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Effect of Lime, Moisture and Compaction on a Clay Soil

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•IN AN EFFORT to use economically material otherwise unsuitable for subgrade construction, the State of Virginia is in many instances adding a small percentage of lime to improve such material. Much of this so-called lime stabilization or lime modification has been done in the southwestern part of the State where Virginia Polytechnic Institute is located. The purpose of this project was to investigate the change in engineering characteristics of a typical clay soil from this area, when different amounts of hydrated lime were added.

The testing program consisted mainly of studying the effect of three variables—percent lime, moisture content, and compactive effort—by fabricating CBR specimens and obtaining values of CBR, density, and swell. In addition, Atterberg limit determinations were made on the soil containing different percentages of lime. Use of the CBR test was particularly appropriate for this study because the State of Virginia bases the flexible pavement design on this test.

TEST SOIL PROPERTIES

The soil, a reddish brown, plastic clay, had approximately 97 percent passing the No. 200 sieve and about 66 percent finer than 0.001 mm. The soil had a liquid limit of 71 and a plasticity index of 35. Standard AASHO density was 86 pcf at an optimum moisture content of 31 percent. Modified AASHO density was 97 pcf at an optimum moisture content of 27 percent. According to the AASHO classification system, the soil was classified as A-7-5(20). X-ray diffraction analysis was run on the soil with the following results: kaolinite group (but mostly halloysite), 30 percent; montmorillonite group, 10 percent; chlorite, 10 percent; illite, 10 percent; and amorphous ferrous and aluminum oxides and hydroxides, 40 percent.

TEST PROCEDURES

CBR tests were performed on soil with the addition of 0, 5, and 10 percent (by weight of dry soil) hydrated lime. The compactive efforts used were 10, 25, and 55 blows per layer on five layers with a 10-lb hammer dropped 18 in. The underlying purpose of this procedure was to develop a family of curves showing the effect of variation in lime content, compactive effort, and moisture content on density, swell potential, and CBR values of the soil, and finally to show the relationships between CBR and density at different moisture contents. Knowing such relationships for subgrade soils would enable better compaction control in the field in order to obtain a required CBR value.

CBR values were obtained immediately after compaction and again after a 4-day soaking period with a 20-lb surcharge weight. Moisture content and density before soaking were determined. Swell measurements were taken during the soaking period and the percent swell after 4 days of soaking determined. Specimens were molded at about six different moisture contents for each level of lime content and compactive effort.

Liquid and plastic limit tests were performed on soil containing 0, 3, 5, 7, and 10 percent lime. In running these tests, the soil and lime were mixed thoroughly in the dry powdered state. Water was added to the mixture in sufficient quantity to approach the plastic limit. The liquid and plastic limit tests were then run in the ordinary manner.

EFFECT OF LIME ON ATTERBERG LIMITS

Table 1 summarizes the results of this part of the experiment and Figure 1 shows the effect of lime content on the liquid limit flow curve.

To illustrate the effect of lime content on the plasticity properties of the soil studied, the values in Table 1 are plotted in Figure 2. It is evident that these properties of the soil were improved by the addition of lime. There was a marked decrease in liquid limit by addition of lime. Also, the plastic limit was decreased from 35 for the natural soil to 18, 16, 13, and 11 with the addition of 3, 5, 7, and 10 percent of lime, respectively. With the addition of a small percentage of lime (3 percent), it was reduced drastically from 35 to 18. However, as the percent added increased, the rate of reduction was decreased.

TABLE 1
ATTERBERG LIMITS FOR VARIOUS
LIME PERCENTAGES

Lime Added (%)	Atterberg Limit (%)		
	Liquid Limit	Plastic Limit	Plasticity Index
0	71	36	35
3	66	48	18
5	65	49	16
7	63	50	13
10	62	51	11

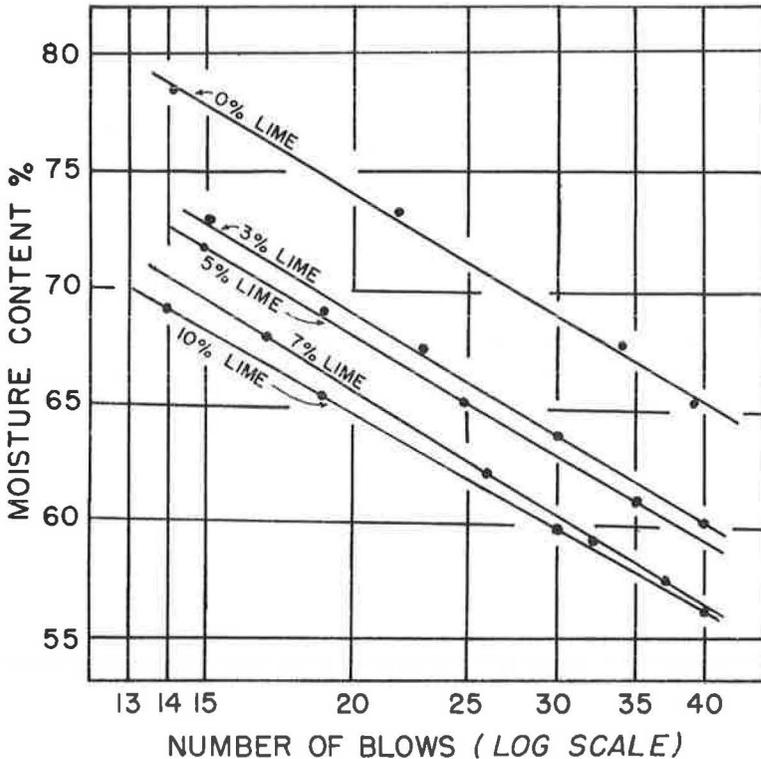


Figure 1. Flow curves for different percentages of lime.

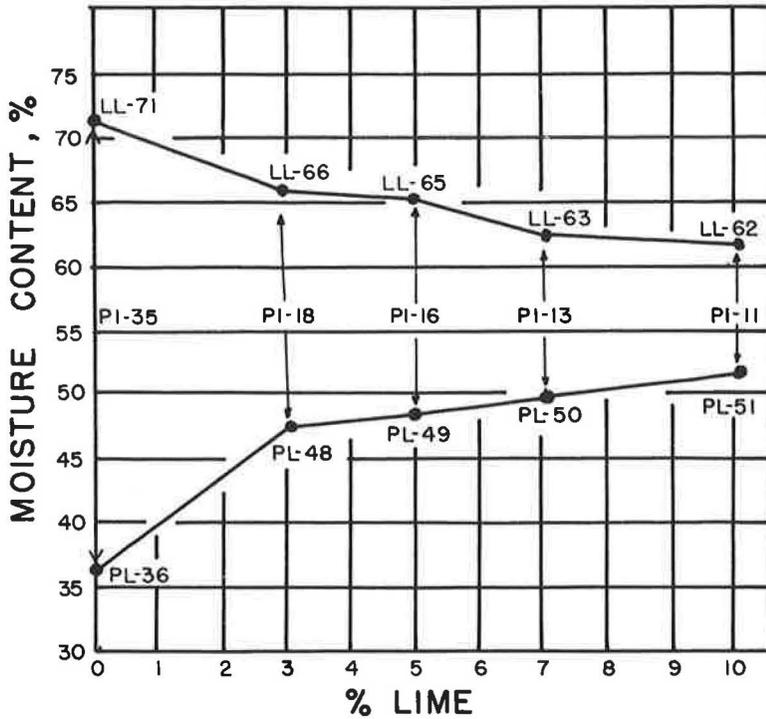


Figure 2. Effect of lime on plasticity indexes of soil.

TABLE 2
CBR TEST RESULTS FOR 0 PERCENT LIME

Compactive Effort (blows/layer)	As-Molded Condition			Soaked Condition	
	Moisture Content (%)	Dry Density (pcf)	CBR (%)	CBR (%)	Swell (%)
10	19.8	73.3	6.7	0.7	4.0
	27.3	76.3	13.3	3.0	3.4
	29.2	82.0	25.0	3.3	2.6
	34.6	85.0	10.0	6.7	0.4
	38.6	80.4	1.7	3.7	0.2
25	22.0	85.8	46.7	3.7	6.7
	27.6	89.4	40.7	8.3	3.8
	32.2	87.2	21.7	10.0	0.6
	35.0	85.6	9.3	10.0	0.4
	37.0	82.5	3.3	7.3	0.2
55	41.9	77.3	1.7	3.7	0.1
	22.2	93.0	61.7	5.0	7.3
	26.9	94.2	75.7	11.7	5.1
	27.7	93.4	56.7	12.0	3.3
	31.4	91.0	20.0	13.3	0.5
	34.4	86.0	6.7	10.0	0.4
	38.6	81.9	3.7	3.3	0.3

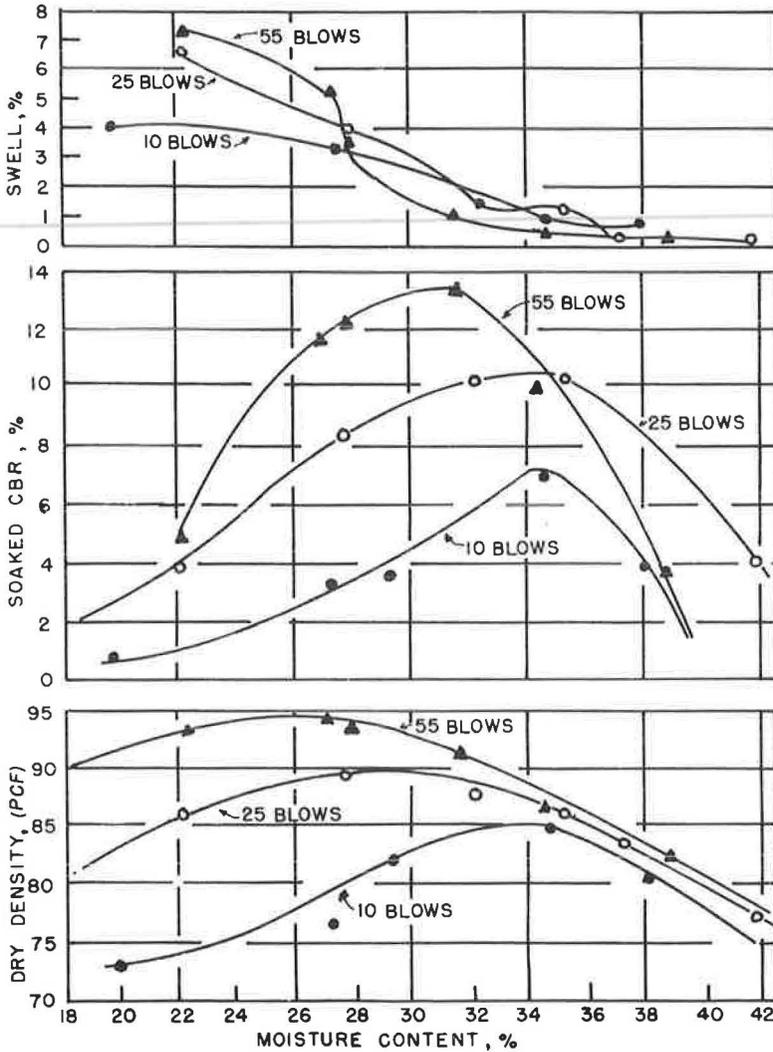


Figure 3. Density, strength, and swell vs moisture content in untreated soil.

CBR TEST RESULTS

CBR tests were performed with the addition of 0, 5, and 10 percent of hydrated lime. The CBR load penetration values were recorded for the soil and soil-lime mixtures before soaking and also after 4 days soaking period.

The results obtained for CBR tests with 0 percent lime are given in Table 2. On the basis of these results, a family of curves was developed by plotting values of dry density, soaked CBR, and percentage swell against moisture content (Fig. 3).

To study further relationships that exist between strength, compactive effort, and moisture content, values of dry density were plotted against CBR soaked for arbitrarily selected moisture contents of 23, 27, 31, 36, and 39 percent in Figure 4.

CBR test results for 5 and 10 percent of lime added to the soil are given in Tables 3 and 4. Figures 5 and 6, and 7 and 8, are similar to Figures 3 and 4 for 0 percent lime.

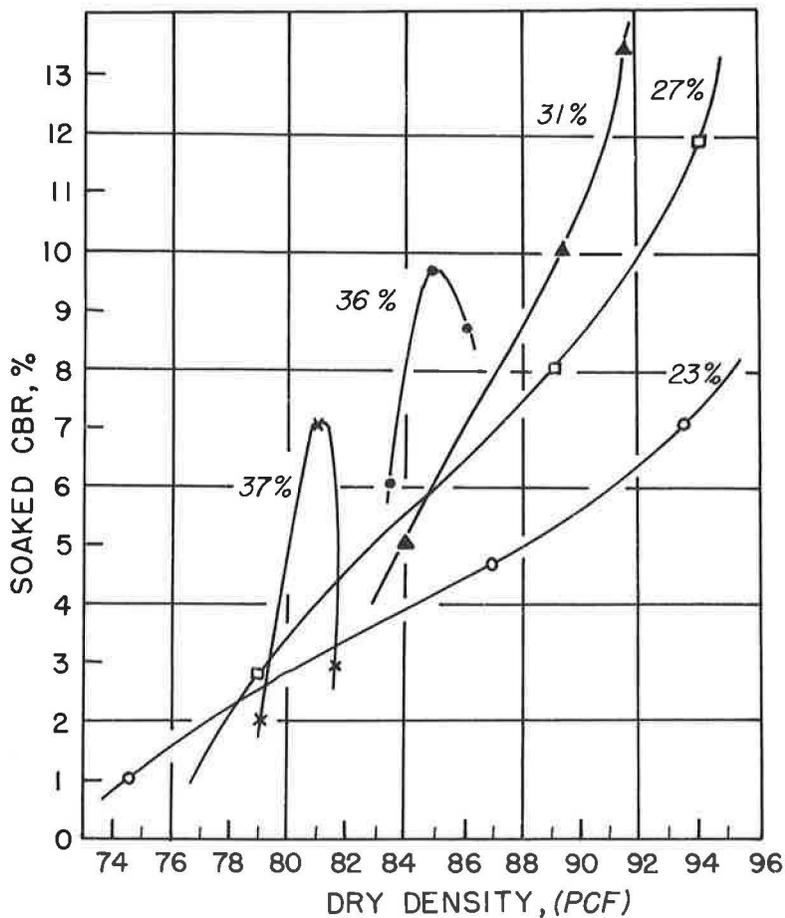


Figure 4. CBR-density relationships in untreated soil.

TABLE 3
CBR TEST RESULTS FOR 5 PERCENT LIME

Compactive Effort (blows/layer)	As-Molded Condition			Soaked Condition	
	Moisture Content (%)	Dry Density (pcf)	CBR (%)	CBR (%)	Swell (%)
10	23.0	75.1	26.0	25.0	2.6
	26.6	79.2	31.7	36.2	2.1
	29.3	80.6	32.7	63.3	1.6
	32.4	83.3	28.3	83.3	0.9
	34.7	83.8	30.0	81.3	0.8
25	37.4	82.6	11.7	45.3	0.4
	18.7	81.2	25.0	26.0	3.3
	20.4	82.3	18.3	33.3	2.8
	26.1	87.4	38.3	44.7	1.1
	28.4	89.0	48.3	60.0	0.5
55	30.0	90.5	48.3	80.0	0.2
	31.8	89.5	31.0	101.0	0.1
	36.1	84.4	19.0	40.9	0.1
	16.4	87.6	41.7	45.0	3.9
	20.7	95.1	81.7	84.0	2.0
	24.8	96.7	81.7	131.7	0.4
	28.1	95.2	44.7	72.0	0.2
34.2	87.2	10.0	39.0	0.0	

TABLE 4
CBR TEST RESULTS FOR 10 PERCENT LIME

Compactive Effort (blows/layer)	As-Molded Condition			Soaked Condition	
	Moisture Content (%)	Dry Density (pcf)	CBR (%)	CBR (%)	Swell (%)
10	20.0	78.9	12.7	23.3	1.3
	23.6	82.5	31.7	30.3	1.0
	27.9	85.2	33.3	66.7	0.4
	32.4	86.3	18.3	70.7	0.2
	36.7	80.0	5.0	28.2	0.0
25	20.8	85.0	53.3	60.0	1.8
	23.3	86.0	35.7	65.0	1.4
	27.0	88.3	33.3	115.0	0.2
	32.0	88.0	25.0	87.7	0.1
	36.8	82.6	6.7	56.7	0.0
55	18.6	89.3	43.3	75.0	2.3
	21.9	91.0	80.0	83.3	1.6
	26.3	93.2	86.7	153.3	0.4
	30.3	91.6	46.7	90.7	0.2
	34.9	86.4	10.0	56.7	0.1

ANALYSIS

Strength-Density Moisture

Using Figure 4 as an example, strength is shown to be a function of both density and moisture content. The optimum strength of the soil (CBR=13.4) can be achieved by using the compactive effort to develop 91.5 pcf dry density at the moisture content of 31 percent. At moisture contents lower than 31 percent, even at high densities (achieved through increased compactive effort), this value of CBR could not be realized.

Again, at moisture contents higher than 31 percent, even with increased compactive effort, values of dry density and CBR soaked continued to decrease. This is due to the fact that, with the increase in moisture content beyond the optimum, the voids are gradually filled with water resulting in a loss of grain-to-grain contact. The structure of the soil is also affected. The flocculent structure is destroyed gradually, and the clay particles are oriented more and more with the increase in moisture content. Further, thickness of the water film continues to increase, causing a reduction in the attraction force between the clay particles.

Such relationships as shown in Figures 4, 6, and 8 can be used as an important tool in controlling compaction in the field by permitting the designer to choose intelligently a range of allowable moisture content and density in order to choose a design CBR.

Effects of Lime Additions

Dry Density and Optimum Moisture Content.—Maximum dry density and optimum moisture content values obtained from the CBR tests on natural soil and soil-lime mixtures with the various compactive efforts are given in Table 5. Unlike many studies, no distinct reduction in maximum density is shown by these data. However, some slight decrease is shown in the optimum moisture content with the addition of lime with only one value contradicting this trend.

Soaked CBR.—The maximum values of CBR soaked for the different percentages of lime and compactive efforts are given in Table 6. The values of soaked CBR are plotted against percent lime added in Figure 9. The following trends are observed:

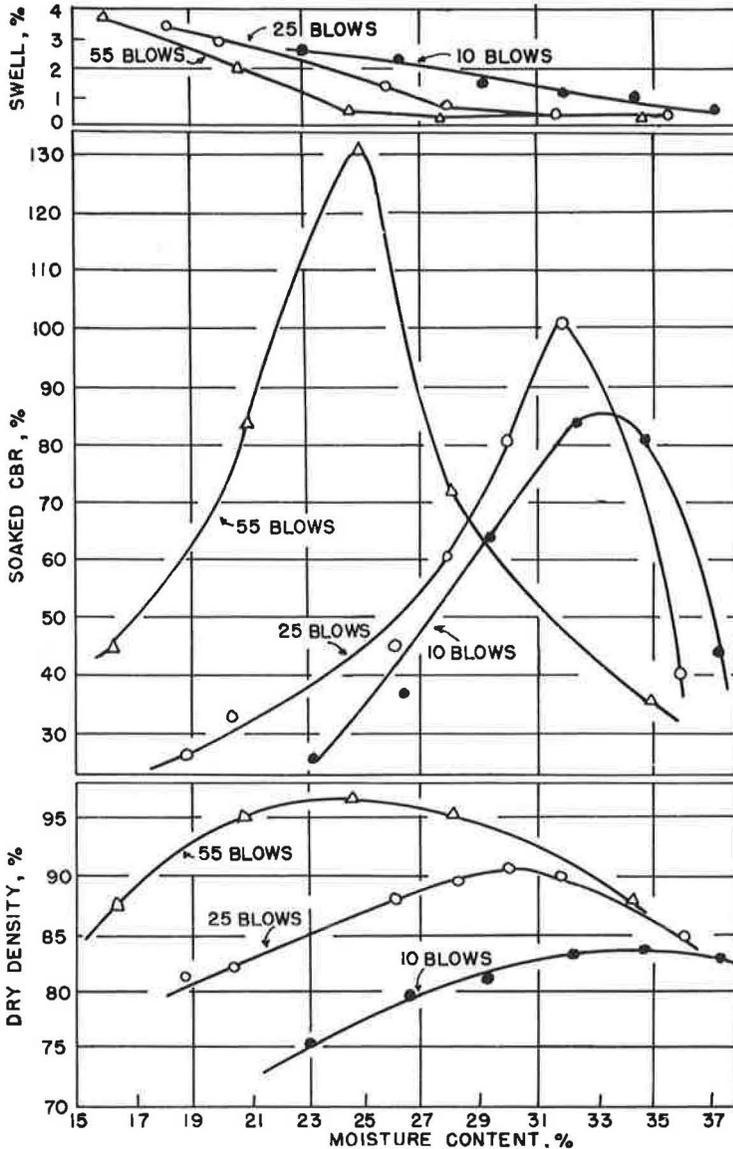


Figure 5. Density, strength, and swell vs moisture content for 5 percent lime.

1. Addition of hydrated lime materially increased the strength as measured by the CBR test. Addition of 5 percent lime increased the CBR value almost ten times, regardless of compactive effort. The CBR values were increased from 6.67 to 83.3, from 10 to 101, and from 13.3 to 131.7, for 10, 25, and 55 blows per layer compactive effort, respectively.

2. With the addition of 10 percent lime there was a further increase in the CBR value, but this increase was not as pronounced as those obtained initially with the addition of 5 percent lime. Figure 9 shows the addition of 10 percent lime relatively did not produce similar results.

3. For the natural soil, change in compactive effort did not increase the CBR value significantly (Fig. 9). But with the increase in lime percentage, the increased

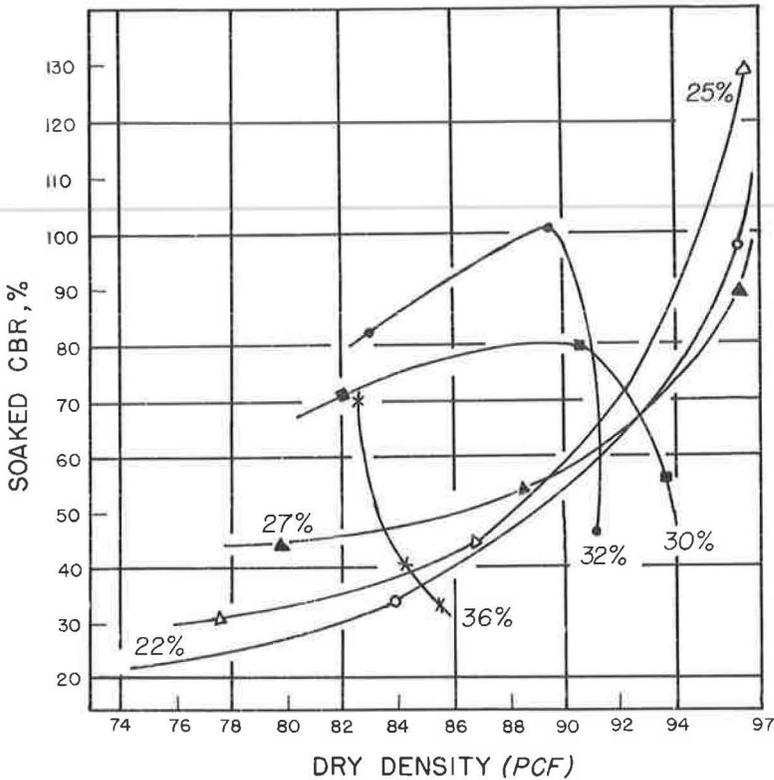


Figure 6. CBR-density relationships for 5 percent lime.

compactive effort increased CBR values significantly. For the natural soil, CBR soaked increased from 6.7 to 10 blows per layer compactive effort to 10.0 and 13.33 for 25 and 55 blows per layer, respectively. With the addition of 5 percent lime CBR soaked was increased from 83.3 for 10 blows per layer compactive effort to 101 and 132 for the next two compactive efforts. Similar increases in CBR soaked, due to increased compactive effort, are observed with the addition of 10 percent lime. Hence, the increased compactive effort produced more favorable results with the addition of lime to soil. Thus, increased rolling of a lime-treated subgrade should be more effective, as far as strength increases are concerned, than of an untreated subgrade.

4. For the development of maximum strength (CBR), compactive effort and moisture contents must be properly controlled. Without the proper density-moisture control, even addition of large amounts of lime (10 percent) will not develop maximum strength. Hence, it is imperative to make certain by proper checks during construction that the designed densities and compaction moisture contents are fulfilled.

