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(As of December 31, 1963)

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Foreword

Progress in the development of knowledge through research seldom results from a single major study. More often it results from a combination of several small research projects, each of which makes a significant contribution. For this reason, six papers, three from the Board's 43rd (January 1964) Annual Meeting and three from the 44th (January 1965) Annual Meeting are combined in this Record for the reader's convenience because each paper is concerned with some aspect of soil-cement stabilization.

The first three papers discuss the physical properties of soil-cement in terms of load-deflection, shrinkage, and the effect of repeated loadings. The remaining three papers pertain to the nature of reactions of cement and lime stabilizers and soils as they are affected by chemical additives, soil-cement construction on loess soil, and rapid testing for determining cement content.

The first paper establishes deflection characteristics and fatigue properties of seven different soils ranging from a non-plastic sand (Group A-1-a) through the plastic silt [A-4(8)] and sandy clay [A-6(2)] groups with K values ranging from 79 to 284. The studies reported on resulted in the development of an equation that predicts load capacity as a function of deflection, thickness of soil-cement, radius of bearing area, and modulus of subgrade reaction.

"Factors Influencing Shrinkage of Soil-Cement" gives the results of one of the first studies on shrinkage of soil-cement and shows that drying after curing is a large factor in controlling shrinkage.

Soils that owe their highly-elastic properties to the presence of mica have been successfully stabilized with cement, but data on the effect of numerous applications of load have not been available. The data presented in the paper "Effects of Repeated Loads in Elastic Micaceous Soils Stabilized with Portland Cement" show that ultimate strengths under repeated loadings are less than for identical samples using conventional loading. This indicates the usefulness of further study of repetitive loading in evaluating stabilization of these soils.

The fourth paper describes a study of the reaction of quartz soil and kaolinite with three stabilizers—hydrated lime, tricalcium silicate, and Type 1 portland cement.

The report entitled "Soil-Cement Construction Using Loess Soil" discusses the design and construction of a 26-mile project in loess soil and its stabilization with portland cement. Data are included from special and routine tests and comparisons are made between preconstruction laboratory test data and data from tests on the completed soil-cement.

The last paper describes a ten-minute test method for determining cement content of cement stabilized mixes. This is only a fraction of the time required by some other tests in current use.

Contents

Load-Deflection Characteristics of Soil-Cement Pavements

P. J. NUSSBAUM and T. J. LARSEN
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A comprehensive research program to develop a thickness design procedure for soil-cement pavements is in progress at the laboratories of the Portland Cement Association. The program has been divided into the following areas of study: (a) the establishment of a relationship between load and deflection for soil-cement pavement, and (b) an evaluation of the fatigue properties of soil-cement. Data from these studies may be correlated by theoretical concepts to formulate a thickness design procedure. This paper reports the findings of the study to develop a relationship between load and deflection for soil-cement. An equation was derived which predicts load capacity as a function of deflection, thickness of soil-cement, radius of bearing area, and modulus of subgrade reaction. Neither the type of material stabilized nor the cement content was statistically significant in determining the parameters of the equation for soil-cement. This suggests that if ASTM procedures are followed and PCA criteria are met, a material with unique structural characteristics is produced. A comparison between the load capacity of soil-cement and a high-quality granular material indicated that if deflections were maintained equal, the capacity of soil-cement was 1.5 times greater for the 4-in. and 3.3 times greater for the 10-in. base thicknesses.

•SOIL-CEMENT is a compacted mixture of pulverized soil, portland cement, and water. It is used in the construction of base courses and subbases for streets, roads, highways, shoulders, airfield pavements, and parking areas to provide a firm, durable pavement layer with considerable bearing strength which distributes imposed traffic loads to the underlying weaker subgrade.

The amount of cement required to produce soil-cement is determined from ASTM (1) procedures for freeze-thaw wet-dry testing and the PCA (2) weight loss criteria. Soils with a cement content less than that required for soil-cement are commonly designated as cement-modified soils. The term cement-treated as used in this report refers to materials containing either more or less cement than is required to meet the minimum criteria for soil-cement.

Mechanically stabilized materials containing an excessively large fine-grain fraction are generally unsuitable for compacted base or subbase construction. However, many of these materials may be used advantageously as soil-cement bases and as cement-treated bases. Some of the advantages of soil-cement are as follows:

1. Protection from failure within the subgrade layer is provided by the load-spreading capability of soil-cement;
2. Volume changes within the base due to freezing and thawing or wetting and drying are minimized; and

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Paper sponsored by Committee on Soil-Portland Cement Stabilization and presented at the 43rd Annual Meeting.

3. The strength of a soil-cement base provides a stable working platform for construction operations.

SCOPE AND OBJECTIVES OF PROGRAM

Data are reported from load-deflection tests on 34 panels, each of which was 10 × 10 ft or larger. Of these panels, 28 were constructed with the cement requirement for soil-cement and six contained higher or lower cement contents. For comparative purposes, tests were also made on six mechanically stabilized bases constructed from a high-quality granular material.

Principal controlled variables of the test program were applied load, magnitude of deflection, thickness of base, strength of subgrade, size and shape of loading plate, and soil type or cement content. Secondary variables were included in indoor and outdoor tests to evaluate the effect of exposure conditions and tests both with and without a 1-in. thick flexible surface to determine the effect of a thin wearing course on load capacity.

The long-range objective of the investigation is the development of a thickness design procedure for soil-cement. This paper is concerned with the load-deflection characteristics of cement-treated materials and has the following specific objectives:

1. To determine the significant variables affecting the load-deflection response of soil-cement test panels;
2. To develop an equation describing the load-deflection relationship for soil-cement bases in terms of the significant variables;
3. To determine the effect of cement content on the load capacity of cement-treated soils constructed with cement contents above and below that required for soil-cement; and
4. To compare the load response characteristics of soil-cement to a high-quality granular material.

TABLE 1

LOAD TEST PROGRAM

Base Type	Base Mat.	Subgrade Type	Group	Thickness (in.)	Plate Radius (in.)[a]				
					6	8	12	15	Ovals
Soil-cement	I	A	a	4	x	x	x		
				7	x	x	x		
				10	x	x	x		
	II	A	b	4	x	x	x		
				7	x	x	x		
				10	x	x	x		
	III	A	c	4	x	x	x		
				7	x	x	x		
				10	x	x	x		
	IV	B	d	4	x	x	x		
				7	x	x	x		
				10	x	x	x		
	V	C	e	4	x	x	x		
				7	(x)	(x)	(x)		
				10	x	x	x		
	V	A	f	4	x	x	x	x	
				7	x	x	x	x	
				10	x	x	x	x	
				13	x	x	x	x	
	V	D	g	4	x	x	x	x	x
				7	(x)	(x)	(x)	(x)	(x)
				10	x	x	x	x	x
	V	D	h	4	x	x	x	x	x
				7	x	x	x	x	x
				10	x	x	x	x	x
				13	x	x	x	x	x
Cement-treated	V	A	i	7	x	x	x	x	x
			j	7	x	x	x	x	x
			k	7	x	x	x	x	x
	VI	A	l	7	x	x	x	x	x
			m	7	x	x	x	x	x
			n	7	x	x	x	x	x
Granular	VII	A	o	4	x	x	x		
				7	x	x	x		
				10	x	x	x		
	VII	B	p	4	x	x	x		
				7	x	x	x		
				10	x	x	x		

[a] (x) = duplicate.

TABLE 2

SOIL PROPERTIES OF BASE MATERIALS

Base Material	Test Panel Group	Atterberg Limits (%)		Std. Dens.[a] (pcf)	Opt. Moist.[a] (%)	Cem. Cont. for Soil-Cement (% by wt.)	As-Constructed Cem. Cont. (% by wt.)	In-Place Dens. (pcf)	AASHO Class.
		L. L.	P. I.						
I	a	NP	NP	128	9.6	4.6	4.6	126	A-1-b
II	b	28	9	107	15.5	13.3	13.3	104	A-4(8)
III	c	25	9	116	12.5	8.7	8.7	116	A-4(1)
IV	d	NP	NP	125	10.5	5.6	5.6	125	A-2-4
V	e,f,g,h,i,j,k	18	NP	128	10.5	5.5	2,4,5.5,8	125	A-1-b
VI	l,m,n	30	11	115	13.6	8.5	3,6,11	115	A-6(2)
VII	o,p	NP	NP	136	10.0	-	None	141	A-1-a

[a] AASHO Designation: T 99.

LOAD PROGRAM AND TEST PROCEDURE

The load program includes test on panels made from soil-cement, cement-treated, and granular base materials. The panels were 4, 7, 10 and 13 in. thick and were constructed from seven different soil types on four different subgrade materials. Loads were applied at the panel interior with circular bearing plates of 6-, 8-, 12-, or 15-in. radius or with a pair of oval plates spaced 14 in. center to center to simulate a dual-tire wheel assembly. The major and minor axes of each oval plate were 11.5 and 7 in., respectively.

Because of the numerous variables, it was not feasible to conduct the program to fit a complete factorial; therefore, the load test program given in Table 1 was designed to emphasize those combinations of variables considered most important.

The load test procedure included the application of seven preloads to eliminate some of the inelastic deformation of the subgrade, thus reproducing better the behavior of an in-service pavement. Deflection under the preload was limited to 0.03 in. Loads for each test were applied in increments to a deflection of 0.05 in. This limit was set to preserve the integrity of the test panels for loading with other plate diameters. In a few cases, the 0.05-in. deflection was not attained because of the load limits of the reaction system. Deflections at each load increment were read when the rate of settlement was less than 0.001 ipm.

MATERIALS AND CONSTRUCTION

Base Materials

Test panels were constructed from soil types I through V at the cement content determined necessary from ASTM test procedures and PCA weight-loss criteria, and from soil types V and VI with cement contents greater and less than those required to meet minimum requirements for soil-cement. The standard density, optimum moisture, and required cement content, together with the placement conditions for each base material, are given in Table 2. The standard density varied from 107 to 128 pcf, the optimum moisture content from 9.6 to 15.5 percent, and the required

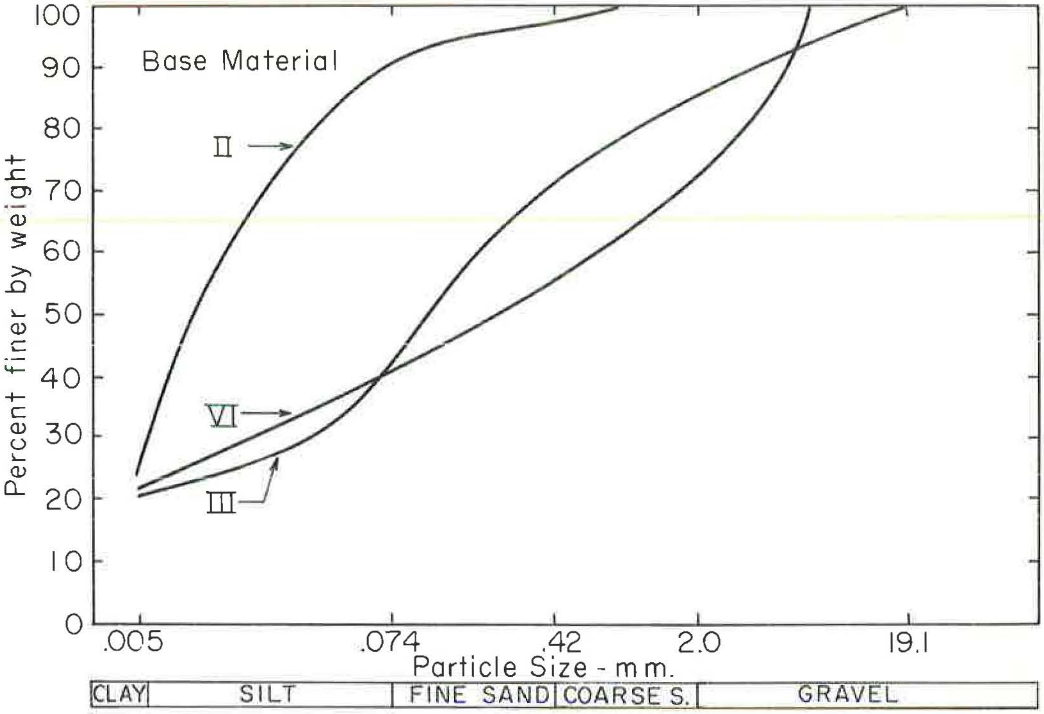

Figure 1. Gradation of cohesive bases.

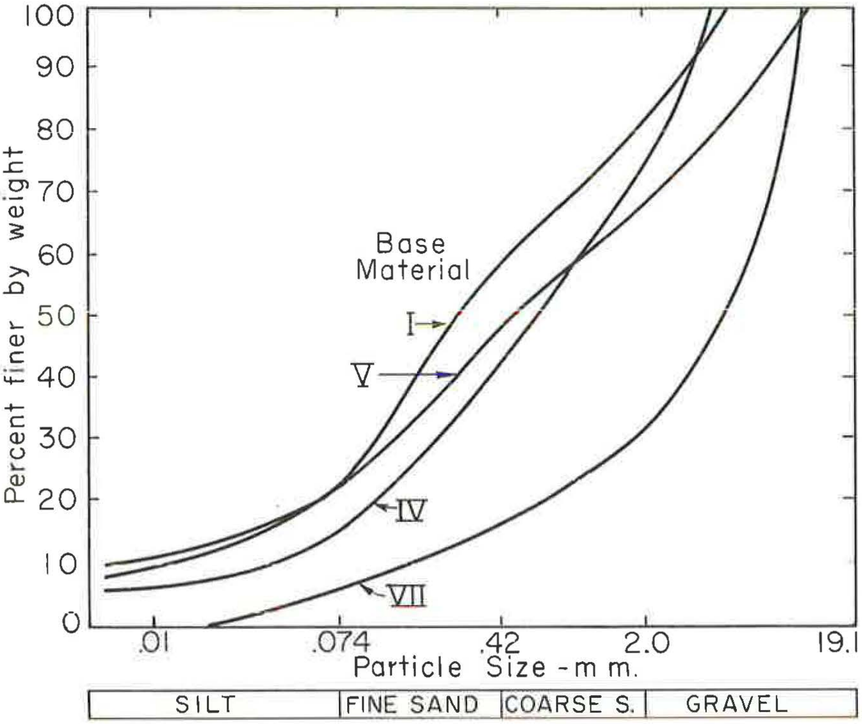

Figure 2. Gradation of cohesionless bases.

TABLE 3

STRENGTH PROPERTIES OF BASE MATERIALS

Base Type	Base Mat.	Cement Cont. (%)	Sonic E (10^6 psi)	28-Day Strength (psi)	
				Flexural	Compressive
Soil-cement	I	4.6	1.15	105	475
	II	13.3	0.57	170	660
	III	8.7	1.0	194	675
	IV	5.6	1.4	210	-
	V	5.5	1.30	118	460
Cement-treated	V	2	0.91	50	260
		4	1.05	107	382
		8	1.32	130	451
	VI	3	0.56	53	193
		6	0.87	76	180
		11	1.18	130	295

cement content for soil-cement from 4.6 to 13.3 percent by weight.

Gradations for each of the untreated soil components are shown in Figures 1 and 2. Two cohesive and three noncohesive materials were used in the test panels, with the amount passing the No. 200 mesh sieve varying from 16 to 91 percent and the gravel component ranging from 0 to 25 percent. The soil type varied from A-1-b to A-4(8), covering a portion of the wide range of soils suitable for soil-cement base construction.

Specimens were fabricated for strength tests at the time of construction of the panels. The specimens were 2.8- × 5.6-in. cylinders and 3- × 11.25-in. beams. All specimens were moist cured for 28 days. Strength data for the base materials are reported in Table 3.

Soil for construction of the panels was mixed in a pug mill for 2 min before and after addition of Type I cement. Sufficient water was then added with a spray bar to bring the mixture to optimum moisture and mixing was continued for 3 min. The treated soil was compacted within 30 min after mixing. Compaction was accomplished with hand-operated vibratory and impact equipment. All test panels were cured for 28 days before loading, either by application of a curing compound commonly used for portland cement concrete pavements or with an asphalt emulsion.

Base material VII was used for construction of the granular test panels. The gradation and soil properties are indicated in Figure 2 and Table 2. A 1-in. thick bituminous surface course was placed on all granular base panels.

Subgrade Materials

Four different materials, designated A, B, C and D, were used as subgrades under the test panels. The gradation and Atterberg limits for each material are shown in Table 4. Materials A and B were placed outdoors to a depth of 4 ft on top of a native silty clay. Materials C and D were used for indoor tests.

Subgrade strength was determined with a 30-in. diameter bearing plate in accordance with the procedures of ASTM D 1195-57. The modulus of subgrade reaction for each panel group was determined at the time of load testing and is reported in Table 5. Most of the panels were constructed on low-strength subgrades to emphasize the load response characteristics of the base for the more critical, weaker subgrade condition. Variation of strength for indoor subgrades was attained by selectively reworking the soils on removal of the test panels. Subgrade strength changes for the outdoor test panels were due principally to the climatic variable.

FACILITIES

Indoor Area

Indoor tests were conducted in a concrete building with temperature control during

TABLE 4

SUBGRADE MATERIALS

Subgrade Material	Gradation (%)						Atterberg Limits (%)	
	Gravel (> 2.0 mm)	Coarse Sand (2.0-0.42 mm)	Fine Sand (0.42-0.074 mm)	Silt (0.074-0.005 mm)	Clay (< 0.005 mm)	Colloids (< 0.001 mm)	L. L.	P. I.
A	0	2	8	32	58	44	38	13
B	29	32	26	8	5	-	NP	NP
C	0	4	16	40	40	20	36	19
D	0	4	9	35	52	40	39	22

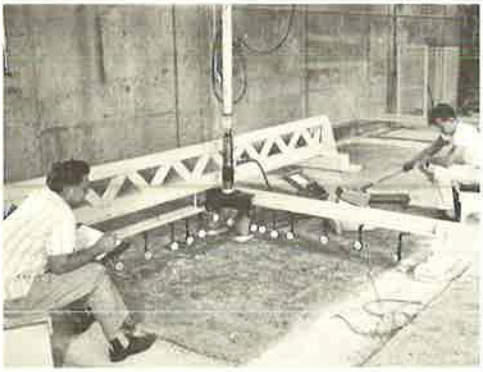

Figure 3. Load test with dual oval plates, indoor.

Figure 4. Load test with 30 in. plate, outdoor.

TABLE 5

MODULUS OF SUBGRADE REACTION k

Group	Subgrade Material	k (pci)
a	A	99
b	A	106
c	A	79
d	B	284
e	C	120
f	A	92
g	D	175
h	D	155
i	A	116
j	A	116
k	A	116
l	A	116
m	A	116
n	A	116
o	A	120
p	B	300

the heating season. Space was provided for testing six soil-cement panels. Overhead steel beams were framed into the building walls and a system of traveling crossbeams and trolleys permitted load application at any location on a test panel. A view of the indoor area with a load test in progress is shown in Figure 3.

Subgrade soils were placed in a 4-ft deep pit excavated within the building. The test pit is 24 ft wide and 50 ft long. A concrete slab isolated from the footings by an expansion joint was placed at the bottom of the pit, and a shielded membrane waterproofing treatment was applied to protect the subgrade from moisture changes.

Outdoor Area

Reaction for outdoor load tests, as shown by Figure 4, was provided by a system of steel beams and trolleys which permitted loading at any location on the test panels. The reaction system was secured by a series of soil anchors placed to a depth of about 10 ft.

An area adjacent to each test panel and at the same elevation as the subgrade was protected with polyethylene and a plywood housing. Thus, a subgrade condition similar to that below the test panels was preserved for determining the subgrade strength at the time of load testing.

EQUIPMENT AND INSTRUMENTATION

Loads were applied with 20-ton capacity hydraulic jacks. The magnitude of load was measured by a strain sensing load cell and a strain indicator. The oval bearing plates seen in Figure 3 were arranged to simulate in gross area and spacing the dual truck tire commonly used with 18,000-lb axle loadings. Equal distribution of load was assured by careful leveling of each oval and by measurements of the magnitude of load transmitted to each. As shown in Figure 4, a housing was placed between the load cell and the bearing plate to contain a dial used to measure deflection directly under the center of the load. A centrally located $\frac{3}{4}$-in. diameter hole afforded access of the dial stem to the test panel below the bearing plate.

Deflections of test panels due to load were measured with 0.001-in. dial gages attached to a wooden bridge. Supports for the dial bridge were outside the area influenced by load. As seen in Figures 3 and 4, dials were arranged to read deflections of the test slab adjacent to the edge of the bearing plate, at the center of load application, and also along longitudinal and transverse lines through the center of loading. Stainless steel plugs cemented to the soil-cement base served as reference points for the dial stems. For panels with thin bituminous surfacing, the stainless steel plugs extended through small holes drilled in the asphalt cover.

LOAD-DEFLECTION DATA

An analysis of variance was made on the accumulated data to determine which of the variables were statistically significant in influencing the relationship between load and deflection. The magnitudes of load and deflection, modulus of subgrade reaction, thickness of base, and radius of bearing area were found to be significant. Variables of soil type, cement content, thin bituminous surface, and exposure were not significant when the minimum criteria for soil-cement were met, although cement content was significant in determining the load capacity if the cement content was less than that required for soil-cement.

Data are presented to indicate the effect of each significant variable on load capacity, and comparisons are made between soil-cement, cement-modified soil, and granular bases. Techniques of nondimensional analysis are used to derive equations for computing pavement thickness in terms of the variables. Since inclusion of the complete record of accumulated data is not feasible, in most cases only sample data are presented.

EVALUATION OF VARIABLES

In the following discussion an evaluation of the variables of thickness, size of bearing plate, strength of subgrade, soil type, and cement content is made to determine the contribution of each to the load-deflection characteristics of soil-cement base construction. Each variable is examined in terms of load capacity with the remaining variables held constant.

TABLE 6

EFFECT OF THICKNESS ON LOAD-DEFLECTION[a]

Group	Thickness (in.)	Deflection under Load (in.)			
		3 Kips	6 Kips	9 Kips	12 Kips
a	4	0.032	0.058	-	—
	7	0.010	0.020	0.030	0.040
	10	0.005	0.010	0.017	0.022
b	4	0.024	0.042	0.063	-
	7	0.012	0.018	0.025	0.035
	10	0.006	0.013	0.020	0.027
c	4	0.020	0.034	0.063	-
	7	0.013	0.027	0.045	-
	10	0.009	0.015	0.027	0.035
d	4	0.008	0.011	0.021	0.023
	7	-	0.006	0.001	0.015
	10	-	0.003	0.005	0.008
e	4	0.020	0.043	-	-
	7	0.007	0.016	0.027	0.038
	10	0.005	0.014	0.023	0.032
f	4	0.024	0.047	-	-
	7	0.010	0.023	0.037	0.056
	10	0.006	0.015	0.025	0.035
	13	0.005	0.010	0.015	0.020

[a] Test data for 8-in. plate radius.

Effect of Thickness on Load Capacity

To evaluate the effect of thickness on load capacity, the variables of subgrade strength and loading plate size were held constant. A sampling of test data for four increments of load applied with a 16-in. diameter plate is presented in Table 6 and in Figures 5, 6 and 7. The data show that load capacity increased with increasing thickness. The load-deflection data shown in Figure 5 are from test panels of Group f and are representative of the load response characteristics obtained from all other tests on soil-cement. For a constant thickness, the load-deflection diagram was linear for the range of deflections shown.

The trend of load capacity with increasing thickness of soil-cement, illus-

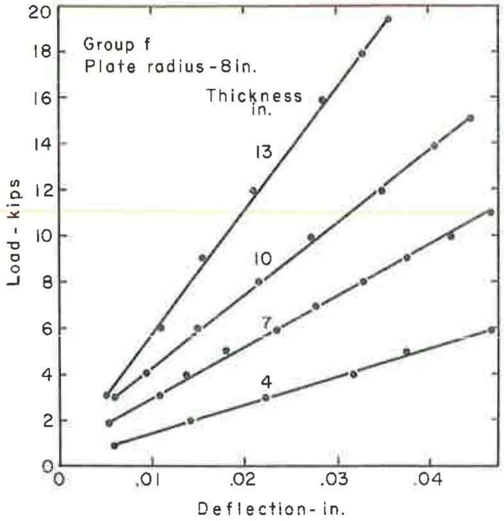

Figure 5. Effect of soil-cement thickness on load capacity.

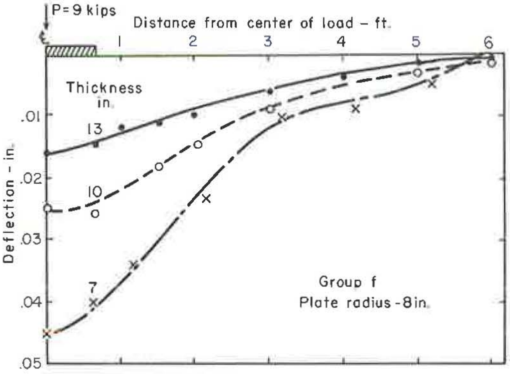

Figure 7. Deflection profile for soil-cement base.

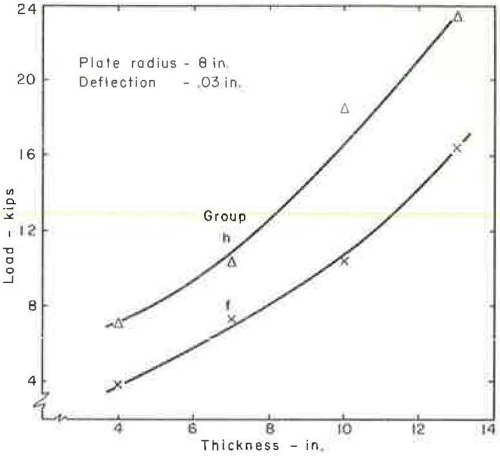

Figure 6. Curvilinear relationship between thickness and load capacity.

TABLE 7

EFFECT OF PLATE SIZE ON DEFLECTION[a]

Group	Thickness (in.)	Deflection at Plate Radius (in.)			
		6 In.	8 In.	12 In.	15 In.
a	4	-	-	0.062	-
	7	0.032	0.030	0.021	-
	10	0.014	0.017	0.015	-
b	4	-	0.063	0.050	-
	7	0.030	0.025	0.019	-
	10	0.021	0.020	0.018	-
c	4	-	0.063	-	-
	7	0.052	0.045	0.032	-
	10	0.028	0.027	0.019	-
d	4	0.024	0.021	0.017	-
	7	0.010	0.011	0.009	-
	10	0.005	0.005	0.006	-
e	4	-	-	0.043	-
	7	0.30	0.027	0.019	-
	10	0.024	0.023	0.017	-
f	4	-	-	0.038	0.038
	7	0.048	0.037	0.033	0.031
	10	0.029	0.025	0.018	0.015
	13	0.016	0.015	0.013	0.014

[a] Test data for 9-kip load.

trated by Figure 6, shows that for a constant deflection the load capacity increased exponentially with increased thickness of soil-cement.

Deflection profiles obtained along two lines at right angles also demonstrated the effect of thickness on load capacity. Data in Figure 7 were taken from tests made on three thicknesses of Group f for a 9-kip load applied with a 16-in. diameter plate. Measurable deflections occurred over a radial distance of more than 5 ft from the point of load application, demonstrating that a considerable slab area participated in carrying and distributing imposed loads.

Effect of Plate Size and Shape on Load Capacity

An evaluation of the effect of plate size on load capacity was made by comparison of deflections for loads applied with four different size bearing plates on four thicknesses

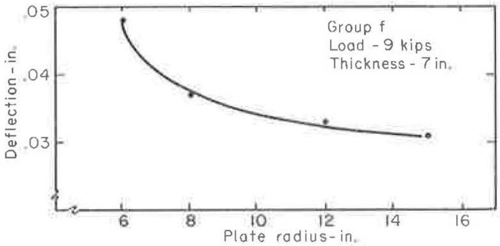

Figure 8. Effect of plate size on deflection.

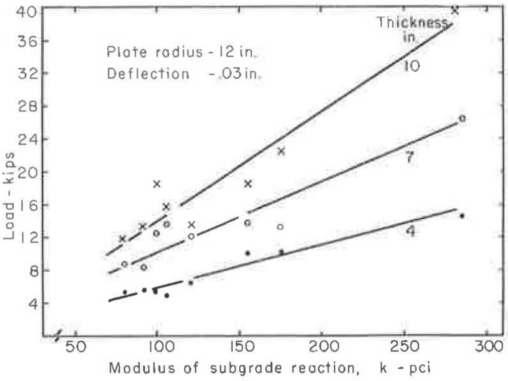

Fig. 10 – EFFECT OF SUBGRADE STRENGTH ON LOAD CAPACITY

Figure 10. Effect of subgrade strength on load capacity.

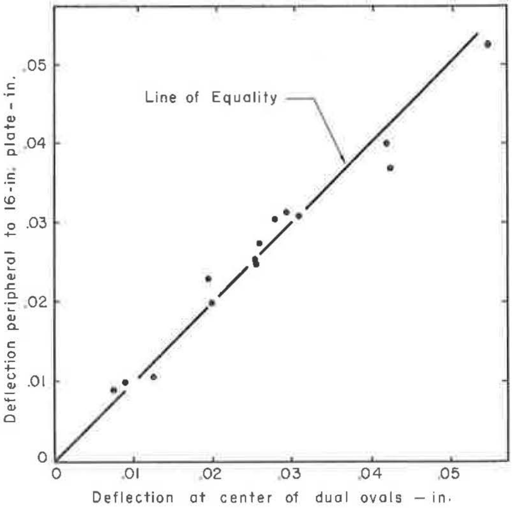

Figure 9. Comparison of deflections for circular and oval plates.

of soil-cement. A sampling of test data given in Table 7 indicates that as the plate size was increased, the magnitude of deflections decreased. It is significant that soil-cement bases are more sensitive to plate size than are the more rigid concrete pavements. However, the effect of increasing plate size was reduced for increased thicknesses of soil-cement. An example of the data for the 7-in. thick slab of Group f is shown in Figure 8. An increase in plate radius from 6 to 15 in. resulted in an exponential decrease in base deflections. For an 8-in. radius plate with a bearing area of 201 sq in., the deflection was 0.037 in. A plate of this size corresponds closely to the area of a dual truck tire commonly used with 18-kip axle loading. A reduction in bearing area to 113 sq in. (6-in. radius plate) resulted in a 30 percent increase in deflection.

Dual oval plate tests were made to compare deflections with those obtained with circular plates. The oval steel bearing plates were arranged to simulate the bearing area of dual truck tires. A comparison is made in Figure 9 between deflections measured at the center of the dual plates and those measured at the periphery of a 16-in. diameter circular plate. Deflections of the oval and circular plates were in good agreement, so it may be concluded that data from the laboratory study are comparable to the vehicle load data obtained from field in-service pavement evaluations.

Effect of Subgrade Strength on Load Capacity

To determine the effect of subgrade strength on load capacity, a comparison was made of the test data with deflection, thickness, and size of load plate held constant. Data in Figure 10 show that load capacity of the soil-cement increased with increasing subgrade strength. The data show considerable scatter, but indicate that a linear relationship is acceptable. It is seen that subgrade strength should be included in a load response equation.

Influence of Soil Type and Cement Content on Load Capacity

Influence of soil type and cement content on load capacity of soil-cement was determined by comparing the magnitudes of load required to obtain a deflection of 0.04 in.

TABLE 8

EFFECT OF CEMENT CONTENT ON LOAD CAPACITY

Soil-Cement Thickness (in.)	Load Capacity[a]		
	Arith. Mean (kips)	Std. Dev. (kips)	Coeff. of Var. (%)
4	4.1	0.5	13
7	8.1	1.2	15
10	12.7	1.3	10

[a] At 0.04-in. deflection, adjusted to k = 79 pci.

when loaded with an 8-in. radius plate. Data presented previously showed that load capacity varied directly with subgrade strength. Therefore, to compare load capacity, a strength ratio was obtained for each test group by dividing the modulus of subgrade reaction, k, of each group by the modulus of Group c, which had the low k-value of 79 pci. Equivalent loads for the uniform subgrade strength condition were then obtained by dividing the actual loads by the strength ratio for the respective group. Variations of equivalent load capacity were observed for the range of soil type from A-1-b to A-4(8) and the required cement content of 4.6 to 13.3 percent by weight. The arithmetic mean load capacity, standard deviation, and coefficient of variation for panel thicknesses of 4, 7 and 10 in. are given in Table 8. The average coefficient of variation for the three thicknesses was about 13 percent, demonstrating that the wide variations of soil type and cement content were of minor significance in influencing the load-deflection response of soil-cement bases. It is demonstrated later that this variation is approximately equal to the replication error.

