reference is made to the percentage of CaO leached in terms of how much could be leached if complete hydration of the cement occurred. The calculations for these percentages were based on  $Ca(OH)_2$  being 25 percent of fully hydrated cement (Brunauer and Copeland, 1). Reference is also made to the theoretical maximum amount of Al<sub>2</sub>O<sub>3</sub> capable of being leached from the mixtures with HCl, provided the clays are not altered by exposure to the hydrating cement environment. This quantity of Al<sub>2</sub>O<sub>3</sub> should be no more than the sum of the quantity of Al<sub>2</sub>O<sub>3</sub> in the cement plus the quantity of Al<sub>2</sub>O<sub>3</sub> leached with HCl from the untreated clay.

 $Ca(OH)_2$ —An attempt will be made to use the quantity of CaO leached from the mixtures as an indication of the relative rates of production of  $Ca(OH)_2$  and thus also as an indication of the relative rates of cement hydration.

A brief discussion of the sources of  $Ca^{++}$  and its degree of availability should prove beneficial to the interpretation of the CaO data. Calcium may be leached from:

1. Crystalline  $Ca(OH)_2$  and  $Ca(OH)_2$  dissolved in interstitial water.

2. Ca(OH)<sub>2</sub> adsorbed on the external surfaces of the clays and other alumino-silicates.

3. Ca<sup>++</sup> adsorbed on the internal and external cation exchange sites of the clays.

4. The hydrated cement compounds.

5. Pozzolanic compounds, from pozzolanic reaction of the  $Ca(OH)_2$  with the clays.

6. The anhydrous cement compounds.

Calcium is easily leached by distilled water from the first two sources only. Although the other sources will undoubtedly contribute  $Ca^{++}$  to the leachate, the quantity contributed would be expected to be minor. The  $Ca^{++}$  held in the cation exchange sites and that consumed in the pozzolanic reactions was taken from the  $Ca(OH)_2$  produced by the hydrating cement. Any interpretation of hydration rate based on the  $Ca^{++}$  removed by the distilled water leachate will yield an erroneously slow hydration rate if some allowance is not made for the  $Ca^{++}$  so consumed.

There was a rough but decided correspondence between the quantity of CaO leached from the cement-clay mixtures and the pH's of the leachates, which, coupled with the magnitude of the pH's, suggests that the CaO was principally derived from  $Ca(OH)_2$ .

The change in quantity of CaO leached from the mixtures (Fig. 10) reflects the change in production and consumption of  $Ca(OH)_2$ . As the quantity increases, production exceeds consumption, and the converse is true. It is interesting to note that from 3 to 112-days curing the relationship of production and consumption of  $Ca(OH)_2$  in the Anna kaolinite and Fithian illite mixtures is approximately the same, as illustrated by the parallelism in the curves. In the Aberdeen montmorillonite-cement mixtures, the relationship of production and consumption of  $Ca(OH)_2$  is different from that of the other mixtures, with the quantity of CaO leached reaching a maximum level at 3 days and diminishing from 14 through 112 days.

Figure 10 also shows the difference in quantity of CaO leached from the several cement-clay mixtures for the various periods of curing. The Anna kaolinite mixtures



Figure 10. CaO (ppm) derived from first distilledwater leachate.

yielded 130 to 215 ppm more CaO than did the Fithian illite mixtures, and 415 to 520 ppm more than the Aberdeen montmorillonite mixtures. These differences in easily leachable CaO may be interpreted as indicating the extent of cement hydration in the several mixtures, provided no processes are active which tend to combine the calcium in compounds only slightly soluble in distilled water. Cation exchange on internal and external exchange sites and pozzolanic reaction are the principal processes known to occur in cement-clay mixtures which will tie up calcium in a relatively insoluble form.

The difference in cation exchange capacity between Anna kaolinite and Fithian illite, and between Anna kaolinite and Aberdeen montmorillonite, can account for 16-26 and 30-38 percent, respectively, of the differences in quantity of CaO leached from their mixtures. If pozzolanic action is considered to have consumed the remainder of the difference in easily leached CaO, it seems that the Fithian illite and Aberdeen montmorillonite mixtures would have developed greater strengths. Should cation exchange and pozzolanic action not account for the differences, it seems the only other plausible explanation is restricted hydration of the cement.

Diamond (5) has shown that silica, in solution, will rapidly react at the surface of  $Ca(OH)_2$  crystals to create an impermeable envelope of reaction product which effectively shields the remaining  $Ca(OH)_2$  from further reaction with the silica. Considering the alkaline nature of the micro-environment about a particle of hydrating cement, it seems possible that the more hydrophilic and/or finer grained the clays, the more they would tend to form relatively impermeable envelopes about the cement particles, thus slowing or virtually stopping hydration.

A second look at the data, based on the percentages of the total potential  $Ca(OH)_2$  that they are, shows that the mixtures yielded the following:

Mixtures	Percent of Total Potential Ca(OH) <sub>2</sub>
Anna kaolinite	56-72 on first leach
Fithian illite	31-55 on first leach
Fithian illite	60-90 through third leach
Aberdeen montmorillonite	8-16 on first leach
Aberdeen montmorillonite	20-34 through third leach

Significant quantities of calcium could not be tied up in slightly soluble compounds and still have the percentages of total potential  $Ca(OH)_2$  leached as were leached from the kaolinite and illite mixtures. The data and the preceding discussion and hypothesis indicate that hydration was very rapid in the kaolinite mixtures; slower in the illite mixtures, possibly because of envelopment of the cement grains; and slowest in the montmorillonite mixtures. The additional CaO yielded through the third leaches is probably due to hydration of cement during the leaching process. The amount of this additional CaO might be interpreted as an indication of the tightness of the relatively impermeable envelope enclosing the grains of unhydrated cement.

Oxides	Aberdeen Mont,	Attapulgite	Anna Kaolinite	Dry Branch Kaolinite	Fithian Illite	Grundite	Wyoming Mont,	White Springs Mont.
SIO	56,66	55,80	57.60	43, 32	56.85	57,60	57.15	55.05
CaO	2.66	1.42	0.24	0.00	0.30	0.30	0.50	0.90
MgO	4.44	12.21	1,26	1,05	3.30	2.58	4,45	3.75
Al <sub>2</sub> O <sub>3</sub>	14.00	10.75	22.50	37.75	9.75	17.00	16.50	14.00
Fe <sub>2</sub> O <sub>3</sub>	4.70	4.25	0.98	0.00	6.00	5.75	4.10	9,50
TIO	0.57	0.48	1,43	1.30	2.22	0.76	0.11	0.63
SO <sub>3</sub>	0.12	0.21	0.46	0.36	0.89	0.31	0.48	0.28
K O	0.77	1.25	0.25	0,05	4.90	0.45	4.58	1.35
Na <sub>2</sub> O	0.85	1.10	0.58	0.78	3.55	3.43	4.28	0.80
Loss on								
ignition	11.51	13.11	9,81	14.42	11.08	8.02	7,02	10.08
Total	96.28	100.58	95.11	99.03	98.84	96.20	99.17	96.34