CBR As Molded.—The values of maximum CBR as molded for various additions of lime percentage and compactive efforts are given in Table 6. The values of CBR as molded against percent lime added are shown in Figure 10. The following trends were observed:

1. For the natural soil, the unsoaked strengths were generally greater than those of soaked for 4 days (Table 6). But for the soil-lime mixture, the values of CBR soaked are greater than those of CBR as molded. This is probably due to the flocculation and aggregation of clay particles and cementing action which occur with lime addition. The absence of these reactions in natural soil results in lower values of CBR after 4 days soaking period. Loss in grain-to-grain contact and increase in

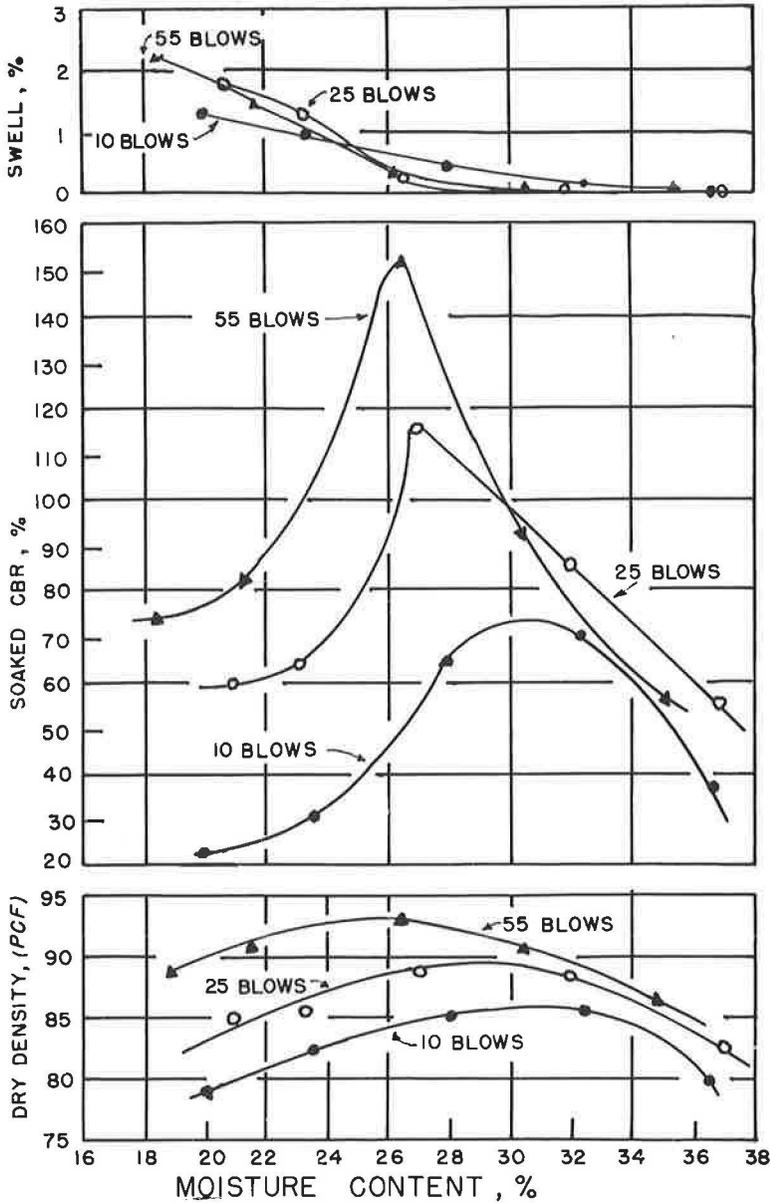


Figure 7. Density, strength, and swell vs moisture content for 10 percent lime.

water film thickness surrounding the clay particles further reduces the strength of the soaked CBR specimens.

2. Although the increase in lime content increases the CBR as molded, the change is not as pronounced as those of CBR soaked. It is probably due to the fact that aggregation of particles and cementation action, with the lime addition, occur most favorably when the soil-lime mixture is soaked in water.

Swell Potential.—The values of maximum swell as observed during the 4-day soaking period of samples for various lime contents and compactive efforts are given in

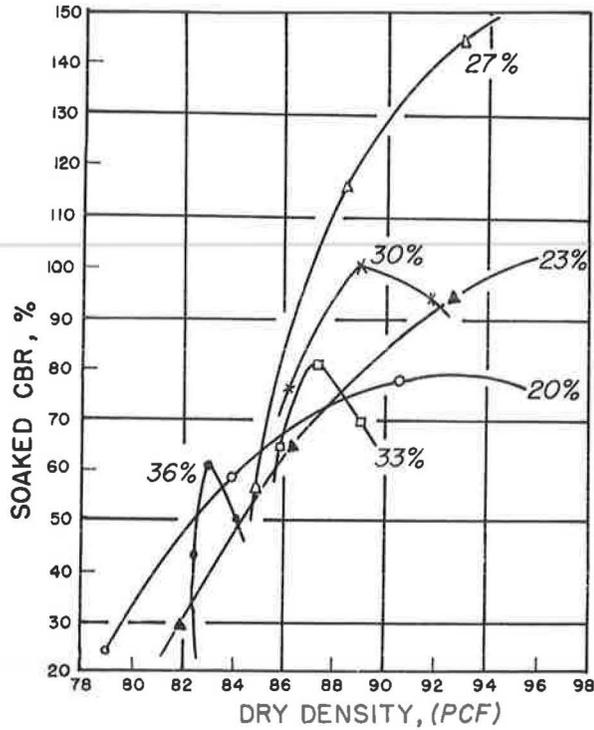


Figure 8. CBR-density relationships for 10 percent lime.

TABLE 5
EFFECT OF LIME ADDITION ON DRY DENSITY AND OPTIMUM
MOISTURE CONTENT

Lime (%)	10 Blows per Layer		25 Blows per Layer		55 Blows per Layer	
	Opt w (%)	Max γ_0 (pcf)	Opt w (%)	Max γ_0 (pcf)	Opt w (%)	Max γ_0 (pcf)
0	34.6	85.0	27.6	89.4	26.9	94.2
5	32.4	83.3	30.0	90.5	24.8	96.7
10	32.3	86.3	27.0	88.3	26.3	93.2

TABLE 6
EFFECT OF LIME ADDITION ON CBR AND
SWELL POTENTIAL

Compactive Effort (blows/layer)	Lime (%)	CBR		Maximum Swell ^a (%)
		As Molded (%)	Soaked 4 Days (%)	
10	0	25.0	6.7	4.0
	5	32.7	83.3	2.6
	10	33.3	78.0	1.3
25	0	46.7	10.0	6.7
	5	48.3	101.0	3.3
	10	53.3	115.0	1.8
55	0	75.7	13.3	7.3
	5	81.7	131.7	3.9
	10	86.7	153.3	2.3

^aGenerally occurs on dry side of optimum moisture; swell percentage at optimum moisture is much lower than these values.

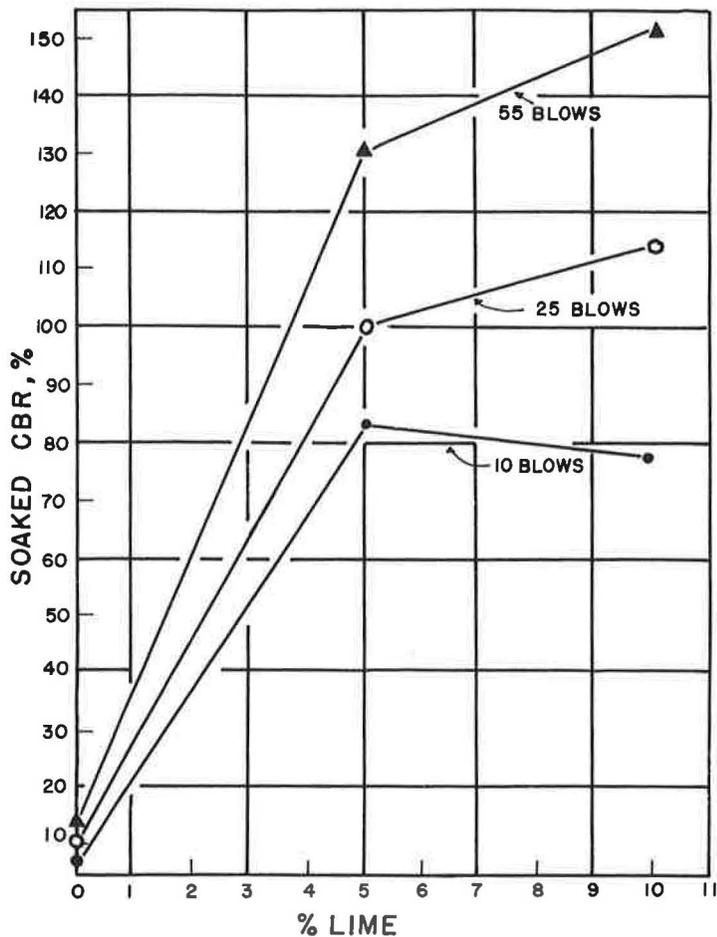


Figure 9. Effect of lime on CBR.

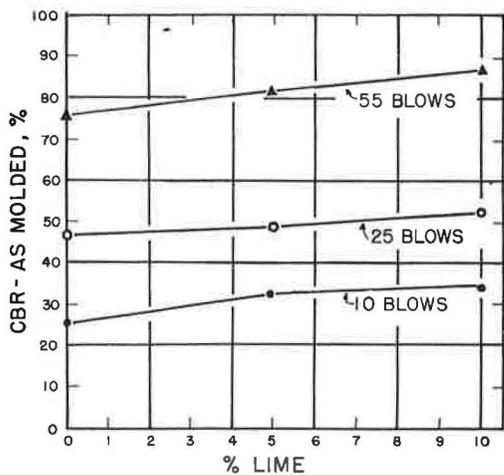


Figure 10. As-molded CBR vs percent lime.

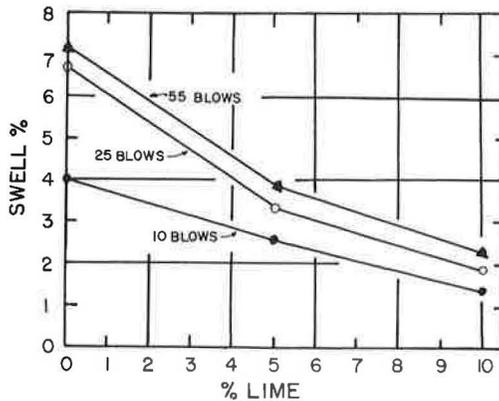


Figure 11. Effect of lime on swell.

Table 6. The values of percent swell against percent lime added are plotted as shown in Figure 11. The following trends are observed:

1. An increase in lime content decreases the percent swell. The reduction in swell percentage is relatively more significant with the addition of smaller percentages of lime.
2. Figures 3, 5, and 7 indicate that moisture content at which compaction is carried out has a very important influence on swell percentage. Appreciable swell is observed on the dry side of optimum moisture content with values decreasing as optimum moisture content is reached. Little decrease in swell results as moisture content is increased beyond optimum. Also, on the dry side of optimum, an increase in compactive effort causes an increase in amount of swell. However, on the wet side of optimum, the increase in compactive effort does not increase swell.

CONCLUSIONS

The following represents a summary of the trends shown by this study:

1. The plastic properties of the soil were improved appreciably by the addition of hydrate lime. There was a marked decrease in liquid limit, and the plastic limit was decreased; consequently, the plasticity index was decreased considerably. Little change in plastic properties was obtained by adding more than 3 percent lime.
2. Addition of hydrated lime materially increased the strength of soil as measured by the CBR method. Five percent lime addition increased the CBR-soaked values more than ten times. With the addition of more lime (up to 10 percent), CBR values increased further. However, the addition of more than 5 percent lime produced relatively less significant increases.
3. Increases in compactive effort were more effective on lime-treated soil than on untreated soil.
4. For the natural soil, the values of CBR as molded were generally greater than those of CBR soaked for 4 days, but with the addition of lime the situation was reversed and the CBR-soaked values observed were greater than those of CBR as molded.
5. Although the increase in lime content increased the CBR as molded, the change was not as pronounced as for CBR soaked.
6. The addition of hydrated lime reduced the swell percentage of the clay soil appreciably. The effect of lime addition on dry density and optimum moisture was found to be negligible in this study.
7. The CBR test on natural soil and soil-lime mixture using varying compactive effort and moisture content indicated that strength is a function of both density and moisture content. Maximum strength can be developed only by exercising proper control over moisture content and density.

Curing Lime-Stabilized Soils

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The study investigated the possibility of establishing a correlation between the strengths of field-cured specimens of lime-stabilized soils and those of laboratory-cured specimens. Several sets of specimens of each soil were subjected to field curing during different parts of the spring, summer, and fall of 1961, and only the temperature variable was investigated. The data were analyzed on a strength vs maturity basis. The results obtained indicate that a strength prediction based on 2 days at 120 F laboratory curing can be made with reasonable accuracy. Temperature data recorded throughout the study suggested seasonal limits for lime stabilization.

•ONE PROBLEM in highway construction today is the scarcity of suitable base or sub-base materials. A solution offered to the highway engineer is the stabilization of soils otherwise classified as "below specification" or "unsuitable." A major field in stabilization is the use of cement as an additive. Soil-cement and cement-modified soils are successfully used as parts of flexible pavements or as a firm bed under rigid pavements. However, it has been observed that cement cannot be used as successfully with cohesive soils as with more granular soils.

Supported by a great amount of laboratory and field data, most researchers agree that hydrated lime or quicklime should be used to stabilize cohesive soils. However, though the results of laboratory and field research on lime stabilization provide the highway engineer with useful knowledge, it is often difficult to compare the work of different researchers because of the wide range of curing temperatures, temperature levels, and periods of time used to cure soil-lime mixtures.

It is agreed that especially for any kind of strength tests, soil-lime specimens should receive a certain amount of curing before being tested. Further, standardizing this curing period would be a major step toward better understanding and generalization of all future testing and research.

It is fairly common knowledge that the use of lime is severely restricted by climatic conditions; this explains why lime is used more in southern States than in northern regions. Virginia, having a moderate climate, shows great possibilities for lime stabilization. However, where lime is used, the construction season should be limited to summer months and a quick method of evaluating the properties of the stabilized mixtures, together with an accelerated curing period, is needed.

PREVIOUS EXPERIENCE

In 1960 a study (1) was initiated to correlate the gain of strength of lime-stabilized soil specimens cured under two conditions: (a) simulated field curing, and (b) accelerated laboratory oven curing. It was thought that specimens of lime-stabilized soil buried under an artificial pavement, at proper depth, would cure in a manner similar to that of a stabilized soil in regular construction.

Two soils were chosen for study. One showed possibilities as a stabilized base, the other as a stabilized subbase. A typical pavement was chosen for the design of the simulated road. Unconfined compressive strength specimens and California Bearing Paper sponsored by Committee on Lime and Lime-Fly Ash Stabilization.

Ratio specimens of each soil were buried at average base and subbase depths of the chosen typical pavement. These specimens were removed from the simulated road at random and tested at 15-day intervals. The values obtained from the unconfined compressive strength specimens were compared with those of similarly molded specimens oven cured at 120 and 140 F. The values obtained from the field-cured CBR's were compared to the values obtained from specimens cured in the moist room.

The conclusions derived from the study (3, 4) indicated the following:

1. There could be a correlation between field-cured and oven-cured unconfined compressive strength specimens.
2. The 120 F oven curing proved to be a more realistic procedure than did the 140 F curing.
3. The results of CBR tests were widely dispersed and no conclusions were derived from them.

On the basis of these conclusions the study was continued on a larger scale to permit generalization of the findings. However, several changes were made:

1. The CBR specimens were omitted.
2. Only 120 F oven curing was used as laboratory accelerated curing.
3. A two-pen temperature recorder was installed at the simulated road and temperatures at assumed base and subbase levels were recorded.

PURPOSE AND SCOPE

It was the purpose of the present study to develop an accelerated curing method under standardized conditions. This, in turn, was to be the basis for a quick laboratory method of determining the suitability of a soil for lime stabilization. The kind of test for the laboratory method of evaluation will be the subject of a separate study (2). This paper reports only the attempts made to find a correlation between an accelerated laboratory curing and simulated field curing.

The study was limited to soils chosen as representative of most Virginia soils under consideration for lime stabilization. Most of the soils sampled were from experimental lime stabilization projects previously installed in Virginia. All soils were stabilized with 5 percent lime. However, to determine if the findings with 5 percent lime were true for another percentage, one soil was stabilized with 3 percent and 5 percent lime and another soil with 5 percent and 8 percent lime.

The study did not consider such variables as movement of moisture or repetition and magnitude of loads, but was concerned with the effect of maturity (temperature \times time) on gain of strength.

PROPERTIES OF SOILS TESTED

The physical properties of the six soils chosen for study are given in Table 1, and a location map is shown in Figure 1.

Soil A is a clayey sand common in the northeastern part of the State of Virginia. Pilot tests showed that the soil reacts very well with lime and produces fairly high unconfined compressive strength and resists freeze-thaw and wet-dry tests satisfactorily (5). The pit contains from 30 to 60 percent plus No. 4 material; however, due to the small size of the strength specimens used only the portion of the soil passing the No. 4 screen was sampled. It is believed that when stabilized with lime and covered with adequate surfacing this soil could be used as an alternate to the locally available crushed aggregate. The streets of several residential subdivisions in Fairfax County that were accepted into the State secondary system were built with unstabilized material similar to this soil and created great maintenance problems. During the summer of 1961 several of these streets were treated with cement and lime to help reduce the maintenance problem. These partly experimental projects, though far from being research projects because of the oversized particles used and the lack of compaction control, can serve as a guide towards the use of this soil as a stabilized base.

Soil B is a micaceous silt regarded as being a "troublesome" soil in Virginia. The soil was sampled from an experimental lime, lime-fly ash, and cement stabilization section built in 1956 on Project 1770-03, US 58 Patrick County (6), where it was used as a stabilized subbase.

Soil C is a clayey sand. It was sampled from an abandoned pit from which nearly all the soil was used for an experimental lime stabilization project on Route 641, Isle of Wight County in 1957-58 (6). The soil is not representative of that used on the experimental project, being considerably more sandy. However, at the time it was thought beneficial to include a sandy soil in the accelerated curing study because sands are known to react less with lime than finer grained soils do.

Soil D, a silty clay, was sampled from Project 0460-035-101, US 460 in Giles County, where part of the subgrade was stabilized with hydrated lime. The project was constructed in 1961.

Soil E, a heavy clay, was sampled from the experimental lime stabilization project in Rockingham County, Route 276, Project 0276-082-005. The project was built in 1959 (7).

Soil F is a clayey silty shale. It was sampled during the construction on Project 0050-034-101, C 501, US 50, Frederick County. The project was built in 1961 and the in-situ soil was stabilized with lime to serve as subbase.

About 600 lb of each soil were air dried, screened through the No. 4 sieve, mixed thoroughly, and stored in closed containers. The amount of material retained on the No. 4 screen was recorded and discarded. The gradations in Table 1, therefore, reflect the properties of the minus No. 4 soil.

TEST PROCEDURE

Size and Number of Specimens

Previous experience (1, 3) had indicated that at least 11 or more specimens had to be used for each period of field curing and oven curing in order to obtain satisfactory precision at the 95 percent confidence level. For this reason the Harvard Miniature Mold size (1.34-in. diameter and 2.78-in. height) was chosen as the size of the unconfined compression test specimens to keep the amount of soil needed to a minimum and to obtain a homogeneous mixture. The ease of molding specimens of this size permitted use of 15 specimens for each curing period.

Molding

The maximum density and the optimum moisture content of each soil, with 5 percent hydrated lime added, were determined in accordance with AASHTO T99-57 Method A.

The Harvard Miniature Mold was used for molding specimens; however, the spring hammer furnished by the manufacturer was replaced with a home made drop hammer. The hammer was constructed to have a 1-lb weight falling 10 in. The diameter of the hammer head was one-half that of the mold.

Using the Harvard Miniature Mold and this hammer it was observed that at optimum moisture content five layers and fifteen blows per layer would produce a density a little above the maximum obtained by AASHTO T99-57 Method A.

All specimens were molded close to their optimum moisture content, wrapped with aluminum foil, and coated with a special wax to preserve the molding moisture content during the curing period. The different stages of preparing specimens are shown in Figure 2.

Field Curing

It was estimated that a lime-stabilized pavement will get a curing of about a month or two before it is opened to traffic. A round figure such as 3,000 degree-days was chosen as basis of comparison. At base and subbase levels this maturity can be reached in about 40-45 days during summer months, and of course it will take longer during winter, fall, and spring. However, it is believed that maturities achieved at different levels (or ranges) of temperature might not affect the lime-stabilized specimens simi-

TABLE 1
PHYSICAL PROPERTIES OF SOILS TESTED

Property	Soil					
	A	B	C	D	E	F
HRB classification	A-2-6 (0)	A-4 (1)	A-2-7 (0)	A-7-6 (20)	A-7-6 (20)	A-4 (2)
Description	Clayey sand	Micaceous silt	Clayey sand	Silty clay	Clay	Clayey silty shale
Origin	Coastal Plain	Piedmont	Coastal Plain	Valley and Ridge Province	Valley and Ridge Province	Valley and Ridge Province
Percent passing:						
No. 4	100	100	100	100	100	100
No. 10	86	99	98	89	100	70
No. 20	68	97	89	87	99	57
No. 40	45	89	58	86	98	50
No. 60	31	76	29	84	98	46
No. 80	23	63	20	84	97	44
No. 100	26	55	16	83	97	44
No. 200	25	39	12	82	96	41
Percent silt (0.05-0.005 mm)	3	29	6.6	12.9	10	22
Percent clay (0.005 mm)	21	4	5.5	63.2	84	16
Specific gravity	2.70	2.75	2.67	2.74	2.77	2.72
Liquid limit (%)	35	40	55	63	75	29
Plasticity index (%)	11	N. P.	26	35	48	8
Max. den. with 5% lime (pcf) ^a	117.0	94.8	122.0	87.5	88.0	110.0
Opt. moist. cont. with 5% lime (%) ^a	13.4	24.4	11.0	30.0	29.0	16.0
Considered for	Base	Subbase	Base	Subbase	Subbase	Subbase

^aAASHTO T-99

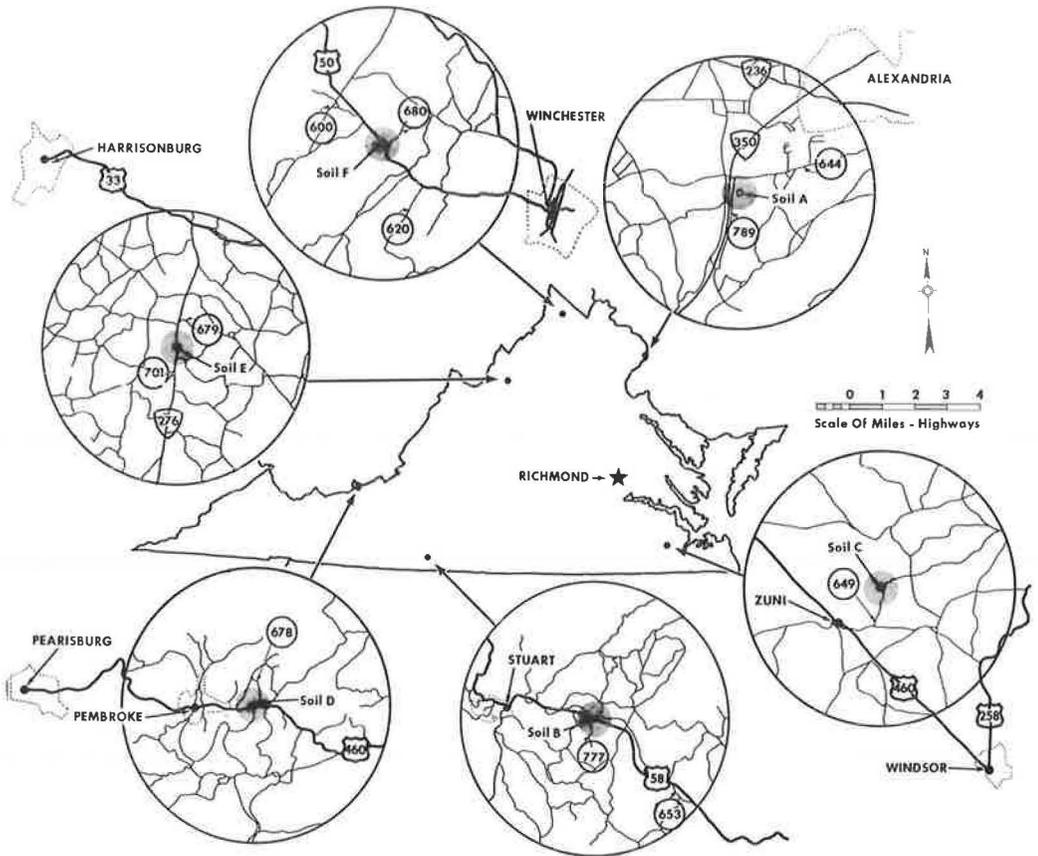


Figure 1. Location of soils investigated.