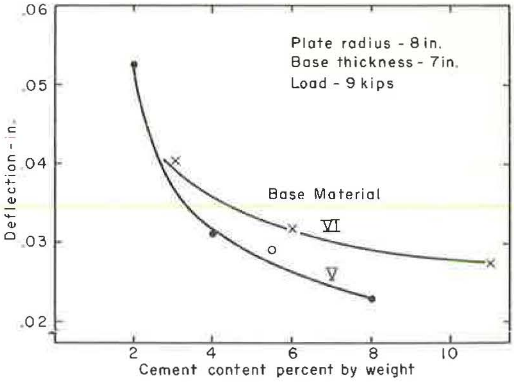

Figure 11. Effect of cement content on deflection for cement-treated soils.

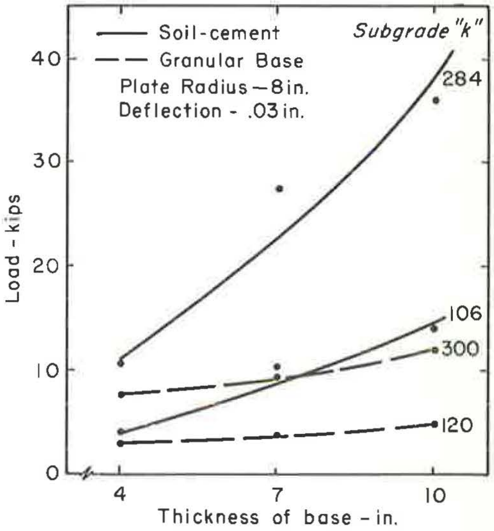

Figure 12. Load capacity of soil-cement and granular bases.

Effect of Higher or Lower Cement Content.—Load tests were made to determine the effect on load capacity of variations in cement content above and below the minimum requirements for soil-cement. Data in Figure 11 show that for the same soil component, deflections decreased significantly as the cement content was increased in the range of cement-modified soils. For the range of cement contents greater than the minimum requirements for soil-cement, the rate of decrease of deflection was smaller. The lower durability of pavements constructed with cement contents less than the minimum amount required to produce soil-cement may be important to field performance. Therefore, environmental factors should be considered in selecting the desired cement content. As noted previously, the required cement contents for soil-cement are 5.5 percent for soil type V and 8.5 percent for soil type VI.

The preliminary data of a further investigation of cement-treated bases indicate that for cement contents substantially different from the minimum amount necessary for making soil-cement, cement content should be considered in the load response analysis.

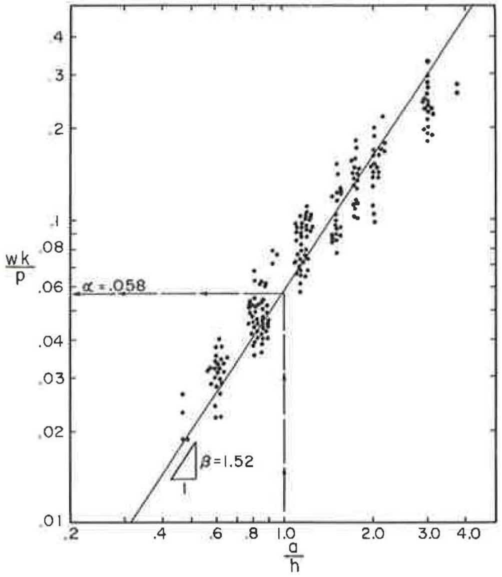

Figure 13. Best-fit line for soil-cement bases.

TABLE 9

REPRODUCIBILITY OF TEST DATA[a]

Group	Bearing Plate Radius (in.)	Load (kips)		Avg. Load (kips)	Dev. from Mean (%)
e	6	8.8	10.3	9.5	8.4
	8	9.7	9.9	9.8	1.2
	12	12.1	13.0	12.6	3.2
g	6	9.8	10.7	10.2	4.9
	8	10.6	12.3	11.5	10.4
	12	13.4	14.1	13.8	2.2
	Dual ovals	10.4	12.6	11.5	9.6
Avg.					5.7

[a] Data for replicate 7-in. soil-cement panels at 0.03-in. deflection.

Comparison with Load Capacity of Granular Bases. — Granular base load tests were made to compare the load capacity of soil-cement with those of a high-quality granular base material. Figure 12 indicates that the load capacity of the granular base is considerably less than that of soil-cement for both strong and weak subgrades. It should be noted that the subgrade under the granular base was slightly stronger than the subgrade under the soil-cement. In addition, the thicknesses refer only to the base material and do not include the 1-in. bituminous surface on the granular materials. Load capacity increased with increasing granular base thickness, but the rate of gain in load capacity was substantially greater for increasing thicknesses of soil-cement than for granular bases. A comparison of load capacity based on equal deflections for soil-cement and granular bases showed that the ratio of soil-cement to granular base load capacity was about 1.5 for the 4-in. and about 3.3 for the 10-in. thicknesses. This was true for both weak and strong subgrade conditions. This demonstrates the effect of the beam strength of soil-cement and its influence on load capacity.

ANALYSIS OF LOAD-DEFLECTION TEST RESULTS

An analysis of the load-deflection test results was made to develop an equation evaluating the influence of the significant variables on load capacity for soil-cement and cement-treated bases.

Soil-Cement Bases

Load response of soil-cement bases is described in terms of the significant variables. A nondimensional logarithmic plot of the test data is shown in Figure 13. Deflection, w, multiplied by the modulus of subgrade reaction, k, and divided by the intensity of applied load, p, is plotted along the ordinate. The ratio of bearing area radius, a, to soil-cement thickness, h, is given along the abscissa.

$$\frac{wk}{p} = \alpha \left(\frac{a}{h}\right)^{\beta} \qquad (1)$$

where the parameter α is the ordinate at a point on the best-fit line corresponding to an abscissa of $a/h = 1$ and β is the slope of the regression line. It is recognized that a curvilinear best-fit line would better describe the test data at a/h of 3 or larger. However, the straight line is used for the range of a/h from 0.5 to 2.0, which covers most of the conditions of load area and pavement thicknesses encountered in pavement design. It is seen that $\alpha = 0.058$ and $\beta = 1.52$. Thus, the specific expression describing the load response of soil-cement is

TABLE 10

BEST-FIT PARAMETERS FOR CEMENT-TREATED BASE COURSES

Base Mat.	Group	α	β	Cement Content (% by wt.)
V	i	0.100	1.52	2.0
V	j	0.066	1.58	4.0
V	k	0.049	1.58	8.0
VI	l	0.085	1.59	3.0
VI	m	0.066	1.57	6.0
VI	n	0.057	1.57	11.0

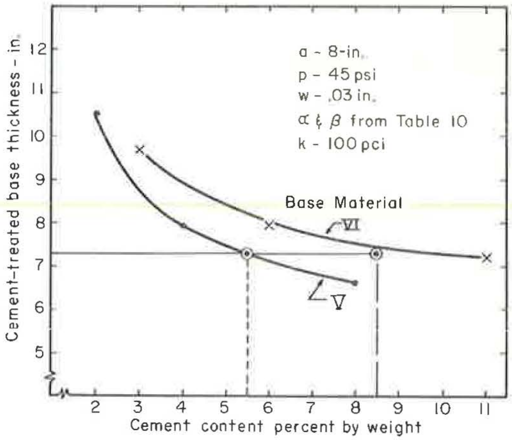

Figure 14. Effect of cement content on required base thickness.

$$\frac{wk}{p} = 0.058\left(\frac{a}{h}\right)^{1.52} \qquad (2)$$

The standard error of estimate at a/h = 1 was 0.007 and the coefficient of correlation was 0.94. When expressed in terms of thickness of soil-cement required for a 9-kip load, an 8-in. radius plate, and 0.03-in. deflection, the standard error was about 0.5 in.

Replicate tests were made on 7-in. thick panels to determine the reproducibility of the data. Presented in Table 9 are comparisons between original and replicate test data. The comparisons are based on the magnitude of load required to produce a deflection of 0.03 in. It is seen that maximum deviation from the mean was 10.4 percent and the average deviation for seven replicate tests was 5.7 percent. For a 7-in. thickness, the replication error would be about 0.4 in., which is approximately equal to the standard error of estimate and to the coefficient of variation attributed to the effect of different soil types and cement contents.

The best-fit equation may be solved explicitly for thickness of soil-cement, h, to yield:

$$h = a\left(\frac{0.058\,p}{wk}\right)^{\frac{1}{1.52}} \qquad (3)$$

and may be restated in terms of the total applied load, P, to read:

$$h = a\left(\frac{0.072}{0.32}\right)\left(\frac{P}{wk}\right)^{0.658} \qquad (4)$$

An increase in plate radius, deflection, or subgrade strength would decrease the required thickness of soil-cement, whereas an increase in total load results in an increase of the required thickness.

Eq. 4 defines the load response characteristics for soil-cement produced from a wide range of soil types stabilized with the cement content required from ASTM tests and PCA weight-loss criteria. A wide range of soil-cement materials were used to determine the load-deflection equation, thus indicating that soil-cement is a unique structural material, and a strength factor is not required in the equation.

Cement-Treated vs Soil-Cement Bases

Load response of cement-treated bases is described in terms of the variables found significant for soil-cement; however, for cement-treated bases the amount of cement was significant in determining load capacity. The parameters α and β were therefore determined for each of the three cement contents used with base materials V and VI. Values of the parameters are given in Table 10. Data were obtained for plotting Figure 14 by substituting these values in Eq. 1 and solving for h, using a 9-kip load, 8-in. radius plate, and 0.03-in. deflection. These data show that variations of cement content within a soil type were significant in determining required thickness. For example, increasing the cement content of base material V from 2 to 8 percent resulted in a decrease in thickness requirements of nearly 4 in.

Agreement of the soil-cement and cement-treated load-deflection response equations is demonstrated in Figure 14. The solution of the equation for soil-cement for the 9-kip load, 8-in. radius plate size and 0.03-in. deflection yields a required thickness of 7.3 in. A plot of this thickness vs the required cement content of 5.5 percent for material V and 8.5 percent for material VI is shown by the large circles. It is seen that these circles fall on the curves of required thickness vs cement content, thus indicating agreement between the load response equations for cement-treated and soil-cement bases.

Soil-Cement vs Granular Bases

A comparison of granular bases with soil-cement is made in terms of the variables used in defining the load response characteristics of soil-cement. As shown in Figure 15, the best-fit line for the granular base was determined for Eq. 1. Parameters α and β were 0.163 and 0.65. Thus, the specific expression describing the granular bases is

$$\frac{wk}{p} = 0.163 \left(\frac{a}{h}\right)^{0.650} \qquad (5)$$

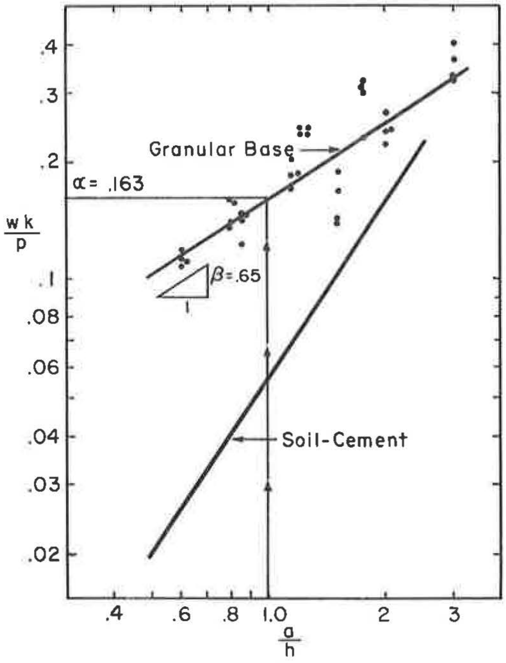

Figure 15. Best-fit line for granular base.

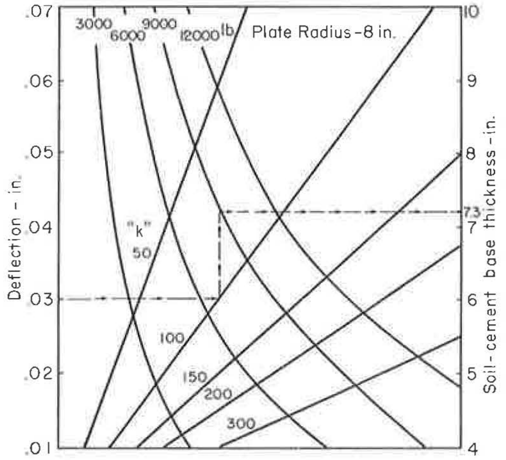

Figure 16. Load-deflection-thickness nomogram.

When expressed as thickness of granular base course for the 9-kip load, the standard error of estimate at a/h of 1 was about 5 in. and the coefficient of correlation was 0.94. Best-fit lines for soil-cement and granular base sections are compared in Figure 15, the lower describing the best-fit line for soil-cement as presented previously in Figure 13.

SOIL-CEMENT THICKNESS DESIGN

Development of a rational method for soil-cement thickness design is the overall aim of the current investigation. Solution of Eq. 4 yields the required thickness of soil-cement for a predetermined magnitude of permissible deflection. In Figure 16 a nomogram of Eq. 4 is presented for a 201-sq in. bearing area, equivalent to an 8-in. radius plate. In this example, the nomogram is entered with an assumed deflection of 0.03 in., progression is horizontal to intercept the modulus of subgrade reaction of 100 pci, then vertical to intercept the curve for a wheel load of 9,000 lb; the required thickness is given as 7.3 in. Similarly, with an assumed deflection of 0.04 in., the thickness is 6.1 in. Thus, the magnitude of deflection assumed has considerable influence on the required thickness. The development of the load-deflection relationship for soil-cement, as described by Eq. 4, is a step forward in developing a thickness design method for soil-cement. A further test program has as its objective the determination of the fatigue characteristics and permissible deflections of soil-cement bases. From the load-deflection characteristics and the permissible deflection, a fully realistic soil-cement thickness design method can then be developed.

CONCLUSIONS

1. Significant variables of the load-deflection response of soil-cement meeting recognized test criteria were the modulus of subgrade reaction, thickness of soil-cement, and radius of bearing area. Variables not significant were type of soil and cement content, climatic exposure variables, and thin bituminous surfacing.
2. A load response equation for soil-cement (Eq. 2) was developed from a statistical analysis of a nondimensional plotting of the significant load-deflection test data. However, use of this equation in the thickness design of soil-cement bases depends on the determination of the fatigue characteristics and permissible deflection limits of soil-cement. It is significant that a factor for strength was not needed in this equation.
3. Load-deflection characteristics of cement-treated bases can be described by Eq. 1. Variations of cement content within a soil type were significant in determining required thickness, as was shown by decreasing values of α with increasing amounts of cement. The effect of increased load capacity with increased cement content was most significant at cement contents less than those required for soil-cement. However, durability considerations may alter the load capacity vs cement content relationship.
4. Load capacity of soil-cement compared to granular bases at equal magnitude of deflection was about 1.5 times greater for the 4-in. thick bases and about 3.3 times greater for the 10-in. thickness.

REFERENCES

1. American Society for Testing Materials. ASTM Standards, Part 4. ASTM Designation D559-57 and D560-57, pp. 1343 and 1349, 1961.
2. Soil-Cement Laboratory Handbook. Portland Cement Assoc., p. 28, 1959.

Factors Influencing Shrinkage of Soil-Cement

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Linear shrinkage of 2- × 2-in. diameter molded soil-cement specimens was measured at prescribed intervals during curing. Four soils were used: a standard graded Ottawa sand with no clay, an Iowa dune sand and an Iowa loess silt with 2.8 and 16 percent montmorillonitic clay, respectively, and a North Carolina clay with 30 percent kaolinitic clay. Effects of varying the cement content, initial water content, and curing method were evaluated. Results show no relation between total shrinkage and cement content in the ranges investigated. The initial water content had some effect on shrinkage of the silt and the clay but not of the sands; compaction below optimum moisture content gave slightly less shrinkage. Montmorillonitic clay in a mix greatly increased total shrinkage, kaolinitic clay increased shrinkage less, and total linear shrinkage ran as high as 1 percent. Shrinkage occurred mainly on drying, and prolonged curing before drying slightly increased total shrinkage.

•SOIL-CEMENT is a hardened material formed by curing a tightly compacted mixture of soil, portland cement, and water. Use of soil-cement as flexible pavement base course is steadily increasing; last year's rate of construction was over 10,000 lane-miles (1), rivaling the construction rate of portland cement concrete pavement.

A soil-cement problem which is probably more damaging to public relations than to the pavement is shrinkage cracking. Soil-cement commonly contracts slightly following construction; the resulting cracks may be as much as ⅛ in. across, although are usually much less. At worst the cracks have about the same effect as joints on the load-carrying capacity; they are a problem only if left unsealed, permitting water to infiltrate and weaken the subgrade. Subgrade pumping through the cracks is not common, perhaps because of small slab size, bridging by slab interlocking, and low traffic volumes. The cracks are usually sealed or are allowed to form before application of the final bituminous surfacing, which acts as a seal. Sealing of individual cracks by maintenance crews is costly, looks unsightly, and affects riding qualities of the surface.

The present study was undertaken to learn the causes of shrinkage and its relationship to fundamental variables including soil, cement content, and method and time of curing and drying.

REVIEW OF PREVIOUS WORK

Factors influencing shrinkage of portland cement paste and concrete have been studied extensively for many years (2, 3). Shrinkage of concrete relates to drying of the cement paste (2). Aggregate reduces shrinkage, theoretically by acting as rigid inclusions in the shrinking matrix, the more inclusions, and the more rigid the inclusions, the less the shrinkage. Based on this assumption is a relationship derived by Pickett (5):

$$S = S_o (1 - g)^{\alpha} \qquad (1)$$

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where S and S_0 are unit linear shrinkages of concrete and cement pastes, respectively, g is the volume fraction of the aggregate, and α depends on Poisson's ratio and Young's moduli of the paste and aggregate. Subsequent tests verified the relationship; α for sand mortars is about 1.7 (5).

Drying shrinkage of neat cement pastes relates to water/cement (w/c) ratio, although shrinkage of concrete is reported to be little influenced by the w/c ratio after a day of hardening (3). High w/c ratios mean higher initial water content and consequent shrinkage; however, as water content is lowered, either through drying or because of initially low w/c ratio, shrinkage per unit loss of water is increased. This is believed to be due to much higher tensile stresses that result from water lost from the gel instead of from the larger capillaries. Increasing the cement content tends to increase shrinkage by providing more gel. However, increasing the cement content without correspondingly increasing the amount of water first increases shrinkage until the w/c ratio is about 0.5 to 0.4, and then decreases shrinkage (9) at lower w/c ratios, perhaps because of incorporation of unhydrated particles. Increasing the size of gel particles into the noncolloidal range, as by steam curing, also reduces tension and shrinkage (2). Use of coarse grind cement reduces shrinkage, perhaps partly because of included unhydrated particles (2). Prolonged curing before drying should increase shrinkage because of the higher proportion of gel; however, this appears to be offset by increasing structural rigidity (4), perhaps causing stress relief by microcracking with correspondingly less relief through shrinkage (2).

Carbonation of the $Ca(OH)_2$ liberated by hydrating cement causes irreversible shrinkage, for reasons not yet understood. However, either water-saturated or ovendry specimens absorb little CO_2 and, therefore, are not affected. Carbonation shrinkage is maximum at a relative humidity of about 55 percent (2).

Application of these factors to soil-cement suggests that the latter should have less shrinkage than concrete, soil-cement contains relatively little cement gel, and its use compared to most uses of concrete should cause it to undergo relatively little carbonation. However, the importance of clay content is indicated by Hveem and Tremper (6), who emphasize the utility of cleaning the clay from aggregates to reduce concrete shrinkage. Shrinkage of mortar bars was found to be in linear relation to the sand equivalent, a sedimentation value showing relative amount of plastic fines (AASHO Designation: T 175-56). In terms of shrinkage, it has been suggested that the clay fraction should be considered part of the shrinking or gel phase rather than of the rigid or aggregate phase (2) because clay aggregate would shrink more than the matrix.

Shrinkage of relatively pure clays in the manufacture of ceramic products is commonly measured for specimens dried from the water-of-plasticity (slightly more than plastic limit) to ovendry (105° C) (10, p. 75 ff). Therefore, shrinkage relates to water-of-plasticity requirements, which depends on the kind of clay mineral, particle size distribution, and adsorbed ions.

T.

PROPER

Soil Class.[a]	AASHO Class.[b]	Sample No.	Horizon	Sampling Depth (ft)	Textural Composition (%)[c]				
					Gravel (>2mm)	Sand (2-0.074 mm)	Silt (0.074-0.005 mm)	Clay	
								(<0.005 mm)	(<0.002
Fine Sand[g]	A-3(0)	S-6-2	C	6-11	0.0	95.5	1.5	3.0	2.
Silt loam[i]	A-4(8)	20-2 VII	C	35-36	0.0	0.4	80.0	19.6	16.
Clay[j]	A 7-6(11)	AR-6	B	2[k]	0.0	45.2	18.3	36.5	30.

[a] Triangular chart developed by U. S. Bureau of Public Roads, using 0.074 mm, instead of 0.05 mm, as the division between silt-size particles and sand.

[b] AASHO Method M 145-49.

[c] ASTM Method D 422-54T.

[d] ASTM Methods D 423-54T and D 424-54T.

[e] Ammonium acetate (pH = 7) method on soil fraction < 0.42 mm (No. 40 sieve) for sand and silt, and on soil fraction < 2 mm (No. 10 sieve) for clay.

[f] Versenate method for total calcium.

PROCEDURE

Soils

Four soils were selected to represent a range in size gradations and mineralogy: graded Ottawa pure silica sand meeting gradation requirements of ASTM Designation: C 109-56; an Iowa A-3(0) dune sand and an Iowa A-4(8) loess silt loam with about 2.8 and 16 percent -2 μ montmorillonitic clay, respectively; and a North Carolina A-7-6(11) clay containing about 30 percent -2 μ kaolinitic clay. Pertinent properties of the natural soils are given in Table 1.

Cement and Water

Type I portland cement for the study (Table 2) was stored in a sealed container until used. Distilled water was used for mixes.

Mixes

Cement requirements and optimum moisture contents for the soils were determined by standard soil-cement freeze-thaw test procedures (7). Additional shrinkage measurements were made with cement contents above and below the estimated minimum requirements (Table 3) and with the moisture contents above and below optimum (Table 4) to determine the effects of these variables.

Specimens

Measurements of soil-cement shrinkage presents problems not encountered in concrete or mortars because of the differing method of sample compaction. Soil-cement is molded relatively dry by ramming into a mold. This procedure makes it difficult to obtain long specimens of uniform density throughout. Greater precision in measurements is required for short specimens, but they are less subject to cracking from shrinkage differentials.

Soil cylinders 2.00 in. in diameter by 2.00 ±0.05 in. in height were molded to approximately standard Proctor density by dynamic compaction procedures using 10 blows from a 5-lb rammer dropping 12 in. (13). Before extrusion with an hydraulic jack, a double-pointed index pin was pushed through a plastic guide tube into the top center of each sample. The sample was then extruded and turned over, and another index pin was pushed into the other end for later optical measurements of the distance between the tips of the two pins.

Measurements

In initial trials, measurements were attempted with a 1/10,000-in. dial gage, but error was introduced by inconsistent spring pressure from the stem of the dial and by

LE 1

ES OF SOILS

	Colloids (<0.001 mm)	Atterberg Limits (%)^{d}			Chemical Properties				Predominate Clay Material
— m)		L.L.	P.L.	P. I.	C. E. C. (m eq/100 g)^{e}	Carbonates (total Ca^{++}, %)^{f}	pH	Organic Matter (%)	
	2.6	12.7^{h}	-	non-plastic	1.0	0.4	6.6	0.1	Montmorillonite (trace)
	14.5	30.8	24.6	6.2	13.4	10.2	8.7	0.17	Montmorillonite
	25.0^{h}	51.0	25.5	25.5	8.4	0.1	5.7	0.1	Mica-kaolinite

[g] Wisconsin-age eolian sand, fine-grained, oxidized, leached; sampling location, Benton Co., Iowa; soil series, Carrington.

[h] Extrapolated data.

[i] Wisconsin-age loess, oxidized, calcareous friable; sampling location, Harrison Co., Iowa; soil series, Hamburg.

[j] Residual soil over medium-grained biotite granite; sampling location, Durham Co., N.C.; soil series, Durham.

[k] Below A horizon.

TABLE 2

PROPERTIES OF PORTLAND CEMENT[a]

Properties	Value
Chemical (%):	
SiO_2	22.14
Al_2O_3	4.90
Fe_2O_3	2.80
MgO	2.68
SO_3	2.04
Loss on ignition	0.64
Insoluble residue	0.14
$3\,CaO \cdot SiO_2$	49.2
$2\,CaO \cdot SiO_2$	26.5
$3\,CaO \cdot Al_2O_3$	8.3
CaO	63.92
Free lime (CaO)	0.15
$4\,CaO \cdot Al_2O_3 \cdot Fe_2O_3$	8.5
Physical:	
Fineness, specific surface (sq cm/g)	
Wagner (turbidimeter)	1,896
Blaine (air permeability)	3,468
Setting time	
Gillmore needles	
Initial	3 hr 20 min
Final	5 hr 25 min
Vicat needle	
Initial	2 hr 40 min
Soundness	
Autoclave expansion (%)	0.060
Compressive strength, 1:2.75 G.O.S. (psi)	
1-day	883
3-day	1,883
7-day	3,221
28 day	5,304
Air entrained in standard mortar (%)	7.7

[a] Penn-Dixie Cement Corporation, West Des Moines, Iowa.

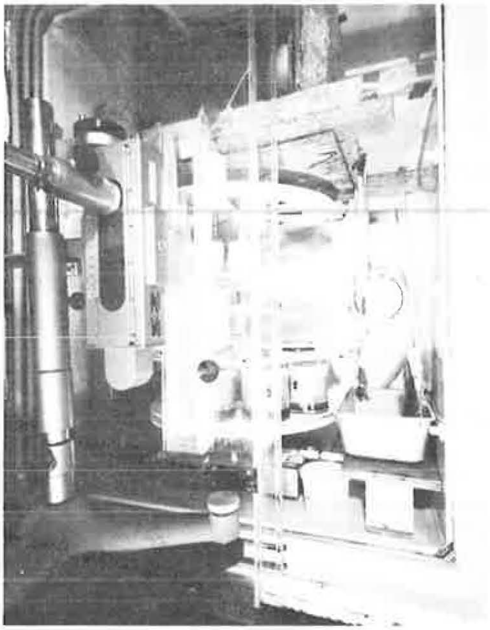

Figure 1. Microscope-slide-cathetometer in place (left) to measure length changes of 2- × 2-in. diameter cylindrical specimens inside constant temperature cabinet (right).

thermal length changes in the gage support. Instability was also found from placing specimens on Plexiglas plates. A micrometer was tried (8) but was inconvenient for measuring 2-in. specimens. The problems from elastic strain suggested an optical measurement method, and a microscope-slide-cathetometer was used. The microscope is focused on an index point at one end of the sample, then it is moved to an index at the other end by a screw arrangement, and the distance was measured to the nearest 0.001 mm (Fig. 1). Later a linear variable differential transformer (LVDT) method was tried with some success.

Curing

As indicated in Table 5, several curing programs were selected:

1. Immediate air drying at 70.5 F and 50 percent RH;
2. Moist curing for 7 days (100 percent RH), then air drying (50 percent RH);
3. Moist curing for 60 days, then air drying; and
4. Moist curing for 3 days, continuous immersion in distilled water for 60 days, then air drying.

Curing chambers were constructed to allow length measurements to be made without disturbing the specimens.

Moist-curing chambers were seal-top polyethylene cups, 5 in. in diameter by 4 in. high, large enough for three specimens. Each specimen rested on a Plexiglas plastic platform with a hole in it for the index pin (Fig. 2a and b). Optical glass windows (made from microscope cover glasses) were cemented in the outside wall of the cups to allow the pins to be viewed for measurements. Nearly 100 percent RH was maintained by having free water in the bottom of the cups.

Specimens for immediate air drying were protected under polyethylene covers during transfer from the molding room to a controlled atmosphere for drying.

TABLE 3

TOTAL SHRINKAGE DURING MOIST CURING[a]

Soil	Cement (%)[b]	Molding M. C. (%)[c]	Linear Shrinkage (%)	
			7 days	60 days
Ottawa sand	8	11.1	0.000(0.006)	0.00
	10	10.4	0.018(0.000)	0.018
	12	10.6	0.010(0.010)	0.018
	10	7.4	0.000	--
	10	11.4	0.000	--
Fine sand	8	10.7	0.000(0.000)	0.000
	10	10.4	0.000(0.000)	0.000
	12	10.3	0.000(0.000)	0.000
	10	7.4	0.00	--
	10	11.4	0.00	--
Silt	10	19.9	0.016(0.016)	0.020
	12	19.5	0.048(0.024)	0.050
	14	19.3	0.024(0.020)	0.024
	12	16.5	0.008	--
	12	22.5	0.060	--
Clay	10	17.3	0.120(0.120)	0.120
	12	17.0	0.122(0.122)	0.120
	14	17.3	0.140(0.140)	0.140
	12	14.0	0.050	--
	12	20.0	0.180	--

[a] Data taken from curing procedures 3 and 2 (latter data underlined).

[b] By weight of ovendry soil.

[c] By weight of ovendry soil + cement.

TABLE 4

TOTAL SHRINKAGE WITH INITIAL MOISTURE CONTENT VARIABLE[a]

Soil	Cement (%)	Mix Water (%)	Shrinkage (%)		
			M. C.	Air Dry	Total
Ottawa sand	10	7.4	0.000	0.022	0.022
		10.4[b]	0.000	0.028	0.028
		11.4	0.000	0.020	0.020
Fine sand	10	7.4	0.000	0.048	0.048
		10.4[b]	0.000	0.064	0.064
		11.4	0.000	0.050	0.050
Silt	12	16.5	0.000	0.636	0.636
		19.5[b]	0.024	0.670	0.694
		22.5	0.064	0.642	0.706
Clay	12	14.0	0.050	0.454	0.504
		17.0[b]	0.122	0.526	0.648
		20.0	0.180	0.820	1.000

[a] Seven-day moist curing, then air drying.

[b] Optimum moisture content for standard Proctor compaction.

TABLE 5

TOTAL SHRINKAGES IN PERCENT WITH VARIABLE CEMENT CONTENTS AND METHODS CURING

Soil	Cement (%)	Envir. 1	Envir. 2			Envir. 3			Envir. 4			
		Air Dry (50% RH)	M.C. 7 Days	Air Dry	Total	M. C. 60 Days	Air Dry	Total	Moist Cure 3 Days	Immerse 60 Days[a]	Air Dry	Total[a]
Ottawa sand	8	0.030	0.006	0.008	0.014	0.000	0.020	0.020	0.000	-0.050	0.040	-0.010
	10	0.046	0.000	0.028	0.028	0.018	0.040	0.058	0.000	-0.040	0.028	-0.012
	12	0.032	0.010	0.026	0.036	0.018	0.038	0.056	0.000	-0.030	0.044	+0.014
Fine sand	8	0.078	0.000	0.050	0.050	0.000	0.080	0.080	0.000	-0.040	0.088	0.048
	10	0.080	0.000	0.064	0.064	0.000	0.080	0.080	0.000	-0.038	0.080	0.042
	12	0.082	0.000	0.068	0.068	0.000	0.054	0.054	0.000	-0.040	0.080	0.040
Silt	10	0.600	0.016	0.652	0.668	0.020	0.790	0.810	0.000	-0.030	0.800	0.770
	12	0.720	0.024	0.670	0.694	0.050	0.860	0.910	0.032	0.000	0.980	1.012
	14	0.610	0.025	0.732	0.756	0.024	0.840	0.864	0.018	-0.068	0.840	0.798
Clay	10	0.688	0.120	0.544	0.664	0.120	0.660	0.780	0.140	-0.070	0.656	0.726
	12	0.656	0.122	0.526	0.648	0.120	0.580	0.700	0.140	-0.080	0.640	0.700
	14	0.652	0.140	0.600	0.740	0.140	0.610	0.750	0.140	-0.050	0.670	0.760

[a] Data erratic.

Specimens for continuous immersion were placed singly in 7-oz polyethylene cups, each with an optical glass window cemented in the side near the bottom. The top pin was allowed to project through a hole in the lid. Another hole in the lid allowed occasional replenishing of the distilled water. Because air bubbles interfered with viewing the lower pin and a carbonate crust sometimes obscured the tip of the upper pin, a glass cup with open top and a reference baseplate was later substituted (Fig. 2c). This proved unsatisfactory for long-term measurements because of excessive carbonation.

A special curing cabinet was built from a 6-cu ft household refrigerator. Constant temperature and near 50 percent RH were maintained with a cooler-heater arrangement and continuous recirculation of air within the cabinet. The door was replaced with a Plexiglas door having a dead air space and a 1.5- × 17.5-in. vertically arranged optical glass window. Inside the cabinet at different levels were three 14-in. diameter revolving steel shelves mounted on a vertical shaft which could be turned by a worm gear. Each shelf could hold 16 air-drying or continuous-immersion specimens. Moist-cured specimens were placed on the middle shelf, which had revolving subtables for each three-specimen container so that each container could be rotated to bring the specimens successively into view.