 $Al_2O_3$  – Previously, the theoretical maximum quantity of  $Al_2O_3$  capable of being leached from the mixtures with HCl was established as being no more than the sum of the quantity of  $Al_2O_3$  in the cement plus the quantity of  $Al_2O_3$  leached with HCl from the untreated clay. Inspection of Tables A-4 to A-6 in the Appendix shows that the total  $Al_2O_3$  leached from the Aberdeen montmorillonite mixtures for all the curing periods exceeded the theoretical maximum leachable  $Al_2O_3$  by 100 to 195 ppm. The total  $Al_2O_3$ leached from the Anna kaolinite mixtures exceeded the theoretical maximum for all the curing periods except the 112-day period. The Fithian illite mixtures did not yield  $Al_2O_3$  in excess of the theoretical maximum, except for the 56-day curing period, for which the data are questionable.

The only possible source of the excess  $Al_2O_3$  was the clays. The only plausible reason for the clays to yield more  $Al_2O_3$  in the presence of hydrating cement is that the alkaline environment established by the hydrating cement causes some degree of deterioration of the clay structure, thus making the  $Al_2O_3$  more susceptible to leaching. The quantities of excess  $Al_2O_3$  leached seem to indicate that the clays suffered deterioration, from greatest to least, as follows: Aberdeen montmorillonite, Anna kaolinite, and Fithian illite.

 $SiO_2$ -Inasmuch as the leachate was acid, it is unlikely that much  $SiO_2$  was removed from the clay structures. The data for CaO show that more than 93 percent of the potential CaO was removed from the mixtures. Therefore, the cement constituents must have been thoroughly broken down. It is possible that the  $SiO_2$  data reflect the extent to which the  $SiO_2$  that was dissolved from the cement compounds had polymerized and been removed from solution between the time of dissolution and analysis for  $SiO_2$ .

Total Chemical Analysis—The results of the total chemical analysis of the clays are presented in Table 4.

#### STRENGTH STUDIES WITH TYPE II PORTLAND CEMENT

Harvard-size specimens were molded and processed in much the same method as they were for the first phase of the study, with the exception that only 10 and 20 percent Type II portland cement by weight of the dry clay was added to the mixtures.

Figure 11 shows that there is not much difference between the strength development in cement-clay mixtures with a Type I and a Type II portland cement. The strength curves for the Type II cement-stabilized mixtures approximately parallel the curves for the Type I cement-stabilized mixtures, and have from 10 to 180 psi lower strengths.

The strengths reported are for 7 days of curing. After 7 days of curing at 21 C, it is expected that the dicalcium silicate and the tricalcium silicate will be approximately 36 and 72 percent hydrated, respectively (Copeland, et al, 2). A comparison of the compound compositions of the Type I and Type II cements (see Table 1) shows that the Type II cement contained only slightly more dicalcium silicate and tricalcium silicate, approximately the same quantity of CaSO<sub>4</sub>, but only 3.12 percent of tricalcium aluminate, whereas the Type I cement contained 11.68 percent tricalcium aluminate. It is unlikely that the difference in strength development between the two cements is due to their content of dicalcium silicate and tricalcium silicate, because they are similar in their

51

TABLE 4



1300

Figure 11. Strength development of clay-cement mixtures.

content of these two compounds. However, inasmuch as tricalcium aluminate is the principal compound contributing to the early strength of cement, the larger quantity of tricalcium aluminate and the higher ratio of tricalcium aluminate to  $CaSO_4$  in the Type I cement could account for the higher strength development.

#### CONCLUSIONS

1. Based on the amount of  $Ca(OH)_2$  produced, the cement in the cement-Anna kaolinite mixtures experienced the most rapid hydration. The hydration of the cement in the cement-Fithian illite mixtures started at a rate of approximately one-half and increased after 3 days to a rate of three-fourths that of the cement in the cement-kaolinite mixtures. The cement in the cement-Aberdeen montmorillonite mixtures was by far the slowest to hydrate. Its rate of hydration started at approximately one-tenth and increased to one-fifth the rate for the cement in the cement-kaolinite mixtures.

2. The strength gain noted in Harvard mold specimens cured for 7 days appears to result from the following conditions:

- a. Early flocculation of the clay particles, which brings them into a state of proximity to each other and thus makes their joint cementation easier.
- b. Hydration of the cement, which creates many minute cementitious particles with a very large surface area, as well as chemical combination of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Ca(OH)<sub>2</sub> to create additional cementitious particles.
- c. Crystallization of Ca(OH)<sub>2</sub>, which cements discrete particles as well as floccules to each other. The microscope study showed that the cement-Anna kaolinite mixtures had the most extensive development of well-crystallized Ca(OH)<sub>2</sub>; the cement-Fithian illite mixtures had moderately well developed but irregular patches of Ca(OH)<sub>2</sub>; and that the cement-Aberdeen montmorillonite mixture, which developed the least strength, had no visible Ca(OH)<sub>2</sub>.

3. The alkaline environment of the hydrating cement caused deterioration of the clays. The clays suffered deterioration, from greatest to least, as follows: Aberdeen montmorillonite, Anna kaolinite and Fithian illite.

4. A mechanism for restricting the hydration of cement grains is encapsulation of the cement grains by the very fine-grained clay and recently crystallized gels of calcium silicate hydrate and, perhaps, calcium aluminate hydrate. This concept of encapsulation is a modification of the concept for encapsulation of  $Ca(OH)_2$  crystals by an amorphous calcium silicate hydrate gel presented by Diamond (5).

5. Up to a curing period of 7 days, the Type I portland cement-clay mixtures developed somewhat greater strengths than the Type II portland cement-clay mixtures, probably because of the larger content of tricalcium aluminate and similar content of CaSO<sub>4</sub> in the Type I portland cement.

6. From the observations presented in this report, it appears that very early in the life of cement-Aberdeen montmorillonite mixtures the cement grains become encapsuled and hydration is slowed to a negligible rate. Diamond (5) has demonstrated that if a large surface area silicate is present in a dilute suspension of  $Ca(OH)_2$ , at a ratio of say 5/1 silicate to  $Ca(OH)_2$ , the adsorption of  $Ca(OH)_2$  onto the silicate surface is so fast that there is no opportunity for encapsulation of the crystalline lime and it is soon depleted. Of course, the preceding reaction might not occur if the silicate and  $Ca(OH)_2$ , in the same proportions, were in paste form. The preceding information suggests that a comparison of a cement-montmorillonite system with a lime-montmorillonite system would be an area for fruitful investigation.