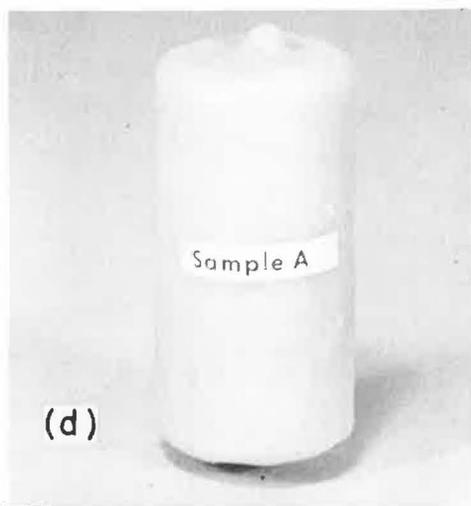
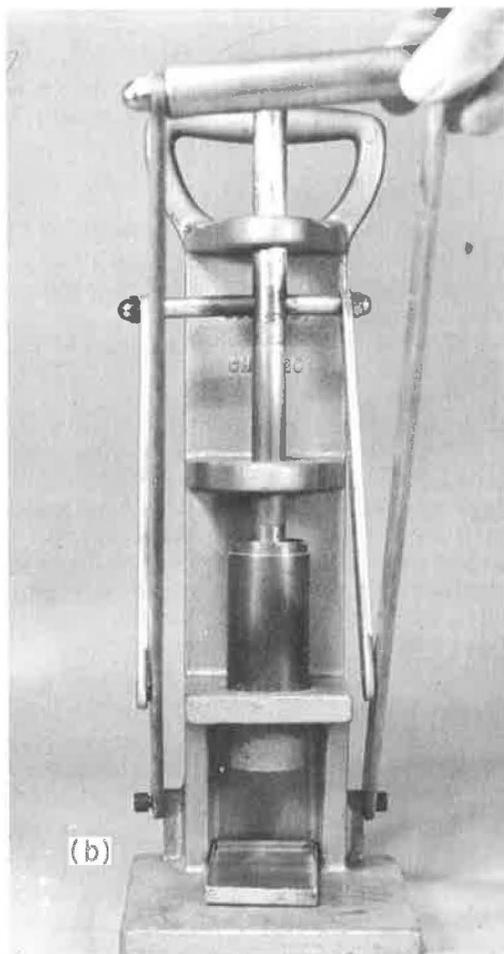


Figure 2. Stages of molding specimens:
(a) molding equipment, (b) ejection of
specimen, (c) waxing, (d) final specimen.

larly. For this reason temperatures at air and assumed base and subbase level were continuously recorded during the study. This subject is discussed later.

Generally, maturity is defined as the summation of the products of temperatures with their durations. However, for each specific purpose, the datum temperature should be specified. Therefore, unless otherwise specified, maturity will mean 0 F as the datum temperature.

Field-Curing Schedule

The study was started in June 1961. Forty-five unconfined compression test specimens of each soil were molded and subjected to field curing. Figure 3 shows the simulated road and the location of test specimens according to their uses as base or subbase materials.

At periods of 30, 45, and 60 days, 15 randomly selected specimens were tested for unconfined compressive strength. The total of 45 specimens is referred to as Series I. When all specimens of one soil had been tested, another set of 45 specimens (Series II) was molded and subjected to field curing and tested in the manner described.

Series III of each soil was molded during the fall of 1961. Because not much strength development was expected due to low temperatures, only 30 specimens of each soil were made and these were randomly tested in two groups of 15 each at maturities around 2,500 and 3,500 degree-days, respectively. A schematic representation of curing periods for each soil and average temperatures and accumulated maturities for each month are shown in Figure 4.

Laboratory Curing and Testing

Forty-five specimens of each soil were molded and cured in a 120 F oven. At periods of 1, 2, and 3 days, 15 random specimens were tested. The temperatures maintained during laboratory curings were within ± 5 F.

All specimens were tested for unconfined compressive strength at room temperature ($70 \text{ F} \pm 5 \text{ F}$) and at a rate of strain of about 0.12 percent per second.

RESULTS

The unconfined compressive strengths of field-cured and laboratory-cured specimens were compared. Figure 5 shows the average unconfined compressive strength and the 95 percent confidence limits are plotted against maturity. From the curves

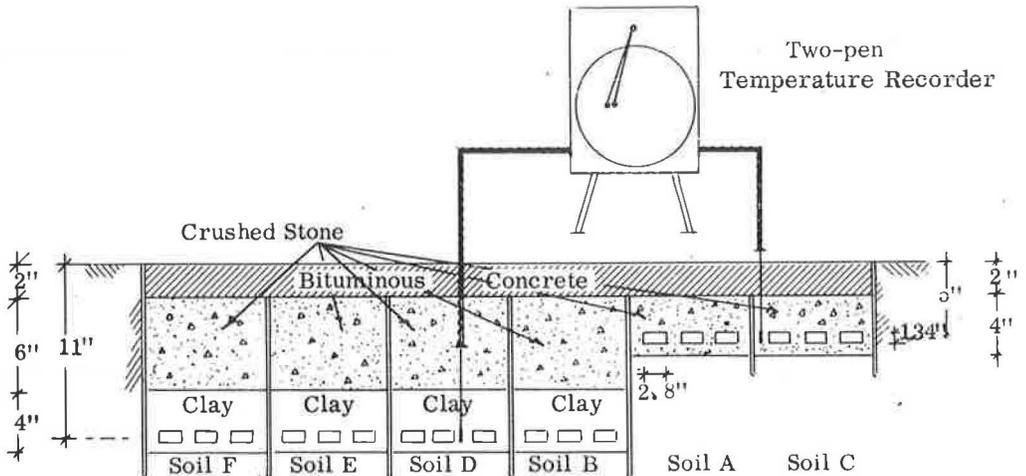


Figure 3. Simulated road and depth of field-cured specimens.

Soil	Month of 1961																				
	June			July			August			September			October			November			December		
A							-----														
B	-----						-----						-----								
C	-----						-----						-----								
D	-----						-----						-----								
E	-----						-----						-----								
F	-----						-----						-----								
	Air	Base	Sub-base	Air	Base	Sub-base	Air	Base	Sub-base	Air	Base	Sub-base	Air	Base	Sub-base	Air	Base	Sub-base			
Ave. monthly temp., deg. F	72	74	72	76	78	77	76	78	78	73	76	77	60	62	64	51	52	54	36	37	38
Maturity above 0 F, deg. days	2074	2139	2096	2359	2423	2368	2356	2414	2411	2192	2278	2296	1856	1937	1983	1515	1573	1631	1111	1136	1190
Maturity above 30 F, deg. days		689	646		873	838		864	861		778	796		387	433		130	159		0	0
Maturity above 60 F, deg. days		399	356		563	528		554	551		478	496		108	135		21	24		0	0

LEGEND: Series I _____ Series II - - - - Series III

Figure 4. Field-curing schedule and maturity data.

obtained, the time required for laboratory-cured specimens to reach a strength equivalent to that of field-cured specimens which received 3,000 degree-days of maturity can be determined. The determinations for Series I and Series II are summarized in Table 2.

In Figure 5, the 95 percent confidence limits and the average values of strength achieved with simulated field and laboratory curing were compared. This approach was considered realistic because the confidence limits reflected the variation in each set of 15 specimens. From the figure it might seem that the range of the accelerated curing period for this soil is rather large (0.7 days). However, the strength values are within 16 psi, or a variation of ± 8 psi. For each soil, the range and variation of strength values are shown in the last column of Table 2. Soil E showed the maximum range and variation, 52 psi and ± 26 psi, respectively. This variation seems reasonable in soils work. The coefficient of variation and the confidence limits for strength values for each type and period of curing are given in Table 3.

The values in Table 2 for the mixtures containing 3 and 8 percent lime indicate that the findings for 5 percent lime are also applicable for other percentages.

For practical purposes the average of all accelerated curing periods (last figure in the accelerated curing day column in Table 2) can be used as a standard. It is believed that this average curing period of 2 days at 120 F is realistic. It will predict, within reasonable limits, the unconfined compressive strength that can be developed by a soil at a time when it is needed; i. e., about 40 to 45 days after construction. As a contrast some of the presently used curing periods for lime-stabilized soils, such as

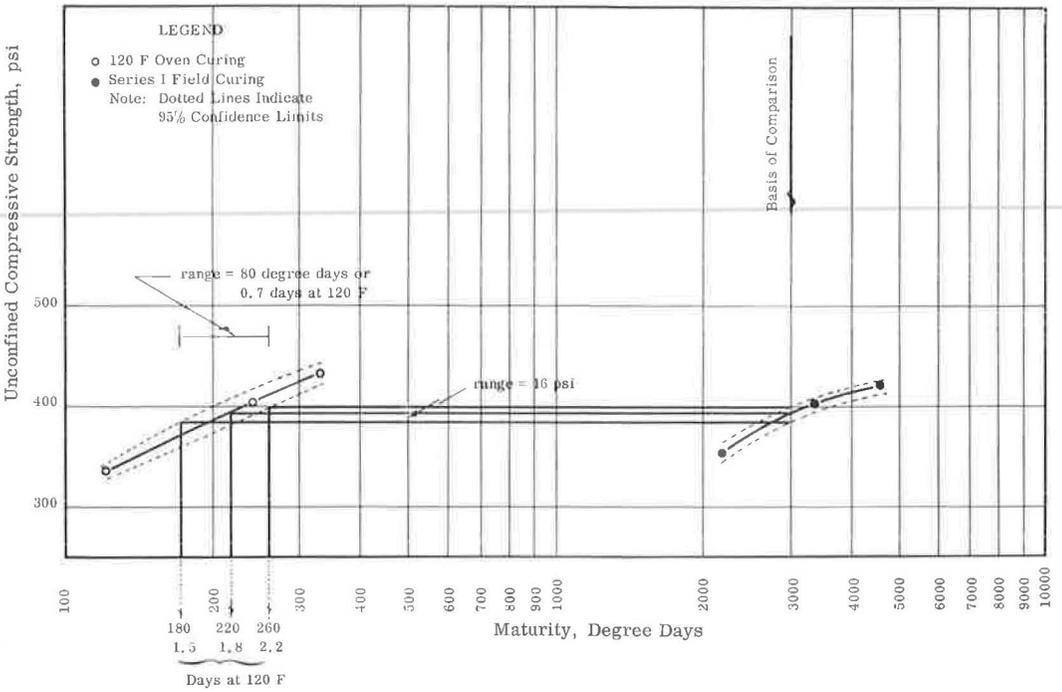


Figure 5. Example of comparison of field and oven curing.

TABLE 2
 TIME OF ACCELERATED CURING AT 120 F TO ACHIEVE 3,000 DEGREE-DAYS FIELD STRENGTH

Soil	Lime Content (%)	Series	Accelerated Curing (days)	Unconfined Compressive Strength (psi)	
				Average	Variation
A	5	I	2.5	450	± 20
		II	2.4	433	± 14
B	5	I	1.9	240	± 8
		II	2.1	249	± 5
C	5	I	2.3	45	± 3
		II	1.9	49	± 3
D	5	I	1.8	395	± 8
		II	1.4	361	± 9
E	5	I	2.2	490	± 26
		II	2.4	500	± 7
F	5	I	2.2	304	± 8
		II	1.4	269	± 5
A	8	I	1.5	217	± 20
D	3	I	2.0	202	± 11
Avg.			2.0		

TABLE 3
COEFFICIENT OF VARIATIONS OF ALL UNCONFINED COMPRESSION TESTS

Soil	Series	Coefficient of Variation					
		Simulated Field Curing			120 F Curing		
		30 Days	45 Days	60 Days	1 Day	2 Days	3 Days
A	I	8.6	5.0	6.3			
	II	5.5	6.5	5.9	5.8	4.3	5.7
	III	-	5.3	3.9			
B	I	9.4	5.6	4.4			
	II	2.9	3.6	3.4	8.7	3.5	2.9
	III	-	11.0	14.0			
C	I	10.1	9.8	-			
	II	15.5	14.0	13.4	7.2	7.7	4.1
	III	-	9.8	8.4			
D	I	4.9	2.9	5.4			
	II	4.4	3.5	4.0	3.7	5.6	4.3
	III	-	2.9	5.0			
E	I	10.0	8.7	4.3			
	II	3.5	2.6	3.7	5.6	4.8	13.3
	III	-	5.6	7.2			
F	I	4.0	3.1	3.2			
	II	3.7	2.8	7.2	3.6	3.3	2.6
	III	-	4.1	3.6			

5 or 7 days at 140 F, might reflect unconfined compressive strength attainable at a year or two after the stabilization process. Data from previous experiments, although limited, might illustrate this point (3).

In Figure 6 the unconfined compressive strengths of a stabilized soil, cured in the simulated road and at 140 F in the oven, are plotted against their ages. From the shape of the curve, after 60 days of field curing the soil specimens reached their maximum strength, whereas the curve for the 140 F oven-cured specimens shows little tendency for leveling off. Even if the laboratory curve did level off after a curing of 5 days at 140 F, the strength reached at that time might never be reached with field curing.

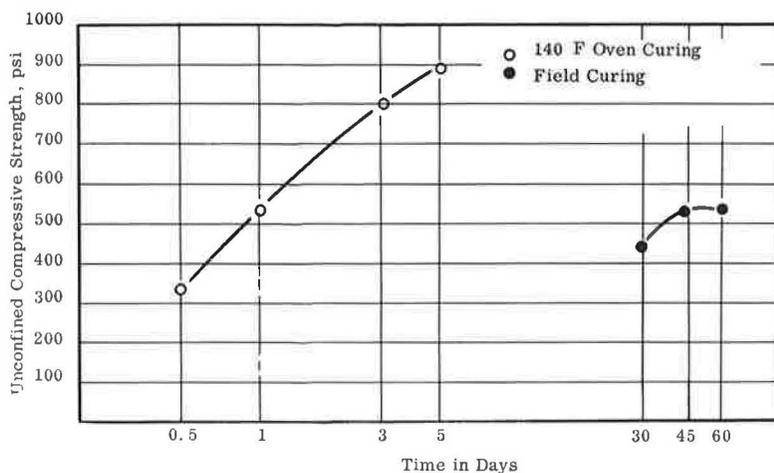


Figure 6. Comparison of field curing and 140 F oven curing.

Season Limits to Lime Stabilization in Virginia

The foregoing portion of the paper deals with the major intention of the study. However, from the temperature and strength data obtained throughout the study, important corollary information was developed. In Figure 7a the strengths of field-cured specimens of a soil are plotted against their maturities. The two curves represent the gain of strength for Series I and III, with Series III of each soil being subjected to field curing after the middle of September 1961 (Fig. 4). From the temperature data, at the lower part of Figure 4, it can be seen that both the average monthly temperatures and maturities are lower for October than for the preceding months, and the same is true for November and December.

In Figure 7a, curves for Series I and Series III are obtained by plotting strength values against their maturities, taking 0 F as datum temperature. With this method the two curves do not follow the same path. However, if a higher datum is taken for calculating maturities (e. g., 50 F) and the strength values are plotted against these maturities, both curves change position and come closer to each other (Fig. 7b). The important point is that the maturities of Series I are decreased by a constant value

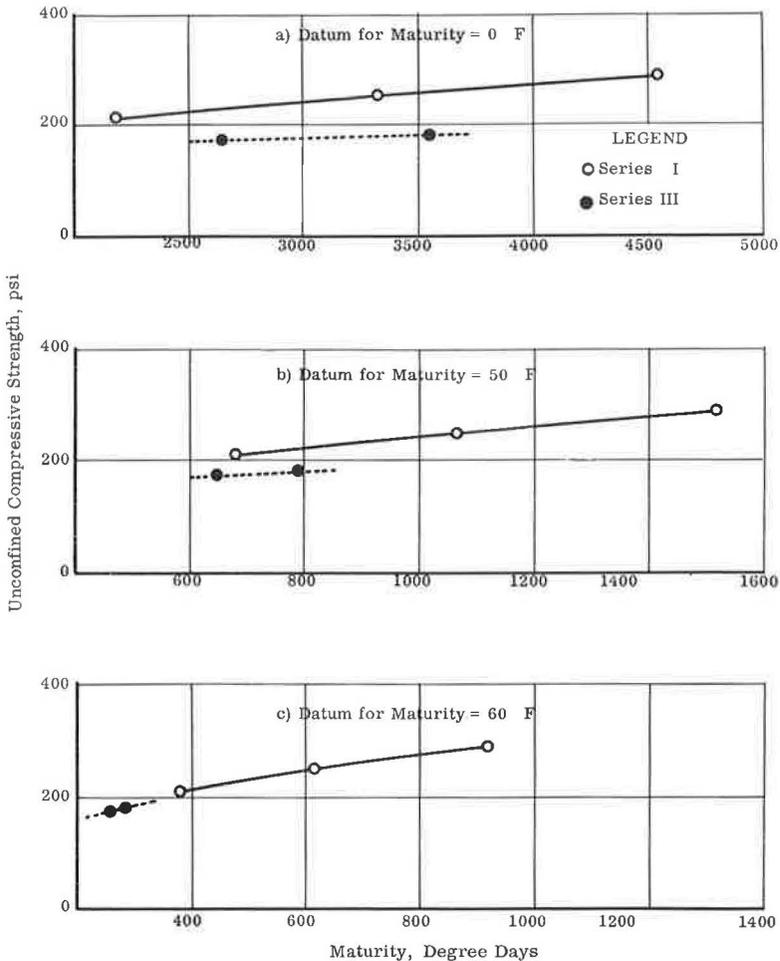


Figure 7. Strength data for Soil B vs maturities with different datum.

TABLE 4
ANALYSIS OF STRENGTH-MATURITY
DATA FOR SEASON LIMITS FOR LIME
STABILIZATION

Soil	Series	Min. Temp. Required for Lime to React ^a (°F)
A	I	55
	II	55
B	I	60
	II	58
C ^b	I	-
	II	-
D	I	53
	II	49
E	I	49
	II	52
F	I	50
	II	50

^aRange for soils tested = 49 to 60 F.

^bResults not conclusive for this sandy soil.

because there were no temperatures below 50 F during its field curing. On the other hand, the curve for Series III is affected more, because its field-curing period did have temperatures below 50 F. Using this method, and taking different datum temperatures for maturity calculations, points for Series I and Series III might be made to lie on the same curve (Fig. 7c).

It is assumed that only one strength-maturity curve exists for each stabilized soil, with the condition that the minimum temperature necessary for lime to react is provided. If this assumption is valid, then the temperature at which the points for Series I and Series III lie on the same curve is the minimum temperature required for lime to react.

The strength-maturity data of each soil and of each series were analyzed by the preceding method. Table 4 summarizes the temperatures for which the points for Series III of each soil will lie on the same curve as those for Series I and Series II. In the table this temperature is referred to as "the minimum temperature required for lime to react."

The data on soils included in this study, with the exception of Soil C which showed very little reaction with lime (Table 2), indicate that not much reaction of lime with soil should be expected at temperatures below 49 or 50 F.

It was mentioned earlier that a curing of 2 days at 120 F would correspond to about 3,000 degree-days, or 40 to 45 days of simulated field curing during summer months. This "3,000 degree-days" refers to a maturity calculated with 0 F as datum temperature. From the preceding discussion it is seen that for limiting the construction season for lime stabilization the datum temperature should be taken as 50 F, if the objective is to obtain a gain of strength. This would mean a reduction of about 2,250 degree-days (45 days × 50 F), leaving about 750 degree-days of maturity calculated with 50 F as datum temperature. Therefore, it is suggested that lime stabilization should not be attempted at a date after which a maturity of 750 degree-days (calculated with 50 F as datum temperature) is not anticipated to accumulate.

Figure 4 shows that in the simulated road, at subbase level, the maturities accumulated during October, November, and December were 433, 159, and 0 degree-days, respectively (50 F as datum temperature). This gives a total of 592 degree-days which, because it is below the needed maturity, indicates that in 1961 lime stabilization should not have been attempted after the middle or end of September.

As a word of caution, it is not intended that lime stabilization be limited to summer months. The hypothesis presented merely urges the construction engineers to be cautious in setting final dates, if any cementation is desired. Local weather forecasts, though applicable only to air temperatures, might be helpful to the highway engineer because temperatures and maturities for air are close to those at the subbase and base levels of the simulated road, as can be seen in Figure 4.

CONCLUSIONS

The data obtained for the six soils investigated and the use of 5 percent hydrated lime of one brand indicate the following:

1. Soil-lime specimens subjected to field curing in a simulated road will show an increase in unconfined compressive strength. However, the amount and rate of gain of strength will depend on the type of soil and climatic effects.

2. At Charlottesville, Va., during the summer of 1961, about 40 to 45 days were required to develop a maturity of 3,000 degree-days if 0 F is taken as datum temperature (or 750 degree-days, if 50 F is taken as datum temperature).

3. The unconfined compressive strength of soil-lime specimens having this maturity could be predicted by an accelerated curing of two days at 120 F.

4. If it is assumed that only one strength-maturity curve exists for each stabilized soil, then not much reaction of lime with soil should be expected at temperatures below 50 F.

ACKNOWLEDGMENTS

The author expresses his gratitude to B. B. Chamblin, G. A. Gatlin, and L. R. Staunton for helping in every phase of the study. Special thanks go to T. E. Shelburne, Director of Research, for his general guidance.

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Discussion

MANUEL MATEOS and JERRY W. H. WANG, Soil Research Laboratory, Iowa State University, Ames, Iowa —The author is to be complimented for the effort of correlating the strength of field cured soil-lime specimens with those cured in the laboratory at high temperature.

The writers' studies show that different limes develop strength at different rates depending on the curing temperature (8, 9, 10). An example of this is given in Table 5, where dolomitic monohydrate lime gives much higher strength than high-calcium hydrated lime at 70 F curing temperature, but high-calcium hydrated lime gives slightly higher strength than dolomitic monohydrate lime at 120 F curing temperature. How is the lime variable taken into account in the 2 day 120 F quick method for strength prediction?

Table 6 shows that a soil stabilized with dolomitic monohydrate lime has about twice as much strength as with calcitic hydrated lime at a curing temperature of 50 F. Should this suggest that different seasonal limits be used for different types of lime in soil-lime stabilization works? It may also be of interest to know the type of lime used in the studies presented in the paper.

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8. Davidson, D. T., Mateos, M., and Barnes, H. F., "Improvement of Lime Stabilization of Montmorillonitic Clay Soils with Chemical Additives." HRB Bull. 262, 33-50 (1960).

TABLE 5
COMPARISON OF STRENGTHS OF SOILS TREATED WITH TWO TYPES OF
LIME AND CURED AT DIFFERENT TEMPERATURES^a

Soil	Lime		Immersed Unconfined Compressive Strength (psi)			
	Type	Amount (%)	Cured at 70 F			Cured at 120 F
			7 Days	28 Days	84 Days	2 Days
Plastic loess	High-calcium hydrated	2	60	65	60	130
		5	85	110	180	330
		8	85	110	210	485
		12	100	130	290	440
	Dolomitic monohydrate	2	43	45	30	170
		5	105	210	280	380
		8	135	230	300	400
		12	150	270	340	370
Friable loess	High-calcium hydrated	2	80	120	210	210
		5	80	130	350	400
		8	80	150	410	370
		12	80	150	360	360
	Dolomitic monohydrate	2	105	170	160	160
		5	150	350	600	310
		8	160	390	580	330
		12	170	400	580	320

^aAnalyses of soils and limes given elsewhere (1).

TABLE 6
COMPARISONS OF STRENGTHS OF A KANSAN TILL SOIL TREATED WITH TWO
TYPES OF LIME AND CURED AT DIFFERENT TEMPERATURES^a

Lime Type	Amount (%)	Immersed Unconfined Compressive Strength (psi)					
		Cured at 50 F		Cured at 70 F		Cured at 100 F	
		7 Days	28 Days	7 Days	28 Days	7 Days	28 Days
High-calcium hydrated	6	86	125	129	179	358	765
Dolomitic monohydrate	6	150	249	223	321	402	642

^aAnalyses of the soil and limes given elsewhere (1).

9. Mateos, M., and Davidson, D. T., "Physical and Mineralogical Factors in the Stabilization of Iowa Soils with Lime and Fly Ash." Iowa State University Engr. Exp. Sta. Bull. 196, 175-272 (1962).
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M. C. ANDAY, Closure—Thanks are due Messrs. Mateos and Wang for their discussion. As explained in the paper, the determination of a curing period that will predict field-cured strengths at a time when the treated pavement will be subjected to traffic was the first step of a general study. This study is being continued. The scope of the study is to establish minimum unconfined compressive strength values for laboratory specimens to qualify soils for base or subbase lime stabilization. This will be accomplished by comparing the performance of several lime stabilization projects in Virginia, with the strength of specimens prepared in the laboratory. These specimens will be pre-

pared from soils obtained from these projects, mixed with identical percentages of lime, and cured for 2 days at 120 F. Once the minimum unconfined compressive strength is specified, then it will be a matter of testing to determine how to obtain this strength, if attainable. The type of lime to be used will be a matter of concern when testing shows that the addition of one type of lime will not give the desired strength and that the addition of another type will. For this reason the lime variable was omitted.

Furthermore, experience has shown that even though different types of limes are used for stabilizing a soil, the strength development will be close enough for practical purposes. The author would like to illustrate this by using the discussor's data. However, it should be kept in mind that it is not the intention of the author to generalize the illustration but just to point out a practical aspect of lime stabilization.