An initial checkout of cabinet temperatures was made with thermocouples feeding a 16-channel recording potentiometer, and temperature was found to be constant with time in all positions to ±0.6 F. Most of this temperature fluctuation was cyclic due to on-off cycles of the refrigerator, and the average temperature in all positions varied from 70.3 to 70.7 F. Checks of the humidity showed an inverse relationship to temperature, the average relative humidity being held in the neighborhood of 50 percent by the heater-cooler closed air circulation system.

During measurements, observation windows in the cups were quite often found to be fogged over, so a small heater was constructed to blow a jet of warm air on a window before measurement.

In general, after each change in environment, measurements were made on a geometric time-increment basis. That is, measurements were usually made after 0, 20, 40, 60, 90, 120, 150 and 180 minutes; 4, 5, 6, 8, 10, 13, 16, 20, 24, 32, 40, 48, and 60 hours; and 3, 4, 5, 6, 7, 9, 11, 14, 17, 21, 28, 35, 42, 56 and 90 days. Moving a specimen to a new environment, as from moist cure to air dry, initiated a new series of measurements starting from time zero.

RESULTS

Shrinkage and Time

Shrinkage-vs-time graphs are shown in Figures 3 to 5. The nominal specimen length of 2.00 in. was used to calculate percent shrinkage. Each point is the average from three specimens molded from a single batch.

Start of Shrinkage.—As shown in Figure 3, the clay soil-cement specimens with no moist curing began to shrink almost immediately on air drying. In other specimens, and in the clay soil-cement after moist or immersion curing, shrinkage was delayed for

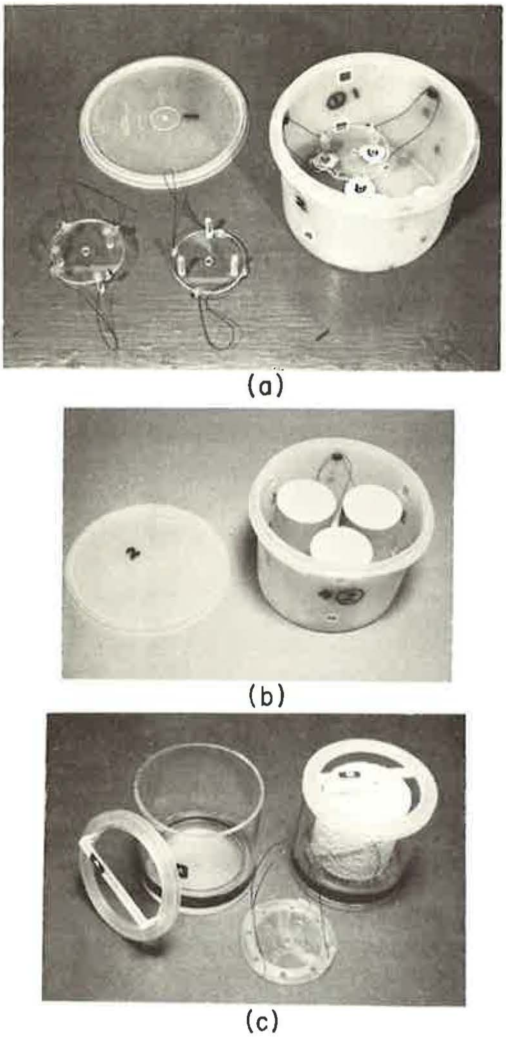

Figure 2. Test equipment: (a) container for moist curing three specimens at 10 percent RH; (b) specimens in place in the moist-cure container (note index pins in tops of specimens, and glass windows for length measurements); and (c) containers for measurements during continuous immersion curing.

periods ranging from several hours to one day, giving an initial flat portion to the shrinkage-log time curves (Fig. 4). Presumably the time delay was necessary for shrinkage tensions to build up and overcome internal restraints in the hydrated cement gel (5). These restraints were apparently absent only in the uncured, high clay content specimens, indicating that the clay with little or no such internal restraint must have participated in the shrinkage.

Rate of Shrinkage. —Before proceeding with the data, we may theorize that in a simplified system, shrinkage should be proportional to amount of evaporation, and drying rate should be uniform. Shrinkage then will be a direct function of time; this can be illustrated by the steady evaporation and shrinkage of a glass of water exposed at constant conditions. It was not found to be the case for the soil-cement.

First, the rate of evaporation probably is not constant, because the binding energy of the water increases with decreasing moisture content, affected by such factors as decreasing size of remaining filled capillaries, increasing concentration of salts in solution, and finally a relatively high-energy function due to decreasing number of water layers adsorbed. This would tend to put evaporation as a function of some root of time.

Second, shrinkage is not proportional to evaporation, because increased bonding energy also means increased internal tensions as water is removed. As has been shown for concrete, less evaporation of held water is required to cause the same amount of shrinkage. This would tend to put shrinkage in proportion to some power function of time.

The two departures from the ideal glass-of-water concept may be expressed by

$$S = ct^{\beta} \qquad (2)$$

where S is shrinkage, t is drying time, and c and β are constants. If β is less than one, reduced evaporation rate is the dominant factor; if β is more than one, increased internal tension and high shrinkage per unit loss of water dominate.

The silt and the clay mixes showed shrinkages large enough to test the equation. Plots are shown in Figures 4 and 5, and there is a considerable straight-line portion in most graphs. The straight-line portions intersect low or zero-shrinkage portions at later ages, presumably as drying rate comes under a new control. Only the clay mixes with no moist curing failed to show the early log time function, perhaps a result of curing changes brought about with drying.

The slope β in the graphs appears to be related to beginning time of shrinkage, which as previously discussed is later for better cured samples. In all except one (0 day m.c.)

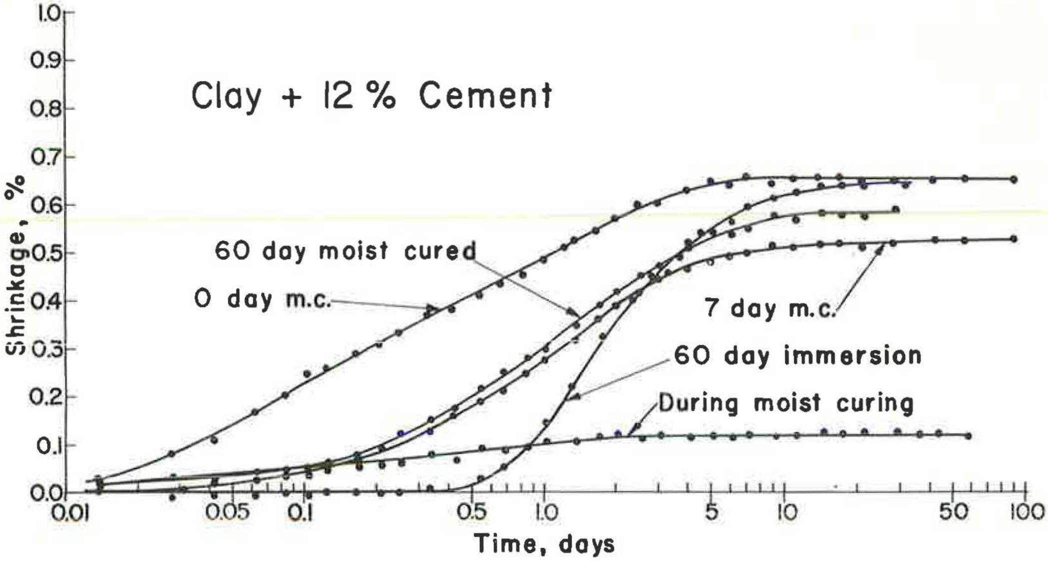

Figure 3. Shrinkage data for typical mix under different curing conditions.

silt mix and in the 60-day-immersed clay mixes, β exceeded 1.0; in others, β was less than 1.0. An inverse relation found between β and C gives

$$\text{Clay:}\quad S = ct^{(2.5 - 2.6c)} \tag{3}$$

and

$$\text{Silt:}\quad S = ct^{(2.0 - 5.6c)} \tag{4}$$

where c is the percent shrinkage after one day. Therefore, low early shrinkage means higher β and a faster rate of shrinkage once it starts. This should be indicative of stronger gel structure, which restricts gel reorganization at early ages until a certain level of tension is reached, then becomes volumetrically very sensitive to water loss because of the high tensions in the well-bonded gel.

Evaluation of c and β in Eq. 2 is readily done by measuring shrinkage of 2- × 2-in. samples after 7.6 hours and after one day, representing, respectively, $\log t = -0.5$ and $\log t = 0$. Application of the shrinkage rate equations to field situations would require consideration of the much lower rate of moisture loss with the lower surface/volume ratio, and particularly under a bituminous cover.

Shrinkage during Moist Curing. —All samples shrank slightly during moist curing; clay shrank the most. The shrinkage delay time was about the same as during straight drying, suggesting that moist-cure shrinkage might be due to partial drying. However, calculation of evaporation necessary to saturate air in the containers (0.003 g H_2O) and account for water absorption by the polyethylene cups (0.024 g) and Plexiglas sample supports (0.15 g) accounts for a maximum linear shrinkage of about 0.02 percent. The amounts measured are shown in Table 3. Repeated runs showed a large variation only in one silt mix, suggesting a container leak. The kaolinitic clay, therefore, appears to undergo a considerable shrinkage without loss of water. As shown in Table 3, this shrinkage was reduced from 0.13 to 0.05 percent by decreasing the mix water, and was increased to 0.18 percent by increasing mix water, perhaps by increasing the mix plasticity and ease of deformation. Possibly cement hydration was robbing clay of water; this could be checked by measuring specimens with zero cement.

Cessation of Shrinkage. —Shrinkage of the 2-in. specimens departs from the log time plot after about 1 to 3 days drying, but appears to continue at a greatly reduced rate. Slight shrinkages may be seen in Figures 4 and 5 at ages up to 100 days, again following a log time relationship.

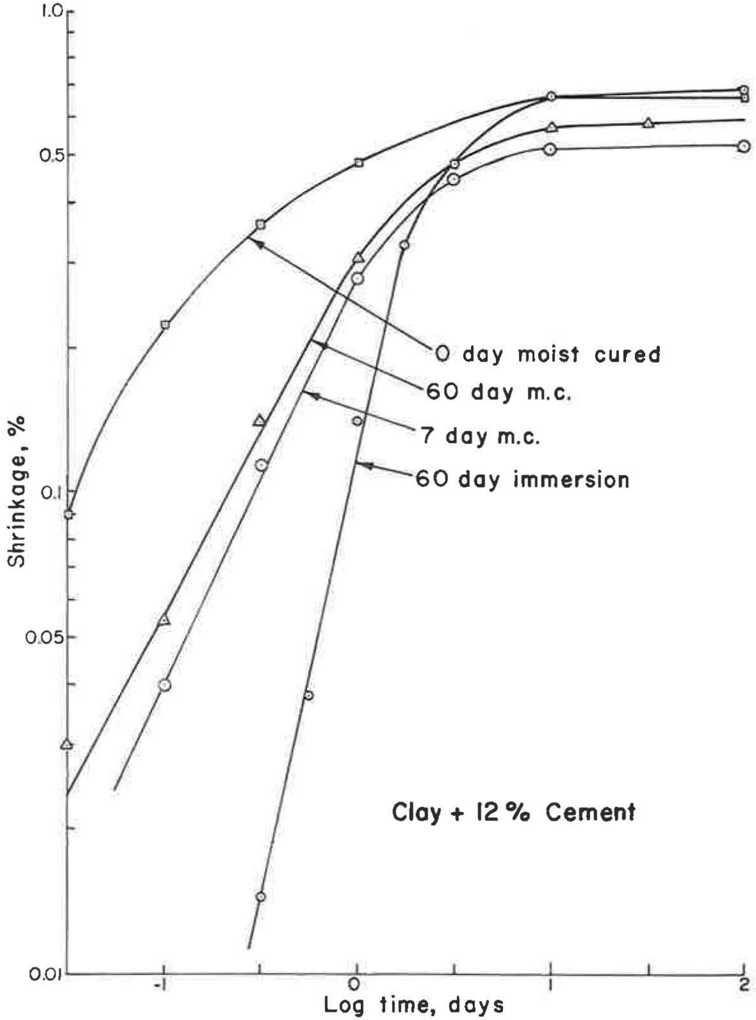

Figure 4. Shrinkage curves for a kaolinitic clay mix; points read from smoothed curves (Fig. 3).

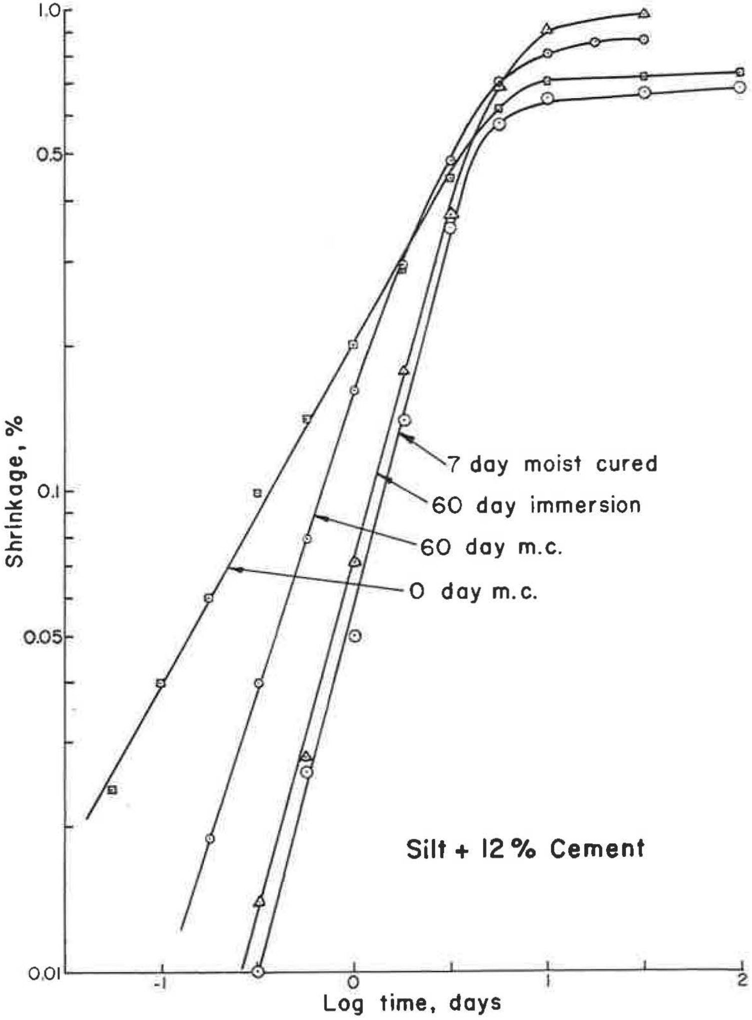

Figure 5. Shrinkage curves for montmorillonitic silt mix.

TABLE 6

AVERAGE SHRINKAGES[a]

Method	Ottawa Sand	Fine Sand	Silt	Clay
No curing	0.04	0.08	0.64	0.66
After 7-days m. c.	0.03	0.06	0.71	0.68
After 60-days m. c.	0.05	0.07	0.86	0.74
After 60-days immersion	0.00	0.08	0.86	0.73

[a] From Table 5.

Total Shrinkage.—Total shrinkages are shown in Table 5. The data are less consistent than in the log time plots, probably because more factors are involved in stopping shrinkage than in establishing its rate during drying.

Shrinkage vs Cement Content.—No consistent or significant trends are shown by shrinkage totals vs percent cement (Table 5). Therefore, the shrinkage percentages for the same soil with different cement contents were averaged to strengthen the data for later comparisons (Table 6). In road bases, cement content in part determines tensile strength at time of shrinkage and, therefore, should influence crack spacing; higher cement contents should give larger cracks farther apart but with about the same total shrinkage per unit length. This trend has been observed in field studies (11).

Shrinkage vs Curing.—Average silt soil-cement shrinkage increased about one-tenth (from 0.064 to 0.71 percent) if shrinkage took place after 7 days of moist curing instead of immediately after molding (Table 6). After 60 days of moist or immersion curing, shrinkage further increased about two-tenths. A similar trend is seen for the clay, and the trend is present but much less pronounced for the sands. Apparently with longer curing before drying, there is more shrinking of the gel and less internal restraint from unhydrated cement particles.

Swelling.—Measurements during continuous immersion were hindered by air bubbles on the container windows, and immersion swelling data in Table 5 are only approximate. Two repeated tests with the newer type of container showed swelling of 0.046 percent for Ottawa sand + 8 percent cement, and 0.080 percent for silt + 10 percent cement. Swelling is expected from hydration of the cement. It is interesting that the silt montmorillonite, with its large potential for swelling, apparently came into equilibrium with water and free lime mixing and exhibited no appreciable further swelling. Swelling during immersion is only partly reversed by later shrinkage.

Shrinkage vs Clay Content.—Shrinkage of the 7-day moist-cured fine sand samples, with about 3 percent montmorillonite, is approximately twice the shrinkage of the similar specimens made with Ottawa pure silica sand. Shrinkage of the silt specimens, with about 16 percent montmorillonite, is over 10 times that of the Ottawa sand. But shrinkage of the clay with 30 percent kaolinite is less than that of the silt. The only correlation to soil property data listed in Table 1 appears to be to plastic limit, which cannot be tested on sands. Total shrinkage in percent about equals P. L. × 0.03.

Shrinkage vs Initial Moisture Content.—Table 4 shows shrinkages of intermediate cement-content specimens molded above and below the optimum moisture content. No consistent trend is shown for the sands, but shrinkage of the silt and clay does relate to the amount of initial mix water; i.e., the more water, the more shrinkage. A 6 percent weight difference in initial water gives only 0.5 percent difference in linear shrinkage for the clay and about 0.07 percent for the silt. These correspond to changes in volumetric shrinkage of about 1.5 and 0.21 percent, respectively, indicating that much of the excess mix water is probably capillary water which may be lost without shrinkage. Control of mix water is, therefore, relatively ineffective for reduction of shrinkage, even for clay, where compacting 3 percent below optimum moisture content reduced total shrinkage only from 0.65 to 0.50 percent.

Cracks.—Shrinkage cracks were observed only in the silt mixes, typically in a rectangular pattern with hexagonal elements near the middle of the specimens. Cracking usually started about 2½ to 8 hours after drying began. The cracks reached a maximum width of 0.04 to 0.12 mm after 1 to 2 days, and usually healed on the third to ninth day. More cracking was observed after longer initial curing and with higher contents of cement. Cracking did not cause any abrupt change in the shrinkage-time curves and probably relates to moisture gradient in the drying samples.

DISCUSSION

Comparison with Portland Cement Mortar

If the two sand mixes are considered to be very lean concrete, 10 percent cement and 10.4 percent water (both by weight of the sand) nominally correspond to 1.8 sk/cu yd and a w/c ratio of 1.04, assuming zero air. Comparison with representative concrete data (4, Fig. 30) shows that the Ottawa sand shrinkage conforms to expectations. The cement paste shrinkage should be relatively large, probably on the order of 0.4 percent at this high w/c ratio, but, by way of counterbalance, there is relatively little paste to shrink. The latter effect from Eq. 1 gives a predicted shrinkage of 0.05 percent for both sand mixes, which is about correct for the Ottawa sand.

In the silt and clay mixes, w/c ratios are 1.6 and 1.4, respectively. If the clay is considered part of the shrinking phase, w/c + cl equals 0.78 and 0.45, respectively, but S from Eq. 1 comes out only about 0.1 percent for the silt and clay soils if S_0 (gel shrinkage) and α stay the same. It is very likely that S_0 is larger for cement-clay pastes than for pure cement pastes. By substituting measured shrinkages in Eq. 1, and assuming the exponent to be 1.7, shrinkage S_0 for the cement + clay gel phase of the silt soil is 2.3 and for the clay soil is 1.9. Assuming a linear relationship to clay content, with 0.4 as the contribution of the cement gel,

$$S = S_0 (1 - g)^{1.7} = S_0 (c + w + cl)^{1.7} \tag{5}$$

where

$$S_0 = 0.4 + 0.12 \text{ Pm} \tag{6}$$

or

$$S_0 = 0.4 + 0.05 \text{ Pk} \tag{7}$$

where c, w, and cl are the volume fractions of cement, water, and clay; Pm and Pk are the percentages of -2 μ montmorillonitic or kaolinitic clay, respectively. Predicted shrinkage of the fine sand mix for Eqs. 5 and 6 is 0.1 percent which approximates data in Table 6. An example of the calculation is shown in the Appendix.

Extrapolation of Eqs. 6 and 7 to 100 percent clay gives $S = S_0 = 12$ percent for montmorillonite and $S = 5$ percent for kaolinite, which represents the clays in the presence of excess free lime. No data have yet been obtained for pure lime-treated clays, but linear drying shrinkage ranges of 3 to 10 percent for kaolinite and 12 to 23 percent for montmorillonite have been reported (10, Tables 3-6). The range reported therein for illite is about the same as that for kaolinite.

In the foregoing calculations, initial air content was assumed to be zero. As can be seen from Table 7, this is not true. The effect of air content should depend on its relation to the system. For example, concrete shrinkage increases appreciably with percent entrained air (14); however air entrained in concrete mainly occurs as tiny bubbles in the paste, probably reducing internal shrinkage restraints and S_0. In soil-cement, air is mainly in capillary-sized voids, surrounded by a skeletal structure of air and cement paste. This would simulate air entering into concrete as the first capillary water is lost during drying and should not influence shrinkage. This was verified for compacted cylinders of calcium and sodium montmorillonite, where percent voids had no effect on percent expansion (12).

Effect of Density

Density or degree of compaction was not investigated as an independent variable. Calculations from the densities obtained with different soils show that the influence of air content must be small compared with that of clay and initial water contents (Table 7).

The effect of higher compaction, such as to modified Proctor density, should be investigated. However, since total amount of shrinkage appears to be mainly related to constitution of the gel, the effect may be indirect, by decreasing the initial moisture content.

TABLE 7

COMPACTED DENSITIES OF SELECTED TEST MIXTURES

Soil	Cement Content (%)	Water Content (%)	Dry Density (pcf)	Volume Percent Air[a]	Shrinkage (%)
Ottawa sand	10	10.4	114	15.0	0.028
Fine sand	10	10.4	112	15.7	0.064
Silt	10	19.9	99	10.3	0.668
	12	19.5	99	11.2	0.694
	14	19.3	99	11.7	0.756
	12	16.5	97	18.5	0.636
	12	22.5	99	6.3	0.706
Clay	10	17.3	99	14.5	0.664
	12	17.0	101	14.3	0.648
	14	17.3	98	17.3	0.740

[a] S. G. of cement = 3.15; S. G. of soil = 2.70.

Shrinkage Control

The data suggest that shrinkage is unavoidable and the main control that can be exercised is timing. Prolonged moist curing or prolonged wetting before drying increase rather than decrease total shrinkage. Compaction below optimum moisture content has some beneficial effect, but not much; however, except with sands, moisture in excess of optimum is damaging and should be avoided.

About the same total shrinkage occurs on drying, regardless of when drying takes place. From the standpoint of riding qualities, many fine cracks may be preferable to a few large ones, but the spacing is a function of tensile strength at the time of drying; therefore, a sacrifice in strength would be required to obtain a better crack distribution. An alternative is to prevent drying until the soil-cement has sufficient tensile strength to crack at intervals scribed as joints. But with linear shrinkages as high as 0.5 to 1 percent, the joints would be rather wide.

A third alternative is to try to reduce shrinkage of the clay. A question needing immediate answer is whether the high shrinkage contribution of montmorillonite relates to its fine particle size or to a slight change in interlayer water. The latter should be more susceptible to control by trace additives or pretreatment. However, the relatively high shrinkage of the kaolinitic soil appears partly due to matrix reorganization taking place during moist curing (Table 4) and probably during drying; this might be more difficult to control.

In summary, minimization of shrinkage requires reduction of the initial water content and, more important, the amount of clay.

CONCLUSIONS

1. Drying shrinkage usually proceeds as an exponential function of time (Eqs. 2, 3 and 4).
2. Longer curing of soil-cement causes a late start in shrinkage, but a faster rate after shrinkage has started.
3. Longer curing generally increases the total shrinkage.
4. The (kaolinitic) clay soil-cement shrinks appreciably during moist curing; the wetter mixes shrink most.
5. Within the cement content ranges investigated (8 to 12 and 10 to 14 percent), no relation was found between rate or total percent of shrinkage and percent cement.
6. Total shrinkage is very sensitive to the amount and kind of clay (Eqs. 5, 6 and 7), montmorillonite contributing most.

7. Compaction at above or below optimum moisture content has no appreciable effect on shrinkage of the sands and only a small effect on shrinkage of the silt and clay.

8. Immersion caused only slightly more swelling of samples high in clay than of those with little or no clay.

ACKNOWLEDGMENTS

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Appendix

EXAMPLE OF SHRINKAGE PREDICTION CALCULATION

Fraction	Wt (per 100 g soil)	S. G.	Rel. Vol	Vol (%)
Soil:				
Sand + cement	97.2	2.7	36.0	69.7
Clay (mont.)	2.8	2.7	1.0	
Cement	10	3.15	3.2	
Water	10.4 × 1.10	1.0	11.4	
			51.6	

$$\begin{aligned} S_o &= 0.4 + 0.12\ Pm && (6) \\ &= 0.4 + 0.12\ (2.8) = 0.736 \\ S &= S_o\ (1\text{-}g)^{1.7} && (5) \\ &= 0.736\ (1\text{-}0.697)^{1.7} \\ &= \underline{0.096\%} \end{aligned}$$

Effects of Repeated Loads on Elastic Micaceous Soils Stabilized with Portland Cement

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Respectively, Engineer, Wiley and Wilson, Consulting Engineers; and Associate Professor of Civil Engineering, University of Virginia

The results of a laboratory study of the effects of repeated load triaxial tests on the stress-deformation characteristics and resilient properties of two elastic micaceous soils stabilized with portland cement are discussed. The ultimate strength and stress-deformation curves for both untreated and cement-treated soils are reported for both conventional and repeated triaxial loadings. The addition of proper amounts of portland cement to these soils greatly increases the strength and stiffness and considerably reduces the resilience. However, the strength and stiffness of these cement-treated soils under the action of repeated loads are not always properly represented by the results of a conventional (gradually applied load) triaxial test. Both the ultimate strength and stiffness of cement-treated soils are less when obtained in a repeated load test. The elastic rebound or resiliency for both untreated and cement-treated soils appears to be directly and perhaps uniquely related to the ratio of the applied repeated deviator stress to the compressive strength as determined in a conventional triaxial test.

•UNTIL A decade or so ago, highway engineers dealing with soil problems involving repeated or dynamic loadings were limited by lack of available information to use of static and gradually applied load parameters. Recent investigations have provided some basic repeated load information and have indicated, for example, that a soil may exhibit different strength-deformation properties when subjected to a series of repeated loads than it would when subjected to statically or gradually applied loads. Most of these studies have been concerned with compacted but untreated soils. Very little work has been reported on the effects of repeated loads on the strength and deformation characteristics of chemically stabilized soils.

Recently, Tate (1) stabilized and compacted a number of samples of a micaceous silt with 5 and 10 percent portland cement and allowed them to cure for 6 days. After testing identical samples under both repeated and gradually applied loads, he found that, except at low levels of deviator stress (approximately 50 to 60 percent or less of the ultimate gradually applied load), the samples would fail suddenly in shear after relatively few repetitions of load. As reported by Ahmed and Larew (2), unstabilized samples of the same type of soil generally did not fail until subjected to repeated loads of magnitudes equal to and greater than 95 percent of the ultimate gradually applied load, even though many thousands of these load repetitions were applied.

Tate also found that the magnitude of the elastic or resilient rebound for a given ratio of repeated load to ultimate gradually applied load was approximately the same for both stabilized and unstabilized soils.

Colley and Nowlen (3) reported studies in which they subjected granular soil-cement subbases to repeated loadings. The soil-cement subbases, 6 in. thick, were placed at AASHO standard density and optimum moisture directly below a rigid concrete slab.

Paper sponsored by Committee on Soil-Portland Cement Stabilization and presented at the 43rd Annual Meeting.

A load of 4,000 lb was transmitted to an 8-in. diameter steel plate resting on a rubber pad astride the joint in a concrete slab. Addition of cement to four granular subbase materials reduced the densification of these materials to an insignificant amount and eliminated pumping from two materials that had pumped before the addition of cement. Moreover, the use of soil-cement greatly reduced the pressure transmitted to the subgrade. These studies by Tate and the Portland Cement Association were the only known published works which dealt with laboratory studies of repeated loadings on stabilized soils.

Studies conducted by the California highway department (4) showed that highway pavements may fail by fatigue as a result of repeated reversals of stress. The fatigue failure is attributed in part to the resilient or elastic deformation of the subgrade underlying the pavement. Since much of the Piedmont Province of Virginia is covered with micaceous silty soils which are highly elastic or resilient and the Virginia Department of Highways has encountered difficulty in the use and performance of these materials, this study was undertaken to determine whether the treatment and stabilization of these soils with portland cement would improve their strength-deformation and resilient properties.

TABLE 1
BASIC SOIL INFORMATION

Soil Property	Glenelg C[a]	Culpeper C[b]
Fraction (% by wt.):		
Clay	7.5	4.0
Silt	53.5	22.0
Sand	39.0	74.0
Spec. gr. of solids	2.74	2.74
Shrinkage limit	24.5	28.50
Plastic limit	27.0	31.5
Liquid limit	35.0	32.0
0 pt. moisture content (%)[c]	21.5	18.5
0 pt. dry density (pcf)	98.5	99.2

[a] AASHO classification A-4.
[b] AASHO classification A-2-4.
[c] AASHO Designation: T99.

The two soils chosen for this study exhibited elastic properties and were obtained from the Piedmont Province of Virginia. The pedological classifications were Culpeper C and Glenelg C, and the AASHO classifications were A-2-4 and A-4, respectively. Index properties for these soils are listed in Table 1 and gradation curves are shown in Figure 1. Both Type I (normal) and Type III (high early strength) cements were employed in the study.

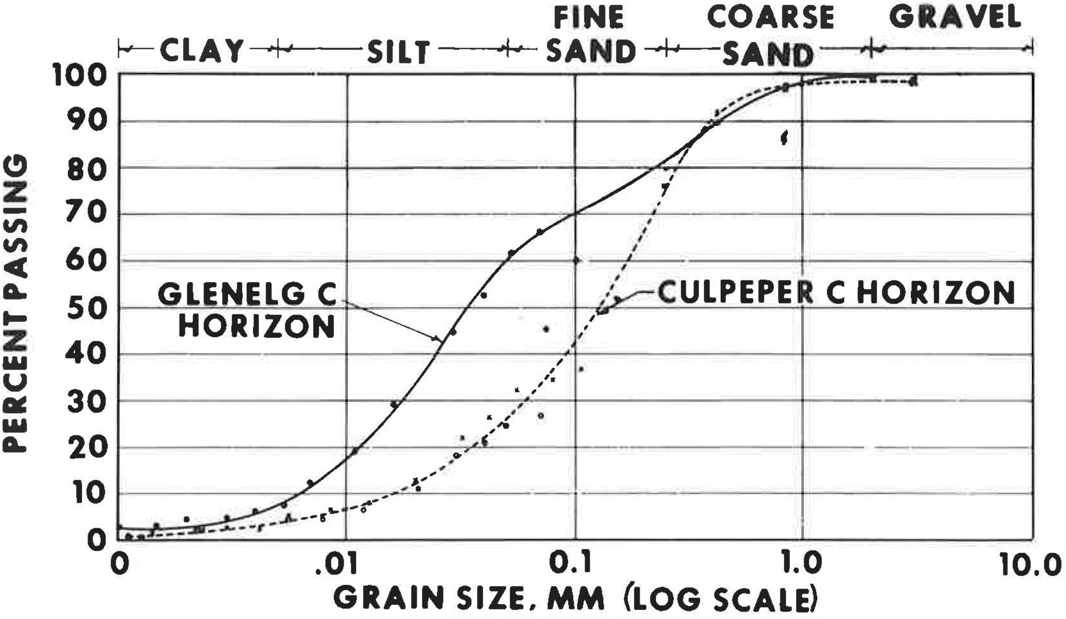

Figure 1. Gradation curves for Culpeper C and Glenelg C.

The purpose of this study was to determine the strength, deformation and resilient properties of these two stabilized soils under the action of repeated loads and to compare these with similar properties of the untreated soils.

SCOPE AND PROCEDURE

Studies were made to determine the required percentage of portland cement to be added to each soil so that Portland Cement Association requirements for soil-cement would be satisfied. For the Culpeper C, 10 percent of either Type I or Type III cement by ovendry weight provided the proper strength and durability for soil-cement. However, the percentage of portland cement required for the Glenelg C yielded sample strengths beyond the testing capability of the repeated load device and, therefore, a 5 percent mixture, which yielded a "cement-modified" soil, was used. All samples tested were compacted to optimum moisture and dry density in a Harvard miniature mold with a modified drop-type rammer. Each molded specimen of both cement-treated and untreated soils was protected by aluminum foil and wax, cured in a humid room at 68 F, and subsequently tested in either a conventional or repeated load triaxial device.