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# Appendix

TABLE A-1 LEACHINGS OF CRO AND pH OF LEACHATE Cement-Anna Kaolinite Mixtures

 	 		_	-
 _	 _	-		-

				Acrid	Total					
Curing Period, Days	1 st		2	nd	3 :	rd	4 th		Leach,	Leached,
	pH	ppm	pH	ppm	pH	ppm	pH	ppm	PP	Ppm
Untreated clay	6.72	0.0	6,62	2.2	6.70	3,4			9.0	14.6
1	12.00	480.5	11.30	122.1	11.27	87.4			1601.6	2291.6
3	12.15	545.4	11.59	149.0	11.35	90.7			1550.1	2335.2
7	12.18	595.8	11.56	153.4	11.31	96.3			1357.4	2202.9
14	12.20	593.6	11.60	150.1	11.32	93.0			1458.2	2294.9
56	12.17	621.6	11.62	190.4	11.38	109.8	11.24	87.4	1344.0	2353.2
112	12.20	567.0	11.70	194.9	11.42	108.6			1395.5	2266.0

TABLE A-2 LEACHINGS OF CaO AND pH OF LEACHATE Cement-Fithian filite Mixtures

		Leach												Acid	Total			
Curing Period, Days	1 /	st	2 1	ıd	3 1	d	4 t	h	5 t	h	6 t	h	7 t	h	8	th	Leach,	Leached,
	pH	ppm	pН	ppm	pH	ppm	pH	ppm	pH	ppm	pH	ppm	pH	ppm	pH	ppm	ppm	ppm
Untreated clay	4.76	19.0	5.23	1.7	5.60	0.0											11.2	31.9
1	11.80	265.4	11.46	132.2	11.30	98.6											1895.0	2391.2
3	12.00	405.6	11.50	152.3	11.36	109.8											1648.6	2317.3
7	12.06	457.0	11,48	155.7	11,25	101.9											1673.2	2387.8
14	12.00	460.3	11.50	170.2	11.30	117.6											1561.3	2309.4
56	12.12	474.9	11.61	175.8	11.44	121.0	11.30	88.4	11,20	67.2	11,00	50.4	10,90	41.4	10.71	35.8	1209.6	2264.5
112	12.03	432.3	11.65	178.1	11.51	137.8	11.39	94.1	11,18	69.4	11.01	50.4	10.88	43.7	10.78	39.2	1377.6	2422.6

Curing Period, Days	1 st			2 nd		3 rd		4 th		5th	Acid Leach,	Total Leached,
	pH	ppm	pH	ppm	pH	ppm	pH	ppm	pH	ppm	ppin	ppin
Untreated clay	8,65	15.7	8,61	12.9	8.35	10,1					147.8	186.5
1	11,13	63.8	11.00	50.4	10,90	53.8					2380,0	2548.0
3	11,50	125.4	11,30	95.2	11,17	71.7					2334.1	2626.4
7	11.46	115,4	11,21	81.8	11.06	63.8					2275.8	2536.8
14	11.58	137.8	11,30	86.2	11.05	66, 1					2266,8	2556,9
56	11.43	101.9	11,24	71.7	11.11	59.4	10.96	48.2	10,82	40,3	2139, 2	2460.7
112	11.40	89,6	11.22	67.2	11.06	53.5	11.04	53, 2	10.92	44.0	2268.5	2576.0

TABLE A-4 LEACHINGS OF Al<sub>2</sub>O<sub>3</sub> Cement-Anna Kaolinite Mixtures

Curing Period,		Leach	, ppm		Acid Leach, ppm	Total Leached,	*0	Total in Mixture,
Daye	1 st	2 nd	3rd	4th		ppm	a	ppm
Untreated clay	0,0	0.0	0.0		134.0	134,0		1251.0
1	13.9	8.0	8.0		496.8	526.7	352,3	1469.0
3	2.4	8.8	9.0		360.0	380.2	352.3	1469
7	1.6	10.4	10.4		420.0	442.4	352.3	1469
14	0.0	2.4	10.4		432.0	444.8	352,3	1469
56	0.0	0.0	0.0	0.0	356.0	356.0	352.3	1469
112	0,0	0,0	0.0		336.0	336,0	352,3	1469

\*a ≈ ppm leached from pure clay plus ppm possible from fully dissolved portland cement.

TABLE A-5 LEACHINGS OF Al<sub>2</sub>O<sub>3</sub> Cement-Fithian Illite Mixtures

Curing Period,				Leach,	ppm				Acid	Total Leached,	*a	Total in Mixture, ppm
Days	1 st	2 nd	3 rd	4 th	5 th	6 th	7 th	8 th	ppm	ppm	a	
Untreated clay	0.0	0.0	0.0						166.4	166.4		
1	5.0	4.8	3.2						348	361	384	760
3	3.2	5.6	1.6						316	326,4	384	760
7	0.0	6.4	4.8						340.4	351.6	384	760
14	2.4	40.0	5.6						320	368	364	760
56	9.5	7.5	10.2	11.2	9.3	5.6	4.8	2.4	473.6	534.1	384	760
112	0,0	3.2	0.0	0,0	0,0	0,0	0.0	0,0	376.0	379.2	364	760

•a = ppm leached from pure clay plus ppm possible from fully dissolved portland cement.

	Cement-Aberdeen Montmorillonite Mixtures										
Curing Period,		Le	each, ppm	L		Acid Leach,	Total Leached.	*a	Total in Mixture,		
Days	1 st	2 nd	3 rd	4 th	5 th	ppm	ppm		ppm		
Untreated clay	0.0	0.0	0,0			172.0	172,0		778,4		
1	0.0	0.0	0.0			552.0	552,0	390.3	997.0		
3	3.2	3.2	1.6			484.0	492.0	390,3	997.0		
7	12.0	6.4	9.6			496.0	524.0	390,3	997.0		
14	15.2	16.8	13.1			524.8	569.9	390,3	997.0		
56	0.0	3.2	0, 0	0.0	0.0	528.0	531,2	390,3	997.0		
112	0.0	0,0	0, 0	0,0	0,0	585,6	585,6	390,3	997.0		

TABLE A-6 LEACHINGS OF Al<sub>2</sub>O<sub>3</sub>

\*a = ppm leached from pure clay plus ppm possible from fully dissolved portland cement.