Figure 8 shows the strength of the "plastic loess," tested by the discussors, with different percentages of two types of lime. The shaded area between the two curves indicates the practical percentages of lime that are used in field work (3 to 7%). Assuming that (a) the samples were randomized and (b) the results were calculated statistically, the differences between mean strengths are not more than 40 or 50 psi. The author does not consider this significant. The many variables involved in conducting an unconfined compression test on a laboratory prepared and cured soil-lime specimen can justify the preceding statement.

In addition to these reasons, Virginia's problem due to the lime variable is not nearly as severe as that of some other States because nearly all the limestone used in manufacturing lime is from the New Market—Five Oaks Limestone Formation. The product is a high-calcium lime, which was used in the study.

No further comments are made here as to the effect of different limes on the seasonal limits for lime stabilization, because this was not investigated in the study.

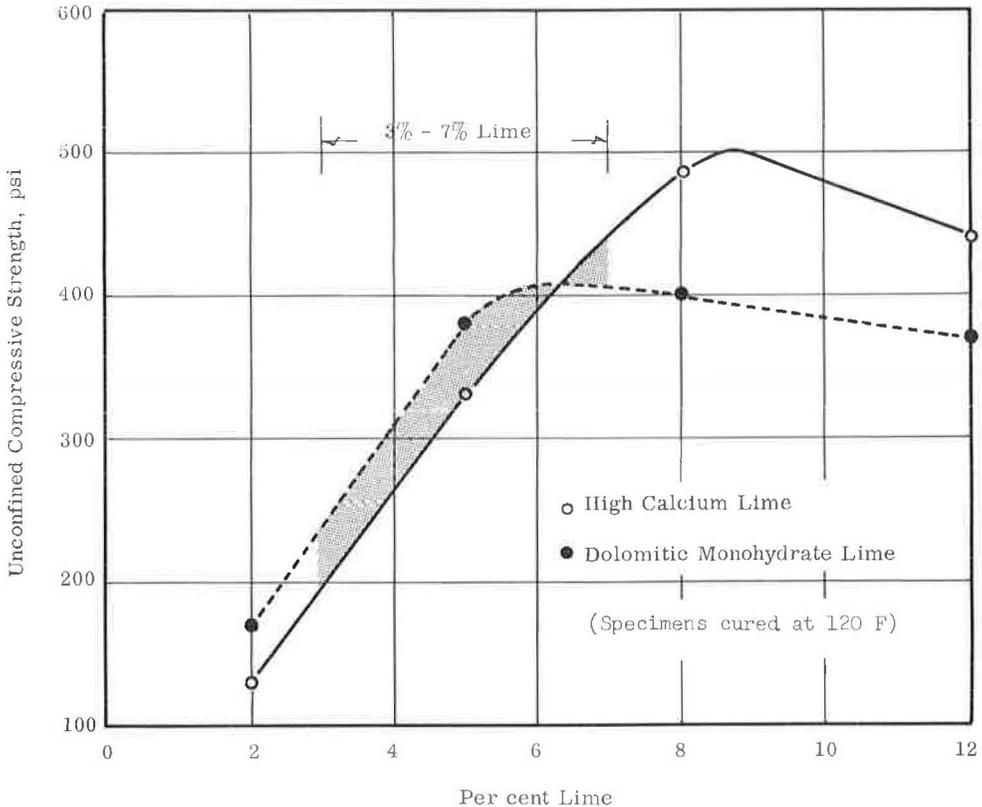


Figure 8. Strength vs percent lime for plastic loess soil.

Compaction Characteristics of Soil-Lime-Fly Ash Mixtures

MANUEL MATEOS and DONALD T. DAVIDSON, Respectively, Research Associate and Director (deceased), Soil Engineering Research Laboratory, Engineering Experiment Station, Iowa State University

Field compaction is one of the most important steps to success in the stabilization of soils. There are several factors that influence compaction, including water content, amount of compaction, temperature of mix materials, and effect of delay in compaction after mixing. A laboratory investigation was made to study these factors using four soils, three fly ashes, and one lime. Specimens were molded near standard and modified AASHO compactive effort and moist cured up to 90 days.

The results indicate that the best compacting moisture for maximum strength is to the dry side of the optimum moisture for maximum density in the sandy soil and on the wet side in the two clayey soils. The temperature of the materials does not have a marked influence on the strength. Of paramount importance is the minimizing of the delay between wet mixing and compaction of the soil-lime-fly ash mixtures when the soil contains clay particles that can react with lime, lowering the density and strength for the same compactive effort. The modified compactive effort gave strengths from 50 to 160 percent higher than the standard.

•FIELD COMPACTION is one of the most important steps in the stabilization of soils. Several factors affect compaction; among them water content, amount and type of compaction, temperature of mix materials, and effect of delay in compaction after mixing. These also affect other kinds of soil stabilization using cementitious compounds (1, 2, 3).

In laboratory investigation, some of the factors that affect the compaction of soil, lime, and fly ash mixtures were studied. The results of this investigation are presented in this paper.

MATERIALS

Dune sand, friable loess, alluvial clay, and gumbotil, all from Iowa, were used. An analysis of the soils and other materials used is given elsewhere (4, 5). The sand was Wisconsin-stage eolian sand, fine grained, oxidized, and leached. The friable loess was a Wisconsin-stage silt, friable, oxidized, and calcareous. The alluvial clay was a recent alluvial fill, plastic, slightly calcareous, with 72 percent 5- μ clay and 1.6 percent organic matter. The gumbotil was a Kansan-stage highly weathered till, plastic, noncalcareous, with 66 percent 5- μ clay. The predominant clay mineral in these soils is montmorillonite.

Three representative fly ashes were selected. Based on the pozzolanic reactivity with lime, fly ash No. 1 is of a medium to good quality, fly ash No. 2 is of a poor quality, and fly ash No. 3 of a very good quality (6).

A commercial grade calcitic (high-calcium) hydrated lime, Ca(OH)_2 was used.

METHODS

The mix proportions used were 76.5 percent soil, 6 percent lime, and 17.5 percent fly ash, on a dry weight basis. Only the soil fraction passing a No. 10 (2-mm) sieve was used.

Molding was started immediately after a batch was mixed, except in the studies on delay of compaction. Test specimens 2 in. indiameter by 2 in. high were molded in the Iowa State compaction apparatus. Five blows on each side with a 5-lb hammer dropping 12 in. were given to approximate standard Proctor compaction (ASTM Designation D698-58T; AASHO Designation T99-57) (7). Ten blows on each side with a 10-lb hammer dropping 12 in. were given to approximate modified Proctor compaction (ASTM Designation D1557-58T and AASHO Designation T180-57) (8).

After being molded, specimens were moist cured at 70 ± 4 F at a relative humidity of over 90 percent. The specimens wrapped in waxed paper and sealed with cellophane tape were placed in the humid room.

After each curing period, specimens were removed from the curing chamber and immersed for one day in distilled water. They were then tested for unconfined compressive strength using a load travel rate of 0.1 in. per min. Tests were run in triplicate; the average strengths are reported.

INVESTIGATION

Moisture-Density and Moisture-Strength Relationships

The most common practice in soil stabilization is to compact specimens at a moisture content as near to the optimum for maximum dry density as possible. Previous tests made at the Engineering Experiment Station of Iowa State University with mixtures of soil, lime, and fly ash showed some differences between the optimum moisture for maximum dry density and that for maximum 7-day strength of a silty soil (7).

Inasmuch as little is known of the effects of molding moisture on the strength of lime-fly ash-stabilized soils, an investigation was conducted to find if there is any correlation between the moisture for maximum strength. Specimens were molded with different moisture contents and were cured for periods of 7, 28, and 90 days.

Two compactive efforts were used—one approximating the standard Proctor and the other approximating the modified Proctor. The soils used were the dune sand, friable loess, alluvial clay, and gumbotil; lime was commercial calcitic hydrated and the fly ashes were No. 3 with all the soils and Nos. 1 and 2 also with dune sand and gumbotil (Figs. 1 to 8).

Dune Sand.—The moisture for maximum dry density and the moisture for maximum 7- or 28-day strength in any of the six sets of mixtures show no correlation (Figs. 1 to 3). The moistures for maximum strength are far to the dry side of the optimum moisture for maximum density. Both moistures of the specimens cured 90 days are closer, but there is a difference of about 2.0 percent for the mixtures compacted at the standard Proctor and 1.0 percent or less for the modified Proctor; the moisture for maximum strength is on the dry side of the optimum moisture for maximum density. The strength curves for 7- and 28-day curing are rather flat, but for 90 days there is a very sharp peak for the maximum strength.

Gumbotil.—The moisture contents for maximum strength for gumbotil contrasted with that for sand are to the wet side of the moisture for maximum density (Figs. 4 to 6). Some of the density and strength curves are rather flat, making it difficult to define the maxima.

Friable Loess.—The moistures for maximum dry density and maximum strength for standard Proctor compaction of friable loess practically coincide (Fig. 7). That is not so for modified Proctor compaction, in which 7- and 28-day curing strength curves, although rather flat, show a maximum strength at moisture contents less than the optimum for maximum density, and a maximum is well defined at a moisture content greater than the optimum for maximum density for 90-day curing.

Alluvial Clay.—The moisture-density curves for alluvial clay do not show a peak for maximum dry density, and the density increases as the moisture content decreases

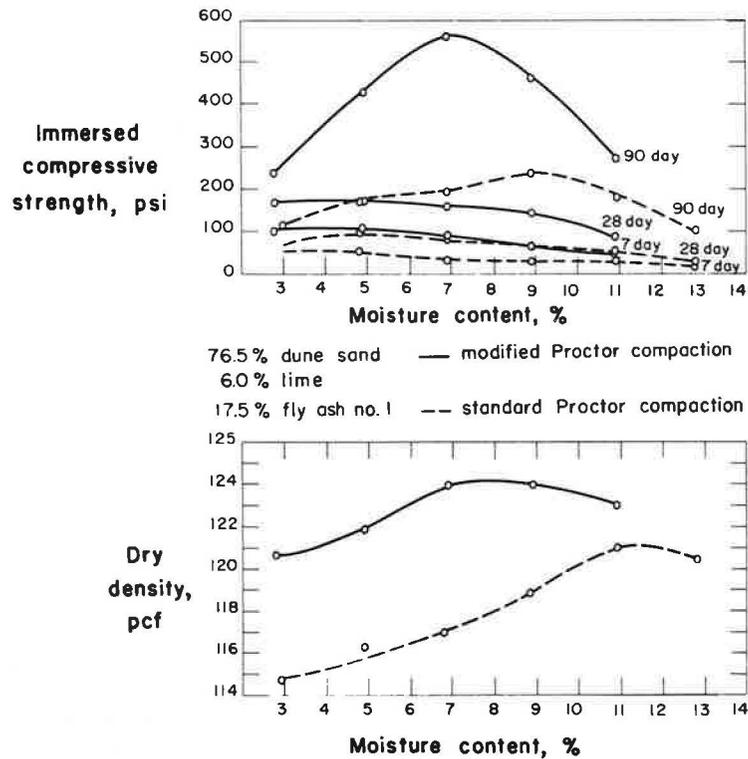


Figure 1. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash No. 1 for standard and modified Proctor compactive efforts.

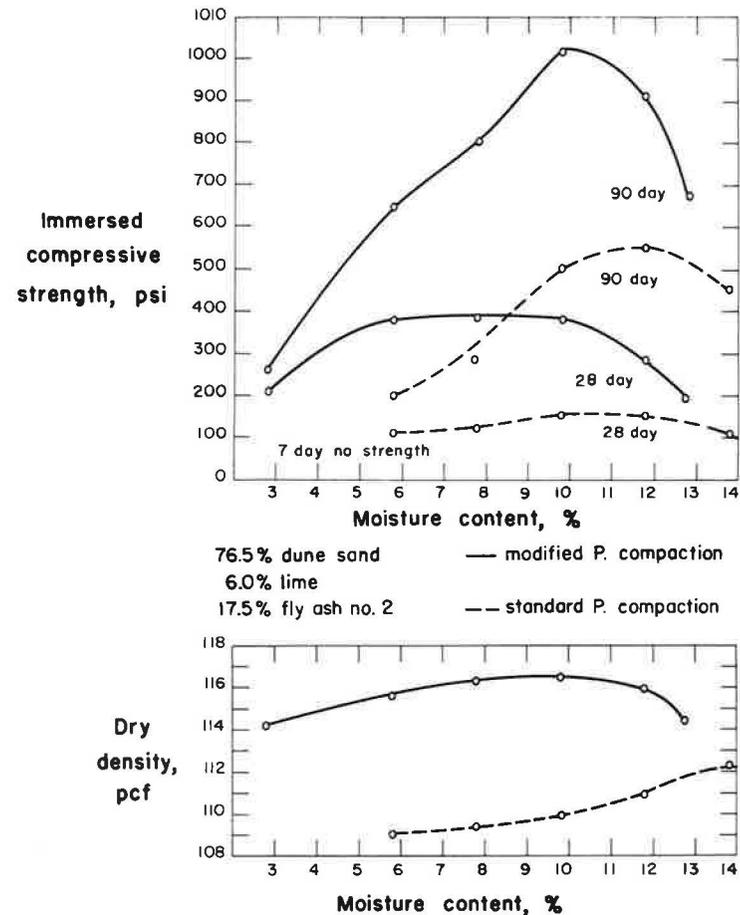


Figure 2. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash No. 2 for standard and modified Proctor compactive efforts.

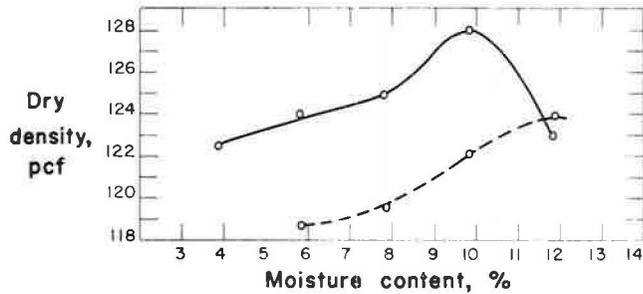
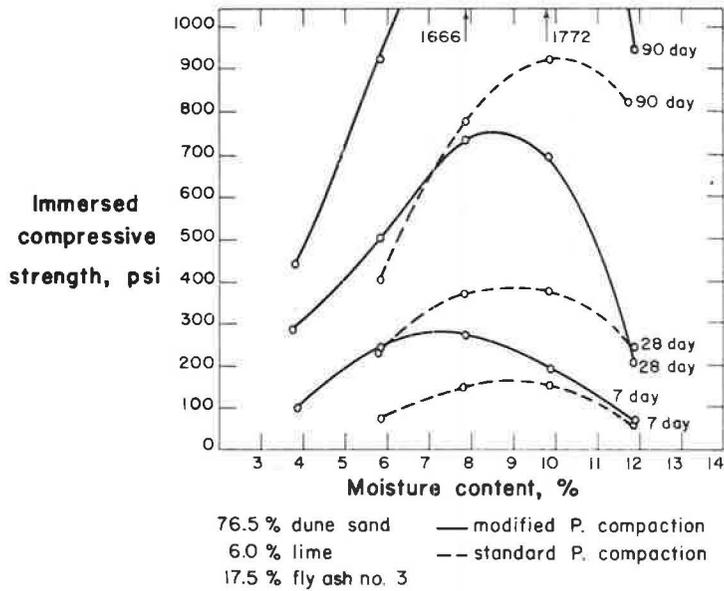


Figure 3. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash No. 3 for standard and modified Proctor compactive efforts.

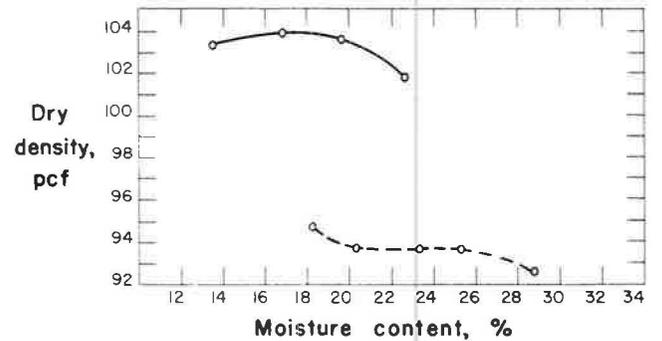
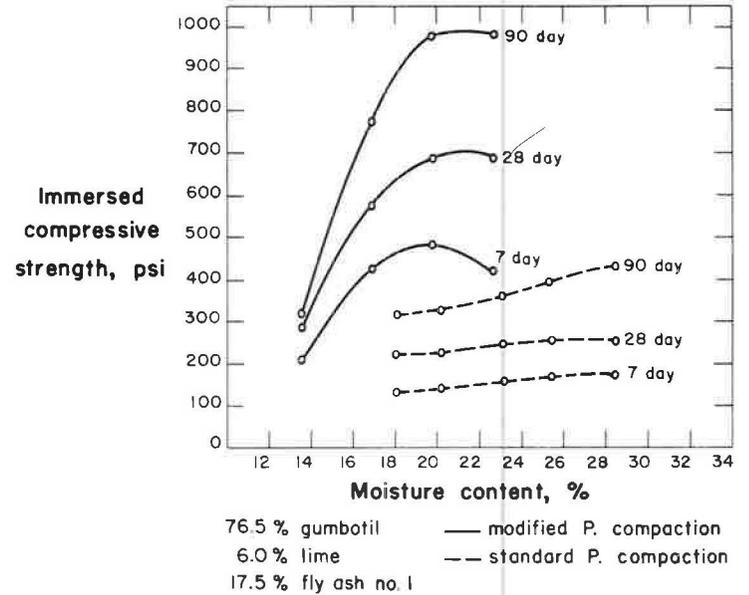
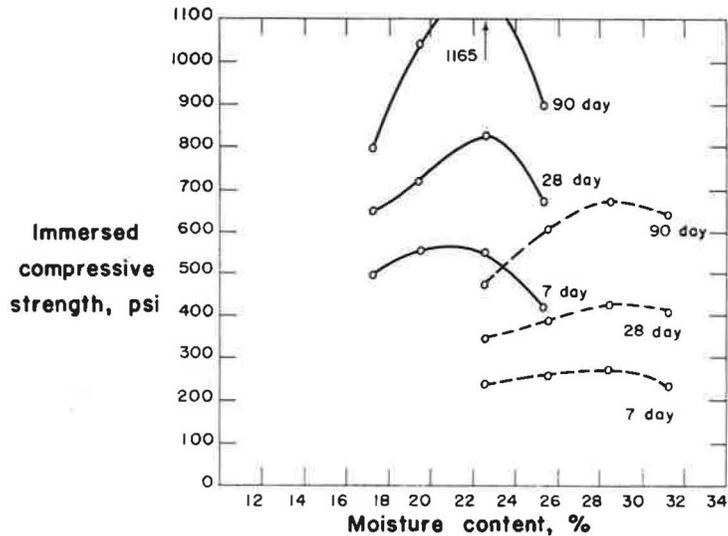


Figure 4. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of gumbotil clay, calcitic hydrated lime, and fly ash No. 1 for standard and modified Proctor compactive efforts.



76.5 % gumbotil — modified P. compaction
 6.0 % lime — standard P. compaction
 17.5 % fly ash no. 2

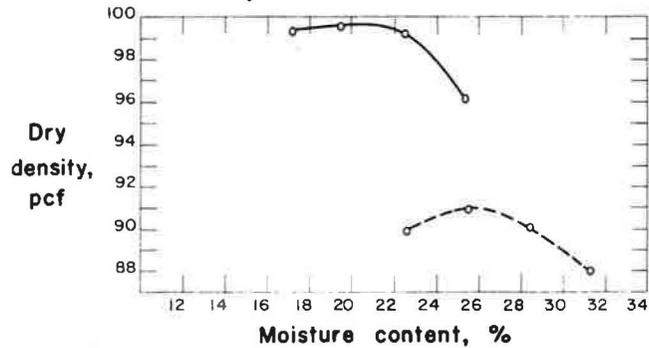
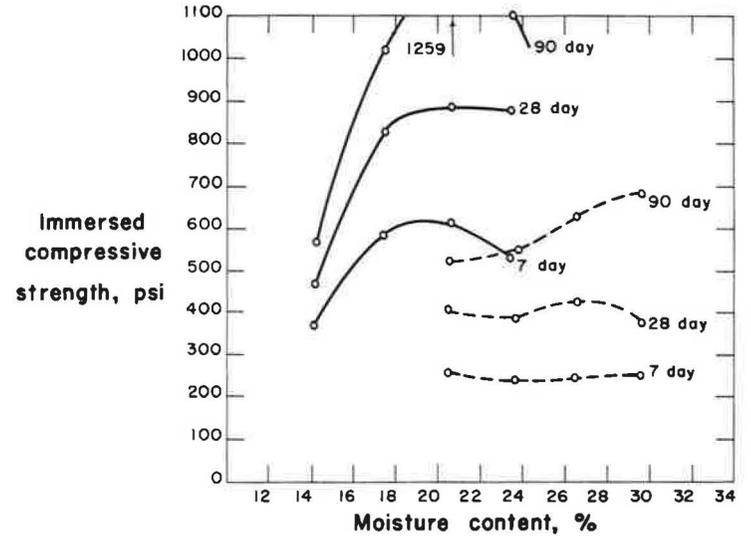


Figure 5. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of gumbotil clay, calcitic hydrated lime, and fly ash No. 2 for standard and modified Proctor compactive efforts.



76.5 % gumbotil — modified P. compaction
 6.0 % lime — standard P. compaction
 17.5 % fly ash no. 3

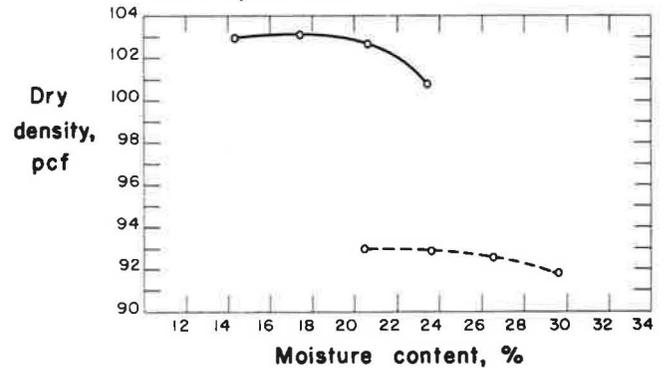


Figure 6. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of gumbotil clay, calcitic hydrated lime, and fly ash No. 3 for standard and modified Proctor compactive efforts.

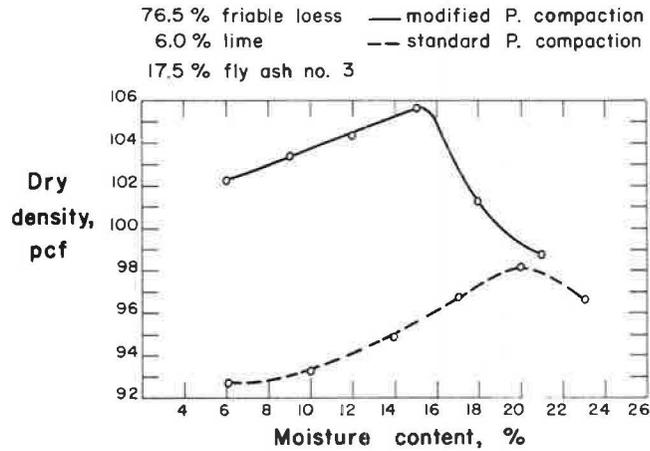
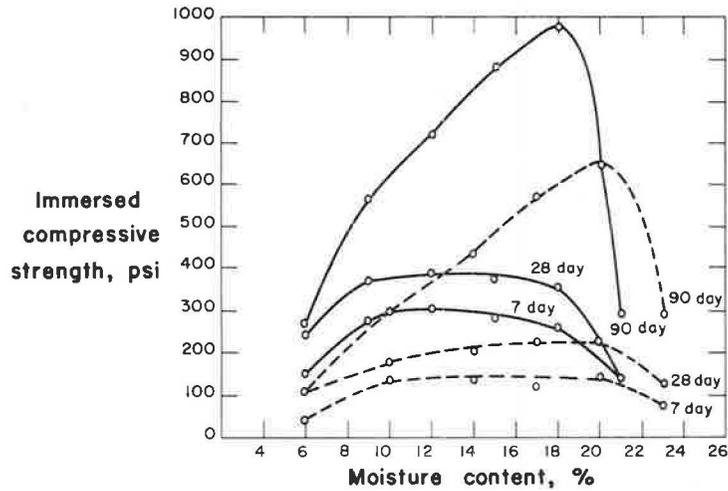


Figure 7. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of friable loess, calcitic hydrated lime, and fly ash No. 3 for standard and modified Proctor compactive efforts.