To reduce the curing time, Type III high early strength cement was used with both soils. A curing period of 10 days was employed for the Culpeper C soil. Figure 2 indicates that for this material there was only a minor strength gain after the 10-day curing period which minimized the effect of strength increase during the testing period. Figure 2 also indicates that the Culpeper C stabilized with Type I cement and cured for 35 days had essentially the same strength in the conventional triaxial test as the Type III cement-stabilized soil with a 10-day cure period. Figure 3 shows the similarity between conventional triaxial stress-strain curves for the Culpeper C stabilized with both types of cement, each type being cured for the previously mentioned lengths of time. Only a 5-day curing period at 68 F was required for the Glenelg C Type III cement-modified samples as shown in Figure 4. No Glenelg C Type I cement-modified samples were prepared.

Conventional and repeated load triaxial units as described by Larew and Leonards (5) were used to obtain the conventional and repeated load stress vs strain curves for both untreated and cement-treated soils. A confining pressure, σ_3, of 10 psi was used for all tests and most specimens were subjected to 100,000 load repetitions applied at the rate of 20 loads per minute. Both total deformation and elastic rebound deformation

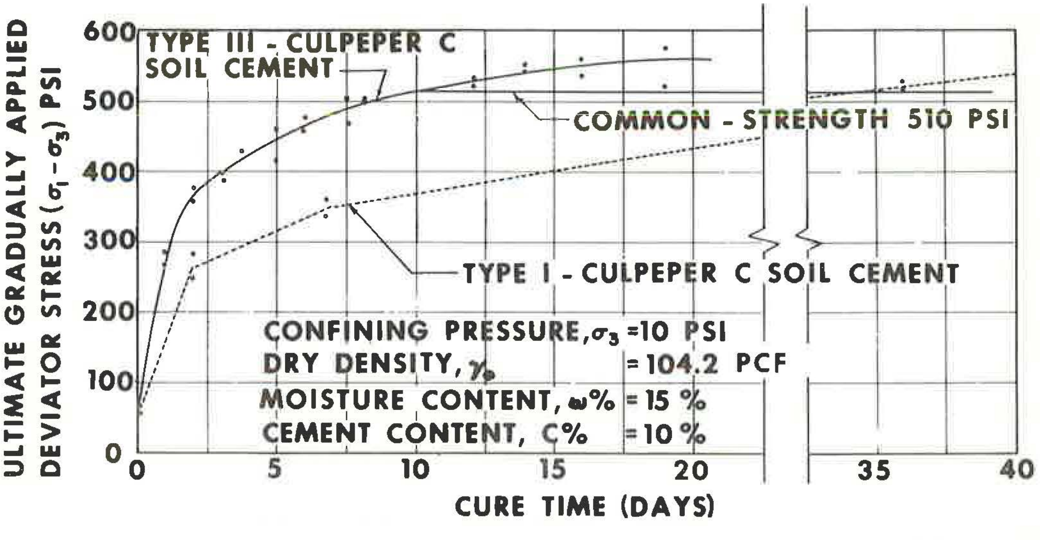

Figure 2. Ultimate gradually applied stress vs cure time for Type I and Type III cement.

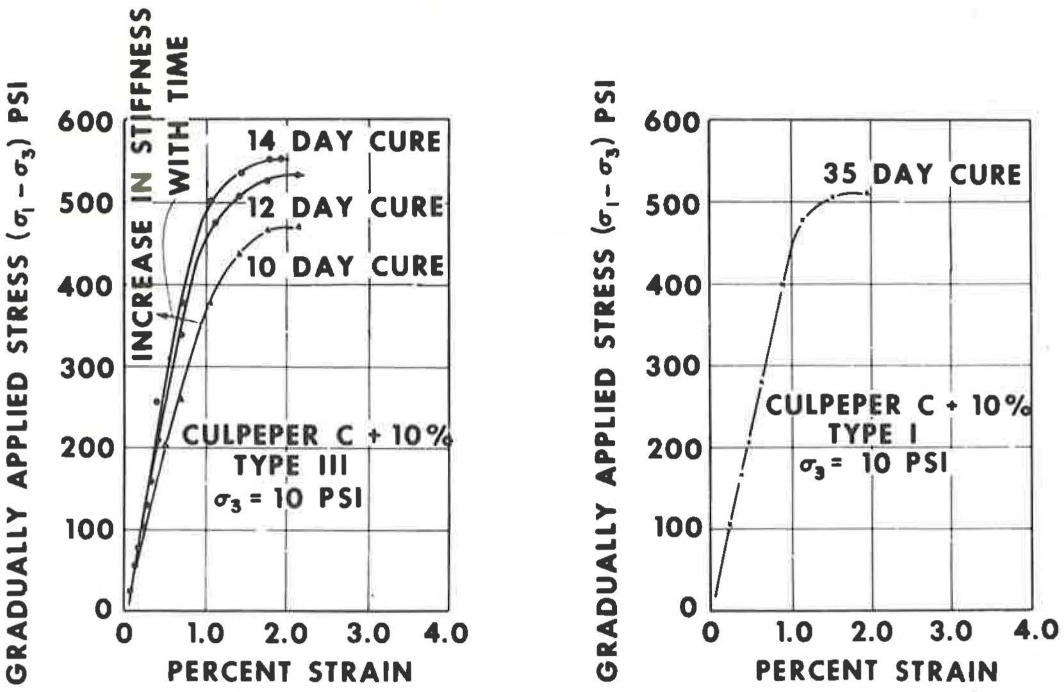

Figure 3. Comparison of conventional stress vs strain curves for Culpeper C + 10 percent Type III cement and Culpeper C + 10 percent Type I cement for various curing times.

readings were obtained for each sample tested in the repeated load device. A typical deformation vs log of the number of load repetitions curve is shown in Figure 5.

The repeated load stress vs strain curves were obtained by essentially the same method first described by Ahmed and Larew (2). As shown in Figure 5, the total

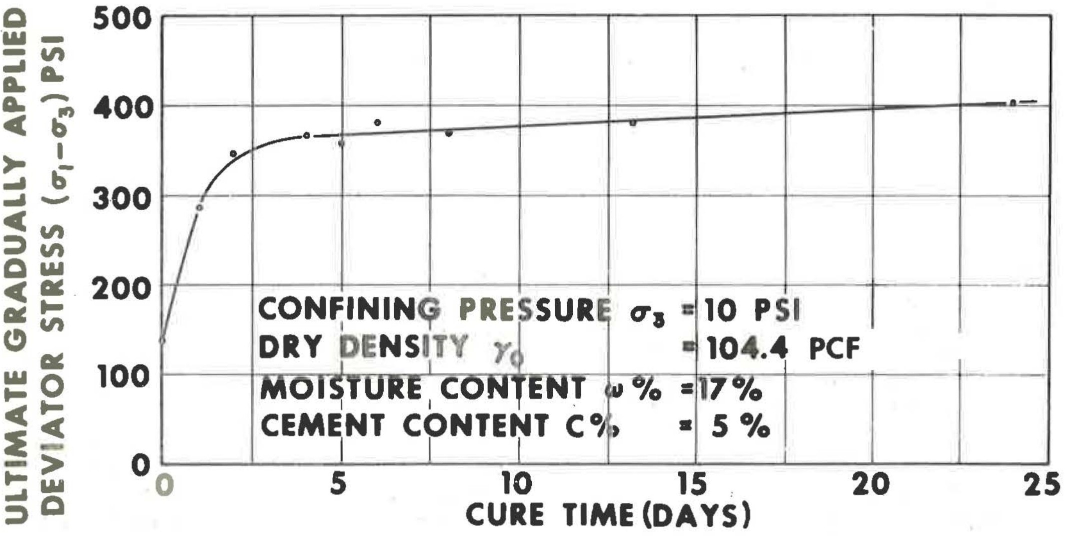

Figure 4. Strength vs curve time for Glenelg Type III cement-modified soil.

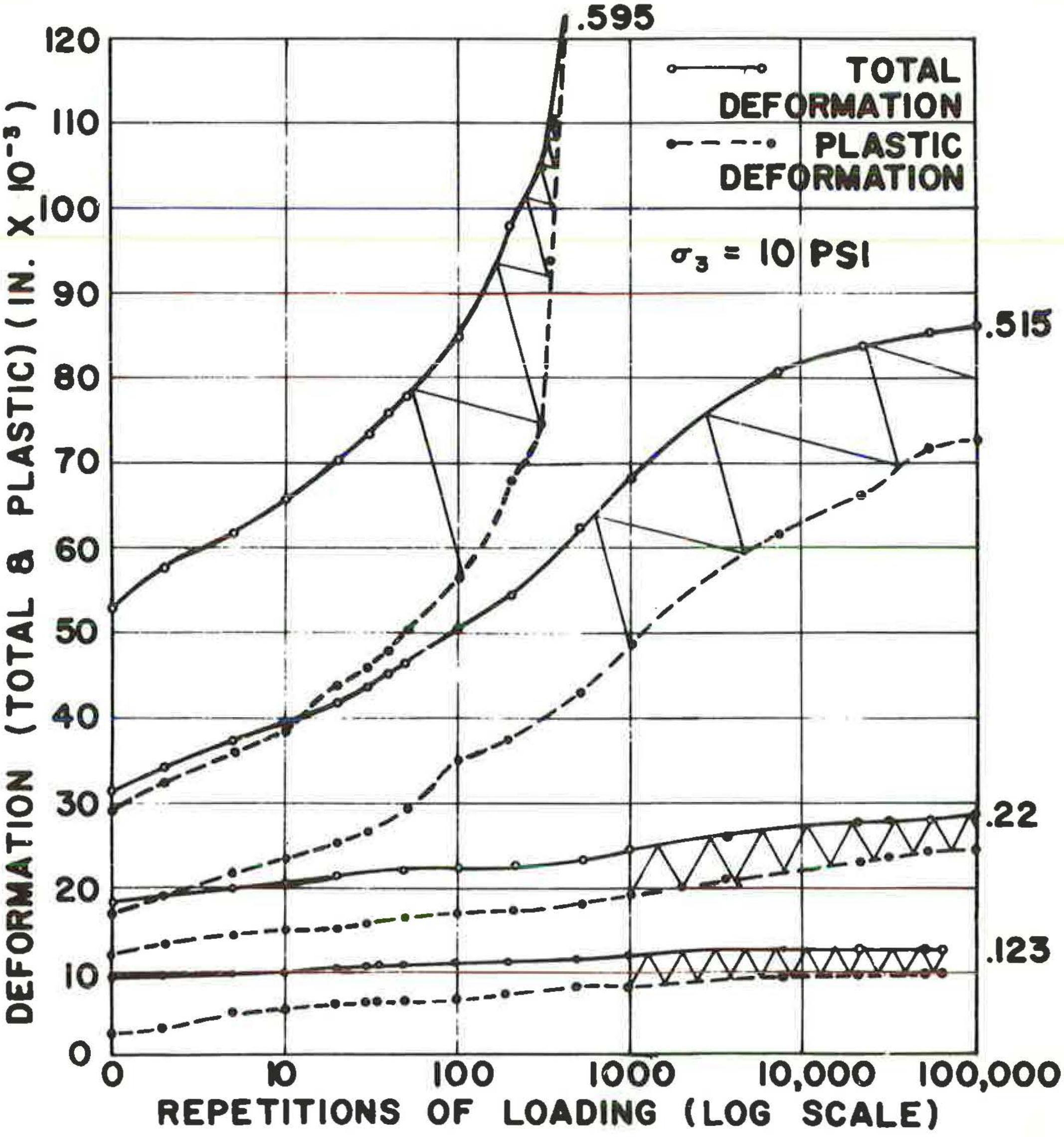

Figure 5. Repetitions of loading vs deformation for Glenelg C + 5 percent Type III cement; numbers to right are $\Delta\sigma_r/\Delta\sigma_s$ values.

sample deformation increased with each increase in the ratio of $\Delta\sigma_r/\Delta\sigma_s$. The term $\Delta\sigma_r/\Delta\sigma_s$ symbolizes the ratio of the stress induced by the magnitude of the repeated stress, $\Delta\sigma_r$, to the ultimate compressive stress, $\Delta\sigma_s$, as determined in the conventional triaxial test. For a given level of the ratio of $\Delta\sigma_r/\Delta\sigma_s$, the total deformation increased with increasing numbers of load repetitions, but at a decreasing rate. After a certain number of load applications, the total sample deformation became constant for each value of $\Delta\sigma_r/\Delta\sigma_s$ below a critical level of the ratio $\Delta\sigma_{rc}/\Delta\sigma_s$, as defined earlier by Larew and Leonards (5). For any level of $\Delta\sigma_r/\Delta\sigma_s$ in excess of this critical value, the sample deformation increased at an increasing rate with each additional load application until the sample failed. The limiting value of deformation for each level of $\Delta\sigma_r/\Delta\sigma_s$ below this critical value was defined as the equilibrium total deformation for that level of $\Delta\sigma_r/\Delta\sigma_s$. These equilibrium total deformation values were used by Ahmed and Larew (2) to calculate the corresponding sample strains. For very low ratios of $\Delta\sigma_r/\Delta\sigma_s$, such as 0.123, the equilibrium deformation was reached within a few hundred load applications in many cases. For ratios of $\Delta\sigma_r/\Delta\sigma_s$ above the critical level, no equilibrium deformation could be established, since the sample continued to deform until it failed.

From a family of curves such as that shown in Figure 5, several levels of $\Delta\sigma_r$ and the corresponding values of limiting strain were obtained. These data were then used by Ahmed and Larew to plot a repeated load stress-strain curve as shown in Figure 6. Both repeated and static load stress-strain curves shown in Figure 6 are for one level of dry unit weight and water content. From repeated load stress-strain curves such as these, static and repeated strength moduli, E_s and E_r, were calculated. The procedures for obtaining the repeated load stress-strain curves employed by the authors differed from the foregoing method in that the limiting total deformation for any level of repeated deviator stress, $\Delta\sigma_r$, was chosen at a constant and arbitrary number of load repetitions rather than for the equilibrium condition.

RESULTS

Families of curves similar to Figure 5 were obtained for Glenelg C stabilized with 5 percent Type III cement, for Culpeper C stabilized with both Type I and Type III cements, and for the untreated Culpeper C. The elastic deformation or resiliency at a point on any one curve is the vertical distance between the total and plastic deformation lines.

Figure 6 compares the stress-strain curve for the Glenelg C soil with 5 percent Type III cement as obtained in the conventional triaxial test with that obtained by

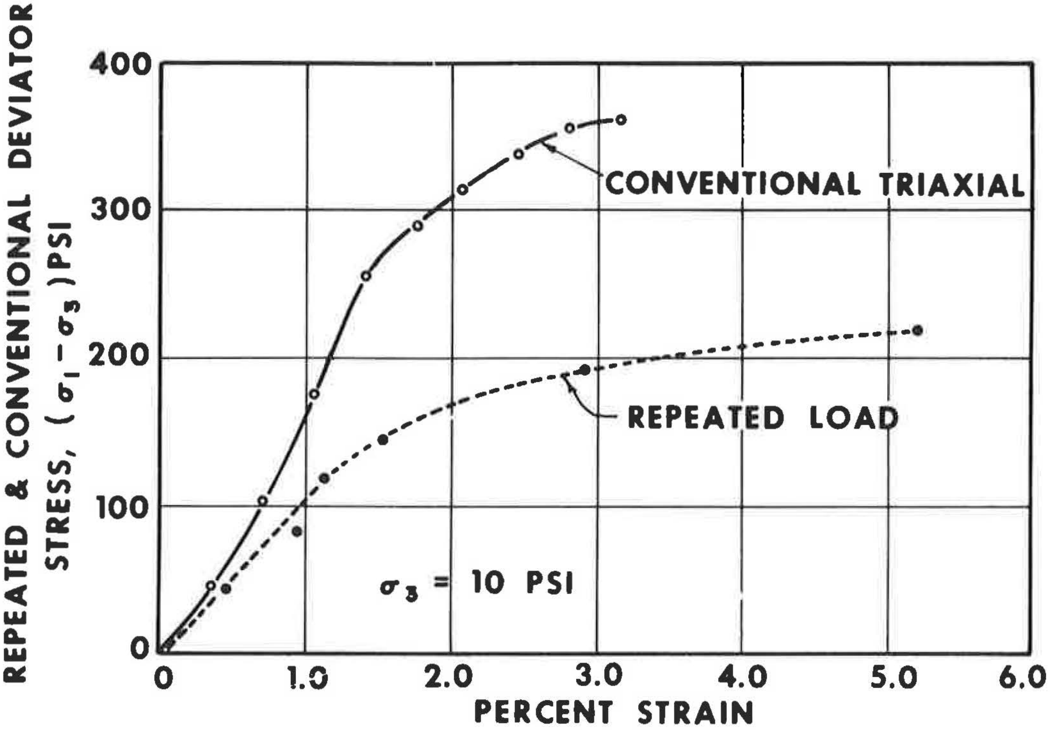

Figure 6. Repeated load and conventional stress vs strain curves for Glenelg C + 5 percent Type III cement.

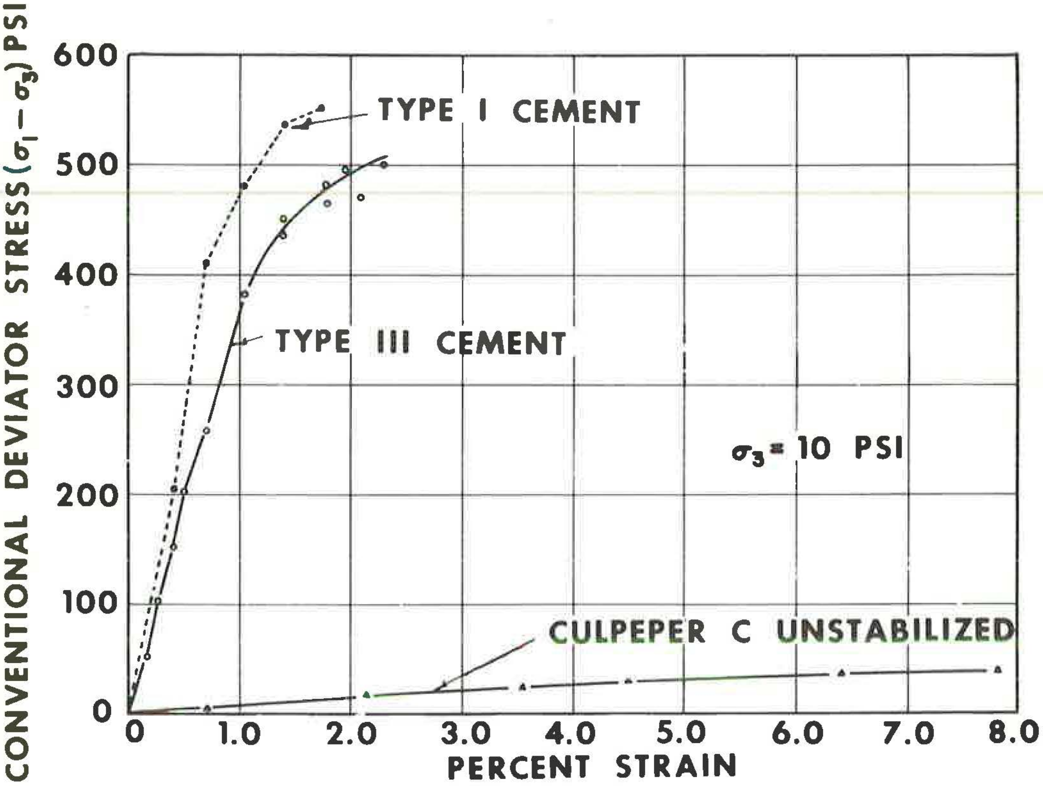

Figure 7. Conventional stress vs strain curves for unstabilized Culpeper C, Culpeper C + 10 percent Type I cement, and Culpeper C + 10 percent Type III cement.

repeated load triaxial tests. From Figure 6 it can be readily seen that the repeated load strength and stiffness for this cement-modified soil is considerably less for repeated loads than for conventionally applied loads; however, the strain at failure is essentially the same in both cases.

Figure 7 shows that the ultimate strengths and stiffnesses of the stabilized Culpeper C soil as obtained in the conventional triaxial test are much greater than that of the

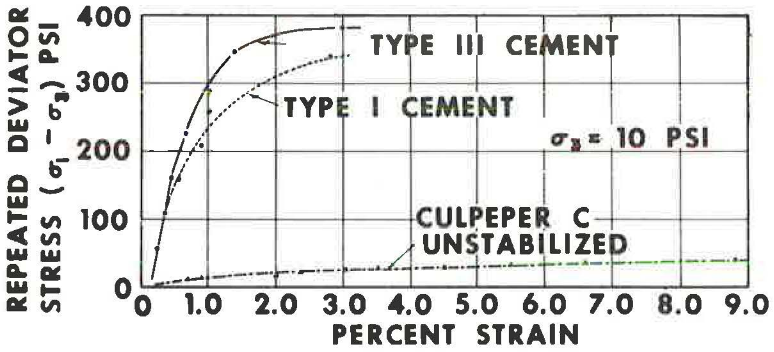

Figure 8. Comparison of repeated load stress vs strain for unstabilized Culpeper C, Culpeper C + 10 percent Type I, and Culpeper C + 10 percent Type III cement.

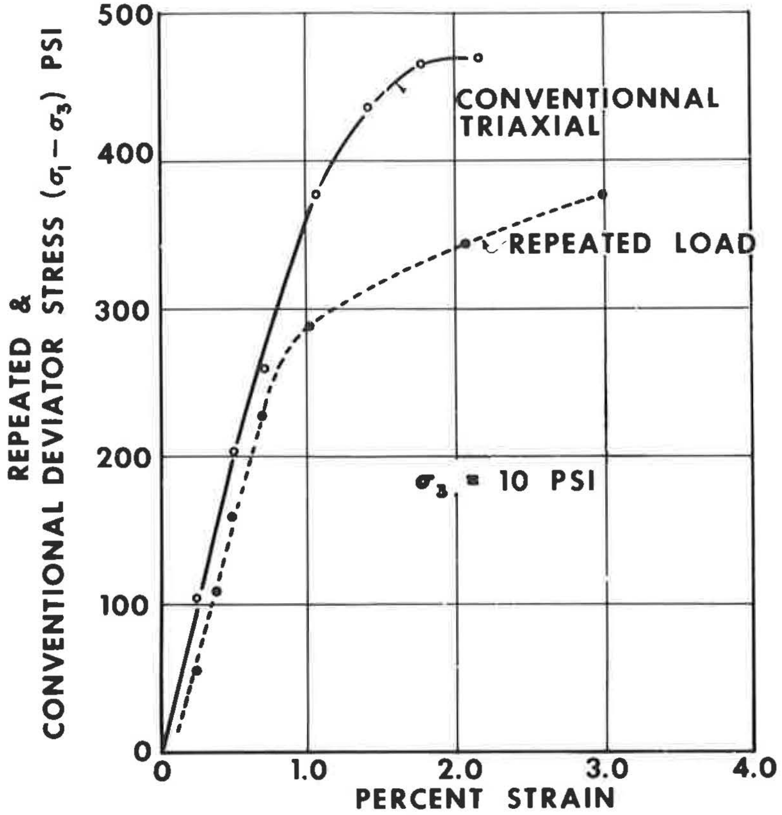

Figure 9. Repeated load and conventional stress vs strain curves for Culpeper C + 10 percent Type III cement.

unstabilized soil. Figure 8 shows the repeated load stress-strain curves for the Culpeper C soil, both untreated and stabilized with 10 percent Type I and Type III cements. Both ultimate strength and stiffness as obtained from repeated load tests are greater for the stabilized soil than for the unstabilized condition.

Figures 9 and 10 show a comparison of the conventional and repeated load stress-strain curves for the Culpeper C soil stabilized with 10 percent Type III and Type I cements, respectively. In contrast to the Glenelg C soil, the initial stiffness of the Culpeper C soil, as measured by an initial tangent modulus and as obtained from the repeated load stress-strain curves, is essentially the same as obtained in the conventional triaxial test. However, the ultimate strength of the Culpeper C soil, as obtained in the repeated load test, is less than that obtained in the conventional test.

Figure 11 is a comparison of the conventional and repeated load stress-strain curves for the untreated Culpeper C soil. In this instance the secant modulus for the repeated stress-strain curve is, at least, equal or greater than the conventional secant modulus. This is not in agreement with the findings of Ahmed and Larew (2) who, for untreated soils, obtained secant moduli in conventional triaxial tests that were always greater than the corresponding repeated load moduli. Differences in the manner of selecting equilibrium deformations from the deformation vs log of number of repetitions curves have caused this apparent disagreement with Ahmed and Larew's results, as explained earlier.

Figure 12 shows the variation of the equilibrium elastic rebound with changing values of the ratio $\Delta\sigma_r/\Delta\sigma_s$, where $\Delta\sigma_r$ is the intensity of the repeated deviator stress and $\Delta\sigma_s$ is the ultimate compressive strength as obtained in a conventional loading

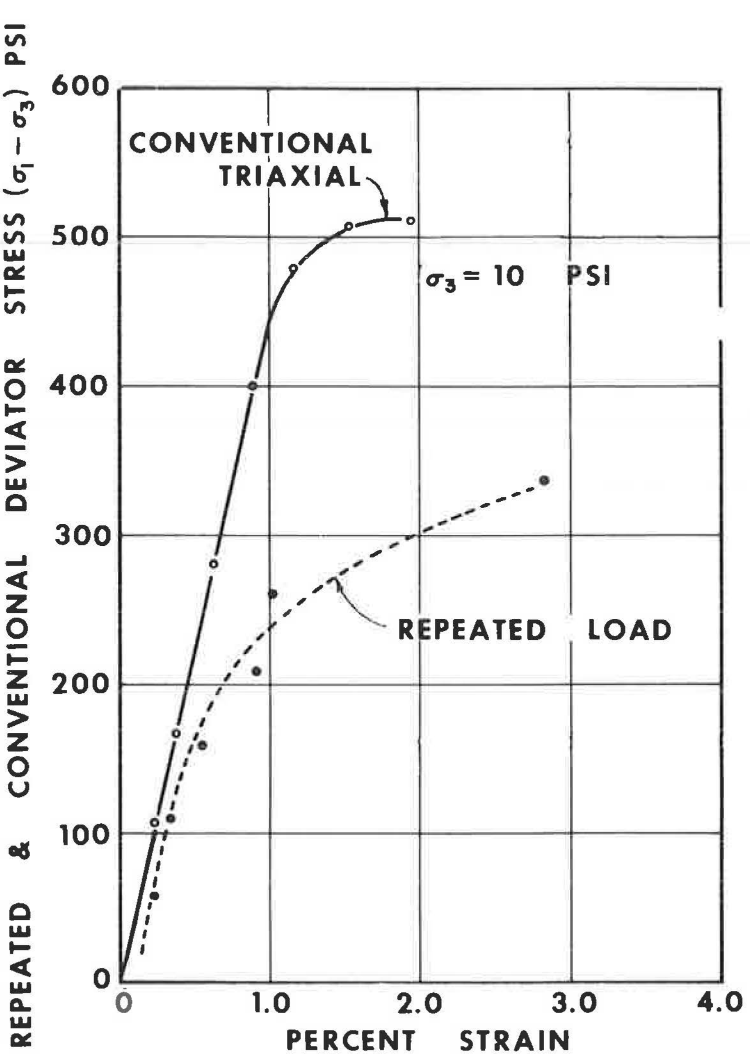

Figure 10. Repeated load and conventional stress vs strain curves for Culpeper C + 10 percent Type I cement.

test. For lower levels of the ratio, the elastic rebound was both essentially linear and unique for both the untreated and stabilized soils studied. This agrees with Tate's (1) earlier findings. Figure 12, therefore, indicates that whereas the addition of portland cement to these soils reduced the elastic rebound caused by a given repeated load, the elastic rebound was no different for stabilized or untreated soils when repeated loads imposing similar ratios of $\Delta\sigma_r/\Delta\sigma_s$ were employed. This indicates that the magnitude of the elastic rebound under the action of repeated loads will not be reduced by the addition of portland cement if at the same time we attempt to take full advantage of increased strength and stiffness of the cement-treated soils.

If the relationship shown in Figure 12 is truly unique for any given soil, then a method for predicting the amount of elastic rebound produced by a given intensity of repeated load can be developed if the conventional stress-strain relationship for either the treated or untreated soil is known. Further studies of this relationship are needed, however.

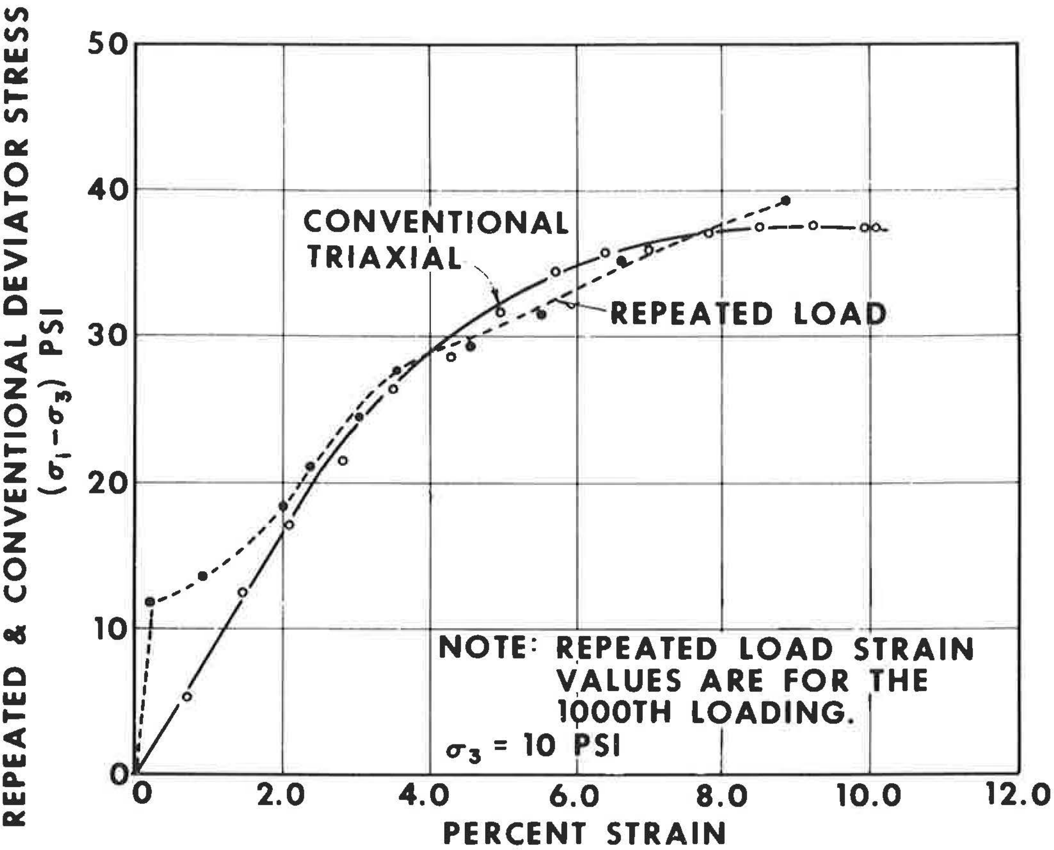

Figure 11. Repeated and conventional load stress vs strain curves for unstabilized Culpeper C soil.

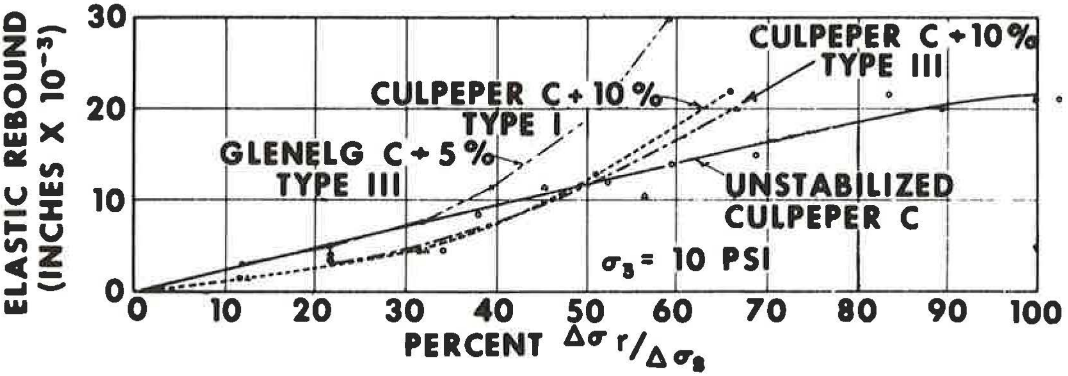

Figure 12. Elastic rebound vs percent ultimate gradually applied stress for soils indicated.