#### TABLE A-7 LEACHINGS OF SiO<sub>2</sub> Cement-Anna Kaolinite Mixtures

Curing Period,	1	Leach, pp	m	Acid Leach,	Total Leached,	Total in Mixture	
Days	1 st	2 nd	3 rd	ppm	ppm	ppm	
Untreated clay	0.0	0.0	0.0	0.0	0.0	3202.0	
1	0.0	4.8	3.2	188.3	196.3	3986.0	
3	0.0	3,2	2.4	202.0	207.6	3986.0	
7	0.0	3.2	0.0	169.6	172.8	3986.0	
14	0.0	3.2	0.0	128.0	131.2	3986.0	
56	0.0	4.8	20.0	103.2	128.0	3986.0	
112	0.0	2.4	3.2	54.4	60,0	3966.0	

TABLE A-8 LEACHINGS OF SiO<sub>2</sub> Cement-Fithian Illite Mixtures

Curing Period,		4	_	Leac	h, ppm				Acid	Total	Total in Mixture,
Days	1 st	2 nd	3 rd	4 th	5th	6 th	7 th	8 th	ppm	ppm	ppm
Untreated clay	0.0	0.0	0.0						12.8	12.8	3160.8
1	0.0	9.6	8.0						152.0	169.6	3945.0
3	0.0	3.2	6.4						145.6	155.2	3945,0
7	0.0	0.0	0.0						153.6	153.6	3945.0
14	0.0	0.0	0.0						160.0	160.0	3945.0
56	0.0	0.0	6.4	4.8	12.8	8.0	11.2	12.8	*N.A.		3945.0
112	3.2	8,0	14.4	8.0	11,2	14.4	20.0	18.4	78.4	176,0	3945.0

\*Note: No analysis.

LEACHINGS OF SiO2	C	ement-	Aberdee	en Mon	tmori	llonite	Mixtures	
			LEA	CHING	S OF	SiO <sub>2</sub>		

Curing Period,		L	each, ppr	n		Acid	Total	Total in Mixture ppm	
Days	1 st	2 nd	3 rd	4 th	5 th	ppm	ppm		
Untreated clay	0,0	0,0	0.0			12.8	12.8	3150.0	
1	11.2	12.8	9,6			201,6	235.2	3935.0	
3	0.0	6.4	11.2			195,2	212.8	3935.0	
7	6.4	10.4	11.2			200.0	228.0	3935.0	
14	0.0	0.0	4.8			196.8	201.6	3935,0	
56	0.0	4.8	4.8	1,6	4.8	*N.A.		3935.0	
112	6.4	4.8	3.2	0.0	0.0	153.6	168.0	3935.0	

\*Note: No analysis.

# **Soil-Bituminous Stabilization**

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The effect of certain cationic surface active agents, commercially sold as antistripping agents, on the waterproofing properties of bitumen cutback used in soil stabilization was studied. For the sake of comparison paraffin wax and rubber were also included in the study. All these chemicals have been found to have a marked effect in enhancing the waterproofing property of bitumen, consequently effecting saving in consumption of the binder for obtaining the desired results. Paraffin wax was found to be the most effective and economical additive for this purpose.

•SOIL stabilization by incorporation of an admixture such as bitumen is becoming the subject of many research projects, as this type of stabilization has important applications in the construction of both pavements and buildings. Investigations were undertaken to determine whether or not the proportions of bitumen could be reduced and the stabilization process by this method made more economical. Some preliminary investigations on the effect of certain cationic chemicals and different methods of mixing have been carried out. The study was divided into three parts:

1. Influence of cationic additives on the waterproofing quality of bitumen cutback used for soil stabilization;

2. Effect of pretreatment of soil with cationic surface active agents on its strength and stability; and

3. Effect of two different methods of mixing bitumen in soil on the properties of the resultant mixture.

As can be seen from these reports, it is possible to economize considerably on the cost of soil stabilization with bitumen by making use of some chemicals or by adopting the proper method of mixing bitumen in soil.

Antistripping agents, generally cationic chemicals, are now widely used as additives in bitumen for improving its bond with road aggregates during surface dressing operations, and also for bitumen coating of wet stones for road construction during the rainy season (3). These chemicals possess the property of displacing water from the stone surface and insuring the coating of the solid with an uninterrupted uniform layer of binder. They can be used as admixtures in the binder as well as for pretreatment of the stone aggregate to be coated. In both cases results are satisfactory, but their incorporation in the binder is more economical and convenient.

Since bitumen is also used for the stabilization of soil for road construction (1), particularly in water logged (2) or sandy areas, and also for the preparation of improved mud mortar (5) and sundried bricks, laboratory experiments were conducted to study the effect of the addition of such chemicals on the waterproofing property of bitumen when used for soil stabilization.

#### Preparation of Bitumen Cutback

In this study, 80-100 penetration bitumen (Mexphalt) was used. Because thinned bitumen is easier to mix with soil and because antistripping agents are more effective

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Figure 1. Specimen blocks in wetting tray.

in a less viscous binder, the bitumen was thinned with 20 percent of its weight of kerosine by heating and thorough stirring. In a separate study kerosine was found to be a better and more economical solvent for making such cutbacks than the other common solvents such as diesel oil, petrol and wood preservative oil. Kerosine oil was used as the cutback in all experiments conducted in connection with these investigations.

#### Soil

The soil used throughout the study was of a graded nature having a plasticity index of about 10 and sand content (fraction passing 40 mesh and retained over 200 mesh) of about 40 percent.

#### Chemicals

Three proprietary antistripping agents (cationic chemicals), designated as Product A, Product B, and Product C, were studied.

Product A was a long chain diamine, smelling faintly of ammonia and having a chemical formula  $C_{18}H35$  NH-(CH<sub>2</sub>)3-NH<sub>2</sub>. It is marketed under the trade name of Duomeen-T in the form of a pale paste which melts to a liquid at 48 C.

Product B was a plyamine derivative, smelling faintly of ammonia. It is marketed in the form of a dark colored liquid of low viscosity under the trade name of Udol.

Product C was a pale yellow flaky opaque (powder) cationic surface-active agent manufactured in India and marketed under the trade name Hiquat T.



Figure 2. Results of blocks made at workable moisture.

In addition to these three antistripping agents, commercial paraffin wax and a rubber solution were also included in the study as these are supposed to improve appreciably the waterproofing quality of asphalts. The rubber solution containing approximately 20 percent rubber was obtained from M/S Rajsathan Electro-Conductor Manufacturing Corporation Jodhpur and was mixed with hot bitumen in proportions to give the required percentage of rubber; viz., 150 gm rubber solution mixed with 850 gm bitumen to give a mixture containing 3 percent rubber; 100 gm mixed with 900 gm to give a mixture containing 1 percent rubber and 25 gm mixed with 975 gm to give a mixture with 0.5 percent rubber.

To study the effect of varying proportions of Products A, B, and C on the properties of the bitumen, 0.5, 1, 2, and 3 percent of each chemical (by weight of the cutback) was mixed in the requisite quantity of the cutback separately by heating to about 150 C. Twenty cutback samples containing different chemicals in varying proportions were obtained besides the original sample containing no chemical.