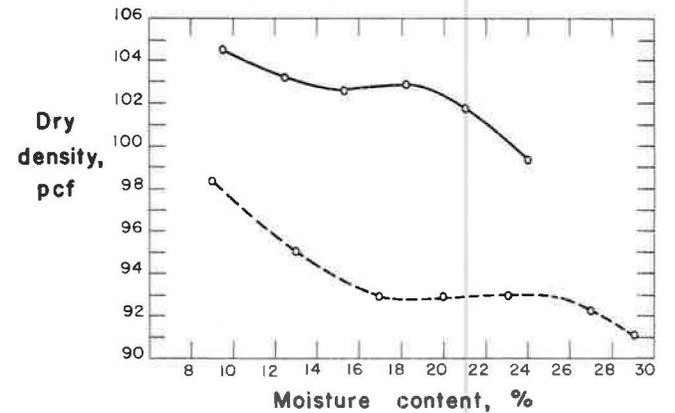
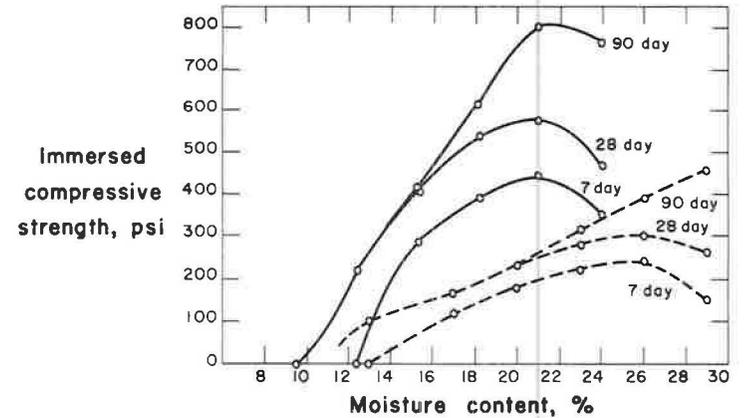


Figure 8. Moisture-density and moisture-strength relationships of 76.5:6:17.5 mixture of alluvial clay, calcitic hydrated lime, and fly ash No. 3 for standard and modified Proctor compactive efforts.

(Fig. 8). The strength curves show, however, a definite optimum moisture that changes conspicuously with curing time for standard compaction and slightly for modified.

Analysis.—The results obtained present new facts on the relations between maximum density and maximum strength in soil-lime-fly ash stabilization. The common practice has been to compact the stabilized soil as the optimum moisture for maximum density. It has been assumed that a maximum density should give a greater strength through a more dense packing of the soil and stabilizer particles, thus putting into contact more surface area for the development of the chemical reactions that lead to the formation of cementitious compounds. But in processes developing cementitious compounds by hydration, as that of the lime-fly ash reaction, the role of the water is of paramount importance.

Analyzing the results, the following have in general been observed:

1. The optimum moisture for maximum strength increased with the increase in curing time.
2. The optimum moisture for maximum strength was to the dry side of the optimum moisture for maximum dry density with the dune sand soil.

With both clayey soils (the gumbotil and the alluvial clay), it was on the wet side. With the friable loess both optimums coincide rather well.

The results indicate that a supply of water is needed for the hydration processes to continue. With dune sand, an amount of moisture two percentages below the optimum for maximum density will develop a maximum, or close to the maximum strength over a long curing period.

The moisture content is critical with friable loess. Reasonably good strengths were obtained at the optimum moisture content for maximum density, but an excess of water brought about a sharp decrease in strength; and amounts of water below the optimum reduced the strength. The optimum moisture for maximum density represents an amount of water sufficient for the chemical process of hydration which, therefore, should be the recommended moisture to stabilize the friable loess. The moisture should be on the dry side of the optimum.

The clayey soils showed great avidity for water. This is because complex reactions take place between the lime and soil particles apart from the lime-fly ash reaction. A rearrangement of the structure of the clay or colloidal particles may take place due to the excess of Ca ions in the stabilized soil. These Ca cations use up H and O ions and H₂O molecules. Based on long-term strengths, amounts of water much greater than the optimum for maximum density are advisable with clayey soils containing high percentages of montmorillonitic clay. It is observed that the shape of the moisture-density curves for both clayey soils is rather flat. In some instances the maximum density is not sharply shown, being undefined. The same happened when lime alone was used. This peculiarity has already been discussed (5).

Effect of Compactive Effort

The trend in compaction of earth embankments, subgrades, and stabilized soils is towards compactive efforts greater than the standard Proctor. The Corps of Engineers and other agencies specify the required density in airfield construction as a percentage of the modified maximum density. Although some work has been done in comparing the strengths obtained at different compactive efforts (8, 9) only one fly ash was used, and the specimens were cured only up to 28 days.

In this work three fly ashes were used with the sand and gumbotil, and one fly ash was used with the alluvial clay and loess. Curing periods were carried up to 90 days. The results for different moisture contents and the maximum strengths vs time are plotted (Figs. 1 to 12).

The modified compaction gave strengths considerably greater than the standard compaction in all eight comparative studies. In all curing periods, the increase ranges from a minimum of 50 percent to a maximum of 160 percent without any correlation.

The rate of strength increase for 7-, 28-, and 90-day curing is almost a straight-line relationship, except for those mixes made with the gumbotil. Greater rate of in-

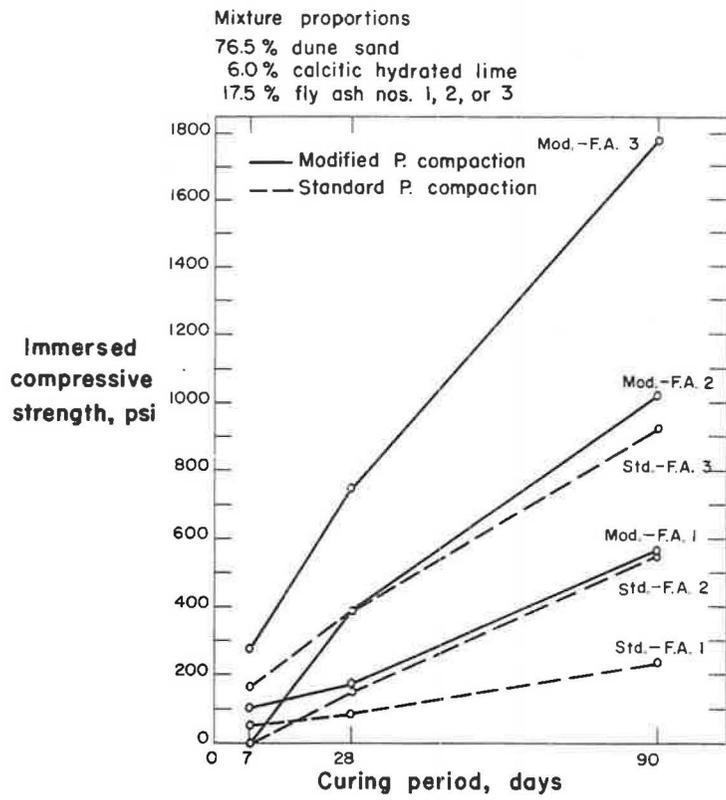


Figure 9. Effect of compactive effort on strength of 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash.

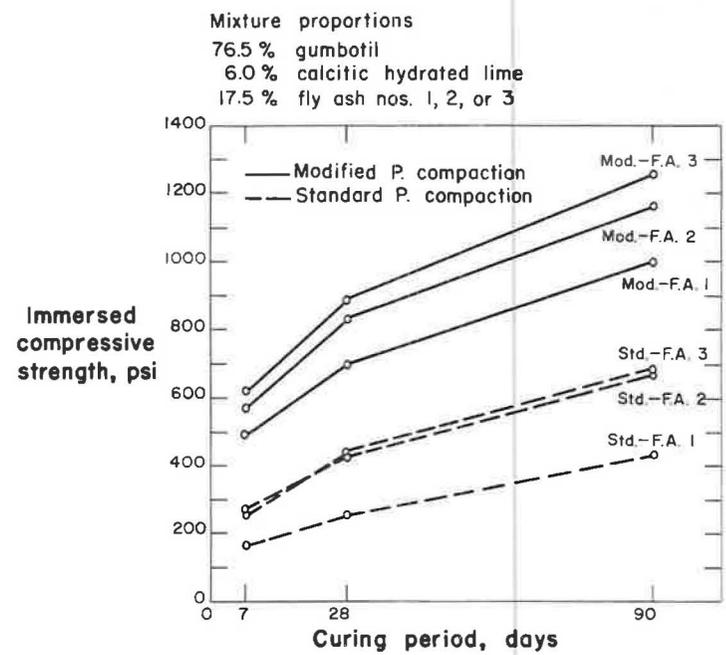


Figure 10. Effect of compactive effort on strength of mixture of gumbotil clay, calcitic hydrated lime, and fly ash.

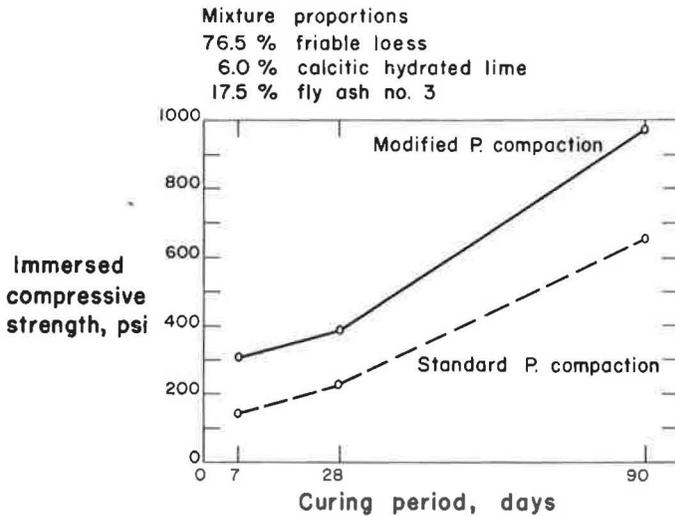


Figure 11. Effect of compactive effort on strength of 76.5:6:17.5 mixture of friable loess, calcitic hydrated lime, and fly ash No. 3.

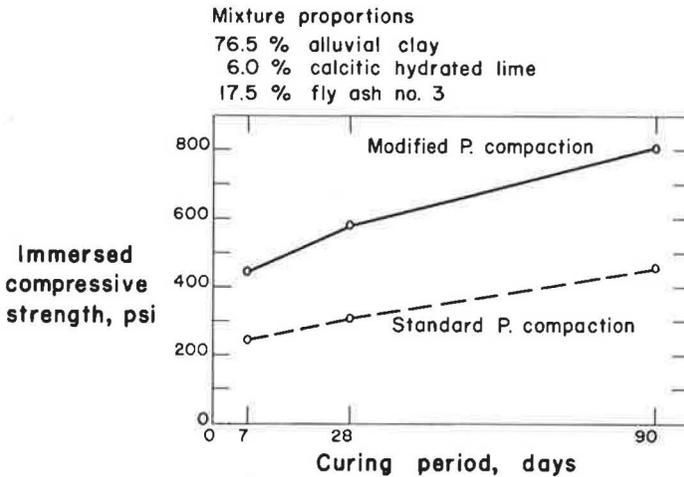


Figure 12. Effect of compactive effort on strength of 76.5:6:17.5 mixture of alluvial clay, calcitic hydrated lime, and fly ash No. 3.

crease with time is found in the friable soils (dune sand and friable loess), in which there is not a break in the rate of increase up to the longest curing period used. After 90-day curing, all the mixtures show that the strength increase also takes place at longer curing periods.

The convenience of compacting the mixtures of soil, lime, and fly ash to the highest possible degree is obvious. By a closer contact of particles at the proper moisture, the surface reactions have more opportunity to develop. This results in the higher strength obtained with the modified compaction.

When lime and fly ash are used to stabilize soils, the steady increase in strength with time has to be accounted for (Figs. 9 to 12). Early strengths may be low, but the continuous gain in strength over long periods of time increases the quality of the pavement made with lime-fly ash stabilized courses. This is desirable when the volume of traffic is expected to increase with time.

Influence of Temperature of Materials at Time of Compaction

So far as known, the influence of temperature of the materials at the time of compaction on soil, lime, and fly ash mixtures has not been studied. The ambient mean temperature between two consecutive days may be as much as 40 F, and that between a cool day in the early working season and another day in the hot part of the summer may be more than 60 F. This work was undertaken to determine the influence of extremes in ambient temperature during the working season on the strength of soil, lime, and fly ash mixtures.

The soils used were dune sand and gumbotil. The very reactive fly ash No. 3 was used because it should accentuate the findings. A series of batches at different moisture contents was mixed and compacted with the soil, lime, fly ash, and water in a cooled state (about 54 F), and another series in a heated one (about 104 F). The soil-lime-fly ash mixtures, molded at several water contents, were stored in the moist room at 70 ± 3 F. The maximum immersed unconfined compressive strength and density values were obtained from the tests of these specimens (Table 1).

Although the data do not show a marked trend, mixing and compacting with hot materials may be detrimental in clayey soils stabilized with lime and fly ash. The density and strength were somewhat reduced.

The results show that the basic reaction between lime and fly ash with sand is not influenced by the temperature, in the range of 54 to 104 F of the materials at the time of mixing. The slight decrease in strength and density in the hot batches made with the clayey soil (gumbotil) is caused by the reaction between the lime and the highly active surface of clay particles before compaction.

Further tests were made in which the materials were mixed at the same temperatures with different water moisture contents and then stored at the same temperatures of mixing for 4 hr before compaction (Table 2). The compacted test specimens were cured in the moist room. Dune sand was the only soil used.

The results obtained further prove that the reaction between lime and fly ash in itself is not affected by the temperature of the materials, (between 54 and 104 F) at the time of mixing. Nevertheless, lime when used in clayey soils reacts in several ways with the clay particles, and some of these reactions may be activated by temperature. These reactions subtract or make inactive part of the lime for the pozzolanic reaction with fly ash and soil particles, causing a decrease in compacted density and in subsequent strength.

Effect of Delay of Compaction After Wet Mixing

When interruptions in road construction occur after lime and fly ash are mixed with

TABLE 1

INFLUENCE OF MIXING TEMPERATURE OF MATERIALS ON STRENGTH OF 76.5:6:17.5 MIXTURE OF SOIL, CALCITIC HYDRATED LIME, AND FLY ASH NO. 3, WITH COMPACTION AFTER MIXING

Soil	Temp. (°F)	Max. Immersed Unconf. Compress. Strength (psi)			Max. Dry Density (pcf)	Optimum M. C. for Max. Density (%)
		7-Day	28-Day	90-Day		
Dune sand	54	154	422	1,004	123.8	12
Dune sand	70	165	390	930	124.2	12
Dune sand	104	158	382	1,010	124.2	12
Gumbotil	54	302	455	620	94.1	25
Gumbotil	70	255	445	685	93.0	25
Gumbotil	104	238	350	492	92.5	25

TABLE 2

INFLUENCE OF MIXING TEMPERATURE OF MATERIALS ON STRENGTH OF 76.5:6:17.5 MIXTURE OF DUNE SAND, CALCITIC HYDRATED LIME, AND FLY ASH NO. 3 IN WHICH COMPACTION WAS DELAYED 4 HR AFTER MIXING

Temperature (°F)	Max. Immersed Unconf. Compress. Strength (psi)			Max. Dry Density (pcf)	Optimum M. C. for Max. Density (%)
	7-Day	28-Day	90-Day		
54	140	369	960	124.0	12
70	141	348	935	122.7	12
104	148	342	973	122.0	12

soil and water and compaction is delayed, the strength of the stabilized soil may be affected. A few tests were made to establish a criterion on the maximum permissible length of time to be allowed soil, lime, and fly ash mixtures between wet mixing and compaction.

Selected mixes using dune sand or gumbotil were made. The mixtures were prepared with different amounts of water to obtain maximum values for strength and density. After mixing the soil, lime, fly ash, and water, one set of mixtures was immediately compacted into specimens; another set was stored for 4 hr in the moist room at 70 F, and then specimens were compacted; another set was stored for 24 hr in the same moist room before compaction of specimens. The maximum values for strength and density are given in Tables 3 and 4. It was found that the longer the compaction was delayed, the higher the moisture content required to obtain a maximum strength.

Dune Sand. --Strength and density of the mixture with dune sand decrease slightly as the time between wet mixing and compaction increases. The greatest decrease in strength is found in mixtures made with fly ash No. 3. For 7-day curing it dropped from 165 psi for no delay in molding to 118 psi for a 24-hr delay; for 28-day curing the drop is from 390 to 243 psi; for 90-day curing there is no difference between the

TABLE 3

RESULTS OBTAINED WITH 76.5:6:17.5 MIXTURES OF DUNE SAND, CALCITIC HYDRATED LIME, AND FLY ASH COMPACTED AFTER DIFFERENT LAPSES OF TIME FOLLOWING WET MIXING

Fly Ash No.	Setting Time ¹	Max. Dry Density (pcf)	Max. Immersed Unconf. Compress. Strength (psi)		
			7-Day	28-Day	90-Day
1	0	121.2	55	90	240
	4	120.3	45	81	219
	24	118.6	41	60	210
2	0	112.3	0	150	560
	4	112.5	0	159	532
	24	110.8	0	141	417
3	0	124.1	165	390	930
	4	122.6	141	348	935
	24	122.6	118	243	945

¹No. of hours elapsed between mixing and molding.

TABLE 4

RESULTS OBTAINED WITH 76.5:6:17.5 MIXTURES OF GUMBOTIL, CALCITIC HYDRATED LIME, AND FLY ASH COMPACTED AFTER DIFFERENT LAPSES OF TIME FOLLOWING WET MIXING

Fly Ash No.	Setting Time ¹	Max. Dry Density (pcf)	Max. Immersed Unconf. Compress. Strength (psi)		
			7-Day	28-Day	90-Day
1	0	Undefined	170	260	440
	4	Undefined	151	260	431
	24	Undefined	136	279	327
3	0	Undefined	255	445	685
	4	Undefined	260	405	596
	24	Undefined	173	244	351

¹ No. of hours elapsed between mixing and molding.

strength of specimens molded after mixing and of those molded after a 24-hr delay. With fly ash No. 2 specimens after 90-day curing there is also a great difference between the strengths of mixtures with no delay in compaction and those with a 24-hr delay, the strength for these two cases being 560 and 417 psi, respectively. The decrease is not very significant with fly ash No. 1, although it is steady with time of delay.

In general, the decrease in strength is very slight in mixtures when compaction was performed 4 hr after wet mixing. The decrease is more accentuated for the mixtures stored 24 hr before compaction.

A delay in compaction after wet mixing also brings about a decrease in dry density of sand, lime, and fly ash mixtures. The decrease amounts to less than 2 percent after a 24-hr delay.

Gumbotil.—A great decrease in strength correlates with the time of delay in compaction after wet mixing of gumbotil, calcitic hydrated lime, and fly ash mixtures. With a 24-hr delay for fly ash No. 3 the strengths were reduced from 32 to 49 percent, depending on the curing period. The reduction in the fly ash No. 1 mixture is less important, showing up in 7- and 90-day strengths but not in those of 28 days.

The density diminished consistently as compaction time was delayed. Because the maximum dry density was undefined, the moisture-dry density relationships in mixtures with gumbotil are plotted for the range in moisture content in which the maximum strengths were obtained (Figs. 13 and 14). The compacted density is lowered to a great extent by a delay in compaction. The drop in dry density is about 2 pcf for a 4-hr delay and about 5 pcf for a 24-hr delay.

Analysis.—The results stress the importance of proceeding with compaction as soon as possible after wet mixing of soil, lime-fly ash mixtures. This is highly recommended with montmorillonitic clayey soils in which strengths may drop by about 40 percent and dry density but about 6 percent for the same compactive effort if compaction is delayed one day after wet mixing. With sandy soils the drop in strength and dry density is not very important, and compaction may proceed the following day after wet mixing without significantly impairing the strength or dry density.

The lowering of strength and density may be for one or more of the following reasons:

1. Formation of carbonates by chemical reaction between lime and the carbon dioxide of the atmosphere.
2. Pozzolanic reactions between lime and fly ash.
3. Reactions between lime and soil particles.

The first two are probable in sandy soils and all three in clayey soils.

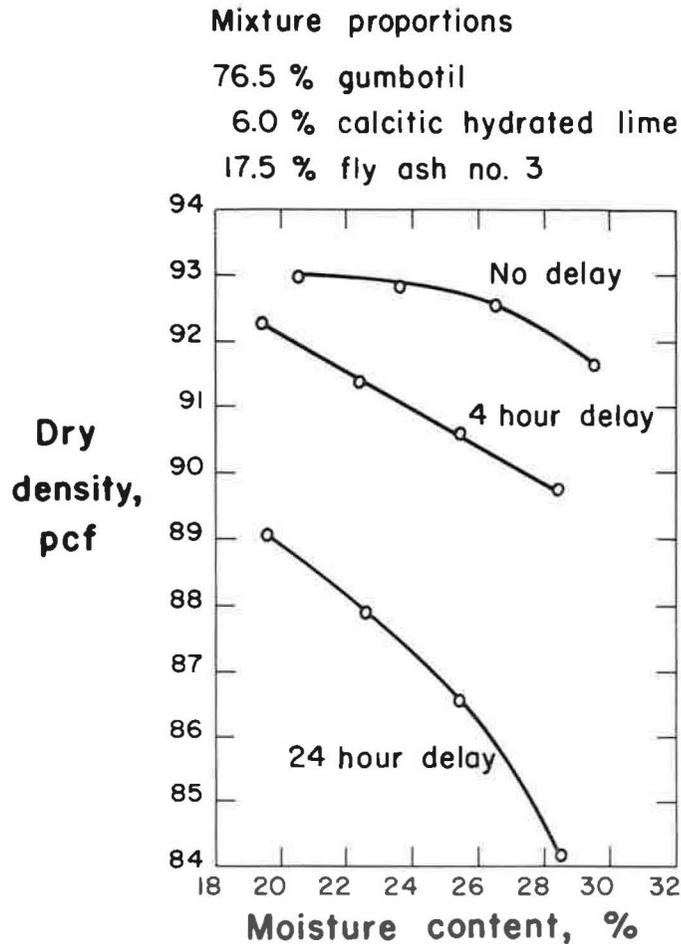


Figure 13. Moisture-density relationships of 76.5:6:17.5 mixture of gumbotil clay, calcitic hydrated lime, and fly ash No. 3, in which compaction was carried at different intervals of time after wet mixing.

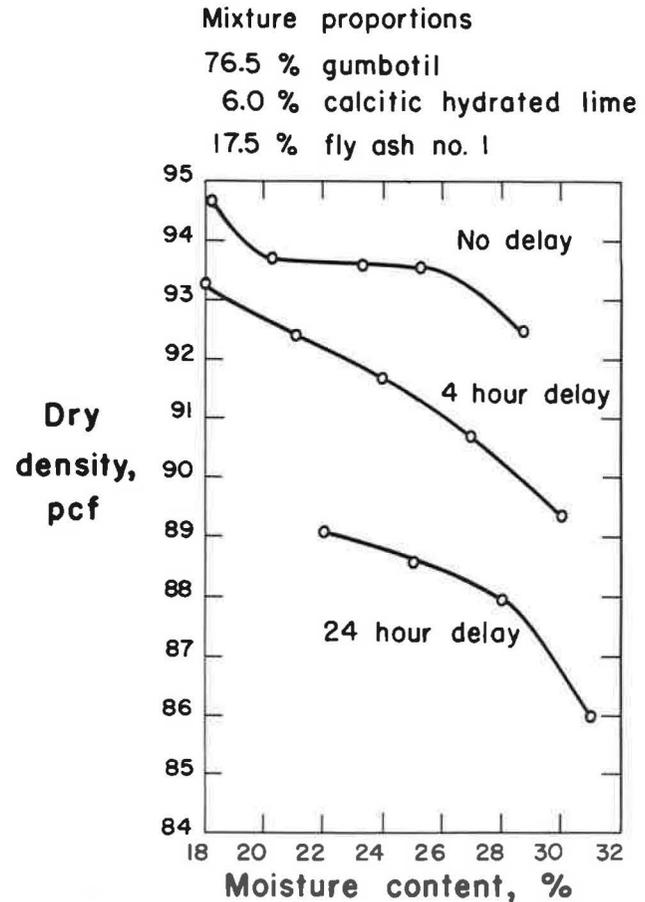


Figure 14. Moisture-density relationships of 76.5:6:17.5 mixture of gumbotil clay, calcitic hydrated lime, and fly ash No. 1, in which compaction was carried at different intervals of time after wet mixing.

A small reduction in strength and density in sandy soils indicates that the first two processes are not developed to a great extent. Because the carbonation of lime takes place at a rapid rate in a moist condition and because of the unlikeliness of quick pozzolanic reactions between lime and fly ash in a loose state, the first reaction is likely mainly responsible for the lowering of density and strength in sandy soils.

The reactions between lime and clay mineral particles are very important in montmorillonitic clay soils. The unbalanced electrical surface forces of the clay particles adsorb calcium cations of lime; calcium ions also produce a crowding action of clay particles; and lime reacts with the soil particles in a pozzolanic action. These reactions account for a great part of the reduction of strength and density when compaction does not soon follow wet mixing of clayey soil, lime, and fly ash mixtures.