CONCLUSIONS

As a result of this study the following conclusions seem justified:

1. The ultimate strengths and stiffnesses of soils employed in this study were increased many times when they were stabilized with portland cement. This was verified by both conventional triaxial and repeated load testing.
2. For unstabilized Culpeper C soil there was no apparent difference between the conventional stress-strain curve and the repeated load stress-strain curve.
3. The ultimate strengths of both the soil-cement and cement-modified soils, as determined with repeated loads, was considerably less than the ultimate strength obtained for identical samples using conventional loading apparatus; however, the strain at failure remained nearly the same for both types of loading.
4. The stiffness of the soil-cement, as measured by a soil modulus, was little different when determined from either repeated load or conventional stress-strain curves. This was not true for the cement-modified soil (Glenelg C + 5 percent Type III cement) in that the stress-strain curve, as determined by the repeated load test, was less steep and, therefore, gave a lower secant modulus. This indicates that gradually applied load moduli may not be indicative of repeated load moduli in cement-stabilized soils. Further research concerning these findings should be undertaken.
5. The amount of elastic rebound increased linearly with increased ratio of $\Delta\sigma_r/\Delta\sigma_s$ up to values of $\Delta\sigma_r/\Delta\sigma_s = 0.3$ for all soils studied, whether treated or untreated. This appears to be a significant finding for if further research corroborates these results, a prediction of the amount of elastic rebound for a soil can be made from the results of only a conventional triaxial load test.

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Reactions of Soil Minerals With Cement and Chemicals

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The nature of reactions accompanying the stabilization of soil with lime and cement and the mechanisms involved in the alteration of soil-cement (or soil-lime) reaction by chemical additives were studied. One of the main objectives was to compare the similarities and differences for lime and cement reactions with two soil minerals. Mixtures of mono-mineral soils and 10 percent cementing compounds, with or without sodium additives, were examined after compaction and various curing periods by a number of analytical techniques, including flame photometry, colorimetry, X-ray diffraction, and differential thermal analysis.

Both mono-mineral soils, quartz and kaolinite, reacted with the three calcium stabilizers (hydrated lime, tricalcium silicate and Type I portland cement). Addition of sodium additives greatly intensified the reaction between the soil and the stabilizer and increased the abundance of reaction products. Reactivity of kaolinite with any one of the cementing compounds was higher than that of quartz. The principal reaction products produced in quartz mixtures were hydrous calcium silicates of various compositions with $CaO:SiO_2$ ratio varying from 0.2 to 1, and in kaolinite mixes were hydrous calcium silicates, hydrous calcium aluminates and a mixed calcium aluminosilicate. Sodium from the additive associated with soluble compounds early in cure and later with insoluble mixed sodium-calcium silicates (or aluminates).

●SOILS ENGINEERS have for many years considered portland cement and hydrated lime the most successful soil stabilizers. The use of cement and lime has increased steadily in recent years in the construction of airfields, roadways and other engineering works in many parts of the world. However, until the late 1950's very little was known about the mechanisms of the stabilization and the nature of the reaction products. Investigators have proposed various hypotheses for soil-lime (9, 12, 20) and soil-cement stabilization (13, 14, 22).

In 1957, the U. S. Bureau of Reclamation (7) initiated an extensive study on the pozzolan-cement reaction by establishing a library of X-ray diffraction patterns and differential thermal analysis (DTA) curves of hydration products of pure compounds used individually and in combination as normal constituents of commercial cement. Benton (1) reported that in the cement-pozzolan reactions all the compounds identified, except a Stratling's compound, are recognized normal hydration products of cement.

Goldberg and Klein (12) concluded from their limited results of X-ray and DTA studies of mixtures of lime and Wyoming bentonite that the calcium hydroxide in the soil-lime mixture is converted to calcium carbonate, not calcium silicates. More recently, Eades and Grim (9) investigated the reaction of hydrated lime with pure clay

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minerals and noted the formation of new crystalline phases, probably tobermorite-like calcium silicate hydrates. Diamond, White and Dolch (8) treated various clay minerals and silicates with 40 to 400 percent (by weight of the mineral) of calcium hydroxide at elevated temperatures. They identified the reaction product as poorly crystallized calcium silicate hydrates of the tobermorite family and various forms of calcium aluminate hydrates. Hilt and Davidson (15) reported the isolation of a crystalline calcium aluminate hydrate reaction product from a mixture of montmorillonite and 40 percent lime. Glenn and Handy (11) found that the products of the reaction in slurry form of several varieties of lime and soil minerals include a 10 Å tobermorite, tetracalcium aluminate hydrates and several compounds of unknown composition. The formation of new products by lime stabilization under field conditions was confirmed by Eades, Nichols and Grim (10). Herzog and Mitchell (14) reported an investigation of the reaction of two clay minerals with 15 and 30 percent of tricalcium silicate and portland cement at the relatively high temperature, 50 C. They suggested a clay-cement structure with "primary" cementitious materials due to the cement hydration and "secondary" cementitious matter resulting from reactions between the calcium hydroxide (a reaction by-product of the "primary") and clay mineral.

Most of these studies employed either a relatively high concentration of the stabilizer or high temperature curing. In several investigations, the reacted samples were subjected to drying (air or oven) before examination by X-ray or DTA. This paper reports the results of an investigation to elucidate further the nature of the reactions accompanying lime and cement stabilization and also the effect of sodium additives on the reactions. One of the primary objectives of the study was to compare the similarities and differences between lime and cement reactions with soil minerals. The stabilizer concentration selected for the study was 10 percent by dry soil weight. All mineral-stabilizer-additive mixtures were humid cured at room temperature for various periods of time and then subjected to examination with no drying.

NOTATIONS

Materials

Q	Quartz
K	Kaolinite
M	Montmorillonite
L, $Ca(OH)_2$	Lime, calcium hydroxide
T, C_3S	Tricalcium silicate
PC	Portland cement

Chemical Terms

C	CaO
S	SiO_2
A	Al_2O_3
H	H_2O
N	Na_2O
X	Anion of sodium additive

X-Ray Diffraction Patterns

I	Relative intensity
d	Diffuse
v, V	Very
b, B	Broad
s, S	Strong
m, M	Medium
w, W	Weak

EXPERIMENTAL INVESTIGATION

The experimental investigation was carried out on specially prepared soil-cement-additive specimens. In view of the extreme complexity of this type of system, mono-mineral soils were used in preference to natural soils. Various combinations of the mono-mineral soils, cementing compounds and selected additives were examined at various stages during the reaction process by a number of special analytical techniques. The solid phase of each system was examined by X-ray diffraction and DTA techniques. Variations in the chemical composition of the solution phase of the system were also analyzed.

Materials

Soil Components.—Two mono-mineral soils, quartz (Q) and kaolinite (K), representing a silicate and an aluminosilicate, were selected for the study of the mechanisms of soil stabilization with cement, lime and sodium additive.

The quartz sample was obtained by grinding Ottawa sand to 100 percent passing No. 200 sieve. The freshly ground mineral was mixed well and allowed to stand in air under room temperature and humidity for at least 3 months before use to minimize any effect of grinding on the reaction pattern of the mineral with the stabilizers. X-ray and DTA data indicated that the quartz sample was reasonably pure. Tests of the kaolinite (Peerless No. 2 kaolin) showed that this product was free of any montmorillonoids and had very little soluble salts. A third mono-mineral soil, montmorillonite (Volclay 325), was used to augment the data on the evaluation of the nature of reactions between the soil minerals and cements. Table 1 summarizes some of the properties of these mono-mineral soils. The X-ray patterns are tabulated in Table 2 and the DTA curves are shown in Figure 1.

Primary Stabilizers.—Reagent grade calcium hydroxide (L), synthesized tricalcium silicate (T) and commercial Type I portland cement (PC) were selected as primary stabilizers. The concentration of 10 percent by dry soil weight used in all experiments represents the average stabilizer content utilized in practical soil stabilization work. Table 3 summarizes the composition of tricalcium silicate and Table 4 shows the composition of the portland cement used. Shown in Figure 2 are thermograms of the three stabilizers in the unhydrated state. ("Cement" is used to denote any of the three primary stabilizers; portland cement is used to denote the particular compound.)

Secondary Additives.—Three alkali sodium compounds (hydroxide, metasilicate and sulfate) were chosen for this study because results from previous studies (20, 22) indicated that alkali metal compounds beneficial to cement stabilization were those forming insoluble salts with calcium and that these three chemicals were the most effective ones on a range of soils investigated. The secondary additive concentration used in the present investigation was equivalent to one normal sodium ion in the molding water.

Procedure

Preparation of Soil-Stabilizer-Additive Specimens.—Individual specimens were prepared for each series of X-ray and DTA tests. Air-dried mineral and stabilizer were first dry mixed; chemical solution or distilled water was then added and mixed by hand until uniform. Samples were compacted immediately after mixing in a specially made miniature mold (to give a sample size just sufficient for one DTA and one X-ray test) with a tamping rod. A second specimen of each combination was prepared for the chem-

TABLE 1

PROPERTIES OF SOIL MINERALS

Soils	Textural Composition (% by wt.)			Physical Properties						Chemical Properties	
	0.074 mm	0.074-0.002 mm	0.002 mm	L.L. (%)	P.L. (%)	P.I. (%)	Spec. Gr.	Max. Dry Density[a] (pcf)	Opt. Water Cont. (%)	Organic Matter (%)	Total Sol. Salt (m.eq.NaCl/100 gm)
Quartz	0	100	—	N.P.	N.P.	N.P.	2.65	96.7	20.1	0	0.3
Kaolinite	2	41	57	52	29	23	2.66	93.8	25.1	0	0.2
Montmorillonite	3	97	—	—	—	—	—	79.1	38.2	-	31.0

[a] Harvard miniature compaction, 40 lb tamper, 3 layers, 25 blows per layer.

TABLE 2

X-RAY PATTERNS OF TWO SOIL MINERALS AND THREE STABILIZERS

Quartz		Kaolinite		Calcium Hydroxide		Tricalcium Silicate		Portland Cement	
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I
4.25	S	7.13	VS	4.91	S	6.06	VW	3.98	W
3.34	VS	4.44	–	3.12	M	3.90	VW	3.63	W
2.45	M	4.34	M	2.63	VS	3.02	S	3.32	W
2.28	M	4.15	–	1.93	M	2.78	VS	3.02	S
2.24	W	3.84	W	1.80	M	2.60	VS	2.76	VS
2.13	W	3.56	VS	1.69	M	2.39	VW	2.61	S
1.98	W	3.32	W	1.63	VW	2.34	W	2.31	WB
1.82	S	2.56	M			2.31	–	2.17	S
1.67	W	2.49	–			2.18	S	2.10	WB
		2.38	M			1.94	M	2.04	WB
		2.34	M			1.77	S	1.97	W
		2.29	M					1.92	MB
		1.99	M					1.76	S
		1.94	VW						
		1.89	VW						
		1.84	VW						
		1.79	W						
		1.66	M						

TABLE 3

COMPOSITION OF TRICALCIUM SILICATE[a]

Analysis	% by Weight	Composition	% by Weight
Total CaO_2	72.74	C_3S	95.12
Total SiO_2	26.30	β-C_2S	1.92
Al_2O_3	0.02	$C_2S \cdot 2H_2O$	1.79
MgO	0.10	$Ca(OH)_2$	0.55
SO_3	0.03	$CaCO_3$	0.25
H_2O	0.46	$C_3A \cdot 6H_2O$	0.07
CO_2	0.11	$CaSO_4$	0.05
Na_2O	—	MgO	0.10
K_2O	—	SiO_2	0.15
Total	99.76		100.00

[a]Data obtained from Brunauer, Hayes and Hass (5).

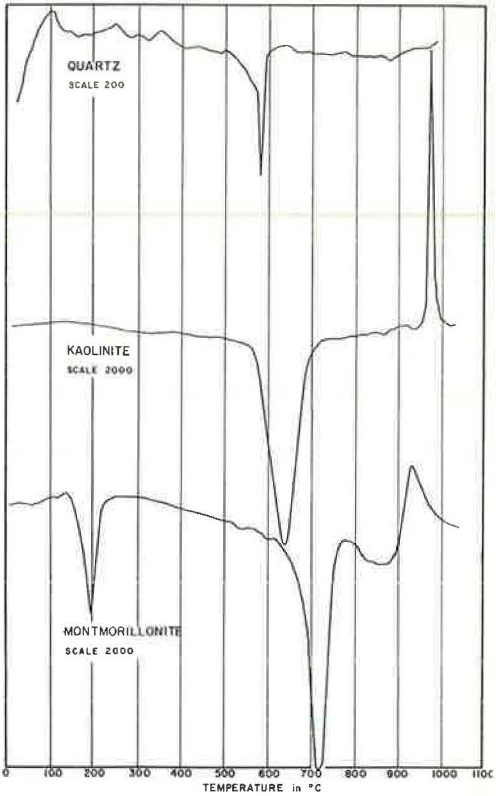


Figure 1. Thermograms of mono-mineral soils.

TABLE 4

COMPOSITION OF CEMENT[a]

Analysis	% by Weight	Pot. Phase Comp.[b]	% by Weight
SiO_2	19.78	C_3S	51.9
Al_2O_3	5.54	C_2S	17.7
Fe_2O_3	3.45	C_3A	8.8
CaO	62.59	C_4AF	10.5
MgO	3.90		
SO_3	2.25		
Na_2O	0.25		
K_2O	0.71		
Loss on ignition	1.30		
Insol. residue	0.08		
Mn_2O_3	0.07		

[a]Spec. surface (Blaine), 3,270 sq cm/g.
[b]Calculated based on equilibrium crystallization (2).

ical analysis. These specimens were compacted statically in a mold 0.625 in. in diameter and 1.600 in. high. The moisture content and dry density of each mineral-stabilizer-additive combination were approximately equal to the optimum moisture content and maximum dry density of the untreated mineral, as determined by the Harvard miniature compaction procedure (1.313-in. diameter 2.816-in. high sample, compacted by a 40-lb tamper in 3 layers with 25 blows per layer). Immediately after molding, specimens were cured at approximately 100 percent RH and room temperature for various periods of time.

X-Ray Diffraction Analysis.—X-ray diffraction patterns were obtained with unfiltered chromium radiation in an evacuated powder camera of 114.6-mm diameter. All cured specimens were crushed at as-cured moisture content (with no drying), and mixed with a droplet of glycerol (used to expand the clay minerals and/or the hydrous calcium silicates to enhance the basal reflections), and worked into a thick

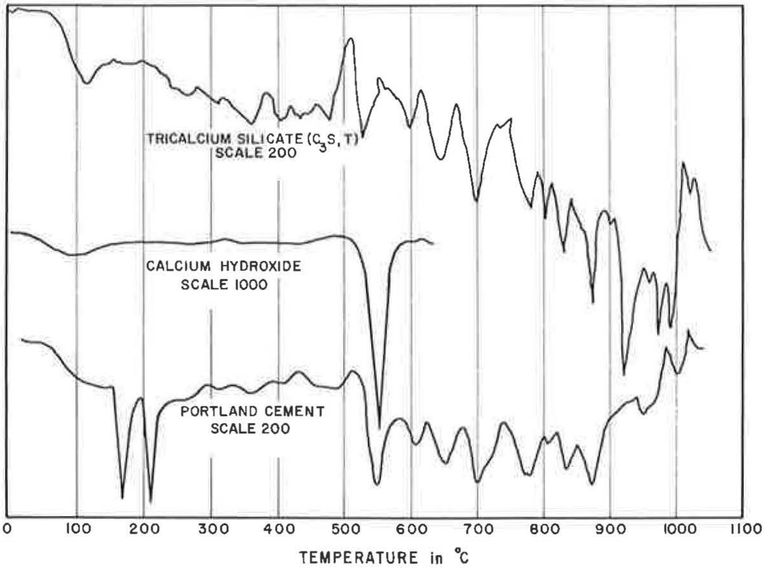


Figure 2. Thermograms of calcium stabilizers.

paste with a spatula. A small amount of collodion was added and the paste was rolled between two ground glass plates to a thin rod approximately 0.5 mm in diameter. The specimen was then mounted in the center of the camera on a rotating shaft. Double-emulsion films were used to record the diffraction patterns. The film extended over half the circumference of the camera. The specimen was exposed for 1.5 hours with continuous rotation to give a random orientation. During the exposure period, the camera was evacuated to reduce air scattering and required exposure time.

X-ray diffraction patterns of the pure materials, e.g., the two minerals and three stabilizers, were checked with a Norelco diffractometer. Excellent agreement was obtained.

Differential Thermal Analysis.—DTA experimental conditions were single-unit nickel steel block with platinum-platinum plus 10 percent rhodium thermocouples, heating rate at 12.5 C/min, sample volume 0.08 cu in. (approximately 1.5 g in weight). A detailed description is given by Lambe (21), except that $^3/_{32}$-in. diameter ceramic tubes were used to protect the thermocouples in the two wells. Calcined aluminum hydroxide (γ-Al_2O_3) was used as reference material. No control of relative humidity of the inert reference material was undertaken.

Specimens were crushed immediately after curing. To avoid the effect of drying on the properties of the test mixture, specimens were crushed as fine as possible (approximately passing a No. 20 sieve) under as-cured moisture condition. The crushed sample and the reference material were firmly packed into the wells. Access to air was limited by a cover.

Amplification of the differential voltage was varied (noted on the DTA traces as "scale") during the test as necessary to provide a maximum degree of sensitivity, since it was desired to study very small changes in the thermal patterns. The same variation in the degree of amplification was used for all DTA tests on the same mineral.

Extraction for Chemical Analysis.—The as-cured specimens were crushed and pulverized to pass a No. 20 sieve after various ages of cure. (The sieve size was chosen somewhat arbitrarily. However, it was difficult to pulverize the wet specimen any further without causing excessive drying of the mixture.) A 10-g portion of the wet pulverized sample was mixed with 50 ml distilled water with continuous stirring for 1 minute, then vacuum filtered immediately. By this extraction procedure, a minimum of time was allowed for the sample to contact a large excess of water. The whole extraction process required less than 2 minutes. The clear filtrate was preserved in capped 50 ml polyethylene containers for analysis of the ionic compositions. A separate sample of the wet pulverized mixture was taken for determination of the moisture content. Results of the chemical analyses are reported on the basis of ovendry weight of the pulverized material.

Chemical Analysis of the Aqueous Extracts.—The clear filtrates were quantitatively analyzed for silica, alumina, sodium and calcium. Silica and alumina contents were determined colorimetrically. Ammonium molybdate was used to develop the blue color for silica determinations (25), and aluminon reagent was used for aluminum determinations (25). Sodium and calcium concentrations were analyzed with a Baird flame photometer. Careful consideration was given to the interference of other ions present in the extract on these determinations. The methods adopted are believed to be completely satisfactory to ± 5 percent, which is adequate for the present study.

RESULTS AND DISCUSSION

The test results obtained indicate that the reaction patterns of one soil mineral with any of the three calcium stabilizers are not greatly different from each other. The major differences are found more in the rate of reaction and the relative amounts of the various reaction products rather than in the formation of entirely different products. Therefore, the following discussion is confined to the results of several selected mixtures. Detailed results and discussion are given elsewhere (26).

Since the primary objective of this research is to attain a better understanding of the fundamentals in the alteration of soil properties with cement and sodium additives, the emphasis of the analysis of the test results is on the examination of the types and trends of the interactions between the various constituents rather than on the formulation of the exact chemical reaction or the final products. Nevertheless, the reaction products are identified whenever possible. Interpretation of these products is largely based on published information concerning similar systems.

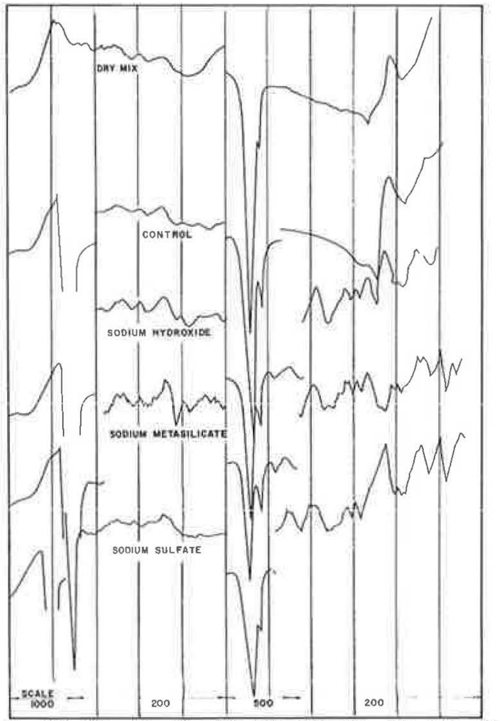


Figure 3. Effect of sodium additives on thermogram of 1-day cured quartz-calcium hydroxide mix.

X-Ray Diffraction and Differential Thermal Analysis

Quartz-Calcium Hydroxide Series.—In the thermograms of quartz-calcium hydroxide mixes, with or without additive, the dehydration endotherm of calcium hydroxide at approximately 550 C and the quartz inversion peak are the two predominant features, as illustrated by the curves in Figure 3. The decrease in amplitude of the two peaks with curing time (Fig. 4) clearly demonstrates the reaction between the two constituents.

X-ray diffraction patterns (Table 5) show that most calcium hydroxide lines diminish after 1-day curing and the only remaining line at 2.62 Å decreases in relative intensity with time as the amplitude of the endothermic peak of this compound in the DTA curves also decreases. A new phase appears to develop, increasing in quantity with curing as indicated by the diffraction line at 3.03 Å. Although the identity of the new reaction product

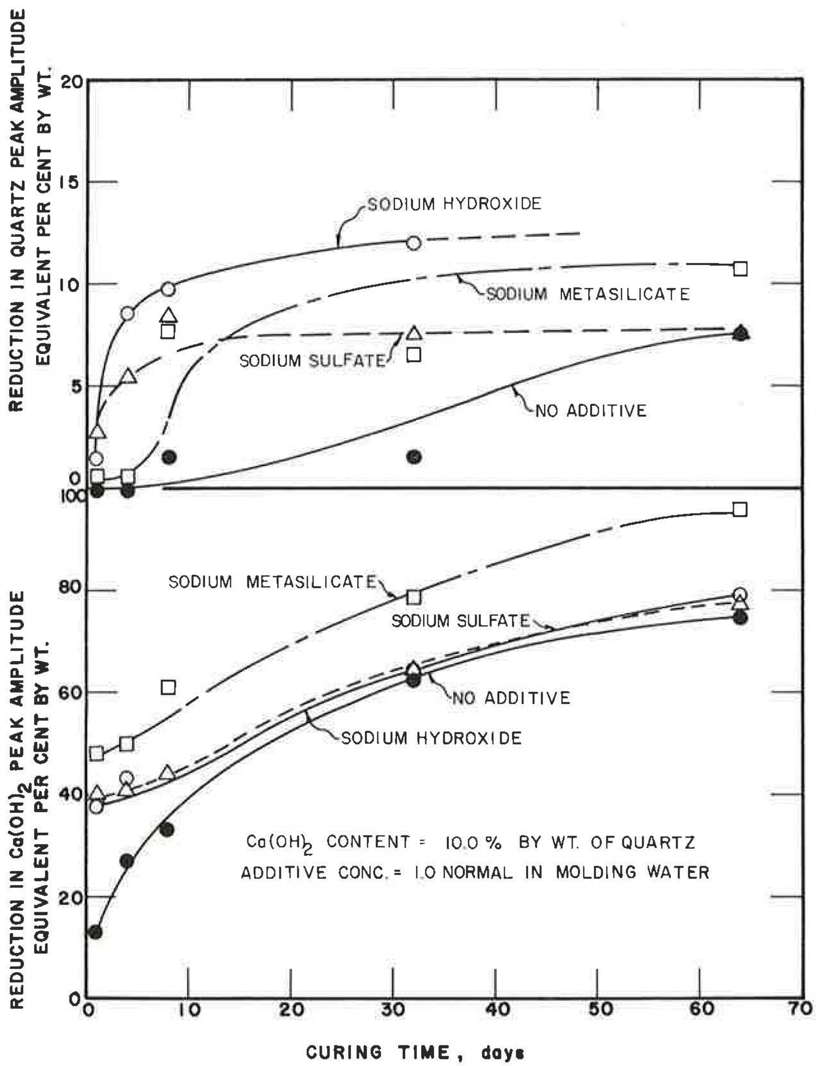


Figure 4. Effect of curing time on DTA peaks of quartz and lime in quartz-lime mixes.

TABLE 5

X-RAY ANALYSES OF QUARTZ-CALCIUM HYDROXIDE MIXTURES

Spacing (Å)	Relative Intensity									Identi-fication[a]
	Dry Mix	No Additive		Sodium Hydroxide		Sodium Metasilicate		Sodium Sulfate		
		1-Day	64-Day	1-Day	64-Day	1-Day	64-Day	1-Day	64-Day	
3.33	10	10	10	10	10	10	10	10	10	Quartz
3.25	—[b]	—	—	—	2	—	—	—	—	CSH
3.03	—	—	2	—	3	1	3	—	3	CSH (I)
2.62	4	2	2	3	1	2	—	2	1	Lime
2.36	—	—	—	—	—	—	1	—	—	CSH

[a] See text for explanation. [b] Line not present in pattern.

is uncertain, the data indicate that the new phase is a poorly crystalline calcium silicate hydrate of CSH (I) type (29). The 3.03 Å line is attributed to CSH, not to calcite, because the X-ray diffraction data indicate that other CSH lines also develop in these mixes. Furthermore, calcite, even at a very small percentage, exhibits very strong endothermic reaction at temperatures above 900 C (23). Examination of the thermograms and X-ray patterns reveals no correlation between the calcite thermal reaction and the observed 3.03 Å line. Since this is the strongest line of a poorly crystalline CSH, it is used to represent this compound in all discussions.

From the amplitude of the thermal peaks in the DTA curves, an attempt is made to estimate quantitatively the amount of materials reacted after various hydration time as shown in Figure 4. At 64 days, only 2.5 percent calcium hydroxide (percentage based on the weight of soil mineral unless otherwise noted) remains in the mix; that is, 75 percent of the original amount of calcium hydroxide has combined into new products or lost its identity. The amount of quartz that disappeared in this sample corresponds to 7.5 percent. If all the missing quartz and calcium hydroxide reacts with each other, a hydrous calcium silicate with lime-silica ratio of 0.8 is formed. This compound is within the range of Taylor's CSH (I) or Bogue's $C_2S_3H_2$ (2) which produces the strongest X-ray diffraction line at 3.03 Å.

As shown in Figure 4, the most obvious effect of a sodium additive is to intensify the reaction between the soil and the stabilizer. The strong alkali additive, sodium hydroxide, greatly increases the reactivity of the quartz mineral toward the lime, particularly early in the cure. However, the general pattern of the DTA curves remains essentially the same as those of the additive-free samples. One additional effect of the caustic is the development of a small endothermic bulge at about 610 to 620 C which is probably a sodium-bearing phase of hydrous calcium silicate (19). The X-ray diffraction data reveal an earlier formation of CSH; the 3.03 Å line first appeared in the 4-day specimen as compared to the 32-day specimen of the additive-free mix.

With sodium metasilicate as additive, the calcium hydroxide is consumed much more rapidly and completely than it is in either the additive-free or the caustic-treated mix. The major reaction occurring early in the cure (about 1 day) is the precipitation by calcium of the highly reactive silicate from the additive to form insoluble calcium silicate hydrate. Thus, very little of the quartz mineral, which is much less reactive than the additive silicate, would enter into the reaction. Gradually, the alkali component of the sodium additive attacks the quartz silica which, in turn, reacts with the remaining lime or with the hydrous calcium silicate already formed. These reaction processes are clearly demonstrated by the very small changes in the quartz thermal peaks for samples cured less than 4 days and the greater reduction in peak size with longer cure. The major reaction product, hydrous calcium silicate, appears to develop after only 1 day of cure and is more abundant than in the additive-free sample. It is also interesting to note that the sodium-bearing phase of CSH formed at early ages gradually diminished after 32 days of reaction as shown by the endothermic reaction between 610 and 620 C in Figure 5. This indicates that in this mix the sodium-bearing phase of the CSH is a metastable phase which transforms to a calcium silicate hydrate with time. The large exotherm at about 875 C of the 64-day sample seems to support this interpretation.

As shown by the DTA and X-ray data, the reactions in the sodium sulfate-treated quartz-calcium hydroxide mixes are essentially analogous to that in the caustic-treated samples, except that a smaller amount of quartz enters into the reaction. The exothermic reaction at approximately 875 C, which corresponds to a calcium silicate hydrate with lime-silica ratio of 1.09 to 1.25 (17), appears on the thermograms of all specimens.

Quartz-Tricalcium Silicate Series.—Assuming that the tricalcium silicate does not react with the soil component during its hydration, the stoichiometry of the hydration of C_3S can be represented by (6):

$$2\,Ca_3SiO_5 + 6\,H_2O \longrightarrow Ca_3Si_2O_7 \cdot 3\,H_2O + 3\,Ca(OH)_2 \qquad (1)$$

The calcium silicate hydrate produced is the high lime end-member of Taylor's CSH (I). It is obvious from Eq. 1 that calcium hydroxide is the major hydration by-

Figure 5. Thermograms of quartz-calcium hydroxide-sodium metasilicate mixes.

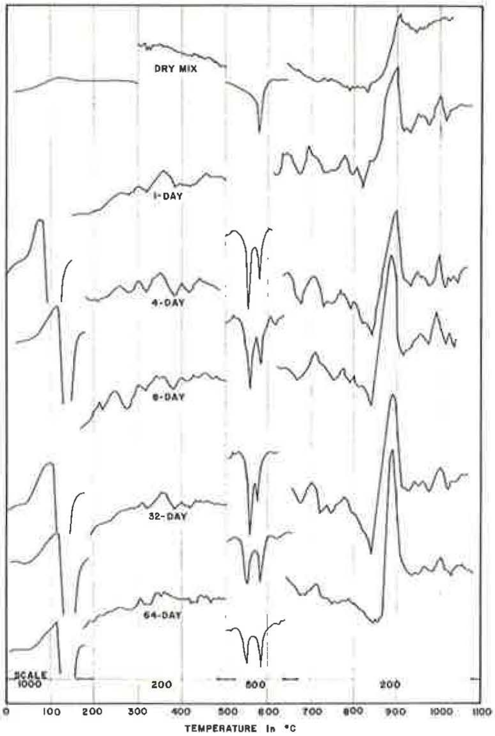


Figure 6. Thermograms of quartz-tricalcium silicate mixes.