# Preparation of Specimen Blocks

Two sets of specimen blocks, one molded at workable moisture and one compacted at optimum moisture, were prepared for testing the effectiveness of the chemicals. The quantity of cutback used varied from 2 to 5 percent by weight of dry soil. For the molded specimens, the requisite quantity of cutback was mixed in the soil after the latter had been well mixed with a sufficient amount of water (about 30 percent by weight of soil) to bring it to molding consistency. The cutback was mixed with a spatula until it was completely dispersed into the mud giving a uniform color and texture. This bitumenized mud was poured into hollow cylindrical split molds (2.5-in. diameter by 2.5-in. high) which were tapped on the sides to remove air pockets and then allowed to set overnight. On the next day the hardened specimen blocks were removed by opening the split molds. The specimens were then allowed to dry.

The compacted specimen blocks were prepared by compaction at optimum moisture (12 percent). For each specimen, 200 gm of dry soil was taken and after mixing with the requisite quantity of water, the designated quantity of cutback was spread over the soil and mixed thoroughly first with a spatula and then by rubbing the mixture between the palms of the hands until a homogeneous mixture was obtained. This mixture was then placed in the modified Abbot compaction apparatus and compacted by 40 blows of the 5-lb hammer dropping from a 12-in. height. The specimen blocks were 2.5 in. in diameter and about 1.25 in. high.



Figure 3. Results of blocks made at optimum moisture.



Figure 4. Results of blocks made at workable moisture.

# **Tests Performed**

The specimen blocks prepared by the two methods were first allowed to dry in the sun and then brought to constant weight in a hot oven maintained at 60 to 70 C. They were then tested for the following properties: (a) moisture absorption by capillary action, (b) resistance to softening effect of water upon complete immersion, and (c) dry and wet compressive strength.

#### Testing Procedure and Discussion of Results

Moisture Absorption by Capillary Action. The dry specimen blocks were weighed and placed over sand constantly kept wet in a tray (Fig. 1). Moisture absorbed by each specimen block was determined at 24-hr intervals for as long as 240 hours. Results of specimens at 144 hours containing 3, 4, and 5 percent cutback are shown in Figure 2 for blocks made at workable moisture and Figure 3 for blocks made at optimum moisture. Specimen blocks made without the addition of bitumen cutback became completely wet and soft within 24 hours and could not be handled for further testing. The addition of only 0.5 percent of all the cationic chemicals considerably reduced the moisture absorption capacity of the cutback and this effect was more marked when 3 percent cutback by weight of soil was used. With the larger quantities of cutback used for soil stabilization the effect of these chemicals was not significant although some improvement was observed with the incorporation of 0.5 percent chemical. With increasing percentages of these chemicals in the cutback, greater water absorption was noticed particularly in the mixtures containing more than 3 percent cutback. Of the three cationic chemicals, Product C gave best results when 0.5 percent was used and the percentage of the cutback in the soil was 4 and 5. Other relationships are shown in Figures 2 and 3.

The rubber solution used as an admixture gave results almost identical to those of Product A. In the specimens containing 3 percent cutback, the water absorption steadily decreased with the addition of rubber solution up to 2 percent. In the case of specimens containing a larger quantity of cutback, 0. 5 percent rubber gave best results and further increase tended to have an adverse effect.

The addition of paraffin wax had a marked effect in enhancing the waterproofing property of the bitumen and confirmed the results of previous investigations (7). In the case of specimens containing 3 percent cutback, water absorption appreciably and steadily decreased with an increasing quantity of paraffin wax up to 3 percent, while in the specimens containing 4 and 5 percent cutback the addition of paraffin wax up to 2 percent gave very satisfactory results. Even with the use of 2 percent cutback, which contained 3 percent paraffin wax, the water absorption was fairly low.

In comparing the results of the two sets of experiments, i.e., the specimens prepared at workable and optimum moisture, the results from the blocks made at workable moisture were more consistent.

Generally, the use of paraffin wax gave much better results than other chemicals insofar as the water absorption by capillary action is concerned. It was also observed that there is an optimum quantity of such additives at which they are most effective.

Resistance to Softening Effect of Water by Actual Immersion of Specimen Blocks. In this test, the blocks were immersed in water contained in a tray and their condition and moisture absorption was observed at 24-hr intervals for as long as 120 hours. Results are shown in Figures 4 and 5. Compressive strength of the wet blocks which remained intact after this test was also determined. The compressive strength of the wet specimens generally increased with the increasing quantity of the cationic chemicals up to 2 percent in the case of Product A, up to 1 percent in the case of Product B, and up to 0. 5 percent in the case of Product C. Further increase in the percentage of the chemicals tended to reduce the wet compressive strength. At their optimum percentages, Product C gave better results than Products B and A. In all cases, the beneficial effect of the chemicals was more pronounced when 3 percent cutback by weight of soil was used, as the wet compressive strength increased from about 15 psi in blocks made with cutback containing no additive to over 100 psi in blocks made from cutback containing



Figure 5. Results of blocks made at optimum moisture.



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Figure 6. Laboratory Carver press.

optimum quantities of the additives studied Specimens of soil prepared with cutback containing 1 percent paraffin wax and 0.5 percent rubber compared favorably with those made from cutback containing 0.5 percent Product C, which was the best fo 3 percent admixture of cutback. When, however, higher percentages of cutback containing these optimum quantities of wa and rubber were used, the results were still better. The optimum quantities of the chemicals which showed highest wet compressive strength showed the minimum water absorption and this effect was again more pronounced in the case of block containing 3 percent cutback.

Dry Compressive Strength. This was determined by the laboratory Carver pre-(Fig. 6). Results are shown in Figures and 5. Dry compressive strength of the specimen blocks made at a higher moistur content generally decreased with increasin quantities of all five additives. However, when these admixtures were used for bituminous stabilization of soil by compaction at optimum moisture, they did not show any marked effect on the compressiv strength with increasing quantities. Speci mens containing higher proportions of al cutback continued to show less strength than those containing lower (3 percent) proportions.

S. No.	Duomeen-T	Cutback	Dry Comp. Strength	Moistu: Immera	re (\$) Abso sion After T	orbed by 'ime (hr)	Wet Comp. Strength After 72-Hr
	(% by wt. of soil)	(% by wt. of dry soil)	(рві)	24	48	72	Soaking (psi)
1.	Nil	Nil	281. 7	Bloc	k disintegi er 15 minu	rated ites	-
2. 2	Nil	1	319.2	14.3	15.4	15.9	14.4
3. ª	Nil	2	398. 5	11.6	14.0	15. 3	24.0
4.	Nil	3	300. 9	6.5	9.2	11.0	49.7
5.	0.05	NII	267.8	Bloc	k disinteg: er 8 davs	rated	-
6.	0,05	1	372.6	10.6	13.9	14.5	28, 8
7.	0,05	2	403. 2	1.7	3.2	4.03	165.0
θ.	0. 05	3	298. 5	0.7	1.8	1.8	237. 2
9.	0.1	NII	244, 5	Partia	lly disinte	grated	Block soft
10.	0.1	1	260. 5	9.4	11.7	12, 4	40.0
11.	0. 1	2	296.6	4. 5	8. 2	10.0	107.4
12.	0. 1	3	332. 7	1.5	3. 5	4.1	176. 4
13. <sup>b</sup>	0. 2	NIL	145. 1	8.08	10.6	10.8	25. 6
14.	0.2	1	154.7	9.5	12.8	13.09	41.7
15.	0. 2	2	127.4	8.6	11, 5	12.5	49.7
16.	0. 2	3	169.9	4.4	6. 2	7.8	72. 1
17. b	0.5	Nil	76.1	10.3	12.9	12.9	19. 2
18.	0, 5	1	119.4	8, 61	12.2	12.7	43. 3
19.	0.5	2	109.7	2.3	4. L	5. 4	67. 3
20.	0. 5	3	114.6	2.0	3.06	4. 3	68. 9

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Bottom somewhat soft.