CONCLUSIONS

1. Maximum strength of soil, lime, and fly ash mixtures is produced by a compaction moisture content that is not necessarily the optimum moisture content for maximum density. The compaction moisture for maximum strength of specimens with sandy soils is to the dry side of the optimum moisture for maximum density. In soils having a high clay content, at least of the montmorillonite type, the compaction moisture is to the wet side. With such other soils as friable loess maximum strength and maximum density may occur at the same compaction moisture.

2. If no water is added during curing, the required compaction moisture content to produce maximum strength changes with the curing period—the longer the curing period, the greater the compaction moisture content needed for maximum strength.

3. Increasing the compactive effort from standard Proctor to modified increases the strength of soil, lime, and fly ash mixtures. The strength increase obtained varies from 50 to 160 percent.

4. If the materials are at high temperature at the time of mixing, the density and strength of clayey soil, lime, and fly ash mixtures are lowered, suggesting pre-compaction reactions. Sandy soils are not affected.

5. Compaction should proceed as soon as possible after wet mixing of soil, lime, and fly ash mixtures; otherwise density and strength may be substantially lowered. With clayey soils, compaction should be completed not later than 4 hr after wet mixing, whereas with sandy soils, compaction can be delayed until the day after wet mixing without appreciable loss of strength.

ACKNOWLEDGMENTS

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Comparative Effects of Hydraulic, Calcitic And Dolomitic Limes and Cement in Soil Stabilization

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The addition of lime to soils brings about changes that are reflected in the plasticity and strength characteristics of the soils. To study these changes, representative samples of different types of lime, hydraulic, calcitic hydrated, dolomitic monohydrate, and dolomitic dihydrate were selected. For comparison, a sample of type I cement was also used. The effects on plasticity were studied with a very heavy clay (gumbotil), and the effects on unconfined compressive strength with two fine-grained soils (friable loess and plastic loess). The results obtained so far indicate that there are marked differences in the plasticity and strength results, depending on the type of stabilizer used.

•MUCH WORK has been done at Iowa State University on the effectiveness of the different types of hydrated limes in soil stabilization (1, 2, 3, 6, 7, 8, 9, 11, 12). The investigation reported herein was undertaken as a further check on the comparative effects of calcitic hydrated, dolomitic monohydrate, and dolomitic dihydrate limes.

Lime is available in several forms (Table 1). One of the varieties is hydraulic lime, which is regarded as an intermediate product between lime and cement. This lime has never been evaluated in soil stabilization. It is believed that this product could be a successful stabilizer of plastic soils by giving better workability than cement and yielding higher strengths than other limes.

In addition, a type I portland cement was also used for comparison. The Atterberg limits and shrinkage tests were used to evaluate the ability of the different additives to modify a soil, and the unconfined compressive strength as an index of the stability after curing for several periods.

MATERIALS

Soils

A highly plastic soil, known as gumbotil was used for the Atterberg limits and shrinkage tests. This soil, considered as a fossil B horizon developed from Kansan stage till, contains about 60 percent montmorillonitic clay. It is one of the most difficult soils to work with in the field.

Two Wisconsin stage loess soils, representative of the major surface deposits in Iowa, were used for strength studies; samples were selected to represent a friable, calcareous loess, and a plastic, leached loess. A field description and physical and chemical properties of each soil are given in Table 2.

Additives

The chemical composition of each additive used in this investigation is given in Table 3.

High-calcium limes are produced from calcareous materials (generally from limestone but also from chalk and oyster shells) containing 95 to 99 percent calcium carbonate. Dolomitic limes are produced from the mineral dolomite, a type of limestone containing from 30 to 45 percent magnesium carbonate and the rest calcium carbonate. Hydraulic lime is a type of cementitious lime that will set and harden under water like portland cement, but retains some of the plastic properties of lime. It is obtained by calcining impure limestone containing 15 to 20 percent silica and alumina so that sufficient calcium silicates and aluminates are formed to give the lime its characteristic hydraulic properties.

TABLE 1
FORMS OF LIME

Quicklime:	High-calcium (calcitic), CaO Dolomitic, CaO and MgO
Hydrated lime:	High-calcium (calcitic), Ca(OH) ₂ Dolomitic monohydrate, Ca(OH) ₂ and MgO Dolomitic dihydrate, Ca(OH) ₂ and Mg(OH) ₂
Hydraulic lime, lime and calcium silicates and aluminates	

TABLE 2
PROPERTIES OF SOILS USED

Property	Friable Loess	Plastic Loess	Gumbotil
Laboratory designation	20-2	528-4	528-8
Sample location	Harrison Co., Iowa	Keokuk Co., Iowa	Keokuk Co., Iowa
Soil series	Hamburg	Mahaska	-
Horizon	C	C	Fossil B
Sampling depth (ft)	39.0-40.0	3.0-6.5	6.5-8.5
Textural composition (%):			
Gravel (above 2mm)	0.0	0.0	0.0
Sand (2-0.074mm)	0.8	3.5	20.1
Silt (0.074-0.005mm)	82.8	55.0	16.5
Clay (below 0.005mm)	16.4	41.5	63.4
Clay (below 0.002mm)	11.6	33.0	60.4
Atterberg limits (%):			
Liquid limit	32	51	68
Plastic limit	27	21	23
Plasticity index	5	30	45
Chemical properties:			
pH ¹	8.4	6.0	7.1
C. E. C., ² (meg/100 g)	15.6	23.5	39.9
Carbonates ³ (%)	10.9	1.2	1.8
Organic matter ⁴ (%)	0.5	0.3	0.2
Predominant clay mineral	Montmorillonite	Montmorillonite	Montmorillonite
Classification:			
Textural ⁵	Silty loam	Silty clay	Clay
AASHO	A-4(8)	A-7-6(18)	A-7-6(20)
Unified	ML	CH	CH

¹Glass electrode method using suspension of 15 g soil in 30 cc distilled water.

²Ammonium acetate (pH=7) method on soil fraction below 2 mm.

³Versenate method for total calcium.

⁴Potassium bichromate method.

⁵From soil texture chart used by U. S. Bureau of Public Roads.

TABLE 3
CHEMICAL COMPOSITION OF ADDITIVES USED^a

Additive	Lab. Symbol	Constituents (% by weight)							Loss on Ignition
		CaO	Ca(OH) ₂	MgO	Mg(OH) ₂	SiO ₂	R ₂ O ₃ ^b	SO ₃	
High-calcium hydrated lime	CL	73.50	97.10 ^c	0.90	TR ^d	1.30	0.25	ND ^e	24.00
Dolomitic monohydrate lime	ML	47.32	62.50 ^c	39.11	TR	1.10	0.75	ND	16.10
Dolomitic dihydrate lime	DL	43.80	57.90 ^c	28.20	40.80 ^c	0.60	0.50	ND	26.90
High-calcium hydraulic hydrated lime	HL	59.06	52.00 ^f	3.98	TR	17.10	5.40	1.27	12.81
Type I portland cement	PC	64.05	TR	2.90	TR	21.62	8.02	2.26	0.58

^aData supplied by respective chemical manufacturers.

^bAl₂O₃ + Fe₂O₃.

^cCalculated hydroxide equivalent.

^dTraces.

^eNot determined.

^fEstimated from loss on ignition.

Portland cement is made by burning a mixture of calcareous (such as marls, chalk, or limestone) and argillaceous (such as clay or shale) material to clinkering temperature, and grinding the resulting clinker to fine powders of calcium silicates and aluminates, which hydrate to give the material its cementing properties.

EXPERIMENTAL PROCEDURES

Preparation and Curing of Specimens

All soils were previously air dried and ground with mortar and pestle to pass the No. 10 sieve. The batches were mixed in a Hobart kitchen mixer, model C-100. The additive was first mixed with the dry soil and then the materials were wet mixed for 2 min.

Cylindrical 2- by 2-in. test specimens were prepared at a density near the standard AASHO using the Iowa State compaction apparatus (1, 10).

The specimens were wrapped in waxed paper and sealed with cellophane tape to prevent carbonation of the additive by carbon dioxide in the air. The specimens were then placed in a moisture room having a relative humidity between 90 and 100 percent and a temperature of 71 ± 3 F.

Strength Testing

Strength determinations were made on mixtures containing 2, 5, 8, and 12 percent of each additive used. For every percentage, additive, moisture-density, and moisture-strength relationships were obtained using five different moisture contents and three specimens molded for every point. One of the three specimens was cured for

7 days, one for 28 days, and one for 84 days. After the specified curing period, the specimens were immersed in distilled water for 24 hr, and were then tested to failure under unconfined compression at a loading rate of 0.1 in. per min. From the moisture-strength curves, the maximum unconfined compressive strengths were obtained.

Atterberg Limits Testing

ASTM Methods D423-59 and D424-59 were followed except that after the soil, additive, and distilled water were mixed together, the mix was scraped into a porcelain pan, covered, and stored in a near 100 percent humidity room for 1 hr to allow for uniform wetting of the sample. After being seasoned, enough of the mixture for four liquid limit tests was placed in an evaporating dish. This procedure was followed on the soil passing the No. 40 sieve with additives of 0, 1, 3, 6, and 10 percent by the oven-dry weight of soil.

A sample weighing about 10 g was taken from the mixture prepared for the liquid limit test. Four plastic limits were rolled for each of the mixture studies (On 1, 3, 4, 6, and 10 percent additive by oven-dry weight of soil). The plastic limit of the mix at each percentage additive was determined as the average of the moisture contents of the four threads rolled.

Shrinkage Tests

ASTM Methods D427-39 were used for mixtures containing 2 and 8 percent of each additive. Shrinkage limit and shrinkage ratio were then calculated to determine the effectiveness of different additives for the modification of a soil.

RESULTS

Modification of Soil

The quick changes in a soil caused by the addition of some stabilizing agents as reflected in such physical properties as plasticity and shrinkage can be considered as a modification of the soil. To study these effects of the addition of limes and cement, a highly plastic gumbotil soil was selected. Atterberg limits and shrinkage properties were measured 1 hr after the soil and additives were wet mixed.

Plasticity Properties.—Plasticity tests were performed to find the relative influence of each additive on a highly plastic soil (Fig. 1; Table 4). The shape of the curves is characteristic of montmorillonitic clay soils treated with lime (5). The liquid limit gradually decreases with the increase in additive, and the plastic limit increases with increase in additive up to a certain point. At that point, further increases in the amount of additive do not further affect the plastic limit.

The decrease of the plasticity index of a highly plastic soil is regarded as an improvement in its engineering properties. The limes as well as the cement, in amount greater than about 2 percent, decreased the plasticity index of the gumbotil soil, and improved its engineering characteristics as a result. The degree of improvement varies somewhat with the kind and amount of additive. The greatest improvement was obtained with high-calcium hydrated lime and the least with cement. The dolomitic limes showed an intermediate degree of improvement.

The soil and soil-additive mixtures were classified under the AASHO soil classification system to show the improvements of soils by the addition of lime or cement (Table 3).

Plasticity tests were also run with the gumbotil and the various main compounds found in hydrated lime to obtain more information on the influence of different types of lime in the modification of the plasticity characteristics of a clayey soil (Table 5).

The compound chiefly responsible for lowering the plasticity index is calcium hydroxide; magnesium hydroxide causes an insignificant reduction; and magnesium oxide lowers the plasticity index to a degree intermediate between calcium hydroxide and magnesium hydroxide. This explains why high-calcium hydrated lime was slightly more effective than dolomitic limes in improving the plasticity characteristics of gumbotil soil.

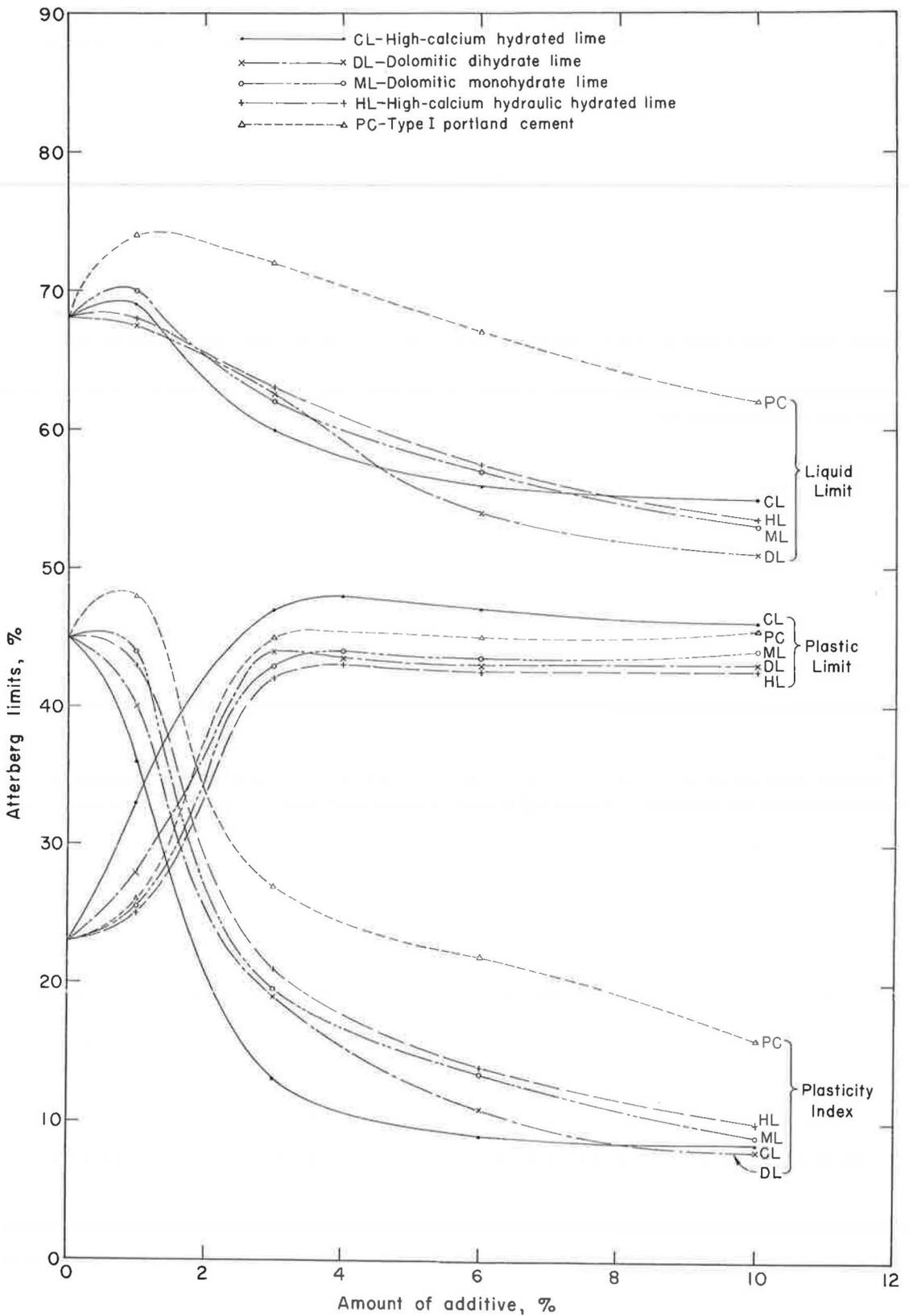


Figure 1. Effect of various additives on Atterberg limits of gumbotil.

TABLE 4
RESULTS OF PLASTICITY TESTS ON GUMBOTIL TREATED WITH
VARIOUS ADDITIVES

Additive		Liquid Limit (%)	Plastic Limit (%)	Plasticity Index (%)	AASHO Class.
Kind	Amount (%)				
None	0	68	23	45	A-7-6(20)
High-calcium hydrated lime	1	69	33	36	A-7-5(20)
	3	60	47	13	A-7-5(13)
	4		48		
	6	56	47	9	A-5(11)
	10	55	46	9	A-5(11)
Dolomitic mono- hydrate lime	1	70	26	44	A-7-6(20)
	3	62	43	19	A-7-5(16)
	4		44		
	6	57	43	14	A-7-5(13)
	10	53	44	9	A-5(10)
Dolomitic dihy- drate lime	1	68	28	40	A-7-6(20)
	3	63	44	19	A-7-5(16)
	4		43		
	6	54	43	11	A-7-5(11)
	10	51	43	8	A-5(10)
High-calcium hydraulic hy- drated lime	1	68	25	43	A-7-6(20)
	3	63	42	21	A-7-5(16)
	4		44		
	6	57	43	14	A-7-5(13)
	10	53	43	10	A-5(10)
Type I portland cement	1	74	26	48	A-7-6(20)
	3	72	45	27	A-7-5(19)
	6	67	45	22	A-7-5(17)
			46		
	10	62	46	16	A-7-5(14)

TABLE 5
RESULTS OF PLASTICITY TESTS ON GUMBOTIL TREATED WITH
CHEMICAL REAGENTS

Chemical		Liquid Limit (%)	Plastic Limit (%)	Plasticity Index (%)	AASHO Class.
Kind	Amount (%)				
None	0	68	23	45	A-7-6(20)
Ca(OH) ₂	2	60	43	17	A-7-5(15)
	8	55	45	10	A-5(11)
MgO: Heavy	2	63	30	33	A-7-5(20)
	8	68	39	29	A-7-5(20)
Light	2	70	39	31	A-7-5(20)
	8	75	45	30	A-7-5(20)
Mg(OH) ₂	2	70	28	42	A-7-6(20)
	8	70	31	39	A-7-5(20)

Shrinkage Tests.—Shrinkage limit is the moisture content of a drying soil below which any decrease in moisture content is accompanied by only a very small decrease in volume. Shrinkage ratio is the ratio of the percentage of volume change to the percentage of moisture loss occurring above the shrinkage limit, which is a measure of the rate of volume change. In general, a high shrinkage limit and a low shrinkage ratio are desired to reduce the shrinkage of a soil, on drying.

The shrinkage characteristics of the gumbotil soil were greatly improved by the addition of lime or cement (Table 6). The lowest amount of additive tried, 2 percent of high-calcium hydrated lime, increased the shrinkage limit in a much greater pro-

TABLE 6
RESULTS OF SHRINKAGE TESTS ON GUMBOTIL TREATED WITH
VARIOUS ADDITIVES

Additive		Shrinkage Limit (%)	Shrinkage Ratio
Kind	Amount (%)		
None	0	7.7	2.08
High-calcium hydrated lime	2	25.4	1.52
	8	37.1	1.26
Dolomitic monohydrate lime	2	13.5	1.86
	8	41.8	1.22
Dolomitic dihydrate lime	2	13.5	1.86
	8	38.5	1.20
High-calcium hydraulic hydrated lime	2	13.3	1.78
	8	35.7	1.28
Type I portland cement	2	15.3	1.65
	8	37.4	1.22

portion than any of the other limes or cement. For 8 percent additive all limes as well as cement cause a comparatively similar increase in the shrinkage limit. The increase in shrinkage limit, which is fivefold when the soil was treated with 8 percent additive, and the decrease in shrinkage ratio indicate that the soil will shrink less on drying when treated with lime or cement.

Cementation of Soils

The gain of strength of soil-lime and soil-cement mixtures while being cured is thought to be caused by the formation of cementitious compounds. To study the cementation properties of lime and cement, a plastic and a friable fine-grained soil were selected. Because this study included the determination of moisture-density and moisture-strength relationships, a discussion of these is given first.

Maximum Dry Density.—The addition of lime or cement to a soil lowers its maximum density for the same compactive effort. Figures 2 and 3 show the greatest decreasing rate in the maximum density is for small amounts of additive—as the amount of additive is increased the maximum density tends to decrease at a slower rate. Additions of calcitic and dolomitic limes decreased the maximum density to a greater extent than hydraulic lime or cement; hydraulic lime is intermediate between cement, which showed the least decrease in maximum density, and the other limes.

The differences in maximum density for the same compactive effort might have some influence in the strengths obtained with the different additives. This influence is not exactly known but appears to be important because the strength of soil-lime and soil-cement mixtures greatly depends on density (4, 11).

The optimum moisture contents of soils with different additives are shown in Figures 2 and 3. Limes as additives increase the optimum moisture contents of both plastic loess and friable loess, and cement increases the optimum moisture content of plastic loess and decreases that of friable loess.

Maximum Strength.—The optimum moisture content for maximum strength differed slightly from the optimum moisture content for maximum density. The difference between both moistures was never greater than one moisture content percentage. The maximum strength was sometimes slightly to the wet side of the maximum density and sometimes slightly to the dry side, depending on the kind and amount of additive, and on the curing period. In practical work the optimum moisture content for maximum density for the compactive effort applied will secure the maximum or very nearly the maximum strength.

Comparative Effects of Additives on Maximum Strength.—Maximum strengths obtained with soil-lime and soil-cement mixtures are shown in Figures 4 and 5. The poor performance of dolomitic dihydrate lime with both soils and for the three curing periods used is clearly seen. After 7 days curing, cement produces greater strengths than any of the limes tested, but after 28 or 84 days curing, some of the limes, for

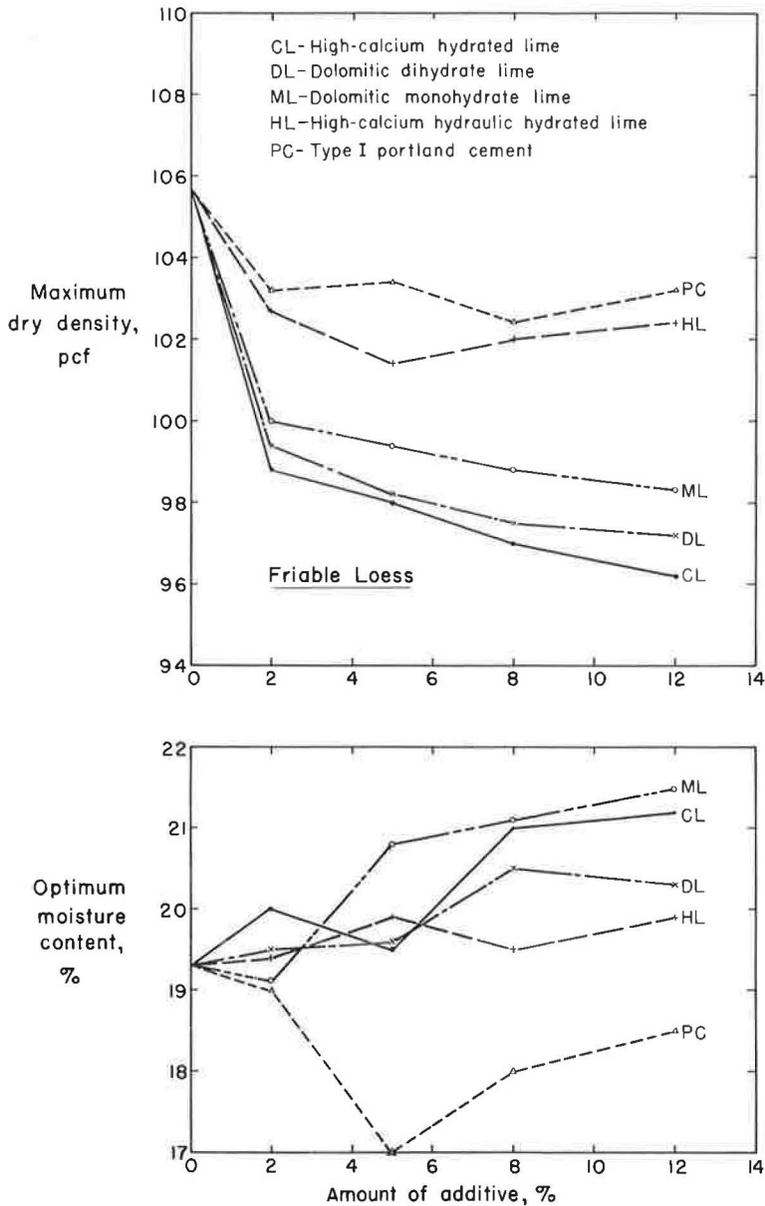


Figure 2. Effect of various additives on maximum dry density and optimum moisture content of friable loess.

lower amounts of additive, performed as well or better than cement. The strengths obtained with 5 percent dolomitic monohydrate lime after 28 and 84 days curing are as high as those obtained with the same amount of cement.

In a general rating of the different additives with the two soils used, cement could be considered the best followed in order by dolomitic monohydrate, high-calcium hydraulic hydrated, high-calcium hydrated and dolomitic dihydrate limes.