TABLE 6

X-RAY ANALYSIS OF QUARTZ-TRICALCIUM SILICATE MIXTURES

Spacing (Å)	Relative Intensity													Identification
	Dry Mix	No Additive			Sodium Hydroxide			Sodium Metasilicate			Sodium Sulfate			
		1-Day	32-Day	64-Day	1-Day	8-Day	64-Day	1-Day	8-Day	64-Day	1-Day	8-Day	64-Day	
8.8	—	—	—	—	—	—	4	—	—	—	—	—	—	CS_2H_2
7.6	—	—	1	1	—	2d	2	—	2	2	—	—	—	$C_2S_3H_2$, Q
7.1	—	—	2	1	—	—	2	—	—	2	—	1	—	$C_2S_3H_2$, Q
3.34	10	10	10	10	10	10	10	10	10	10	10	10	10	Quartz
3.25	—	—	—	—	—	2	—	—	—	—	—	—	—	CSH
3.03	—	—	2	2	—	vw	2	1	1	2	—	—	2	CSH (I)
3.01	2	1	—	—	vw	—	—	—	—	—	—	—	—	C_3S
2.84	—	—	—	—	—	—	—	—	—	—	—	—	—	CS_2H_2
2.80	—	—	—	vw	vw	—	—	—	vw	—	—	—	—	CSH (I)
2.62	—	1	vw	vw	2	1	—	—	—	—	1	1	—	Lime
2.60	2	1	—	—	—	—	—	vw	vw	—	—	—	—	C_3S

product of the C_3S. Thus, the amount of calcium hydroxide present in the sample may be utilized as an indication of the rate of C_3S hydration. By assuming complete hydration of the C_3S present in these mixes, a maximum of 4.2 percent of calcium hydroxide will be produced. Both DTA curves (Fig. 6) and X-ray data (Table 6) show the presence of this compound at all curing ages. The calcium hydroxide increases from 3.6 percent

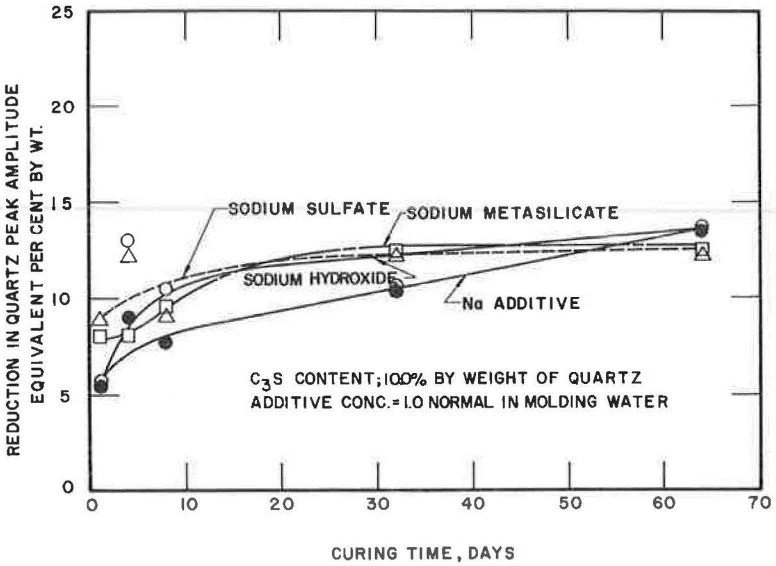


Figure 7. Effect of curing time on DTA peak of quartz in Q-C_3S mixes.

at 1 day to 4.1 percent at 8 days, which corresponds to almost complete hydration of the C_3S, and gradually decreases with further cure. The presence of the maximum amount of $Ca(OH)_2$ in the 8-day sample indicates that during that time practically no reactions occurred between the lime released from the C_3S hydration and the quartz mineral. However, approximately 5 percent of the quartz is lost after only 1 day of cure, as determined from the size of the quartz thermo-peak shown in Figure 7. This quartz must then either react with the CSH or be fluxed by the CSH. The new product (or products) formed in this mix, as revealed by the progressively stronger exothermic reaction at 890 to 900 C, is a calcium silicate hydrate with lime-silica ratio of 1.33 to 1.6 (18). This product is very close to the product shown by the stoichiometric equation of the C_3S hydration. If all the missing quartz, as indicated by the thermo-reactions, truly reacts with the calcium stabilizer to form calcium silicate hydrate, then the corresponding lime-silica ratio in the new product would be much lower. The ratio further indicates that not all the missing quartz (at all curing ages) is truly reacted with the lime and/or CSH; part of it is probably adsorbed, enters into the structure of the hydration product, or is masked by the reaction products during the thermal analysis. The CSH formed appears to be a poorly crys-

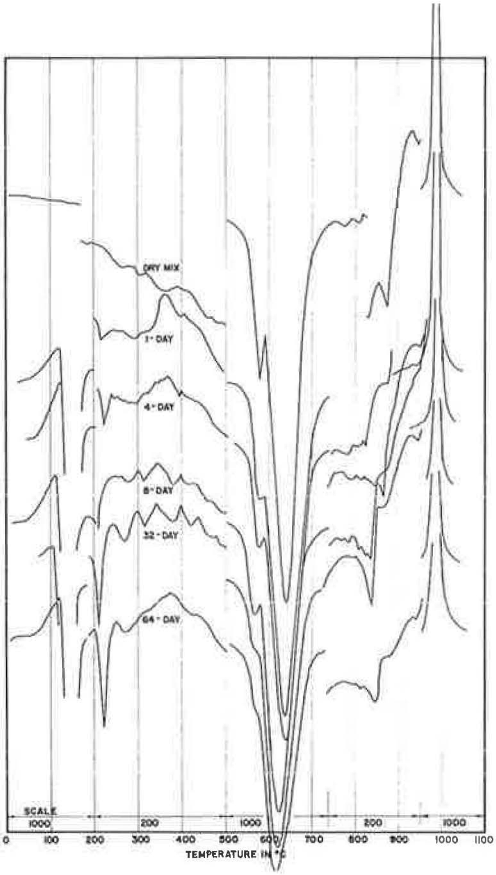


Figure 8. Thermograms of kaolinite-calcium hydroxide mixes.

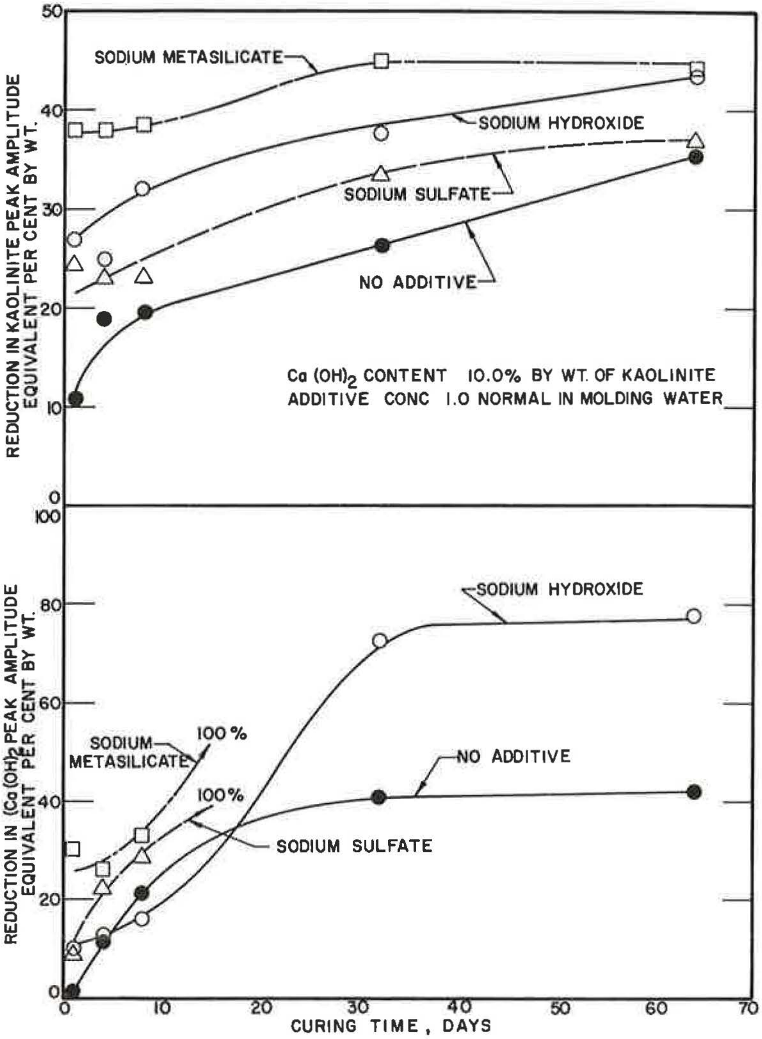


Figure 9. Effect of curing time on DTA peaks of kaolinite and lime.

talline variety at early cure and gradually develops a more orderly structure with time as shown by the X-ray patterns.

The most obvious effect of the addition of sodium hydroxide to the quartz-C_3S mix is the acceleration of the C_3S hydration and hydrolysis, which is virtually completed after only 1 day of curing. The X-ray data show an early appearance (at 4 days as compared to 32 days for the additive-free mix) of the major CSH (I) line at 3.03 Å and also indicate a somewhat more abundant quantity. The appearance of a diffraction line at 8.9 Å in the 64-day sample shows that in addition to the high-lime member of the CSH, there is also some low-lime member of CSH formed. And since part of the quartz mineral would become more reactive due to the addition of the strong alkali, it is reasonable to conclude that most of the missing quartz in these mixes probably truly participates in the reaction. The DTA curves further prove that the CSH formed is of lower lime-silica ratio than that in the alkali-free mixes. The two exotherms at 830 and 870 C belong to the CSH with lime-silica ratios of 0.8 to 1.0 and 1.25, respectively. The gradual increase in the size of the lower temperature exotherm and the decrease in size of the other with time definitely indicate that the high-lime member of CSH is transformed to a lower lime member.

TABLE 7

X-RAY ANALYSES OF KAOLINITE-CALCIUM HYDROXIDE MIXTURES

Spacing (Å)	Relative Intensity											Identification
	Dry Mix	No Additive			Sodium Hydroxide			Sodium Metasilicate		Sodium Sulfate		
		1-Day	32-Day	64-Day	1-Day	32-Day	64-Day	1-Day	64-Day	1-Day	64-Day	
14	-	-			-			-	-	-		
13	-	-			-			-		-	2bd	C_2ASH_X
11	-	-	2b	3b	-	3bd	4bd	2	2bd	-		CSH (I)
9.8	-	-			-			2		2		
9.6	-	-			-			-	-	-	-	
8.7	-	-			-			-	-	2	-	CS_2H_2
7.2	10	10	10	10	10	10	10	10	10	10	10	Kaolinite
3.03	-	-	2	2	vw	3	2	1	1	1	2	CSH (I)
2.87			-	-				-	-	-	-	
2.81	1b	1b	2b	2b	vw	1b	1b	2b	2b	2b	2b	C_2ASH_X
2.73												
2.62	2	2	-	-	2	-	-	2	-	2	-	Lime
2.09	-	-	-	-	-	1	1	-	-	-	-	$C_4A_3H_3$

Sodium metasilicate appears to delay the C_3S hydration, whereas sodium sulfate affects the reactions in essentially the same manner as does the hydroxide. The reaction products formed appear to consist of several varieties of hydrous calcium silicate as illustrated by the X-ray data in Table 6.

Kaolinite-Calcium Hydroxide Mixes.—In the kaolinite-calcium hydroxide series, the reactions are considerably more complex. The X-ray diffraction patterns are more difficult to interpret because of the presence of large amounts of water in the specimens which tend to diffuse and broaden the diffraction lines. Experimental results indicate that the reaction products consist of a mixed calcium aluminosilicate (C_2ASH_X) identified as the Stratling's compound, hydrous calcium silicates, and probably a hydrous calcium aluminate of the composition C_4AH_{13}.

The thermograms in Figure 8 show that both the lime endotherm at about 565 C and the kaolinite dehydration endothermic peak decreased considerably in size as reaction proceeded. Interpretation of the changes in the intensity of the thermal peaks quantitatively, as summarized in Figure 9, indicates that at the end of 64 days, 42 percent of the initial lime is consumed and approximately 35 percent of the kaolinite has lost its identity. The fact that the 32-day sample consumes about the same amount of lime as the 64-day specimen, whereas the amount of kaolinite lost increases, tends to indicate that a portion of the lime is probably adsorbed on the surface of the clay at early ages of reaction. The dehydration characteristics of the adsorbed lime are different from the normal lime which exhibits an endothermic reaction at about 565 C. The endotherm at about 210 C is probably caused by both C_4AH_{13} and the Stratling's compound. The amplitude of this endotherm increases in size with hydration time of the mixture.

The X-ray diffraction lines, as summarized in Table 7, tend to be diffused and broad. The 2.62 Å line of lime diminishes after 32 days of cure. The diffraction lines of the Stratling's compound (C_2ASH_X) appear in all patterns and seem to be stronger with curing time. A line at 3.04 Å appearing in the patterns of 32- and 64-day samples indicates the rather late development of the CSH compound. This CSH compound has been shown (1) to be capable of holding a considerable amount of alumina in its lattice structure.

Although the DTA curves indicate that a rather large amount of kaolinite has lost its thermal identity after various periods of curing, the X-ray data do not show such a big change. It must be pointed out that the experimental procedure used in the X-ray diffraction tests does not allow any reliable quantitative analysis of the extent of reaction. Moreover, other investigators have shown that much higher stabilizer concentrations and longer curing periods are required to affect the intensities of the basal reflections of the clay (9, 14).

Addition of the strong alkali additive sodium hydroxide accelerates the decomposition of the clay and also encourages the earlier formation of CSH. X-ray diffraction patterns (Table 7) show the presence of the 3.03 Å line in the 1-day sample; this line also tends to increase in intensity with curing time. Figure 9 also indicates that addition of caustic soda decreases the initial consumption of lime. This is due to the high alkalinity of the additive which depresses the solubility of calcium hydroxide and therefore retards the rate of reaction of the calcium ions and the clay. However, the total consumption of lime after 64 days of cure is much higher in the mixes with additive.

As noted before, sodium metasilicate not only is a strong alkali additive capable of attacking the soil minerals, but also provides additional reactive silica to the system which would greatly accelerate the formation of calcium silicate hydrates. The X-ray diffraction patterns summarized in Table 7 show the development of 11 and 10 Å lines, as well as the familiar 3.03 Å line of the CSH. The lime and kaolinite endotherms are reduced even more as compared to the caustic-treated samples, particularly in the early ages of cure. As estimated from the DTA peaks and as revealed by the X-ray patterns, no lime can be detected after 8 days of reaction.

The salt sodium sulfate produces a similar effect on the kaolinite-lime reaction as do the other two additives but has somewhat less influence on the kaolinite endotherm. In these mixtures, the formation of Stratling's compound is questionable.

<u>Kaolinite-Tricalcium Silicate Series</u>.—In the kaolinite-tricalcium silicate mixes, as in kaolinite-calcium hydroxide samples, hydrous calcium silicates of various compositions are the major reaction products (Table 8). Judging from the relative intensity of the dehydration endothermic reaction of calcium hydroxide and the X-ray data, it appears that the lime liberated during the early hydration of tricalcium silicate combines with the clay mineral more rapidly than in the quartz-C_3S mix.

As in the kaolinite-lime series, the three sodium additives appear to intensify the reaction between the clay mineral and the stabilizer and also to produce a larger quantity of reaction products, mainly calcium silicate hydrates. The X-ray data given in Table 8 indicate that the various hydrous calcium silicates formed in the additive-treated samples are of lower lime-silica ratio than those in the additive-free samples. In the latter, the lime-silica ratios in almost all CSH are close to a molar ratio of 1.

As shown in Figure 10, the amplitudes of the kaolinite dehydration endotherms are considerably smaller than those of the kaolinite-calcium hydroxide samples at all curing ages. This could be due to more intensive reaction between the clay and the C_3S stabilizer or to a greater fluxing effect because of the different reaction products. However, it should be pointed out that the thermal behavior of these mixtures gives consistent trends for various treatment as in the kaolinite-lime series.

TABLE 8

X-RAY ANALYSES OF KAOLINITE-TRICALCIUM SILICATE MIXTURES

Spacing (Å)	Relative Intensity													Identification
	Dry Mix	No Additive			Sodium Hydroxide			Sodium Metasilicate			Sodium Sulfate			
		1-Day	4-Day	64-Day	1-Day	4-Day	64-Day	1-Day	4-Day	64-Day	1-Day	4-Day	64-Day	
13.8	-	2		2				-	-	-	-	-	-	$CSH_{1.5}$
12.8	-	-		-	2d	1d		1	-	-	-	-	-	CSH, C_2ASH_X
11.8	-	-	1bd	- 2d				-	-	-	-	-	-	CSH
11.0	-	2		2				-	1	-	2	2	3	CSH
10.0	-	2		2		3	2	2	2	2	2	2	2	CSH (I)
8.9	-	1		1	3	3	2	1	2	2	-	-	-	CS_2H_2
7.1	10	10	10	10	10	10	10	10	10	10	10	10	10	Kaolinite
4.18	4b	6b	5b	7b	5b	6b	4b	5b	8b	5b	5b	4b	6b	Kaolinite, C_2ASH_X
3.05	-	vw	1	1	-	-	-	-	-	-	-	-	-	C_2SH
3.04	-	-	-	-	-	-	-	-	-	-	-	-	-	C_2SH (C)
3.03	-	-	-	-	1	1	1	vw	1	1	-	-	-	CSH (I)
3.01	1	-	-	-	-	-	-	-	-	-	1	vw	-	C_3S
2.60	1	2	1	-	1	2	-	-	-	-	-	-	-	C_3S

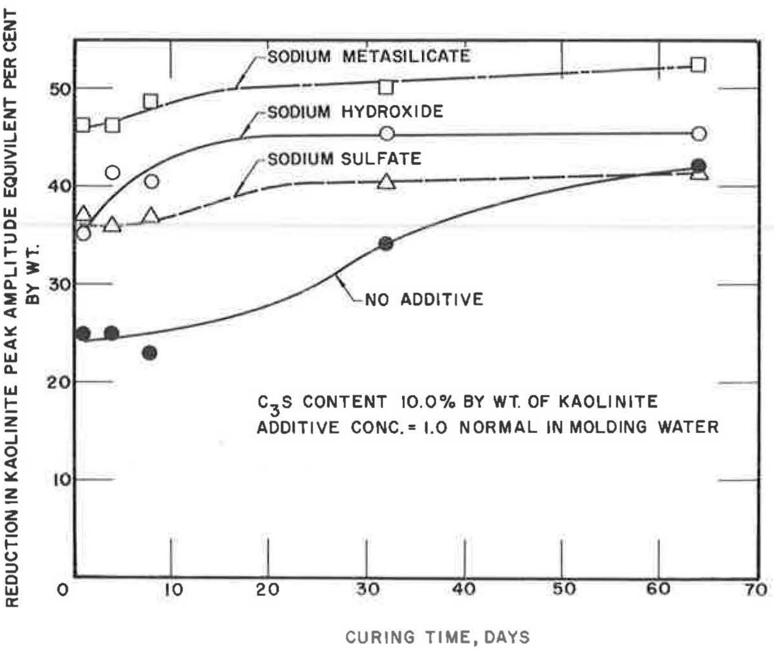


Figure 10. Effect of curing time on DTA peak of kaolinite in K-C_3S mixes.

Extent of Reaction.—The DTA results obtained in this study reveal that the major thermal reaction peaks of the two mono-mineral soils decrease considerably after curing with calcium stabilizer, with or without sodium additive. This apparent change in the thermal behavior of the mixture indicates that certain reactions or interferences must have occurred among the constituents. However, the DTA records only the net heat effect which may result in a seriously distorted thermogram when several reactions proceed simultaneously. Martin (24) has reported anomalous thermal behavior carbonates. Therefore, it is necessary to verify the observation that the disappearance of the mineral constituent, as indicated by the reduction in the intensity of its thermal reaction, is due to true reaction with other constituents in the mixture and not to merely a flux effect. Three series of tests have been made in an attempt to evaluate the extent of reactions of the soil minerals with other constituents.

Quartz-Acid Digestion Tests.—Investigators at the U. S. Bureau of Reclamation (7) have reported that no reaction was evidenced between calcium hydroxide and quartz by either X-ray or DTA. On the other hand, Diamond, White and Dolch (8) reported complete reaction of quartz mineral in clay size with a large excess of calcium hydroxide (400 percent based on the weight of the quartz). Therefore, it is important to confirm the reactions observed in the present investigation between the lime and quartz are true reactions, not just surface absorption phenomena, particularly since in the present investigation the ratio of calcium hydroxide and quartz was much lower than in any of the other studies.

In this study, hydrous calcium silicates are identified as the principal reaction products. These compounds are alkali in nature and thus will dissolve in strong acid solution. A series of experiments were conducted by digesting three samples, approximately 9 g each in 100 ml of 1.0 N hydrochloric acid at 70 C for one hour with intermittent agitation. The three samples tested were a pure quartz, an 8-day cured quartz-lime mixture and a 4-day cured quartz-C_3S-NaOH mix. After the acid digestion, each sample was washed with distilled water five times and then oven-dried and weighed. The weight lost due to this treatment should be an indication of the extent of reaction between the quartz mineral and the stabilizer. The results in Table 9 show that the pure quartz sample has lost only 0.1 percent of its original weight after the acid treat-

TABLE 9

ACID-DIGESTION TESTS OF QUARTZ MIXTURES[a]

Stabilizer (%)	Additive (%)	Weight (g)		Wt Loss (% of total wt.)	Loss of Quartz (%)	Loss of Quartz From DTA[b] (%)	Curing Days
		Before Test	After Test				
None	None	9.94	9.93	0.1	0.1	0	0
10 $Ca(OH)_2$	None	9.04	8.03	11.8	3.0	1.5	8
10 C_3S	0.97 NaOH	8.58	7.45	13.2	4.0	8.5	4

[a] Acid-digestion test: sample after curing mixed with 100 ml of 1.0 N HCl, placed in 70 C water bath for 1 hour, then washed with distilled water 5 times, oven dried at 105 C; all percentages, except where noted, based on weight of quartz.

[b] Estimated from the change in quartz endothermic peak amplitude.

ment, which is certainly within the experimental accuracy of this test. The lime-treated and the C_3S-NaOH-treated samples have lost 3.0 and 4.0 percent of quartz by weight, respectively. The amount of quartz lost in the C_3S-NaOH-treated sample as determined by the acid-digestion test is only about half of the amount estimated from DTA curves. This further substantiates the previous conclusion that in cement-treated samples, part of the quartz lost in the DTA records has not been truly reacted with the calcium stabilizer. All these observations (the DTA, X-ray and acid-digestion test) tend to suggest that the nature of reactions occurring in lime-stabilized and cement-stabilized quartz mixtures may be quite different.

Thermal Reactions of Mixtures of Kaolinite and Hydrated Cementing Compounds.—Since in a kaolinite-calcium stabilizer system, with or without sodium additive, the major constituents are the raw ingredients and their hydration products, they are principally hydrous calcium silicates and hydrous calcium aluminates. Two mixtures of kaolinite and partially hydrated cementing compounds have been prepared for DTA investigation. The cementing compounds (C_3S and portland cement) were hydrated in paste form with a water-solid ratio of 0.7 by weight. Figure 11 shows the thermograms of (a) 50 percent kaolinite and 50 percent oven-dried 32-day hydrated tricalcium silicate, and (b) 57 percent kaolinite plus 43 percent 4-day as-cured hydrated cement. Comparison with the thermograms of their constituents (the clay and the partially hydrated cementing compounds) shows no anomalous thermal behaviors. These test results lead to three possible explanations for the large reduction in kaolinite thermal peak observed in the thermograms:

1. The loss of kaolinite after curing as identified by the thermal behavior is due to true decomposition of the clay mineral. However, the missing kaolinite underwent significant changes in its thermal behavior without complete destruction of its crystalline structure. Several other investigators (9, 14) have reported that a much higher stabilizer content and longer curing period are required to cause any significant effect on the basal reflections of this clay mineral.
2. The reaction products responsible for the fluxing effect are different from those added in these two tests.
3. The fluxing effect is different when the reaction products are mixed in separately from what occurred in the mixture.

Reactions between Montmorillonite and Calcium Hydroxide.—The test results indicate that the reactivity (reactivity of the mono-mineral soils is compared on the basis of the amount of the mineral participating in reaction with the stabilizer in a particular system) of the clay mineral kaolinite is much higher than that of quartz. Obviously, therefore, much stronger reaction could be anticipated when montmorillonite is the soil component. Since the basal spacing of montmorillonite crystals will expand by glycerol treatment and collapse by heating, these characteristics are thus utilized to study the changes in the character of the soil component due to stabilization.

Four mixtures were prepared for X-ray and DTA tests: (a) a pure montmorillonite, (b) montmorillonite plus 10 percent calcium hydroxide dry mix, (c) montmorillonite plus 10 percent calcium hydroxide humid cured for 1 and 8 days, and (d) montmorillon-

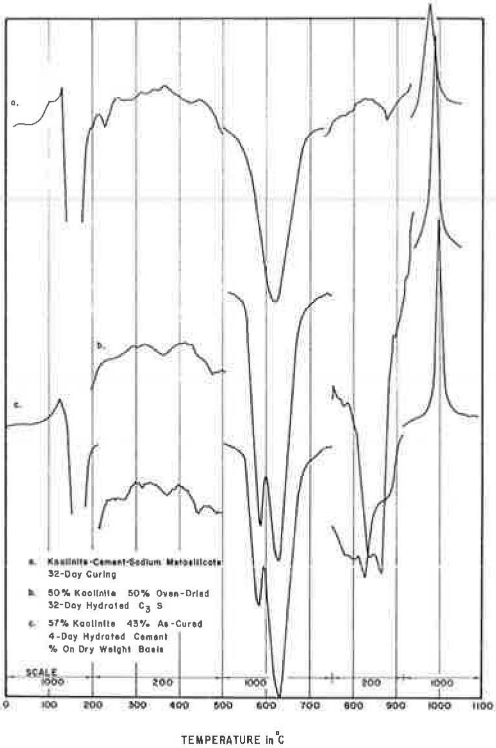


Figure 11. Thermograms of kaolinite-hydrated cementing compound mixes.

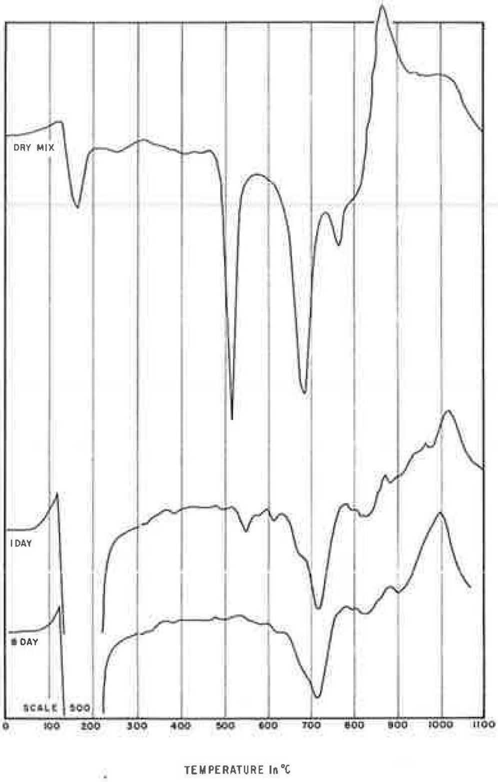


Figure 12. Thermograms of montmorillonite-calcium hydroxide mixes.

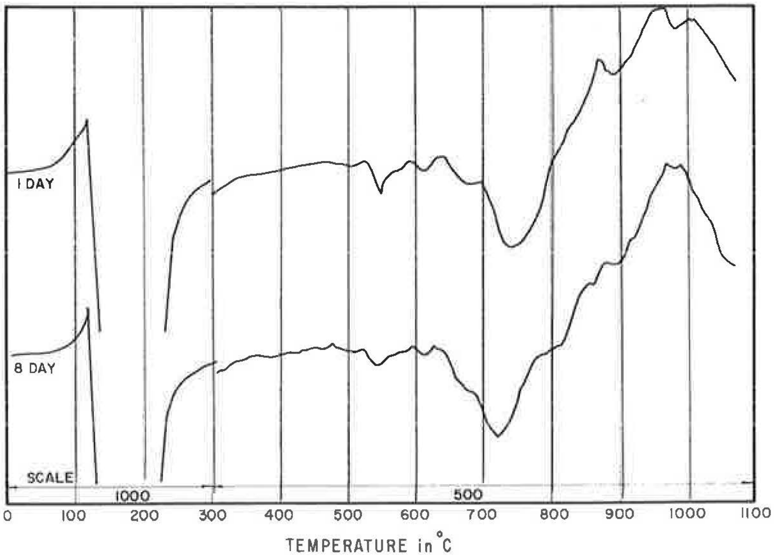


Figure 13. Thermograms of montmorillonite-calcium hydroxide-sodium hydroxide mixes.

TABLE 10

d_{001} X-RAY SPACING OF MONTMORILLONITE-CALCIUM HYDROXIDE MIXTURES[a]

Treatment Temperature (C)	Montmorillonite	Montmorillonite-Lime			Montmorillonite-Lime-Sodium Hydroxide	
		Dry Mix	1-Day	8-Day	1-Day	8-Day
25[b]	18 vvs[b]	18 vvs	18 m 14 vs	14 vvs	18 vvs 14 w	18 vs 14 vs
400	10 vs	10 vs	10 vs	10 vs	10 vs	10 vs
600	10 vs	10 vs	10 vs	10 vs	10 vs	10 vs
700	10 vs	10 s	10 m	10 vvw	10 m	10 gone
800	10 s	10 s	N.D.	10 gone	N.D.	10 gone
900	VW Pattern	10 vw	N.D.	10 gone	N.D.	10 gone

[a] Numbers refer to d_{001} spacing in angstroms, and letters refer to relative intensity; d values are approximate; N.D. indicates no data.

[b] Glycerol treated.

ite plus 10 percent calcium hydroxide and 1.0 N sodium hydroxide solution cured for 1 and 8 days.

A portion of each mixture was first analyzed by DTA and X-ray as for all other mixes. Another portion of the sample was heated in the DTA furnace to various temperatures at the same heating rate as for DTA, i.e., 12.5 C/min. Oriented aggregate specimens of the heat-treated samples were then prepared for X-ray examination. The specimens were mounted in a horizontal position to enhance the 001 lines.

Figure 12 shows the thermograms of montmorillonite-lime mixes and Figure 13 shows the thermograms of NaOH-treated montmorillonite-lime mixtures. Even the dry mix shows evidence of reaction between the clay and lime. This reaction must occur during the heating process. The 725 C endothermic peak of the montmorillonite is lowered to 680 C, a second endothermic peak appears at 760 C, and the exothermic reaction of the clay is lowered from 930 to 860 C. The shifting of the thermal peak temperatures could, nevertheless, be due to some fluxing effect. However, reaction between the clay and lime after curing is apparent and intensive. The lime endotherm practically disappears after only 1 day of cure. Comparison of the thermogram of the 8-day specimens with and without NaOH indicates some difference between the reaction produced by lime alone and that by lime plus caustic soda. Furthermore, there appear to be more reaction products produced with time and with additive; both the DTA and X-ray data are consistent in this respect.

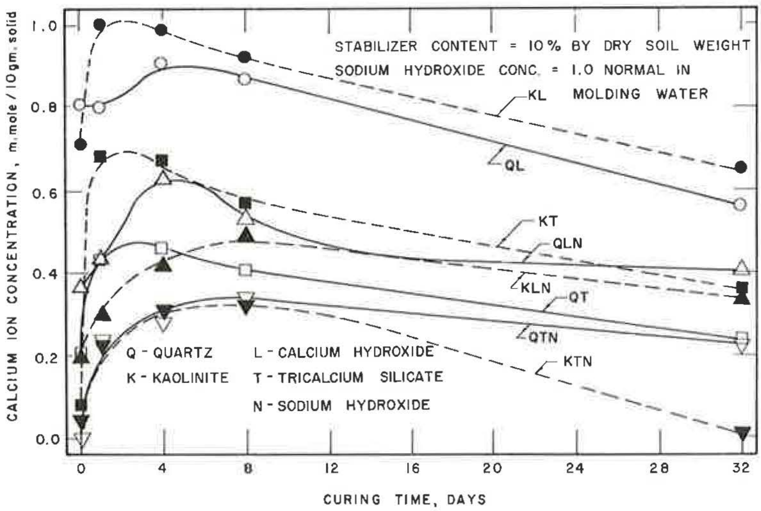


Figure 14. Calcium ion concentration in aqueous extracts of stabilized soil mixtures.

Table 10 summarizes the most obvious and important differences of X-ray data of these mixes. Variations in the basal expansion (glycerol treated at room temperature) give consistent trends for the different treatments and must be real. The reasons for these variations are not clear but they probably reflect the structural differences of various reaction products. Apparently, sodium hydroxide alters the reaction between the clay and lime because 1-day cured specimens in the absence of additive give 14 Å as the strongest line, whereas the caustic-treated 1-day specimen gives 18 Å as the strongest line. Addition of the strong alkali may retard the formation of the reaction product obtained from montmorillonite and lime.

The remaining X-ray data show the effect of the reaction due to stabilization on the decomposition temperature of the montmorillonite mineral. The clay-lime reaction lowers the clay decomposition temperature and addition of sodium hydroxide lowers it still further. Thus, it can be concluded that clay-lime reaction produces real marked changes in subsequent thermal behavior of the mixture and sodium additive further alters the reaction.

Chemical Analyses of Aqueous Extracts

As illustrated by the calcium ion concentrations in the aqueous extracts of various stabilizer-mineral mixtures in Figure 14, the trends of variation of the ion concentration with curing time are similar for the various soil-stabilizer systems. Furthermore, the effect of any one of the sodium additives on the extractable ion concentration is generally the same, regardless of the soil mineral and the kind of the calcium-bearing raw ingredient. Figure 14 also shows the effect of sodium hydroxide on the calcium ion concentration. The following presentation is a generalized summary of salient results with specific reference to one mixture, the kaolinite-tricalcium silicate series.

Soil Mineral-Cement Mixes.—In a mineral-cement mix, any calcium ions liberated from the cement (in the particular case discussed here, tricalcium silicate), or directly from the lime, during hydration and hydrolysis react rapidly with the silicates and aluminates from the soil mineral to form insoluble products. Therefore, the amount of extractable silica (Si) and alumina (Al) will be low throughout the curing period. Figure 15a shows the results obtained from the analyses of the aqueous extracts of kaolinite-tricalcium silicate mixtures. Since the clay contains some sodium (Na) in its natural state, the Si concentration (and possibly that of Al) is somewhat higher initially due to the higher solubility of the sodium silicates (or aluminates).

The extractable calcium (Ca) is the portion of the lime Ca in excess of that reacting with Si and/or Al. Therefore, it is apparent that the amount of extractable Ca at any given time is controlled by the balance between the rate of release of Ca and the rate of Ca consumption by precipitation to form insoluble silicates and/or aluminates. The former is determined by the rate of cement hydration and the pH of the system, and the latter depends on the nature of the soil in the system. Thus, the Ca concentration in extract, as shown in Figure 15a, is low at the start, increases to a maximum after a short period of cure, and then decreases slowly to nothing with time. This variation of the extractable Ca concentration follows the same trend as the variation of the amount of lime present in the mixture as shown in the DTA curves in Figure 10.