Bottom slightly soft.

#### Conclusions and Economics of the Additives

The following conclusions can be drawn from the results previously discussed.

1. The cationic surface active agents, commercially sold as antistripping agents, have a marked effect on the waterproofing property of the bitumen-soil mixture.

2. There is an optimum quantity of each chemical (by weight of bitumen) at which it gives best results.

3. There is an optimum ratio of cutback to soil at which such chemicals are most effective.

4. Soil blocks prepared with 3 percent cutback (by weight of soil) containing 0.5 percent to 1 percent of the chemicals under study compared very favorably in their resistance to water absorption and in their wet compressive strength to those having 4 to 5 percent cutback, containing no chemical. In this way, 1 to 2 percent cutback could be saved by using 0.015 to 0.03 percent chemical by weight of soil (0.5 to 1 percent by weight of cutback) costing less than 40 percent the price of bitumen cutback.

5. Use of paraffin wax is more effective and the most economical of all the additives tried.

# EFFECT OF PRETREATMENT OF SOIL WITH CATIONIC SURFACE ACTIVE AGENTS ON STRENGTH AND STABILITY

It was found in the previous investigation that the incorporation of certain cationic additives, in certain proportions improves considerably the resistance to softening effect and water absorption of the resultant soil mass and helps in preserving its compressive strength in the wet state. An attempt was then made to study the effect of pretreatment of a soil with certain chemicals on the soil's properties when stabilized with bitumen cutback containing no additive. Accordingly, a graded type of soil having a plasticity index of about 10 and a sand content of about 40 percent, was selected and treated with 0.05, 0.1, 0.2 and 0.5 percent stearyl diamine. The required quantity of the chemical was first dissolved in an amount of water just sufficient to wet the soil when thoroughly mixed. The soil was then dried for use in these investigations.

The specimen blocks were prepared by molding these treated soils in suitable cylindrical molds after mixing with a sufficient amount of water (about 30 percent by

TABLE	2
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» by wt. of ary soll/	2			Percent Moisture Absorbed by Capillary Rise in Hours							
		24	48	72	120	144	168	240	(psi)		
Nil	Block became soft after 20 min										
1	2.3	9.5	13. 12	14.1	14.9	-	15.6	15.6	20. 8		
2	1.02	6. 4	10.7	13. 3ª	15.1	-	16.1	16.1	24.0		
3	0.25	3.1	5.6	7.4	10.82		13.1	13.1	35. 2		
NI	Absorption could not be noticed							_b	13.6		
1	1 2	3 7	6 0	7.0	9.0	10.02	10.5	10.7	40.0		
2	0 2	1.0	2.5	2.7	4.3	5.6	6.6	8.4	72.1		
3	0.3	0.7	1.5	1.5	2.8	2.8	3.0	3.0	190. 8		
Nil	Absorption could not be noticed _t								20, 0		
1	1 2	4.0	6.4	7.3	9.1	9.2	9.4	9.4	39.2		
2	0.2	1.5	3. 5	4.0	6.1	7.4	7.6	8.4	71. 3		
3	0.0	0. 2	1.2	1. 2	2, 5	2. 5	3, 0	3. 0	160. 3		
NIL	1.6	4.1	4.9	5. 4	6.0	-	6. 4	6.4	36.1		
1	1.36	8.9	11. 5a	12.0	12.7	-	13.0	13.0	36.8		
2	0.45	4.0	7.9	10. 32	13.0	_	13.3	13.3	40.1		
3	0. 25	1. 29	2.6	4. 2	6. 5	-	9.4	11.8	60. 1		
NIL	3.1	6, 7	7.9	8.8	9.2	_	9.9	9.9	16.8		
1	0.8	8.6	12. 42	12.8	13.5	-	13.9	14.1	40.0		
2	0.2	1.0	1.4	2. 2	3.9	-	6.9	10. 12	45.6		
3	0, 1	0.7	0.7	0.9	1. 3	_	1.7	1.7	75.3		
	3 Nil 1 2 3 Nil 1 2 3 Nil 1 2 3 Nil 1 2 3 Nil 1 2 3 3 Nil	3 0.25   NII Absor   1 1.2   2 0.3   Nil Absor   1 1.2   3 0.3   Nil Absor   1 1.2   2 0.2   3 0.0   Nil 1.6   1 1.36   2 0.45   3 0.25   Nil 3.1   1 0.8   2 0.2   3 0.1	3 0.25 3.1   Nil Absorption co.   1 1.2 3.7   2 0.2 1.0   3 0.3 0.7   Nil Absorption co. 1   1 1.2 4.0   2 0.2 1.5   3 0.0 0.2   Nil 1.6 8.9   2 0.45 4.0   3 0.25 1.29   Nil 3.1 6.7   1 0.8 8.6   2 0.2 1.0   3 0.1 0.7	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$		

weight of soil) and 1, 2 and 3 percent bitumen cutback consisting of 80 parts of 80 to 100 penetration bitumen and 20 parts kerosine. The blocks were then allowed to set and dry. The compressive strength of the blocks in both the dry and wet condition, the rate of water absorption by capillarity, and the amount of water absorbed by the blocks on actual immersion in water were determined. The method used was the same as previously described. The results are given in Tables 1 and 2, and shown in Figures 7 and 8.

#### **Discussion of Results**

From the results given in Table 1 and plotted in Figure 7, the dry compressive strength of the specimen blocks increased slightly when the soil was treated with 0.05 percent of the cationic chemical but decreased with increasing percentages. On the whole, the treatment of soil with the chemical tended to decrease the dry compressive strength of the blocks. On the other hand, the wet compressive strength generally improved when the soil was treated with the chemical, and it was maximum in the case of soil treated with 0.05 percent. Similarly, moisture absorbed by the specimen blocks on actual immersion in water was much lower in the case of treated soil and was the lowest in the case of specimen blocks of soil treated with 0.05 percent of the chemical and stabilized with 2 to 3 percent cutback.