The differences in strength obtained for the different additives are due chiefly to the chemical compositions of limes and cement. Calcium silicates and aluminates (present in cement and to a lesser extent in high-calcium hydraulic lime) are responsible

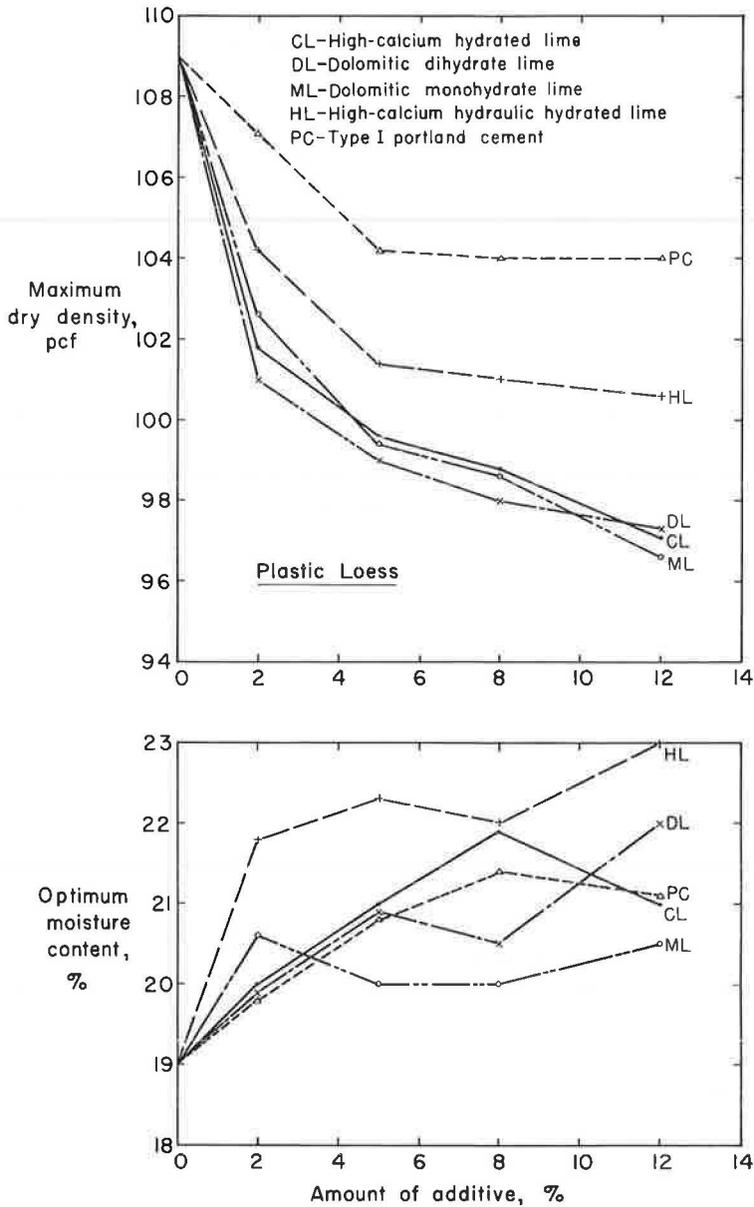


Figure 3. Effect of various additives on maximum dry density and optimum moisture content of plastic loess.

for a great part of the strength obtained. Magnesium oxide as well as calcium hydroxide is also important in the development of strength. Magnesium hydroxide does not contribute much to strength.

In previous studies made with synthetic limes with different ratios of calcium to magnesium, specimens treated only with magnesium oxide developed a strength comparable with that obtained with the same amount of calcium hydroxide alone (7). However specimens treated only with magnesium hydroxide did not show any strength. The study also showed that there is an optimum amount of calcium hydroxide plus magnesium oxide to produce a maximum strength.

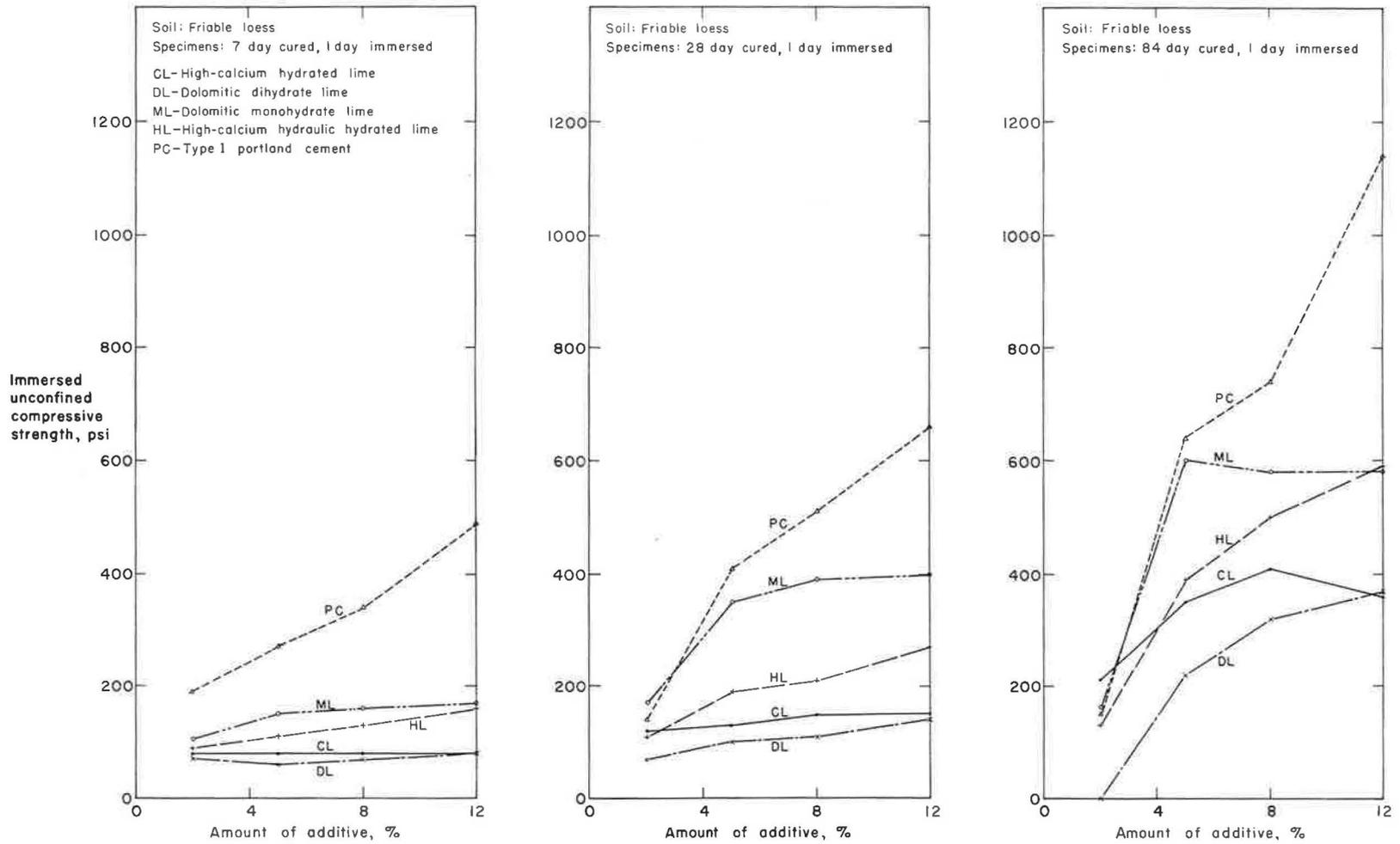


Figure 4. Effect of various additives on immersed unconfined compressive strengths of friable loess.

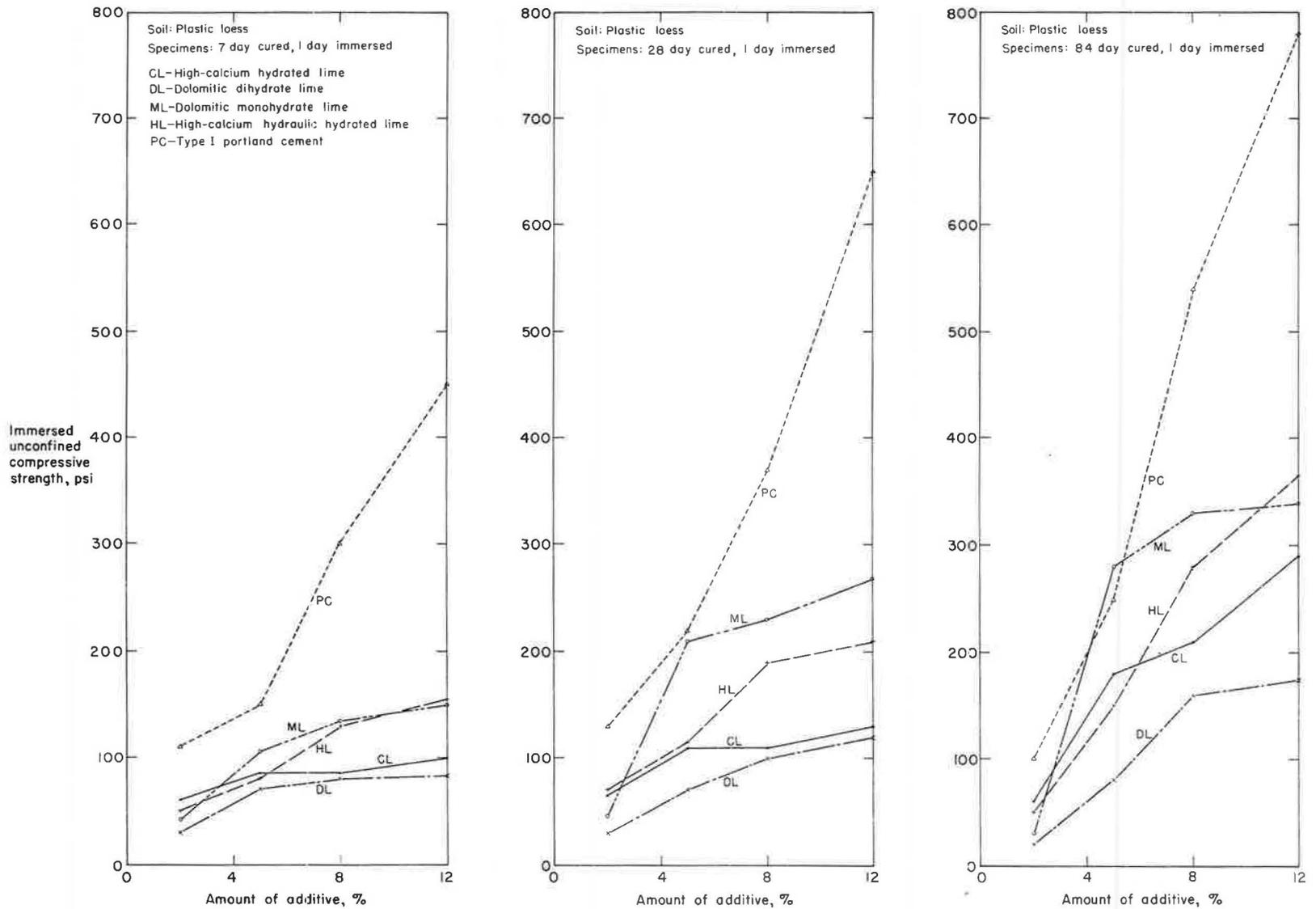


Figure 5. Effect of various additives on immersed unconfined compressive strengths of plastic loess.

The data obtained show that dolomitic monohydrate lime is as good or better than high-calcium hydrated lime in the development of strength of fine-grained soils with predominant montmorillonitic clays and for curing temperatures near 70 F. It also shows that hydraulic lime can also be used in soil stabilization and that hydraulic lime may be better than non-hydraulic limes in larger amounts and longer curing times.

CONCLUSIONS

Based on this investigation, the following conclusions can be drawn:

1. Lime is a more effective stabilizer for reducing the plasticity of a soil than cement.
2. Among the limes tested, high-calcium hydrated lime rated first in lowering the plasticity. The other three types of limes (dolomitic monohydrate, dolomitic dihydrate, and high-calcium hydraulic hydrated) gave a similar performance.
3. Among the major constituents of hydrated limes, calcium hydroxide, followed by magnesium oxide, is mainly responsible for the lowering of plasticity. The influence of magnesium hydroxide is practically negligible.
4. The shrinkage properties of a soil were markedly reduced by the addition of different types of lime and cement. High-calcium hydrated lime showed slight advantage over the others at lower percentage of content.
5. Portland cement was found more effective in increasing soil strength on curing, followed in order by dolomitic monohydrate, high-calcium hydraulic, high-calcium hydrated and dolomitic dihydrate limes. For low additive levels, up to about 5 percent and for curing periods of 28 days or longer some of the limes were as effective as cement in improving the strength of the soils tested.
6. Hydraulic lime can be used effectively in soil-lime stabilization. Hydraulic lime is equivalent to other types in reducing soil plasticity. It also is better in larger amounts and longer curing time than monohydraulic lime in producing strength.
7. Based on the trends found in this study, the best all-round stabilizer for clayey soils seems to be a high-magnesium hydraulic monohydrate lime.

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Characteristics of Lime Retention by Montmorillonitic Clays

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Previous work has shown that hydrated lime added to clayey soils reduces their plasticity; above a certain percent lime there is a leveling off and no further reduction. The point at which this leveling off begins may be referred to as the lime retention or lime fixation point. The amount of lime needed to satisfy the lime retention requirement of clays must be exceeded if later pozzolanic cementitious reactions are to proceed. Lime retention occurs in clays already calcium saturated, although sodium clays take more lime. The total calcium retained exceeds the cation exchange capacity as measured by the ammonium acetate method.

Lime retention by clays was studied by measurements of plastic limit, pH, viscosity, exchangeable calcium, and X-ray diffraction. The lime retention point was most pronounced in montmorillonitic clays, and coincides with a gradual increase in pH and a sharp rise in viscosity of suspensions as measured by a Stormer paddle-rotation type of viscosimeter. The rise in viscosity was observed with natural sodium- and calcium-saturated bentonites and with Iowa gumbotil and plastic loess soils. Viscosity depends on pH as well as on calcium ion concentration, and reaches a maximum at around pH 11.8. Hypotheses of lime retention are presented.

•TWO TYPES of reactions occur between lime and clayey soils: (a) an almost immediate reduction in plasticity on addition of lime to soil, and (b) pozzolanic cementation reactions, resulting in a long-term strength gain of compacted soil (1). This paper is primarily concerned with the first type.

The plasticity reduction effect of lime was long attributed to cation exchange phenomena (2, 3, 4). In 1952 Goldberg and Klein recognized that lime also reduces plasticity of clay that is already calcium saturated (5). That lime is used in excess of the cation exchange capacity, as measured at pH 7, suggested a "combination or fixation" of excess sodium and calcium ions in insoluble and perhaps in non-crystalline form.

In a discussion to this paper, Johnson (6) suggested that saturation of the clay micelle by OH groups may alter the cation exchange capacity and thus change the system. Hilt and Davidson (7) noted that the plastic limit maxima and minimum quantities of lime required for pozzolanic reactions coincide, and termed this coincidence the "lime fixation point." They demonstrated that lime used for reduction in plasticity is not available for pozzolanic reactions. The lime fixation points of the montmorillonitic soils investigated were found to be

$$L_M = \frac{\% \text{ of } < 2\text{-}\mu \text{ clay}}{35} + 1.25. \quad (1)$$

Whether the lime was fixed in the sense of ammonium or potassium fixation by clay was not indicated. Later work by Pinto et al. (8) showed that the floc sedimentation rate of a gumbotil (sample 528-8) suspension reached a maximum near the lime fixation point and leveled off, indicating that increments of lime below this percentage go to increase the size of the flocs (Fig. 1).

PROCEDURES

Lime retention (or fixation) in natural sodium and calcium bentonites was studied by means of physical tests, such as plastic limit and suspension viscosity, and by means of physico-chemical tests, such as pH, X-ray diffraction, exchangeable cation determinations, and differential thermal analysis (DTA). Exchangeable cations were measured both at neutral and at alkaline pH's to ascertain the relationship between the pH and exchange capacity. Selected tests were then made on two montmorillonitic clay soils to show if trends were consistent. Methods and samples used in this study are as follows.

Plastic Limit.—Plastic limits were determined according to ASTM method D424-59 (9). Most were measured independently by three operators, and the results were averaged.

pH.—The pH's of the sample suspensions prepared in 50 percent concentration for soil samples, 30 percent for Ca-bentonite, and 3 percent for Na-bentonite were measured by a Beckman glass electrode pH meter.

Viscosity.—Viscosities of the clay suspensions were measured by Stormer paddle-type viscosimeter at 25 C (Fig. 2). The paddle is actuated by a 25-g weight; results are expressed in minutes required for 100 revolutions of the rotor. Details of the method are presented in Appendix A.

X-Ray Diffraction.—Aliquots of most clay-lime suspensions were allowed to air-dry on glass slides for X-raying. Autoclaved samples were packed moist into 1- by 2-in. bakelite holders and X-rayed. A General Electric XRD-5 diffraction unit with copper $K\alpha$ radiation was used. The incident beam angle of 4° , detector slit of 0.2° , beam slit of 1° , scan rate of 2° per min, and time constant of 3 sec were used.

Differential Thermal Analysis.—Samples equilibrated in a CaCl_2 desiccator were ground to pass a No. 200 mesh sieve before packing into the DTA apparatus. In this apparatus, an automatic controller provides for a heating rate of 10 C per min. A vertical furnace arrangement is used. The sample block of 18-8 stainless steel, $\frac{3}{4}$ in. high by $\frac{1}{4}$ in. diameter is supported by a hollow ceramic pedestal. Two vertical $\frac{3}{8}$ -in. in diameter by $\frac{1}{2}$ -in. deep sample holes are symmetrically located in each block with centers 1 in. apart. No. 22 Pt-Pt 10 percent Rh differential thermocouples are used; the furnace temperature couple is a separate chromel-alumel junction inserted in a

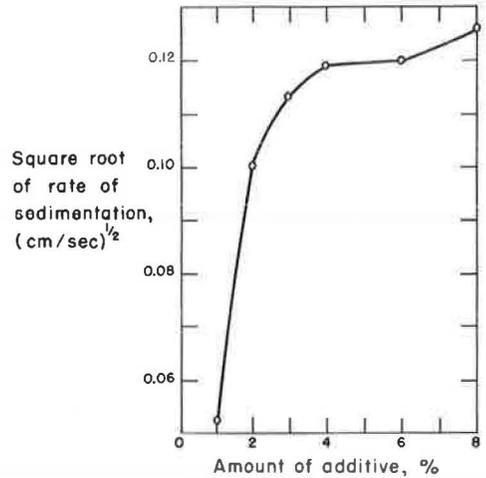


Figure 1. Square root of rate of sedimentation of gumbotil flocculated by additive of lime (12).

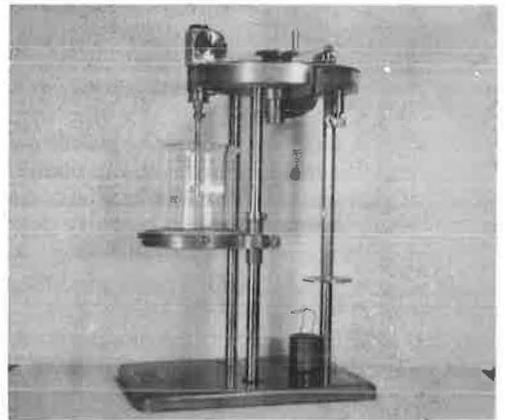


Figure 2. Stormer viscosimeter.

$\frac{3}{8}$ -in. diameter by $\frac{1}{2}$ -in. deep hole drilled up into the bottom of the block. The inert sample is powdered alumina.

Autoclaving.—Mixtures containing 100 g of Ca-bentonite, various amounts of reagent grade $\text{Ca}(\text{OH})_2$, and 110 percent distilled water were autoclaved in air-tight polypropylene bottles at 126 C and 22 psi pressure for 8 days to accelerate pozzolanic reaction. Part of each autoclaved sample was dried in a CaCl_2 desiccator and was ground to pass a No. 200 mesh sieve for DTA analysis.

Exchangeable Ca^{++} and/or $\text{Ca}^{++} + \text{Mg}^{++}$.—Exchangeable Ca^{++} and/or $\text{Ca}^{++} + \text{Mg}^{++}$ displaced by normal KCl was titrated with standard EDTA in presence of pH 10.5 $\text{NH}_4\text{OH} - \text{NH}_4\text{Cl}$ buffer, using Eriochromeschwartz T as an indicator (Appendix B). The determination of exchangeable Ca^{++} in presence of free $\text{Ca}(\text{OH})_2$ and CaCO_3 is difficult because no salt solution could be found to displace the exchangeable Ca^{++} without dissolving $\text{Ca}(\text{OH})_2$ and CaCO_3 . Water was added and free $\text{Ca}(\text{OH})_2$ was removed by mechanical shaking. The quantity of excess water needed was calculated from the solubility of $\text{Ca}(\text{OH})_2$ at 25 C (0.165 g per 100 ml). The hydrolysis of the exchangeable Ca^{++} and the solution of CaCO_3 are not likely to be appreciable. Displacement of Ca^{++} can be performed by leaching with 1 N KCl in about 15 min.

Cation Exchange Capacity.—Cation exchange capacities were determined by an ammonium acetate method using a microdistillation apparatus designed by Bremner (Appendix C).

Samples.—The Panther Creek bentonite and volclay are naturally Ca- and Na-saturated montmorillonites, respectively. Both were ground to pass a No. 200 mesh sieve.

To check if results were consistent in montmorillonitic soils, two Iowa soils, gumbotil 528-8 and plastic loess 528-4 from subhorizons were tested. The soils were passed through a No. 40 sieve and are essentially free of organic matter. Clay in these soils is predominantly Ca-saturated. Brief physico-chemical properties of the clays and soils are given in Table 1.

TABLE 1
PROPERTIES OF SAMPLES

Property	Gumbotil (528-8)	Plastic Loess (528-4)	Panther Creek Bentonite	Volclay
Source	Keokuk Co., Iowa	Keokuk Co., Iowa	American Colloid Co.	American Colloid Co.
Series	Paleo	Mahaska	--	--
Horizon	B	C	--	--
Textural composition (%)				
Sand (2-0.074 mm)	16.0	0.2	--	--
Silt (74-5 μ)	13.5	60.8	--	--
Clay (5 μ)	70.5	39.0	90.0	90.0
Predom. clay mineral	Mont.	Mont.	Mont.	Mont.
Physical properties (%)				
Liquid limit	75.6	52.1	--	--
Plastic limit	25.6	20.0	70.2	--
Plastic index	50.0	32.1	--	--
Chemical properties:				
pH	6.6	5.9	8.4	9.8
CEC (meq/100 g)	34.3	23.3	82.5	88.3
Predominant cation	Ca^{++}	Ca^{++}	Ca^{++}	Na^+

RESULTS

Viscosity vs Percent Lime

Na-Bentonite.—The viscosity of Na-bentonite slurries was markedly changed by small additions of lime (Fig. 3). A sharp increase in viscosity occurred at about 2.5 percent Ca(OH)_2 ; the viscosity reached a maximum at about 6.1 percent lime, then decreased and leveled off. There was a corresponding rapid increase in pH up to 2.5 percent Ca(OH)_2 , followed by a gradual increase. The pH was 11.85 when the sharp change in viscosity occurred.

The amount of Ca(OH)_2 causing the sudden change in viscosity coincides with the lime fixation point predicted by Eq. 1 if allowance is made for additional Ca^{++} to replace exchangeable sodium (7). Exchangeable Na^+ of the Na-bentonite is 63 meq per 100 g, the $<2\mu$ clay is roughly 90 percent, and the milliequivalent weight of $\text{Ca(OH)}_2 = 0.037$ g. Then $63 \times 0.037 = 2.33$ g, or 2.33 percent Ca(OH)_2 would be required to make a Ca-clay. Additional Ca(OH)_2 used for the development of maximum plastic limit would be $L_M = \frac{90}{35} + 1.25 = 2.57 + 1.25 = 3.82$.

Unfortunately the lime retention point for the Na-bentonite could not be verified by the plastic limit method owing to the high swelling property of this clay, which made testing difficult.

The suspension tended to form distinct, large flocs when the concentration of Ca(OH)_2 was around 6 percent and higher. This was most noticeable in flow patterns of the suspensions on wall of flasks, and is an indication of the lime retention point (Fig. 4).

Ca-Bentonite.—Viscosity and pH data for Ca-bentonite with addition of Ca(OH)_2 showed a sharp increase in viscosity at 1.5 percent Ca(OH)_2 (Fig. 5). Viscosity reached a maximum at about 2.2 percent lime; additional Ca(OH)_2 up to 5.2 percent (140 meq per 100 g) decreased viscosity. Plastic limit data also showed the coincidence between

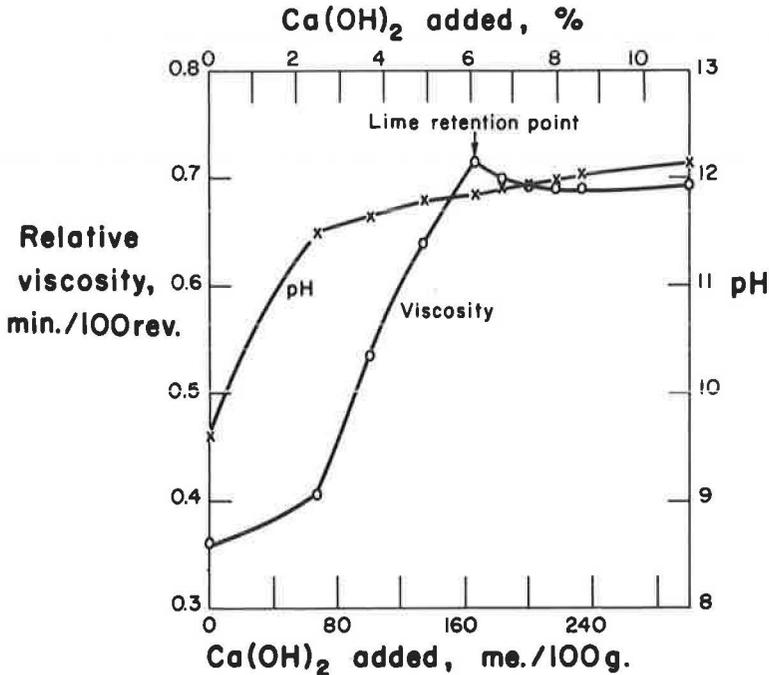


Figure 3. Viscosity and pH changes of 2.7 percent Na-bentonite suspensions with addition of Ca(OH)_2 .

percent lime for maximum viscosity with that for maximum plastic limit. The pH at this point was about 11.85. The $\text{Ca}(\text{OH})_2$ content at the lime retention point for the Ca-bentonite is much less than that for the Na-bentonite. The flow pattern shown in Figure 4 was also seen with the Ca-bentonite.