The reaction scheme of the foregoing process can be represented in a simplified fashion by the following equations:

$$C_3S + H_2O \longrightarrow C_3S_2H_X \text{ (hydrated compounds)} + Ca(OH)_2 \tag{2}$$

$$Ca(OH)_2 \longrightarrow Ca^{++} + 2\,(OH)^- \tag{3}$$

$$Ca^{++} + 2\,(OH)^- + \begin{array}{l} SiO_2 \text{ (soil silica)} \longrightarrow CSH \\ Al_2O_3 \text{ (soil alumina)} \longrightarrow CAH \end{array} \tag{4}$$

$$Ca^{++} + \begin{array}{l} NS \text{ (soil silicate)} \longrightarrow CSH + Na^+ \\ NA \text{ (soil aluminate)} \longrightarrow CAH + Na^+ \end{array} \tag{5}$$

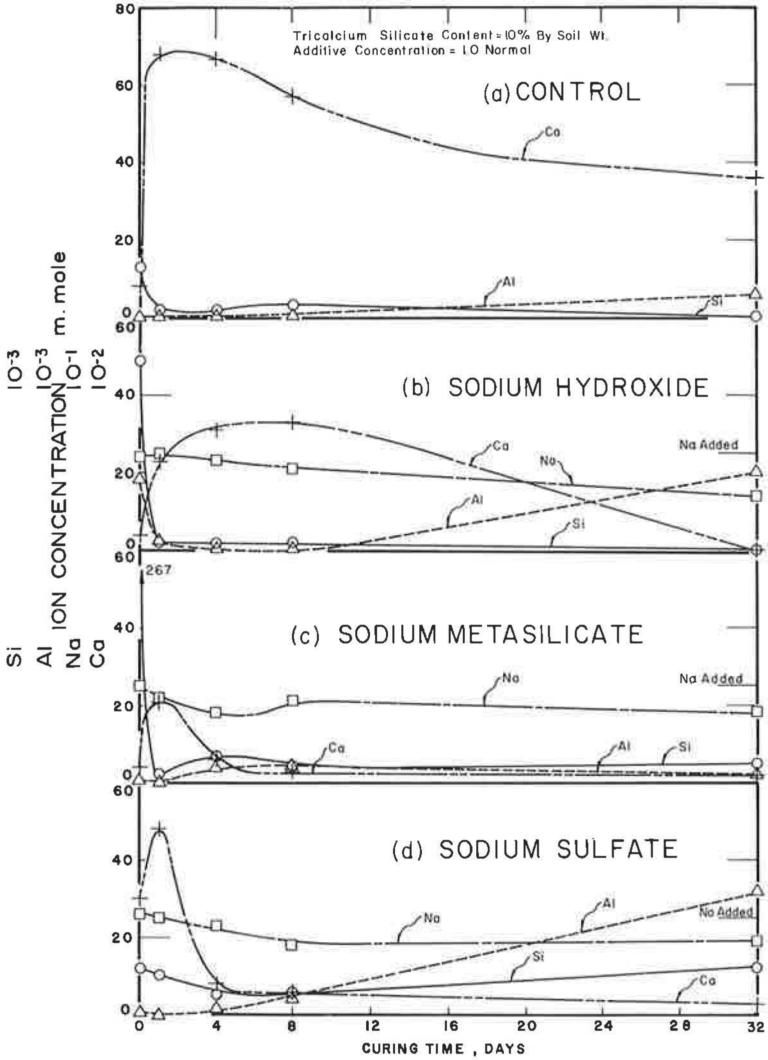


Figure 15. Composition of aqueous extracts of kaolinite-tricalcium silicate mixes.

These equations are for illustration only; therefore, they are not necessarily in equilibrium, and the symbols used are not necessarily the correct ones. When lime is the initial Ca-bearing raw ingredient, Reaction 2 is bypassed. As Reactions 2 and 3 proceed, Ca in solution increases; as Reaction 4 proceeds, Ca decreases; and as Reaction 5 progresses, extractable Ca decreases with the increase in extractable Na. The last reaction occurs only when the soil mineral contains considerable amount of Na in its natural state.

Effect of Sodium Hydroxide.—A comparison of Figure 15b with Figure 15a indicates that the addition of sodium hydroxide to a mineral-cement system causes the following reactions:

1. Suppression of Ca concentration in the pore fluid by the common ion effect, and
2. Acceleration and intensification of the solubilization of Si and Al as the hydroxyl (OH) is consumed by the reaction

$$Na^+ + OH^- + SiO_2 \longrightarrow NSH \text{ (soluble)} + H_2O \qquad (6)$$

With curing, Reactions 2 and 3 proceed and are followed promptly by

$$\begin{aligned} Ca^{++} + NSH &\rightarrow CSH + Na^{+} \\ &\text{or } CNSH + Na^{+} \end{aligned} \tag{7}$$

Initially at zero cure, due to the common ion effect, the Ca ion concentration in the pore fluid is much lower than in fluid without caustic. As curing progresses, the suppression of Ca in the pore fluid is reduced due to the consumption of (OH) in Reaction 5; in addition, the cement continues to hydrate to release calcium hydroxide, thereby increasing the Ca concentration. Later in cure, soluble silicate is precipitated as insoluble product with Ca, and also the Ca reacts with the soil silicates; thus, the extractable Ca concentration decreases with time, as shown in Figure 15b.

The Si concentration in the extract of cured specimens is limited by the solubility of calcium silicate. However, it is high initially due to the immediate solubilizing effect of the caustic.

Early in cure, the sodium primarily associates with soluble silicates and aluminates as indicated by the extractable Na content which is equal to that added. With curing, the soluble Na:Ca ratio increases as Ca is decreased by precipitation. This favors the formation of mixed Na-Ca silicates. Thus, the extractable Na is high early but lower later.

Inasmuch as the solubility of calcium aluminate is probably higher than that of calcium silicate, if calcium is consumed as insoluble silicates, any aluminate must end up as soluble salt. Hence, there is rapid increase in the extractable Al content with decrease in extractable Ca and Si.

Effect of Sodium Metasilicate.—With sodium metasilicate as an additive, the preliminary step of solubilization of silica is bypassed. Therefore, whatever Ca is available from cement can precipitate immediately with $SiO_3^{=}$ present in the pore fluid, as shown by the very low Ca content at zero cure immediately after mixing in Figure 15c. Also, the initial extractable Si concentration is much less than the amount of soluble silicate added. As the soluble silicate (from the additive) in the pore fluid is insolubilized by the Ca, hydroxyl attack of the soil silicates takes place. However, the low solubility of calcium silicate reduces the Ca concentration in the pore fluid, and thus tends to delay the transport of Ca from cement to soil. Nevertheless, the rate of consumption of free Ca should be much faster in the mix with sodium silicate than with sodium hydroxide, as shown by the lower Ca concentration in the former mix (Fig. 15c) compared to the latter (Fig. 15b) at same age of cure.

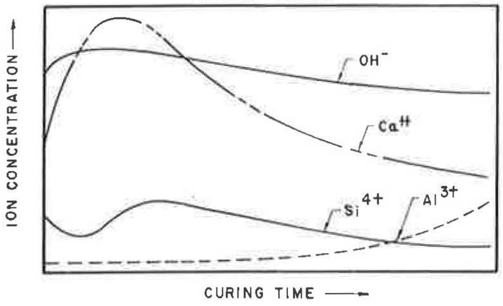
Effect of Sodium Sulfate.—On addition of sodium sulfate to the soil-C_3S system, the following caustization reaction takes place after the cement hydration process shown by Eq. 2:

$$Na_2SO_4 + Ca(OH)_2 \rightarrow CaSO_4 + NaOH \tag{8}$$

Since the silica-attack caustic is formed by this reaction, the rate of attack of the soil silicate or aluminate will be less rapid at start than that with sodium hydroxide, and thus less soluble sodium silicate will be formed as illustrated by Eq. 6.

Because of the higher solubility of calcium sulfate and lower alkalinity of the system compared with the caustic-treated mixtures, the initial Ca concentration in the extract is higher and the initial Si concentration is lower. Soon afterwards, hydroxyl attack of soil silicates takes place, greatly aided by the solubilizing action of the sodium ions. Since the Ca concentration in the pore fluid is high initially, the rate of transport of the Ca from cement to soil silicates is markedly accelerated as compared to that in the sodium hydroxide mixes. Therefore, the free Ca in cement rapidly depletes and the extractable Ca content drops rapidly with curing. This is indicated by the Ca concentration after 4 days of cure in Figure 15d.

(a) SOIL- CEMENT



(b) SOIL-CEMENT- SODIUM ADDITIVE

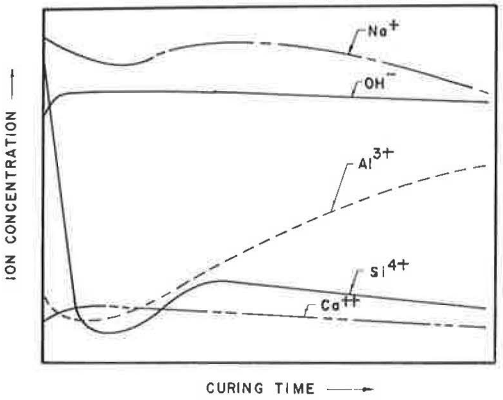


Figure 16. General variation of composition of aqueous extracts of soil-cement mixes.

SUMMARY AND CONCLUSION

This paper describes an experimental investigation aimed at delineating the nature of reactions accompanying the stabilization of soils with cementing agents and elucidating the mechanisms by which a group of alkali metal compounds improves the stability of stabilized soils. Mixtures of two mono-mineral soils (quartz and kaolinite), three cementing compounds (calcium hydroxide, tricalcium silicate and portland cement), and selected sodium additives (hydroxide, metasilicate and sulfate) were examined during the course of cure. The solid phases were examined by employing X-ray diffraction and DTA techniques. The pore fluid phases were analyzed for soluble calcium, sodium, silica and alumina contents. The following summarizes some of the salient results and conclusions:

1. Both mono-mineral soils react with the three calcium stabilizers under conditions of approximately 100 percent RH and room temperature. Addition of sodium additives greatly intensifies the reaction between the soil and the stabilizer and increases the abundance of reaction products.

2. The reaction patterns between any one soil mineral with the three calcium stabilizers are generally the same, the effect of various sodium additives on the reactions is also similar, regardless of the kind of initial calcium-bearing ingredient. The major differences are more in the relative amounts of the various reaction products and the rate of their formation than in the formation of entirely different products.

3. The reactivity of the two mono-mineral soils to any one of the cementing compounds is higher in kaolinite than in quartz.

4. With one particular mineral, the reaction is most intensive when the stabilizing agent is tricalcium silicate and least intensive with calcium hydroxide.

5. The principal reaction products produced in quartz mixtures are hydrous calcium silicates (CSH) of various compositions with lime-silica ratio varying from 0.2 to 1. In kaolinite mixes, the principal products are hydrous calcium silicates, hydrous calcium aluminates and a mixed calcium aluminosilicate.

6. The reaction scheme of a soil-stabilizer system and the effect of sodium additive can be represented by the following simplified equations:

Soil-cement

$$\text{Cement} + H_2O \longrightarrow \text{CSH} + Ca(OH)_2$$

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

$$\begin{aligned} Ca^{++} + 2\,(OH)^- + {} & SiO_2 \text{ (soil silica)} \longrightarrow \text{CSH} \\ & Al_2O_3 \text{ (soil alumina)} \longrightarrow \text{CAH} \end{aligned}$$

Soil-cement plus sodium additive

$$\text{Cement} + H_2O \longrightarrow \text{CSH} + Ca(OH)_2$$

$$Na_2X + Ca(OH)_2 \longrightarrow CaX + 2\,Na^+ + 2\,(OH)$$

$$2\,Na^+ + 2(OH)^- + SiO_2 \text{ (soil silica)} \longrightarrow NSH$$
$$Al_2O_3 \text{ (soil alumina)} \longrightarrow NAH$$
$$NSH + Ca^{++} \longrightarrow CSH + Na^+ \text{ or } CNSH + Na^+$$
$$NAH + Ca^{++} \longrightarrow CAH + Na^+$$

7. All sodium additives depress the Ca concentration in the pore fluid but do not decrease the hydroxyl availability, and may actually increase it.

8. The variations of the ionic composition in the solution phase of a soil-cement system with curing age can be simplified as shown in Figure 16a. Figure 16b illustrates the effect of sodium additive on the extract composition.

9. The rate of reaction between the soil mineral and stabilizer depends on the nature of the sodium additive and the initial calcium-bearing ingredient.

10. The sodium in the additive-treated mixture primarily associates with soluble constituents early in cure, and later with insoluble mixed sodium-calcium silicates (or aluminates). The amount of sodium retained in the insoluble compounds after 32 days of cure decreases in the order of initial sodium additive, hydroxide, sulfate and metasilicate.

ACKNOWLEDGMENTS

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Soil-Cement Construction Using Loess Soil

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Construction of approximately 26 miles of soil-cement base composed entirely of cement, water and loess soil is reported. Two projects are involved; the first, on Iowa 37, was built in 1961, and the second, on Iowa 333, in 1963. Information obtained during the construction of Iowa 37 brought about changes in construction procedure and specifications, including: (a) changing the required density from 90 to 95 percent of the maximum density as determined by AASHO Designation: T 99; (b) eliminating the 0.05-ft tolerance on the finished base; (c) disallowing use of a spike drag; and (d) changing surface treatment from a single to a double bituminous seal. Data relative to routine and special testing are included. A comparison is made between the results of freeze and thaw testing of laboratory specimens and cores taken from the 12 experimental base sections on Iowa 37. A similar comparison is made for compressive strength.

•THE WESTERN PART of Iowa contains an area in which granular materials suitable for highway construction are not readily available from local sources. This area is covered almost entirely by a thick deposit of loess soil.

The stabilization of Iowa loess with portland cement was investigated by Handy, Davidson and others (1, 2). Their studies strongly suggested that the loess soil of western Iowa can be used effectively for soil-cement base construction. This favorable conclusion was a major factor in the decision to use soil-cement on two pavement projects constructed in western Iowa during the past few years.

This report is concerned with the general design and construction procedures and problems, and with laboratory and field data obtained in connection with the soil-cement construction.

DESIGN

The first of the two soil-cement bases was constructed on 12.8 miles of Iowa 37 in 1961; the second was constructed on 13 miles of Iowa 333 in 1963. Location of the projects is shown in Figure 1.

On both projects, the soil-cement base is 7 in. thick and 23.5 ft wide. No subbase was constructed. Design requirements included placement of a 3-in. hot-mix asphalt concrete mat 1 to 3 years after completion of the base. An interim surface consisting of a single bituminous surface treatment was placed on Iowa 37; a double surface treatment was used on Iowa 333.

The design cement content of the base was determined from the results of freezing and thawing tests of compacted soil-cement mixtures made according to AASHO test method T 136-57. The maximum permissible loss during 12 cycles of freezing and thawing was set at 10 percent, which is the maximum recommended by the Portland Cement Association for A-4 soil (3).

Paper sponsored by the Committee on Soil-Portland Cement Stabilization and presented at the 44th Annual Meeting.

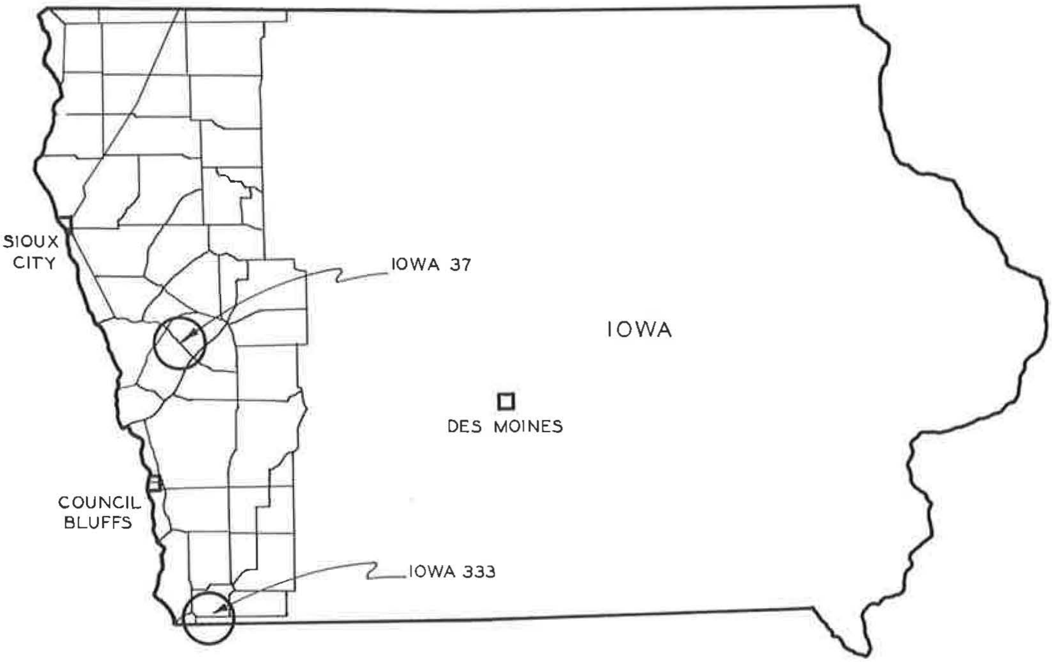

Figure 1. Location of soil-cement projects.

Figure 2 contains curves relating weight loss to the number of freeze-thaw cycles for compacted mixtures of soil from the Iowa 37 borrow area and four different amounts of cement. Similar curves were prepared for mixtures containing the soil used in the base on Iowa 333.

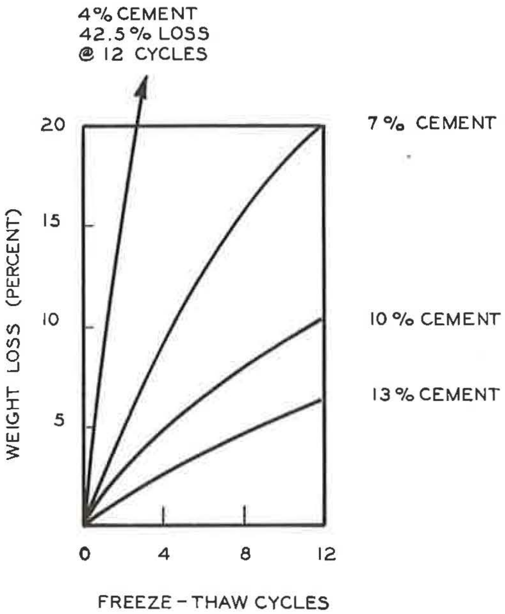

Figure 2. Soil-cement design curves, Iowa 37.

The design cement content for the base on Iowa 37 was set at 11 percent of the dry weight of the soil; for Iowa 333 it was set at 10.75 percent. Iowa 37 also contains 12 experimental base sections in which the cement content was specified to be 7, 9, 11 or 13 percent.

MATERIALS

The design for both projects required that the soil for the base be obtained from borrow areas selected and purchased by the state. Classification of the borrow soil varied from A-4(8) to A-6(10). Table 1 shows additional soil data.

TABLE 1

SOIL DATA INFORMATION[a]

Soil Characteristics	Iowa 37	Iowa 333
Percent passing No. 200 sieve	98-100	99-100
Percent 5-μ clay	18- 24	20- 28
P.I.	9- 15	10- 16

[a] Type I cement was used in both projects.

CONSTRUCTION

The fact that the base contained soil from borrow areas dictated many of the construction procedures. In each project the soil was obtained from a single borrow area located adjacent to the roadway, and a stationary mixing plant was set up at that location.

Although separated in time by 2 years, the Iowa 37 and Iowa 333 pavements were built by the same contractor. To a large extent, the same equipment was employed on both projects. Construction procedures were similar, but those used on Iowa 333 reflected some changes which were made in an effort to overcome the finishing problems encountered on Iowa 37.

The soil-cement was mixed in a continuous flow-type pug mill. The mixture was delivered to the grade in trucks and was placed by two Jersey spreaders. The spreaders were mounted on crawler-type tractors equipped with scarifying teeth to break up the material compacted by the tracks. Compaction was accomplished with sheepsfoot rollers, pad-type vibrators and pneumatic rollers. On the first project, Iowa 37, the contractor attempted to obtain the required compaction with pad-type vibrators alone. This proved unsuccessful; however, these vibrators were used effectively to compact the sloping edge of the base. On Iowa 333, the contractor began operations with a regular sheepsfoot roller and a vibrating sheepsfoot roller. Use of the vibrating roller was discontinued after the first few days because it did not compact to the required density and because it had large, smooth feet which caused compaction planes in the base.

On Iowa 37 the base was cured with RC-0 or MC-3 applied not later than 24 hours after completion. The application rate was 0.25 gal/sq yd. On Iowa 333, the material used for curing was MC-0 or MC-1 at the rate of 0.2 gal/sq yd. On both projects it was necessary to spread blotter sand on the surface before allowing limited traffic at the end of 7 days.

DISCUSSION

Accepted practices, such as those described in PCA publications (3, 4), were generally followed in the design and construction of the soil-cement base. The Iowa 37 project was purposely experimental, whereas Iowa 333 was planned as a standard highway improvement representative of the type of soil-cement construction possible in areas of the state where loess soil is prevalent. The experience and data gained from both projects has proven valuable, although much remains to be done before optimum design and construction procedures can be prescribed.

Soil Pulverization

PCA (4) reports that most specifications require that a minimum of 80 percent of the soil-cement mixture pass a No. 4 sieve, exclusive of any gravel or stone; the final pulverization test is made at the conclusion of mixing operations.

The specifications for Iowa 37 and Iowa 333 required that at least 80 percent of the soil pass a No. 4 sieve before the addition of the cement. Since the cement content was 11 percent of the dry soil weight, this specification was equivalent to requiring that 82 percent of the soil-cement mixture pass the No. 4 sieve.

Pulverization of the soil for Iowa 37 was not a serious problem. The top 1 ft of soil in the borrow area was removed and wasted. The underlying soil was disced and then pushed by a bulldozer into a bulkhead feeder. Large lumps of soil were either broken or removed by a single vibrating screen. The in-place moisture ranged from 16 to 19 percent. This was reduced about 2 percent by aeration during discing.

Pulverization of the soil for Iowa 333 proved to be difficult. The difficulty may have been caused by the slightly larger clay content of this soil plus a greater in-place moisture content, which was found to be as high as 25 percent. The contractor tried several combinations of mechanical processing and aeration in his efforts to pulverize the soil. The most successful method involved making several passes with a P & H stabilizer in the borrow area, but even following this treatment, only about 70 percent of the soil would pass the No. 4 sieve. It was found that the soil was most friable when it contained about 18 percent moisture, and that extensive processing and drying promoted the formation of firm clay balls which would not break up in the mixer.

The effect of the degree of pulverization was reported by Felt (5) in 1955. He concluded that the quality of silty and clayey soil-cement mixtures was highest when 100 percent of the soil (exclusive of gravel and stones) was pulverized to pass a No. 4 sieve. He further stated, however, that the quality was not seriously affected by as much as 30 percent of unpulverized soil, provided the soil lumps were at optimum moisture or slightly above when compacted.

The purpose of pulverization is to insure an intimate mixture of soil grains and cement. The end result is a product of both the initial pulverization and the efficacy of the mixing operation.

Mixing

The soil-cement for both Iowa 37 and Iowa 333 was mixed in a 10-ft, twin-screw pug mill. Production varied from 150 to 180 tons of mixture per hour. To attain this rate, the mixing time was limited to about 15 sec. This may not have been the optimum length of time; PCA (4) reports that a 30-sec mixing time is customary.

A pertinent question is whether or not soil-cement containing fine-grained, cohesive soil can be mixed effectively in a pug mill type mixer. It was observed that the mixing action was considerably influenced by the initial moisture content of the soil and that balls forming where the soil and cement were introduced into the mixer did not always break up during passage through the pug mill. These became coated with cement on the outside and thereafter seemed to act as individual aggregate particles.

British practice (6) is to require a single-pass mix-in-place plant for cohesive soils and to permit stationary plants only for mixing granular soils with low cohesivity. Experience thus far in Iowa does not dictate adoption of the British practice, but it does suggest that there is room for improvement in the mixing action of stationary plants for producing soil-cement from fine-grained soil.

Cement Content

The cement was introduced into the pug mill through a vane-type feeder. A major problem with the cement feed was caused by the fact that the cement feeder and the soil feeder were not interlocked. In spite of close attention by the contractor and the inspectors, the two feeders were not always started and stopped simultaneously.

The average cement content of the soil-cement mixture was determined by observing the total tons of soil-cement produced from one or more carloads of cement. This method is subject to error due to the difficulty of determining the precise beginning and end points for any particular carload of cement passing through the mixer. Nevertheless, it appears to be a reasonably reliable method for estimating the average cement content of the base mixture on normal construction projects.

On Iowa 37, the design cement content was 11 percent of the dry weight of the soil, except in the experimental sections. During construction of the non-experimental sections, 46 cement checks were made, each involving one or more carloads of cement. The results of these tests are summarized in Figure 3. The average of 46 tests was 11.32 percent with a standard deviation of 0.65 percent.

The design cement content of the base on Iowa 333 was 10.75 percent of the dry weight of the soil. There were no experimental sections and no planned variation in cement content. The results of the field checks for cement content are summarized in Figure 4. The average of 71 tests was 11.30 percent; standard deviation was 1.18 percent.

On the Iowa 37 project, only four of the 46 field checks indicated an average cement content different from the design amount (11 percent) by more than 1.0 percent. It was expected that the same degree of control would be exercised on Iowa 333. The results there were disappointing because 27 out of 71 checks showed an average cement content different from the design amount (10.75 percent) by more than 1.0 percent.

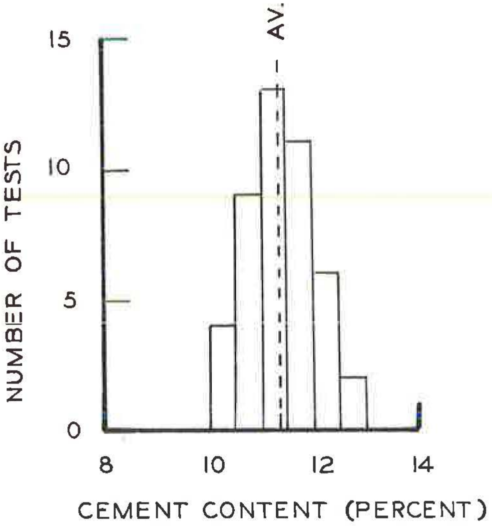

Figure 3. Field checks of cement content, Iowa 37.

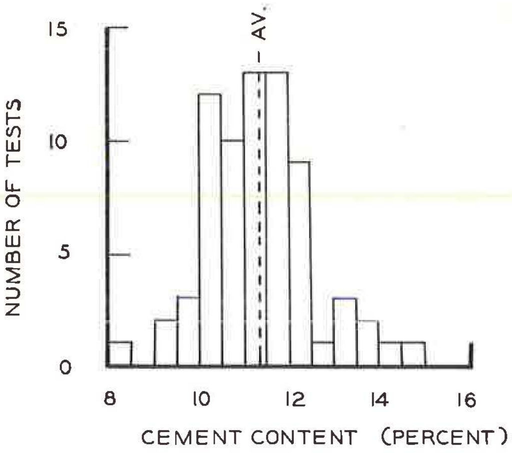

Figure 4. Field checks of cement content, Iowa 333.

It must be recognized that estimates of cement content made from the records of the materials passing through the plant are of little more than historical value. If the cement content is below the amount specified, it can be corrected only by drastic action, such as tearing up and replacing a portion of compacted base.

Before the construction of Iowa 37, a brief laboratory investigation was made of the California (6) and Washington (7) methods for measuring cement content. The California method is essentially a chemical titration procedure; the Washington method depends on the change of conductivity of water with the addition of cement. Neither method gave satisfactory results, perhaps because of the fine-grained nature of the soil. The possibility that the conductivity may be influenced by the presence of clay was suggested by LeClerc and Sandahl (7) in their report on the Washington method.

The principal objective of the experimental base construction on Iowa 37 was to observe the physical properties and performance of a soil-cement base containing quantities of cement other than the design percentage. The 12 experimental sections were scattered throughout the 12.8 miles of base construction.

The cement feed was calibrated to provide an intended cement content of 7, 9, 11 or 13 percent. Field checks were made as previously described, and cement content by chemical analysis was determined from samples sent to the laboratory.

The first cement tests by chemical analysis were made on samples taken from the pug mill during construction. The results varied considerably from the intended cement percentages and were suspected to be in error. It was demonstrated, however, by means of laboratory prepared samples that the chemical analysis test could give results accurate to 0.2 percent of the actual cement content. Consequently, when cores were later obtained from the experimental base sections, a portion of each core was analyzed for cement content.

Figure 5 compares for each experimental section the intended cement content, the cement measured by field checks, and the cement determined by chemical analysis. In 9 of the 12 experimental sections, the field check results showed a higher cement content than that obtained by analysis.

Density

The required density of Iowa 37 was 90 percent of maximum field density determined according to AASHO standard method T 134-57. It was customary for the plant inspector to determine maximum density at least once a day. Throughout most of the

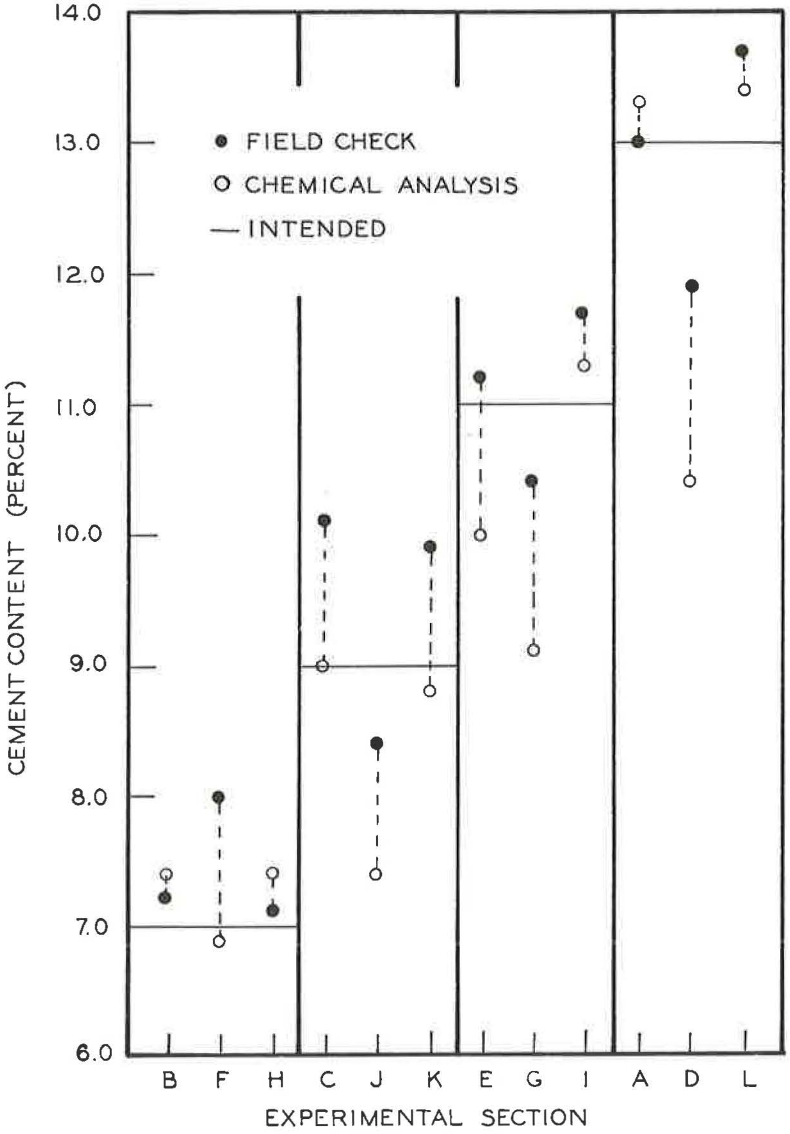

Figure 5. Comparison of intended cement content, field checks, and chemical analysis, Iowa 37.

job the maximum dry density was 98.6 pcf at an optimum moisture content of 21.8 percent.

The density of the compacted base was determined by the oil method. Figure 6 summarizes the results of tests by this method. The average of 121 tests was 93.5 percent of maximum density, with a standard deviation of 3.2 percent.

The density of cores drilled from the experimental base sections at the age of 5 or 6 days is shown in Figure 7. The average of 71 cores was 94.8 percent; standard deviation was 3.6 percent.

Since the average field density was 93.5 percent of maximum and the average core density was 94.8 percent of maximum, it was concluded that compaction to 95 percent of maximum could have been readily obtained. Even this is less than the 100 percent recommended by PCA (4) and some state highway departments (8).