Examination of results given in Table 2 and shown in Figure 8 will also show that the rate of moisture absorption by capillary action in the case of soil treated with 0.05 percent chemical was almost the lowest. Also the wet compressive strength of blocks tested after 240 hours was highest in the case of soil treated with 0.05 percent chemical. Treated soil stabilized with only 1 percent bitumen cutback in every case showed even a better wet compressive strength than that which was obtained from the untreated soil stabilized with 3 percent bitumen cutback.

#### Conclusions

From the preliminary results of this study, it is obvious that some engineering properties of soil stabilized with bitumen can be considerably improved by pretreating the soil with cationic surface-active chemicals, and also considerable savings can be effected in the quantity of bitumen. By using 0.05 percent chemical by weight of soil,


Figure 8. Moisture absorbed when soil was treated with cationic chemical.

S. No.	Nature of Soil	Method of Mixing Cutback	Cutback Added (\$)	(	Moisture After 72-Hr	
				Dry	Wet (after 72-hr soaking)	Soaking (⊀)
I.	Sandy	1	0	911, 4	-	-
	PI = 4, 64		3	980.7	72.03	7.0
	SC = 61.9		4	725.0	88. 04	10.78
	SO4 = 0.0		5	718.6	124.8	6.33
	OM = 10%	п	3	592.9	32.01	14.85
			4	420.1	92.8	8, 29
			5	438. 5	201.6	5. 24
п.	Clayey	I	0	1201.4	-	÷- 1
	PI = 18.10		3	1015.0	Block disintegrated :	after 30 min
	SC = 9.2		4	1180.7	Block disintegrated :	after 1 hr
	SO4 = 0.0		5	890.0	Block disintegrated a	after 41/2 hr
	OM = 15≴	п	3	1015.0	Block disintegrated a	after 6 hr
			4	1118.6	Block disintegrated :	after 8½ hr
			5	870.0	Block disintegrated :	after 24 hr
ш.	Silty	1	0	141.6	-	-
	PI = 9.43		3	144.8	Block disintegrated a	after 4 hr
	SC = 10.5		4	189.6	32.01	22.06
	SOY = 0.0		5	224.9	49.9	18.62
	OM = 10%	п	3	194.4	56. 0	23. 76
			4	156.8	57.6	12.56
			5	142.4	76. 8	8.17

S. No.	Nature of Soil	Method of Mixing Cutback	Cutback Added (\$)	Moisture Absorption (\$) by Capillary Rise After Time (hr)					
				2	24	48	72	120	168
I.	Sandy	I	3	0.02	6.02	11. 5ª	12.0 <sup>a</sup>	12.0a	12.03
			4	0, 49	0.99	1.48	1.48	1. 48	1. 48
			5	0.5	0, 58	0.97	0.97	0.97	0.97
		п	3	0.98	1.95	12.25	13.72	14.7	14.7
			4	0.5	1.45	2.91	2.91	3.8	3.8
			5	0.0	1.41	2.83	2.83	3.77	3, 77
п.	Clavey	I	3b	19. 34 <sup>C</sup>	27.3	28, 77	29.24	29.27	29, 27
			4b	6.8	26, 21	27.66	29.12	29,12	29.12
			5b	23. 0C	35.0	36. 5	36. 5	36.5	36. 5
		п	3b	5.31	31.40	31.40	31.40	_e	_e
			4d	5.94	16.33	22. 77	23.76	23, 76	23. 76
			5 <sup>d</sup>	0, 0	17.00	22. 5	22. 5	22. 5	22. 5
ш.	Silty	I	3 <sup>b</sup>	22. 5 <sup>c</sup>	_e	_e	_e	_e	_e
		- T	4b	17.07C	20.97	21.94	21.94	21.94	21.94
			5	2.44	12.19	13, 17	13.65	13.65	13. 65
		п	30	0.48	10. 59	20, 19	22.11	25.0	25. 0
			4	0.0	0.0	0.97	0.97	0.97	0.97
			5	0.0	0.0	0.94	0.94	0.94	0.94

m + m + m +

Wet but hard.

Block soft.

<sup>C</sup>Top wet. Block somewhat saft.

Loss of soil.

about 2 percent bitumen by weight of soil can be saved without adversely affecting engineering properties of the soil.

In this a similar savings can be made to that previously discussed.

# EFFECT OF TWO METHODS OF MIXING BITUMEN IN SOIL ON THE PROPERTIES OF THE MIXTURE

Stabilization of soil with bitumen for various engineering works is becoming more popular because of its properties of binding and waterproofing. Besides imparting structural stability to soil, the method of soil stabilization has an important application as a capillary moisture cutoff in water-logged areas.

The conventional method of mixing bitumen with soil is to bring the soil to about optimum moisture content and then add the requisite quantity of bitumen (converted to a form of cutback) either by hand or in mechanical mixers. In this way, the soil



Figure 9. Influence of method of mixing on compressive strength.





Figure 10. Influence of method of mixing on moisture absorption.

particles are coated with bitumen, and become water resistant. The mixture is then spread and compacted by means of hammers or suitable rollers to the maximum density.

Another way of mixing bitumen in soil is to add a sufficient amount of water and mix to a mortar consistency, then add the requisite quantity of bitumen cutback and again mix manually or in suitable mechanical mixers such as concrete mixers. This method is actually used for the preparation of waterproof mud mortar for masonry or plaster work where a greater amount of water is required for mixing.

For soil stabilization by compaction, this method is not favored because the mixture has to be again brought back to the optimum moisture by evaporating the excess moisture. Some preliminary trials with this method showed that it gives more homogeneous and water resistant soil-bitumen mixtures and also needs less labor than when bitumen is mixed with the soil at optimum moisture. Therefore, in these investigations a comparative study of the effect of two methods of mixing soil-bitumen on the properties of the resultant mixture was made and the results are given in Tables 3 and 4 and shown in Figures 9 and 10. Method I represents the blocks prepared directly at optimum moisture, whereas in Method II, the initial mixing was done at a higher moisture, the mixture was allowed to dry to near optimum moisture and then compacted. Three soils (sandy, clayey, and silty) were used separately. The bitumen was used in the form of cutback consisting of 80 parts of 80 to 100 penetration bitumen and 20 parts of kerosine, which was mixed hot and then cooled. Three proportions (3, 4 and 5 percent) of the cutback by weight of dry soil were used. Methods adopted for preparation of specimen blocks were the same as described previously.

## Discussion of Results

From Table 3, the dry compressive strength, which generally decreased with an increasing quantity of bitumen in both cases, was appreciably lower in the case of specimens made from the mixture prepared at a higher moisture content and then brought to optimum moisture for compaction. But the results of the wet compressive strength test were just the reverse. The wet compressive strength increased with the increasing quantity of bitumen cutback in both cases and was generally higher in the case of blocks made by Method II. The moisture absorbed by these blocks was also generally less than that absorbed by the corresponding blocks made at optimum moisture. In the case of clayey soil, although all the blocks had disintegrated during immersion in water, the blocks prepared by Method II resisted the disintegrating effect of water much longer than those prepared by Method I.

Similarly from Table 4, except in the case of sandy soil, the rate of moisture absorption and the quantity of water absorbed by the Method II blocks was much lower and less than the corresponding Method I blocks. In the case of sandy soil there was not much difference in the moisture absorbed by capillary rise in the blocks prepared by the two methods.

It can be concluded that mixing the soil with water first at mortar consistency, and then incorporating the required quantity of bitumen and bringing back to optimum moisture (Method II) yields better mixing and uniform coating of the soil particles, thereby increasing resistance to water absorption. As the loss in the soil strength with an increasing quantity of bitumen is generally due to its lubricating effect, the lower dry strength of blocks made by Method II confirms the better mixing of bitumen in soil by this method.

## ACKNOWLEDGMENT

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## Discussion

HANS F. WINTERKORN, Department of Civil and Geological Engineering, Princeton University—The purpose of this discussion is to place the interesting results obtained by Dr. Uppal within the larger content of factual and theoretical knowledge on bituminous soil stabilization developed within the past forty years, and to point out certain facts that are of particular interest.

## The Beneficial Effect of High Water Contents

The better ultimate wet strength and water resistance of soil-bitumen, achieved whe mixing and molding were conducted on the wet rather than the dry side, had been recog nized in the earliest field and laboratory studies and reported in the pertinent literatur (10, 11). This was before the concept of the optimum moisture content and maximum density in compaction had become widely known through the work and publications of Proctor (9). In early field work on clay soil stabilization with bitumen, the water content used exceeded even that needed for plastic mixtures since the available pug mill mixers could not handle the soil in the truly plastic state. Although such mixes required a considerable time to reduce their moisture content sufficiently for compaction with sheepsfoot rollers, the final soil-bitumen systems were of excellent quality.

Actually, many experienced soil engineers hesitate to use the term "optimum" wate content for compaction methods that involve more energy than that employed in the original and in the ASTM and AASHO standardized procedures which is about 12, 375 ftlb/cu ft. Significantly, the optimum moisture for this energy of compaction usually fall within a few points of the plastic limit. At this limit, the water affinity of a clay soil is considered to be sufficiently satisfied to allow the water molecules most distant from the mineral surfaces to approach the state of free water.

There are a number of good reasons for having a sufficiently high water content, at the time of incorporation of the bitumen, to do more than just satisfy the water affinity of the clay minerals. First, below this condition, the water is likely to be nonuniformly distributed and even if its total amount is theoretically sufficient, there are likely to be mineral surfaces or portions of such that are not satisfied and provide energy potentials for later water adsorption. Second, hydrophilic materials are also oxophilic. This means that in a dry condition they have films of strongly adsorbed oxygen-molecules which are not easily wetted or displaced by bitumen. After these oxygen films are replaced by water films, wetting by bitumen can take place. Diffusion and evaporation of the less strongly held water molecules in the outer layers permits the bitumen and its more active constituents to come ever closer to the mineral surfaces until they interact with the latter and the very strongly held water molecules by physical and/or chemical interchange. Third, in the presence of water-bitumen interfaces, molecules of sizes up to the colloidal that possess affinity for both water and hydrocarbons on different parts of their structure tend to collect at the water-bitumen interface where they can satisfy both affinities. The extent and rate of this phenomenon depends among other factors on the total amount of water-bitumen interface and on the distance the molecules have to travel to reach this interface. Both depend on the amount of water present in the system. The same phenomenon also takes place with adhesion promoting agents that have been admixed either to the aqueous or the bituminous phase, such as fatty and resinous acids, soaps, amines, and their salts, and even more "inert" materials such as long chain paraffines and asphaltenes.

## Dangerous Overemphasis on Maximum Dry Density

Unwarrented and actually unscientific emphasis has been placed by too many engineers within recent years on obtaining as high a dry density of soil-bitumen and other stabilized soils as possible with available equipment at the price of an insufficient water content. While the inherent danger of this was first recognized for soil-cement where water is required as a reactant as well as for initial lubrication of the system, it was more easily overlooked in the case of soil-bitumen where water was supposed to be the enemy. The results of many investigations, especially those on the effectiveness of various anionic and cationic additives, were falsified by the use of too low a water content combined with too high a compactive effort. This is well illustrated by the test results obtained by Dr. Uppal on the dry-mixed specimens. The water content employed was a so-called "optimum" of 12 percent; however, this "optimum" went with a compacting effort of more than  $4\frac{1}{2}$  times that of the standard Proctor method. For the latter and the type of soil employed, the optimum moisture content should have been between 20 and 25 percent. The fact that the dry mixed specimens behaved as well as they did is due to the high temperature range 60 to 70 C or 140 to 158 F at which they were cured to constant weight. This high temperature reduced the viscosity of the bitumen and allowed it to spread more uniformly over the mineral surfaces. This spreading and homogenizing was facilitated by the fact that the higher temperatures had cleansed surfaces, left uncoated by the bitumen, of adsorbed oxygen-molecules and of the more loosely held water molecules some of which had migrated to previously unsatisfied locations of high water affinity. The lowered viscosity of the bitumen also enhanced the diffusion of active molecules contained in it to the bitumen-mineral interfaces. In view of the determinant role of a sufficient water content in allowing important physicochemical reactions to proceed in a soil-bitumen system, it was very fortunate that the monumental work on "Physico-Chemical Factors of Importance in Bituminous Soil Stabilization" sponsored by the Missouri State Highway Department and the U. S. Bureau of Public Roads during the 1930's, was planned and started before Proctor's publications, and had provided for mixing water contents in the normal plastic range. This insures the continued validity of the results obtained and of the conclusions drawn from them (14).

# **Results Obtained With Additives**

In the absence of specific data on the surface-chemical characteristics of the soil employed, the results obtained with the particular additives used (cationic, rubber and paraffine) do not lend themselves to physicochemical analysis. The existence of optimum percentages for particular additives in particular bitumen-soil-water combination has been found previously and recorded in the literature (11). Of particular interest is the good showing of paraffine which was previously reported by Endersby (8) for certain soil-bitumen combinations.

It would be a worthwhile job to study the range of soil types for which paraffine is of equal or greater effectiveness than cationic and anionic additives. The reason for this importance transcends the possible economies derived from the lower cost of the paraffines. Many cationic agents have irritative effects on skins and mucous membranes of living beings. This poses special problems in warm climates and particularly under conditions where plastic soil-bitumen mixes are densified by the action of bare feet. Use of paraffine as additive would avoid this health hazard as would, where applicable, the use of such anionic agents as resinous and fatty acids and soaps. Use of irritative cationic agents can be justified only if they are unmatched in performance and if the entire mixing and compaction process is so mechanized that no health hazard exists.

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