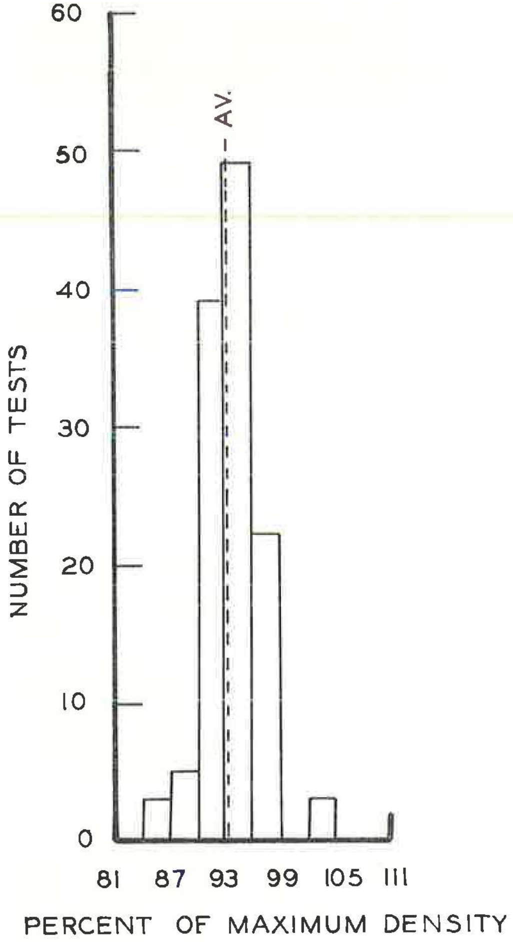

Figure 6. Field density tests, Iowa 37.

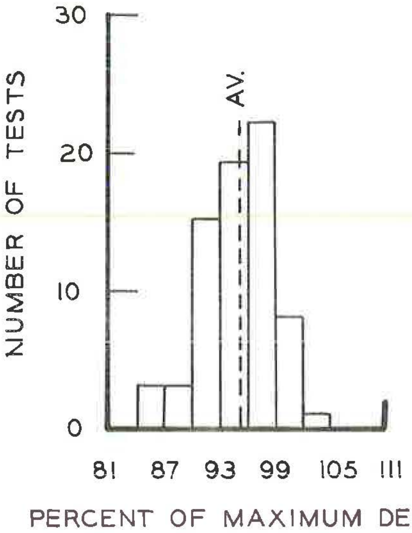

Figure 7. Density of soil-cement cores, Iowa 37.

The specified minimum density for the Iowa 333 base was 95 percent of maximum. Because the soil in the borrow area was not uniform, the maximum field density varied from 97.0 to 101.8 pcf. The field tests of density are summarized in Figure 8. The average of 150 tests was 97.6 percent, with a standard deviation of 4.0 percent.

Finishing

In bringing a soil-cement base to grade and profile, it is frequently necessary to work with very thin cut and fill sections. The specifications for Iowa 37 required that the grade and profile of the base be true to 0.05 ft or less. In meeting this specification, the contractor spent considerable time and effort on finishing operations.

The finishing operations normally require the use of a spring-tooth harrow, spike drag, or similar device to roughen and scratch the surface of the compacted material. On Iowa 37 the contractor used two devices for this purpose. One of them was equipped with large teeth which became smooth and round on the ends (Fig. 9). The teeth left slick, shallow grooves in the compacted base, such as are shown in Figure 10. After the base had cured for about 7 days, the top ½- to 1-in. became separated from the lower portion in many areas, such as the one shown in Figure 11.

When Iowa 333 was constructed, an effort was made to reduce the amount of work needed to obtain an acceptable surface, and the 0.05-ft requirement for grade and profile was eliminated. During construction of the first 6 miles of the project, a heavy drag was used along with a spring-tooth harrow. This heavy drag had thin blade-like teeth with sharp points. It was reasoned that this drag would scarify to a greater depth than the harrow, but that it would not cause the slick grooves produced by the heavy spike drag used on Iowa 37. Unfortunately, compaction planes did develop much as they had on Iowa 37, and use of the heavy drag was discontinued. This reduced the incidence of compaction planes, but did not eliminate them entirely.

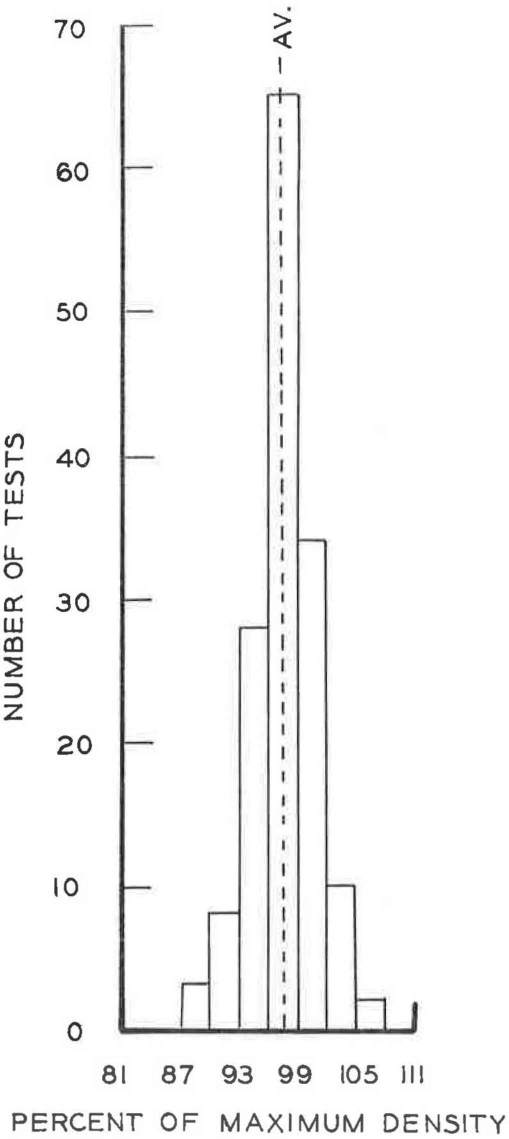

Figure 8. Field density tests, Iowa 333.

Wearing Surface

When Iowa 37 was constructed in 1961 it received a single-course bituminous seal, consisting of 0.28 gal/sq yd of MC-4 and 31 lb/sq yd of cover aggregate. A hot-mix asphalt concrete surface was programmed for 1964. It was expected that most of the shrinkage cracks in the base would appear during the 3-year interval. Previous experience in Iowa and elsewhere (8) has suggested that cracking in the asphalt concrete will be retarded,

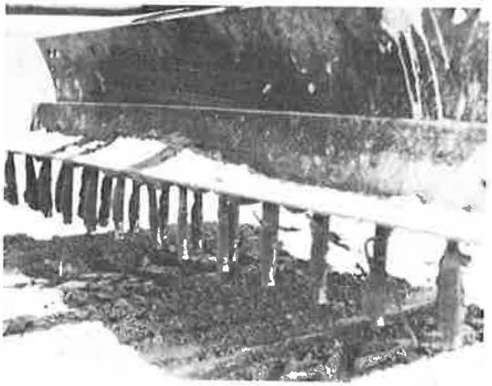

Figure 9. Spike drag with large blunt teeth used on Iowa 37.

Figure 10. Core with top 3/4 in. broken off during drilling (note slick grooves made by spike drag), Iowa 37.

Figure 11. Soil-cement base with top 3/4 in. broken away, Iowa 37.

or possibly reduced, if application of the mat is delayed until 2 or 3 years after construction of the base.

During the spring of 1962, and again in 1963, maintenance of the surface seal became a serious problem. Investigation revealed that moisture had penetrated through closely spaced cracks in the seal and destroyed the bond between the asphalt and the soil-cement. At first only small bits of the surface were picked up or otherwise removed by traffic. These small areas grew rapidly, however, and it soon became necessary to make hundreds of surface patches throughout the entire project. Figure 12 shows an extensively damaged area before patching.

On Iowa 333 a double bituminous seal was applied at the time of construction. The MC-4 was applied at the rate of 0.35 gal/sq yd for the first course and 0.30 gal/sq yd for the second. One-half-inch cover aggregate was placed at an average rate of 30 lb/sq yd for the first course and 25 lb/sq yd for the second. No surface damage has been observed on Iowa 333.

Moisture Content of Base

The moisture content of the soil-cement mixture was checked frequently during construction of Iowa 37. The objective was to insure production of a mix having a uniform moisture content about 2 percent greater than Proctor optimum (21.8 percent) when it left the pug mill. The moisture content of the base was measured again when the density was determined following compaction. It was also determined from cores and samples taken at various times after construction. The results of all moisture tests are summarized in Figure 13.

The moisture content of the samples taken from the pug mill indicated that good moisture control was exercised during mixing. Moisture measurements made in conjunction with the density tests showed that the base was compacted at an average moisture content slightly above optimum. The tests made at age 8 days revealed that adequate moisture was retained in the base during initial curing of the soil-cement.

The average moisture content of the base at ages 8 and 12 months was over 24 percent, which was surprisingly high, especially since the average moisture content of the soil in the first 6 in. below the soil-cement was 16.7 percent.

Figure 12. Surface damage due to failure of single bituminous seal, Iowa 37.

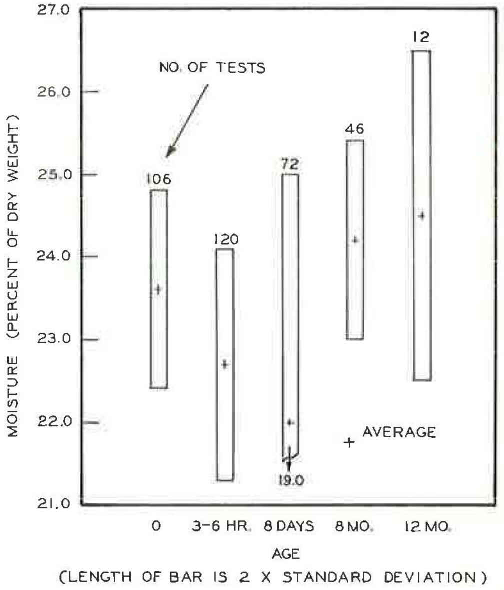

Figure 13. Moisture content of soil-cement, Iowa 37.

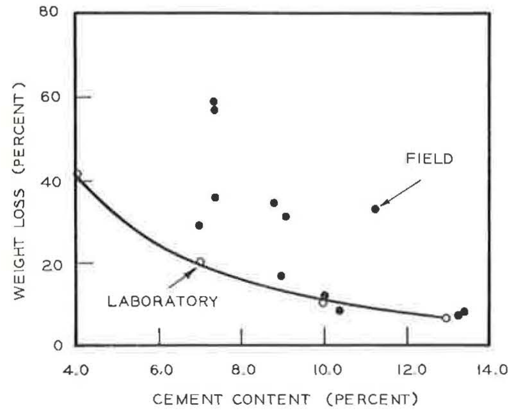

Figure 14. Freeze-thaw resistance of laboratory specimens and cores, Iowa 37.

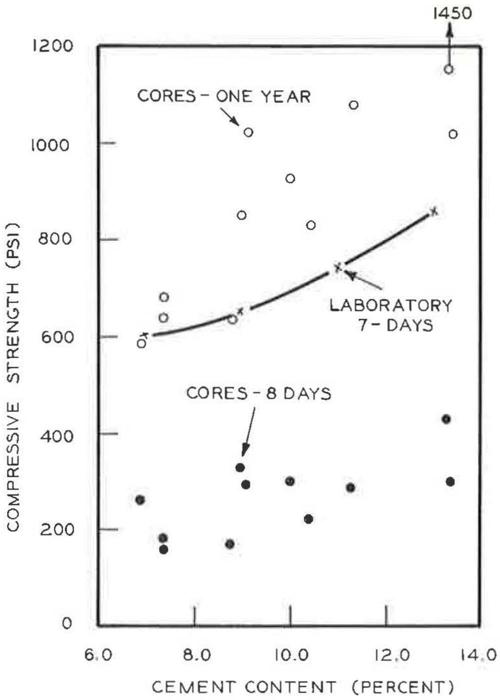

Figure 15. Compressive strength of laboratory specimens and cores, Iowa 37.

Resistance to Effects of Freezing and Thawing

Resistance to the effects of freezing and thawing is usually determined from specimens of soil-cement prepared in the laboratory under the best possible conditions relative to mixing, compaction, and curing. It has been suggested that field operations produce soil-cement which is inferior to that produced in the laboratory.

In connection with the Iowa 37 project, a comparison was made between the freeze-thaw resistance of laboratory specimens and that of cores drilled from the experimental sections at age 6 days and tested at age 8 days (Fig. 14).

Compressive Strength

Figure 15 shows compressive strength and cement content of soil-cement from the Iowa 37 project. The points on the curve represent the strength of specimens prepared in the laboratory. The closed circles represent the average strength of cores from each experimental section drilled at age 6 days and tested at age 8 days. The open circles represent the average strength of cores drilled and tested at the age of approximately 12 months. Both the laboratory specimens and the field cores were immersed in water for 24 hours before testing.

CONCLUSIONS

In general, the construction of a soil-cement base containing loess soil requires the same equipment and workmanship that is needed for any soil-cement pavement construction. Some of the problems are accentuated by the characteristics of the soil; consequently, special attention must be given to soil pulverization, mixing, and finishing. The following specific conclusions apply only to the soil-cement construction discussed in this report:

1. Soil Pulverization—The clay content and the moisture content of the soil seemed to control the degree of pulverization that could be attained. There appeared to be a particular moisture content at which the soil was most friable.
2. Cement Content—On the basis of field checks, control of the cement proportioning on Iowa 37 was fair to good; on Iowa 333 it was poor. Much work remains to be done

in Iowa on the effect of cement variation in soil-cement mixtures and the use of rapid methods for determining cement content.

3. Finishing—The use of a heavy spike drag with blunt teeth produced compaction planes in the soil-cement on Iowa 37. A similar heavy drag with sharp pointed teeth caused compaction planes on Iowa 333.

4. Surface—A single bituminous surface treatment on Iowa 37 was inadequate to protect the soil-cement base. A double surface treatment on Iowa 333 appears to have been satisfactory.

5. Freeze-Thaw Loss—The average freeze-thaw loss of cores drilled from the experimental sections of Iowa 37 was substantially greater than the loss suffered by corresponding laboratory specimens except for sections containing at least 10 percent cement.

6. Compressive Strength—The average compressive strength of cores from Iowa 37, when tested at age 8 days, was about 400 psi less than the strength of laboratory specimens tested at age 7 days. Cores drilled and tested after 1 year showed strength equal to or greater than the 7-day laboratory specimens.

ACKNOWLEDGMENTS

The Iowa 37 project was constructed with Federal participation. Appreciation is expressed to the U.S. Bureau of Public Roads for help in planning the experimental sections. The authors also wish to acknowledge the assistance of design, materials, and construction personnel of the Iowa State Highway Commission in obtaining the material for this report.

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A Rapid Method for Determination of Cement Content of Cement-Treated Base Materials

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Evidence is presented to indicate that an EDTA titration under controlled conditions offers improved precision and accuracy over the presently accepted California method for the determination of the cement content of cement-treated base materials. Exclusive of sample preparation time, the new method permits a complete determination in 10 min or less as opposed to 1 hr or more for the California method. The new procedure uses ammonium chloride for the solvent system, the disodium salt of EDTA as titrant, and hydroxynaphthol blue as indicator.

•THE INCREASING USE of portland cement for the treatment of plastic-base materials has emphasized the need for developing a quick and moderately accurate method for determining the cement content of such materials. Accordingly, a study was initiated by the Nevada Department of Highways during the winter of 1963.

Curtis and Forbes (1) discussed and evaluated six methods for determining the cement content of treated base materials. They concluded that the California (2) and the conductivity methods (3) were the most practical for field use. For laboratory use they recommend the ASTM (4), the Versene (5, 6), or the California methods. The time required for a complete determination by the methods considered varied from 1 to 8½ hr. With present construction equipment having a capacity of 500 tons/hr or more, the time required for testing becomes extremely critical.

In an effort to decrease substantially the required testing time, the Materials and Research Laboratory of the Nevada Department of Highways studied possible modifications of the EDTA method for the determination of calcium for use as an index of the cement content of such materials.

PRELIMINARY

The analytical use of EDTA (ethylenedinitrilotetraacetic acid) and its disodium salt as reagents for the titrimetric determination of polyvalent cations through the formation of strong 1:1 complexes has been well established (7; 8, pp. 847-862). One of the chief disadvantages in the use of these methods has been the lack of specificity or selectivity of the available indicators. In 1963, a highly specific indicator for calcium was introduced, i.e., hydroxynaphthol blue (Mallinckrodt Chemical Works, Code No. 5630).

Preliminary experiments were performed using this new indicator and various solvent mixtures to find a combination that would permit either direct titration or titration of an aliquot of a simple solution. The method finally adopted (Appendix) was chosen for its simplicity, adaptability to field use, and accuracy, as well as for the ready availability and low cost of reagents.

Paper sponsored by Committee on Soil-Portland Cement Stabilization and presented at the the 44th Annual Meeting.

Early experiments indicated that the use of the common laboratory acids, either singly or in combination, would be unsatisfactory due to the necessity of using heat or filtration subsequent to solution. Organic solvents were tried and discarded as either being too costly for volume work or too difficult to handle safely in the field. The solubility of calcium compounds in solutions of ammonium salts (9) was then exploited to develop the method as finally adopted. Ammonium chloride was chosen because of cost, availability, and ease of use in laboratory or field.

EXPERIMENTAL

Principle

The calcium compounds present in cement-treated base materials are taken into solution with ammonium chloride. An aliquot is removed from the resulting suspension and titrated with the disodium salt of EDTA after adjusting the pH between 13 and 13.5 to precipitate interfering substances such as iron, magnesium or aluminum. Cement content is determined from a standard curve after subtracting a blank for the calcium present in the aggregate.

TABLE 1

COMPARISON OF CALIFORNIA AND EDTA METHODS, 2 PERCENT CEMENT ADDED[a]

Sample No.	California Method					EDTA Method					Operator
	Cement Found	d	d^2	p	p^2	Cement Found	d	d^2	p	p^2	
1	2.07	7	49	3.5	12.25	2.00	0	0	0	0	BR
2	2.00	0	0	0	0	1.95	5	25	2.5	6.25	BR
3	1.85	15	225	7.5	56.25	2.00	0	0	0	0	BR
4	2.11	11	121	5.5	30.25	2.00	0	0	0	0	BR
5	2.05	5	25	2.5	6.25	2.02	2	4	1.0	1.0	BR
6	2.17	17	289	8.5	72.25	2.01	1	1	0.5	0.25	PC
7	2.13	13	169	6.5	42.25	2.00	0	0	0	0	PC
8	2.05	5	25	2.5	6.25	2.00	0	0	0	0	PC
9	2.02	2	4	1.0	1.0	1.98	2	4	1.0	1.0	PC
10	1.87	13	169	6.5	42.25	2.01	1	1	0.5	0.25	RM
11	2.04	4	16	2.0	4.0	1.95	5	25	2.5	6.25	RM
12	2.08	8	64	4.0	16.0	1.93	7	49	3.5	12.25	RM
13	1.92	8	64	4.0	16.0	1.98	2	4	1.0	1.0	RM
14	1.95	5	25	2.5	6.25	2.05	5	25	2.5	6.25	JC
15	1.85	15	225	7.5	56.25	2.00	0	0	0	0	JC

[a] California method: s = 7.0, CV = 3.5; EDTA method: s = 2.1, CV = 1.1.

TABLE 2

COMPARISON OF CALIFORNIA AND EDTA METHODS, 3 PERCENT CEMENT ADDED[a]

Sample No.	California Method					EDTA Method					Operator
	Cement Found	d	d^2	p	p^2	Cement Found	d	d^2	p	p^2	
16	3.15	15	225	5.0	25.0	2.98	2	4	0.66	0.44	BR
17	3.07	7	49	2.3	5.29	3.00	0	0	0	0	BR
18	2.95	5	25	1.7	2.89	2.98	2	4	0.66	0.44	BR
19	3.00	0	0	0	0	2.98	2	4	0.66	0.44	BR
20	3.12	12	144	4.0	16.0	3.05	5	25	1.66	2.75	BR
21	3.17	17	289	5.7	32.49	2.98	2	4	0.66	0.44	PC
22	3.28	28	784	9.3	86.49	3.00	0	0	0	0	PC
23	3.50	50	2500	17.0	289.0	3.02	2	4	0.66	0.44	PC
24	3.14	14	196	4.7	22.09	3.00	0	0	0	0	PC
25	2.85	15	225	5.0	25.0	2.95	5	25	1.66	2.75	RM
26	3.00	0	0	0	0	3.05	5	25	1.66	2.75	RM
27	2.95	5	25	1.7	2.89	3.00	0	0	0	0	RM
28	3.08	8	64	2.7	7.29	3.02	2	4	0.66	0.44	RM
29	2.78	22	484	7.3	53.29	2.00	0	0	0	0	JC
30	2.78	22	484	7.3	53.29	3.05	5	25	1.66	27.5	JC

[a] California method: s = 13.5, CV = 4.5; EDTA method: s = 2.0, CV = 0.67.

Procedure

Following the preliminary survey and determination of optimum conditions, an experiment was designed comparing the California and the EDTA methods at four different cement concentrations, keeping moisture, aggregate, and type of cement constant; operators were varied at each concentration level of cement. A supply of aggregate was obtained from a contract pit that would be used for actual construction. The aggregate was thoroughly blended and weighed into 120 portions. Cement was added in the amounts indicated in Tables 1 through 4. After thorough dry mixing, water was added to a moisture content of 8 percent, and the cement content was determined by both methods. Test results, estimated standard deviation, and coefficient of variation are as indicated in the tables.

DISCUSSION

As may be determined from the tables, the estimated standard deviation (s) and the coefficient of variation (CV) are substantially lower for the EDTA method than for the California method. Other than relative experience in laboratory technique, there seems to be no noticeable operator bias.

TABLE 3

COMPARISON OF CALIFORNIA AND EDTA METHODS, 4 PERCENT CEMENT ADDED[a]

Sample No.	California Method					EDTA Method					Operator
	Cement Found	d	d^2	p	p^2	Cement Found	d	d^2	p	p^2	
31	4.12	12	144	3.0	9.0	4.03	3	9	0.75	0.56	BR
32	4.18	18	324	4.5	20.25	4.00	0	0	0	0	BR
33	4.05	5	25	1.25	1.56	4.00	00	0	00	0	BR
34	4.13	13	169	3.25	10.56	4.00	0	0	0	0	BR
35	4.07	7	49	1.75	3.06	4.02	2	4	0.50	0.25	BR
36	4.18	18	324	4.50	20.25	4.02	2	4	0.50	0.25	PC
37	4.30	30	900	7.50	56.25	4.00	0	0	0	0	PC
38	4.07	7	49	1.75	3.06	4.05	5	25	1.25	1.56	PC
39	4.17	17	289	4.25	18.06	4.00	0	0	0	0	PC
40	4.05	5	25	1.25	1.56	4.02	2	4	0.50	0.25	RM
41	4.04	4	16	1.00	1.00	3.98	2	4	0.50	0.25	RM
42	3.92	8	64	2.00	4.00	4.00	0	0	0	0	RM
43	3.84	16	256	4.00	16.00	4.00	0	0	001	0	RM
44	3.95	5	25	1.25	1.56	3.99	1	1	0.25	0.63	JC
45	3.97	3	9	0.75	0.56	4.00	0	0	0	0	JC

[a] California method: s = 9.4, CV = 2.36; EDTA method: s = 1.3, CV = 0.35.

TABLE 4

COMPARISON OF CALIFORNIA AND EDTA METHODS, 5 PERCENT CEMENT ADDED[a]

Sample No.	California Method					EDTA Method					Operator
	Cement Found	d	d^2	p	p^2	Cement Found	d	d^2	p	p^2	
46	5.13	13	169	2.60	6.76	5.00	0	0	0	0	BR
47	5.25	25	625	5.00	25.00	5.05	5	25	1.0	1.0	BR
48	5.18	18	324	3.60	12.96	5.00	0	0	0	0	BR
49	5.23	23	529	4.60	21.16	5.00	0	0	0	0	BR
50	5.12	12	144	2.40	5.76	5.05	5	25	1.0	1.0	BR
51	5.28	28	784	5.60	31.36	5.00	0	0	0	0	PC
52	5.19	19	361	3.80	14.44	5.00	0	0	0	0	PC
53	5.40	40	1600	8.00	64.00	5.10	10	100	2.0	4.0	PC
54	5.20	20	400	4.00	16.00	5.05	5	25	1.0	1.0	PC
55	5.08	8	64	1.60	2.56	4.95	5	25	1.0	1.0	RM
56	5.03	3	9	0.60	0.36	4.98	2	4	0.4	0.16	RM
57	4.98	2	4	0.40	0.16	5.01	1	1	0.2	0.04	RM
58	5.05	5	25	1.00	1.00	5.00	0	0	0	0	RM
59	5.00	0	0	0	0	4.95	5	25	1.0	1.0	JC
60	5.20	20	400	4.00	16.00	4.97	3	9	0.6	0.36	JC

[a] California method: s = 13.5, CV = 2.69; EDTA method: s = 2.8, CV = 0.56.

Under normal experimental conditions, it was found desirable to add 20 percent triethanolamine solution as an additional precaution against interfering cations. As of the date of writing, more than 1,000 determinations have been made in the field with no difficulties encountered. The figures obtained by titration agree to 1 part in 100 with the theoretical amounts from plant operation; i.e., cement consumption, water usage, aggregate consumption, and truck weights.

As only a 10-ml aliquot is used for the titration, minor variations in moisture and calcium contents of the cement are of small moment. It is important to run blanks frequently, however. In actual field use, the calcium content of mixing water and aggregate can vary rather widely. Under the conditions existing in Nevada, the maximum daily variation in blank has been of the order of 1 ml EDTA. With the reagent strength we are using, this is the equivalent of 0.3 percent in the cement content. The most effective procedure we have been able to devise is that of belt sampling the aggregate immediately before sampling the cement-treated material and then running the two determinations simultaneously. This procedure, however, has been necessary on only one contract. On three other contracts, the blank never varied more than 0.1 ml, thus necessitating only one blank determination per day.

Experience has shown that time is a critical factor in the use of this procedure. With low cement contents and aggregate containing small amounts of calcium compounds, a stirring time of 1 min and a settling time of 3 min is quite satisfactory. With increasing cement content and/or greater amounts of calcium compounds present, it is necessary to increase the stirring time to 2 or 3 min and the settling time to 6 or 7 min. In no instance has a hydration period in excess of 10 min been necessary. Our experience has been limited to Nevada aggregates; accordingly, it may be necessary or desirable to experiment with the time factor in dealing with aggregates in different parts of the country.

Adding calcium compounds (e.g., oxide, carbonate, and sulfate) to aggregate blank and cement-treated material caused no difficulty, since the added material was compensated for in the blank determination.

By increasing hydration time, the method has been found applicable to materials up to 6 months old.

The method has not been applied to lime-stabilized materials, but theory indicates that it should apply equally well. It has been used with fresh and hardened concrete by changing the concentration of EDTA to 0.5 M. The main difficulty in dealing with wet concrete lies in obtaining a true sample. The extra moisture is readily compensated for by simple calculation.

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Appendix

DETERMINATION OF CEMENT CONTENT OF CEMENT-TREATED BASE MATERIALS

Principle

A 10 percent ammonium chloride solution is used as the solvent system for the calcium compounds present in cement-treated base materials. The solution is titrated with the disodium salt of EDTA (ethylenedinitrilotetraacetic acid) after adjustment of pH, using hydroxynaphthol blue as indicator. Cement content is determined from a standard graph after subtracting the aggregate blank.

Equipment for Field Use

6 - 10-ml volumetric pipettes;
2 - 25-ml burettes;
1 - Magnetic stirrer and stirring bar;
1 - Burette stand;
6 - 250-ml Erlenmeyer flasks;
6 - 2-qt polyethylene jugs;
6 - Stainless steel stirring rods (12 to 14 in.);
1 - Harvard trip balance with tared pan;
1 - 25-ml graduated cylinder;
1 - 1,000-ml graduated cylinder;
1 - Plastic funnel (12 in. diam.);
2 - 5-gal polyethylene jugs for distilled water;
1 - 5-gal polyethylene jug for ammonium chloride;
6 - Medicine droppers; and
1 - Pkg. pH indicator paper, range 10 to 14.

Reagents

1. EDTA, 0.1 M—Weight 116.88 g of EDTA (acid), A.C.S. Reagent grade, into a 3-l beaker. Add approximately 1.5 l of distilled water and place on stirrer. Add sodium hydroxide pellet by pellet until the acid is completely dissolved. Make up to exactly 4 l and transfer to a 1-gal polyethylene bottle. This solution must be stored in plastic. (For field use this solution will be made up and distributed by Headquarters Laboratory.)

2. Ammonium chloride solution, 10 percent—Transfer 1,893 g of U.S.P. granular ammonium chloride to a 5-gal plastic bottle. Make up to 5 gal with distilled water with thorough mixing.

3. Sodium Hydroxide, 50 percent—Dissolve 500 g of sodium hydroxide pellets in distilled water and dilute to 1 l. Use caution; store in plastic when cool. Dilute 1:1 with distilled water for use.

4. Triethanolamine, 20 percent—Dilute 100 ml of triethanolamine to 500 ml with distilled water.
5. Hydroxynaphthol blue—Obtain from Headquarters.

Caution

All equipment must be kept scrupulously clean and thoroughly rinsed with distilled water. All reagents must be stored in polyethylene containers.

Procedure

Preparation of Curve.--Prepare three duplicate samples as follows:

Set 1. Transfer two 300-gm samples of aggregate at the planned moisture content for the job to separate plastic containers. (The water used in bringing up the moisture to the correct amount should be that used at the job site.)

Set 2. Prepare two samples containing 2.5 percent cement of the type to be used on the job.

Set 3. Prepare two samples containing 5.0 percent cement.

Proceed with each sample as with production samples. Construct a graph showing milliliters EDTA vs percent cement (Fig. 1) using the average figures from Sets 1, 2 and 3. This should yield a straight line. Set 1 corresponds to the blank for the aggregate being used. If the aggregate blank changes, appropriate corrections must be made.

Production Samples.--Weigh into a 2-qt plastic container exactly 300 g of cement-treated material. Add 600 ml ammonium chloride solution and stir exactly 2 min with a stainless steel stirring rod. Allow to settle exactly 4 min and then pipette a 10-ml aliquot of the supernatant solution into a 250 ml Erlenmeyer flask. Add approximately 75 ml distilled water and with thorough mixing add the diluted NaOH dropwise until a pH between 13.0 and 13.5 is obtained using the pH indicator paper. Add 4 drops 20 percent triethanolamine solution and then add hydroxynaphthol blue indicator. Titrate with EDTA to a pure blue endpoint. Determine cement content from the previously prepared graph (Fig. 1).

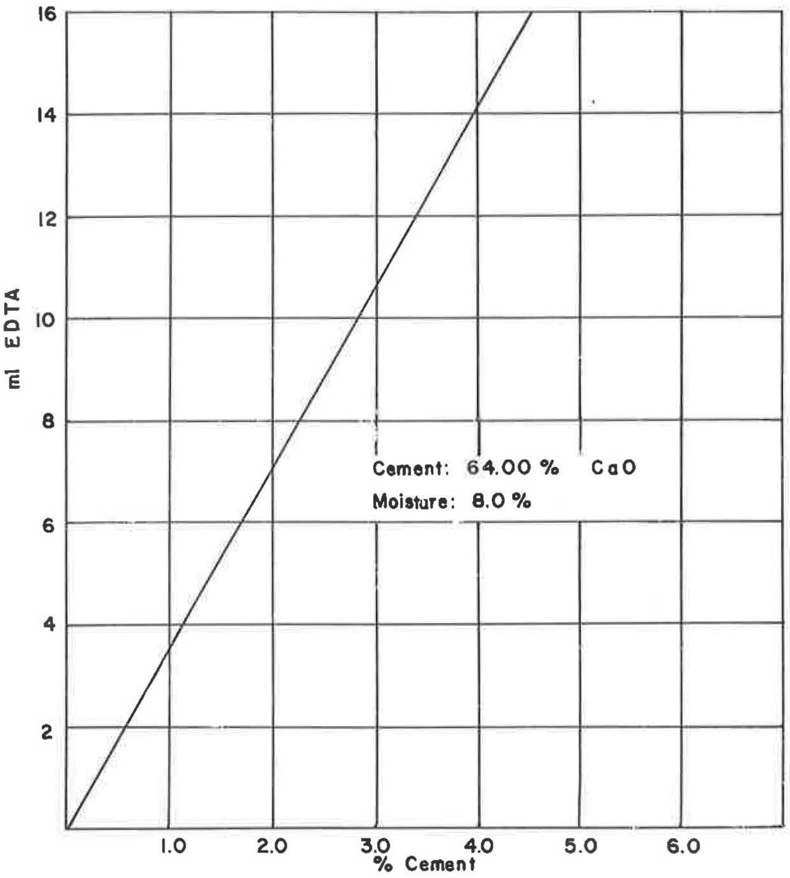
16
14
12
10
8
6
4
2
ml EDTA
Cement: 64.00 % CaO
Moisture: 8.0 %
1.0
2.0
3.0
4.0
5.0
6.0
% Cement

Figure 1.