Viscosity of Ca-Bentonite + Lime vs Time.—The percent lime for maximum viscosity was found to change with time (Table 2). A clay thixotropy phenomenon should be most in evidence in a dispersed suspension, and this is difficult to relate to the shift in lime retention point, unless lime platelets are actually participating in the set.

Another possibility is that the character of the suspensions is changing due to pozzolanic reaction. If only suspensions containing lime in excess of the retention point react pozzolanically, removal of lime from the high-lime content systems would shift the viscosity vs lime curve to the right.

For the preceding reasons, all viscosity and plastic limit tests were conducted within two hours after mixing clay or soil with lime.

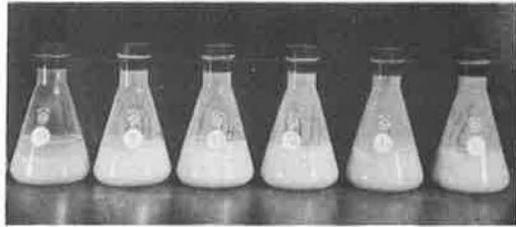


Figure 4. Flow patterns of lime-Na-bentonite suspensions.

TABLE 2
RELATION OF PERCENT LIME
TO TIME

Setting Time (days)	Lime Retention Point (% lime)
0	2.2
1	3.0
2	3.7

Viscosity vs pH

The pH's of 30 percent Ca-bentonite suspensions were adjusted to various values by adding N NaOH, with the volumes of added liquid kept constant. A sharp increase in viscosity took place at pH 11.0 (lower curve, Fig. 6). Viscosity reached a maximum at pH 11.6, where a further increase in pH reduced viscosity.

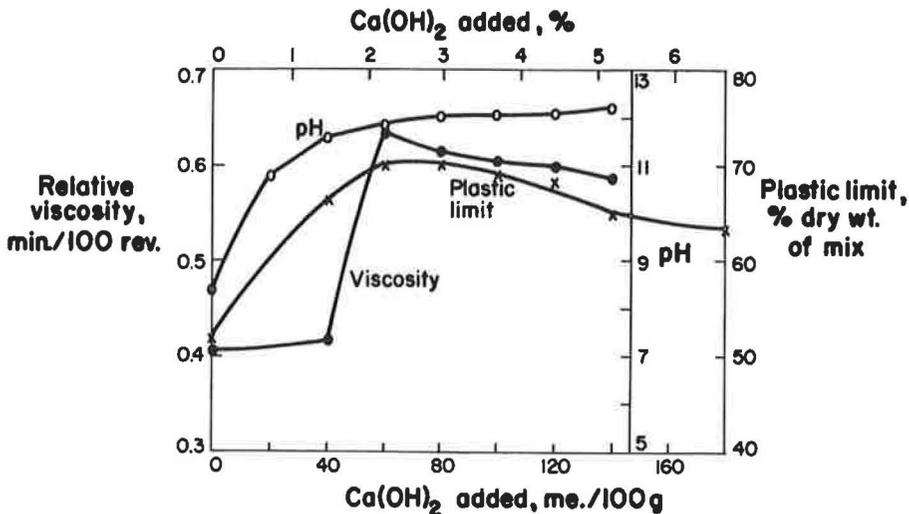


Figure 5. Changes in plastic limit and viscosity (30 percent suspension) of Ca-bentonite with addition of $\text{Ca}(\text{OH})_2$.

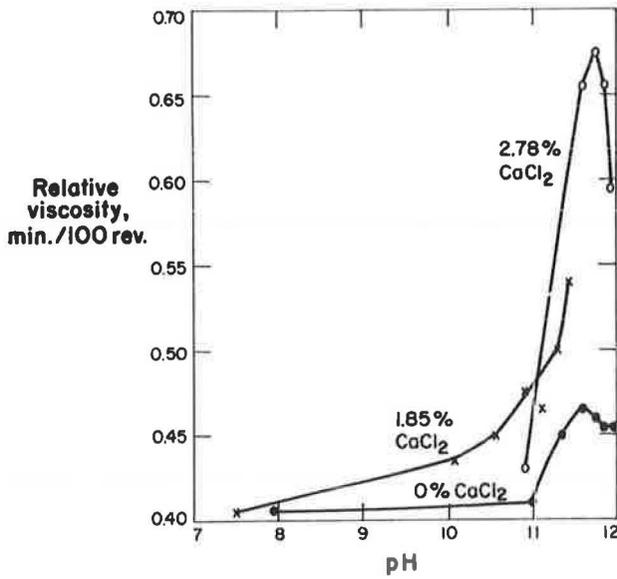


Figure 6. Effect of various pH values and constant CaCl_2 concentration on viscosity changes of 30 percent Ca-bentonite suspensions (pH adjusted by additions of NaOH).

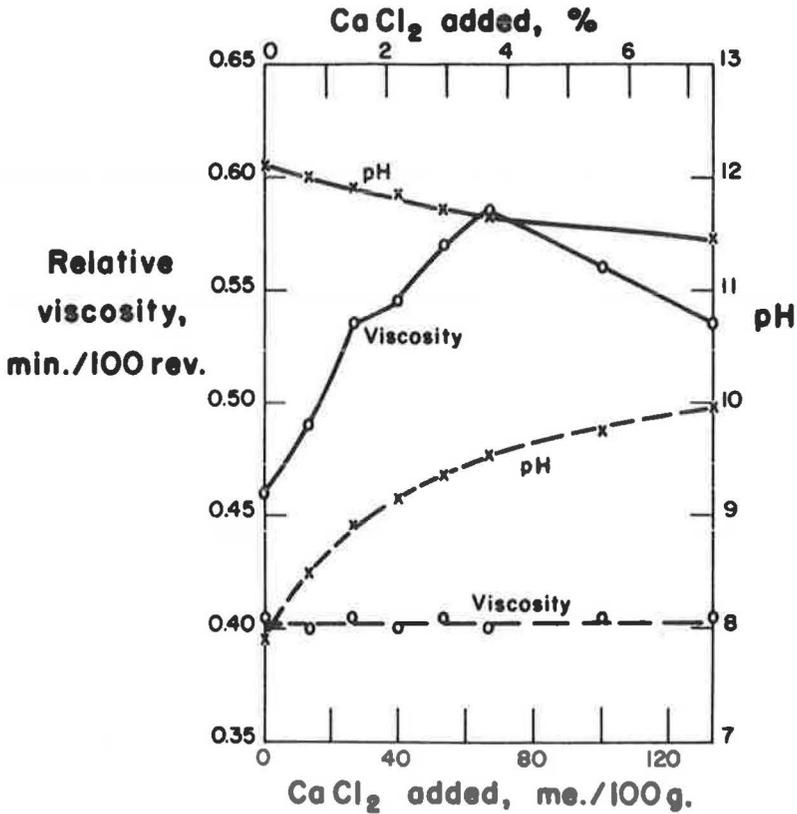


Figure 7. Effect of pH and CaCl_2 concentration on viscosity of 30 percent Ca-bentonite suspensions.

Figure 6 also shows viscosity results obtained with addition of CaCl_2 , the pH's being adjusted by addition of N NaOH. The viscosity of each suspension was increased considerably, particularly at pH's above 11.00. With 2.78 percent CaCl_2 , which corresponds to the lime retention value of 50 meq per 100 g, viscosity increased sharply and reached a maximum at pH 11.75.

To test for an optimum Ca^{++} ion content at high pH, the pH of 30 percent Ca-bentonite suspensions with various additions of CaCl_2 was maintained at 11.5 to 12.1 by adding N NaOH (top two curves, Fig. 7). The viscosity increased with CaCl_2 concentration to 3.6 percent (64 meq per 100 g) and then decreased with additional CaCl_2 . The CaCl_2 concentration at which the maximal viscosity occurred is roughly in agreement with the lime retention point obtained previously—60 meq per 100 g.

The lower curves in Figure 7 show the results of tests that repeated with various pH's below 10. No viscosity change is shown, which indicates that high pH is a requirement for Ca^{++} ions to be effective at increasing viscosity.

Exchangeable Ca^{++}

Measurements of exchangeable Ca^{++} in the lime-bentonite slurries show that exchangeable Ca^{++} increased with addition of $\text{Ca}(\text{OH})_2$, indicating pH dependence of the cation exchange capacity. There is no break in the exchangeable Ca^{++} curve at the lime retention point (Fig. 8).

The amounts of exchangeable Ca^{++} extracted were considerably below the amounts of lime added. Excess lime had been leached by water; that part was carbonated during the leaching process is indicated by X-ray diffraction.

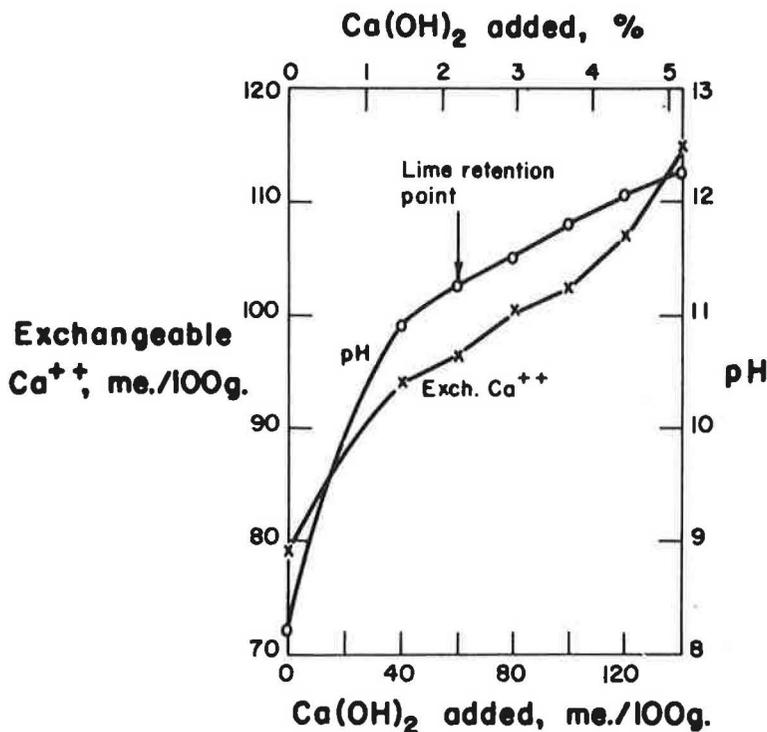


Figure 8. Relationships between $\text{Ca}(\text{OH})_2$ added, pH, and exchangeable Ca^{++} in Ca-bentonite.

Influence of Added Na^+ at High pH.—Effectiveness of Na^+ for increasing the suspension viscosity was checked by addition of NaCl or Na_2SO_4 to 3 percent Na -bentonite suspensions of pH 11.85. Viscosity did not increase significantly. The same was true with Ca -bentonite (Fig. 9).

Influence of Mg^{++} .— MgO is a constituent of dolomitic monohydrate lime. Addition of MgO to 30 percent Ca -bentonite suspensions increased their viscosities only slightly, even when the latter were measured the second day after sample preparation (Fig. 10). This could be a result of the low solubility of MgO , which gave low pH's (from 8.4 to 10.40) as well as a low Mg^{++} ion concentration. However, raising pH's to the range of 11 to 11.6 by addition of NaOH did not increase viscosities of the systems to any significant extent, indicating that the availability of Mg^{++} was probably further reduced by precipitation of $\text{Mg}(\text{OH})_2$ at high pH's.

To summarize, Ca^{++} ions increased suspension viscosity but only at high pH, the optimum Ca^{++} concentration and pH approximately coinciding with those obtained by addition of $\text{Ca}(\text{OH})_2$ alone. Na^+ ions were relatively ineffective. Mg^{++} ions were ineffective, perhaps because of low solubility at high pH.

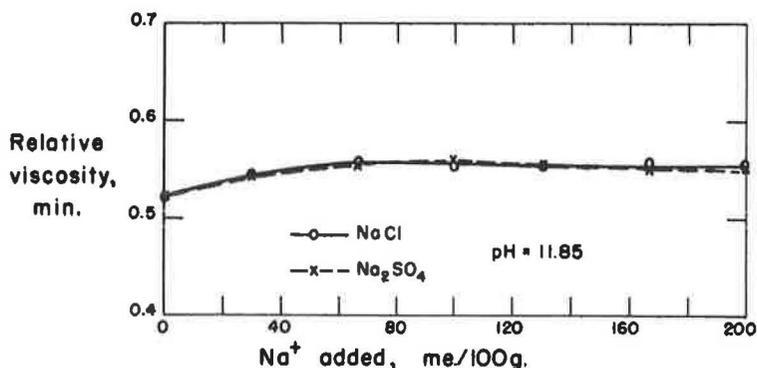


Figure 9. Effect of NaCl and Na_2SO_4 on viscosity changes of 3 percent Na -bentonite suspensions at pH 11.85.

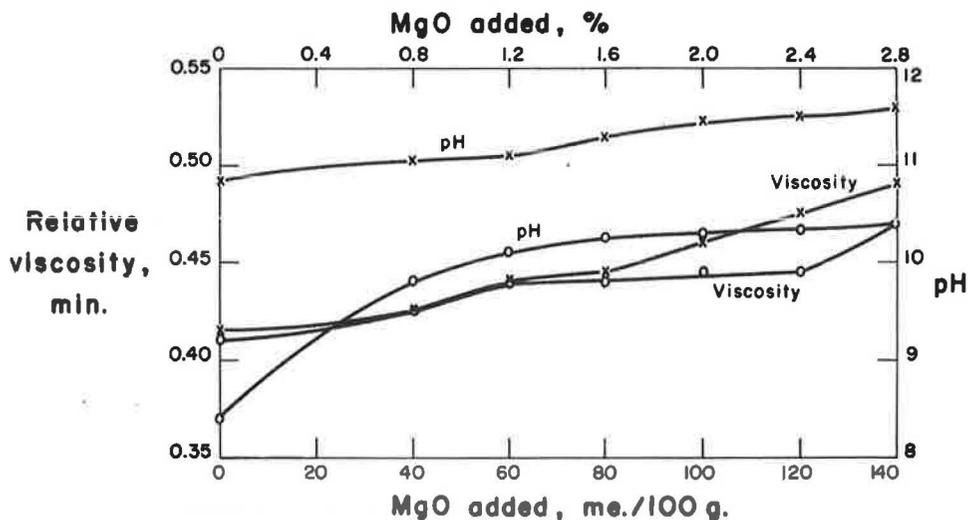


Figure 10. Effect of MgO on viscosity changes of 30 percent Ca -bentonite suspensions at various pH values.

Effect of Anions on Viscosity

As previously shown in Figure 9, increments of NaCl or Na₂SO₄ added to Na-bentonite and Ca-bentonite suspensions together with a constant amount of NaOH to bring the pH of the systems to 11.85 had no significant effect on viscosity. A great increase in viscosity was observed with small additive of Na₃PO₄ to Ca-bentonite suspensions, even at pH as low as 9.8 (Fig. 11). This abrupt increase in viscosity is clearly different from that observed in the clay-lime system, and probably resulted from the reaction between Ca⁺⁺ and phosphate ions to form a Ca-phosphate gel, because addition of Na₃PO₄ to Na-bentonite suspensions did not increase their viscosities significantly.

Pozzolanic Reaction

The availability of lime for pozzolanic reaction was evaluated by means of X-ray and differential thermal analysis of the clay-lime slurries after autoclaving. X-ray diffraction showed no new peaks indicative of reaction products, but intensity of the clay mineral (001) peak diminishes with increasing lime content. This could be influenced by other variables, such as preferred orientation or crystal perfection.

Perhaps more reliable are the differential thermal analyses (Fig. 12). Curve A' is for a dry clay-excess lime mix not autoclaved, and shows the characteristic Ca(OH)₂ and montmorillonite dehydration reactions.

Where lime was added in amounts less than the lime retention point, the Ca(OH)₂ peak no longer shows, even with no autoclaving (curve E', Fig. 12), indicating decomposition and utilization of the Ca(OH)₂ in the lime retention reaction. The Ca(OH)₂ peak also is absent in all autoclaved samples. Interesting features of the curves are first that the endothermic peaks due to dehydration of lattice OH groups (650 to 850 C) decreased with increasing Ca(OH)₂ added above the lime retention point. That is, the first 60 meq of lime had no effect on this peak, whereas the next 20 meq and the additional increments reduce it markedly. This further supports the hypothesis that pozzolanic reaction can proceed only when the quantity of lime added exceeds a threshold quantity; i. e., the lime retention point. The exothermic peak (850 C to 1,000 C) increases with increasing Ca(OH)₂ added above the lime retention point, indicating greater availability of ions for recrystallization. Furthermore, the peak gradually shifts to a lower temperature and thus becomes more clearly defined.

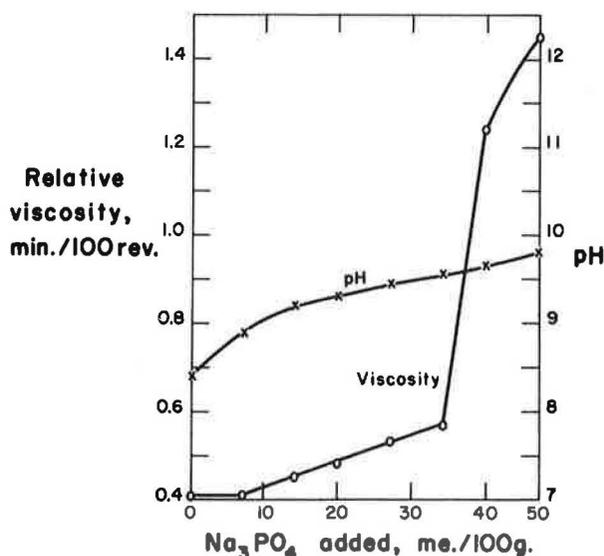


Figure 11. Effect of Na₃PO₄ on viscosity and pH of Ca-bentonite suspensions (22.4 percent).

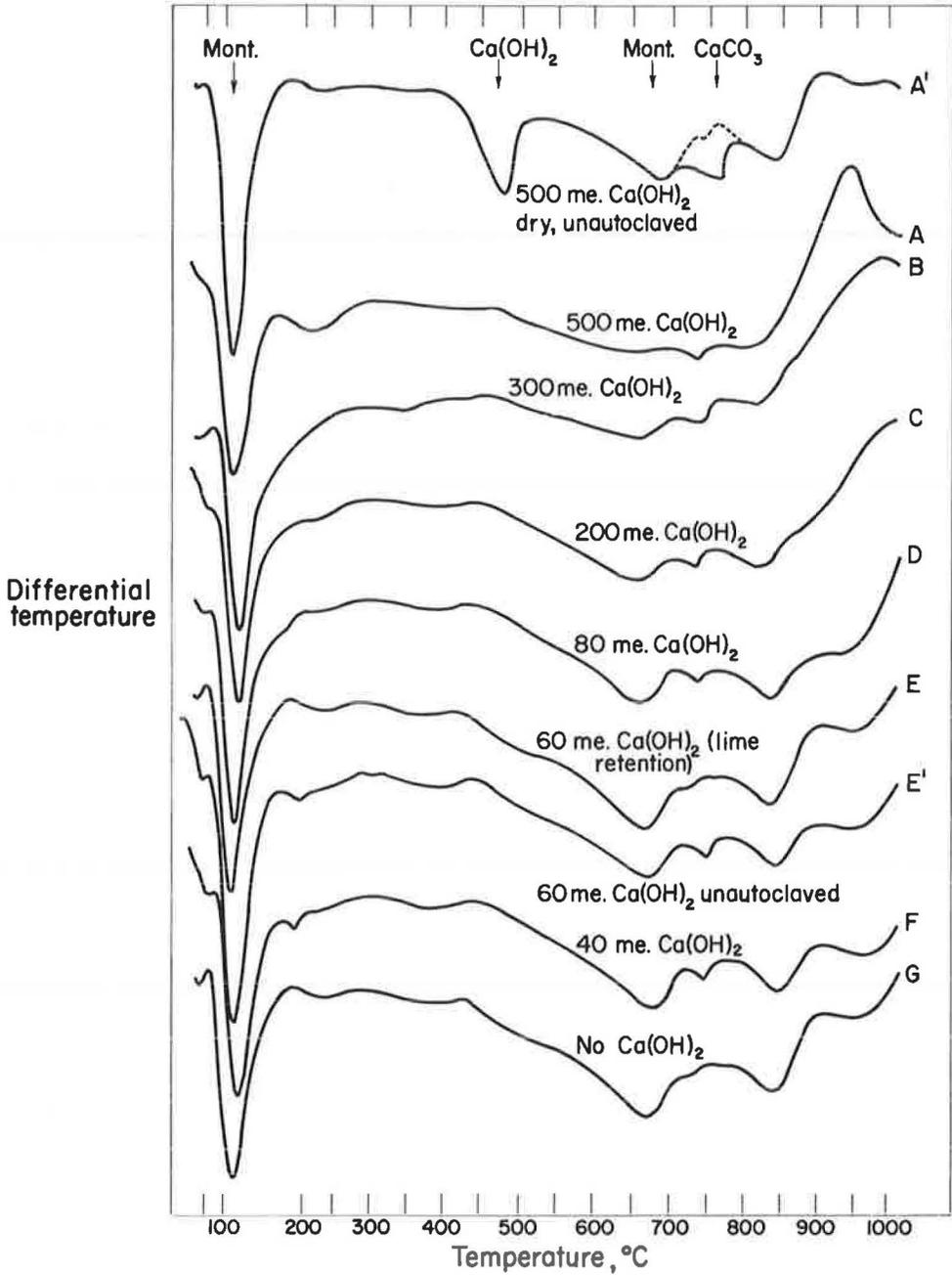


Figure 12. Differential thermal curves for Ca-bentonite treated with various amounts of lime (milliequivalents per 100 g) and autoclaved (126 C and 22 psi pressure for 8 days).

Soils

Selected tests were utilized with two Ca-montmorillonitic Iowa soils to see if relationships were consistent. Plastic limit and viscosity results from 50 percent soil suspensions indicate that the lime retention point is consistent with the percent clay and Eq. 1 (Figs. 13 and 14).

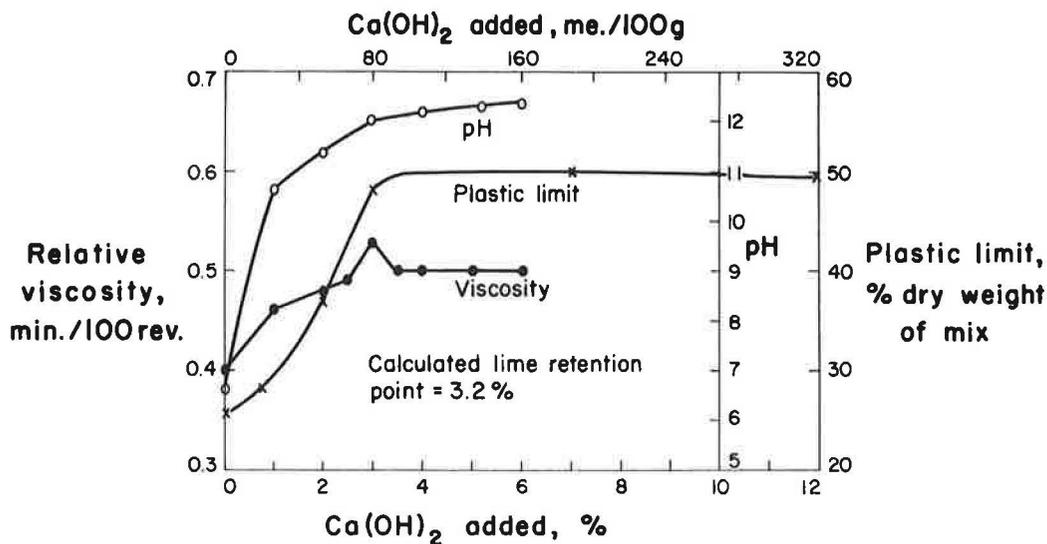


Figure 13. Changes in plastic limit and viscosity (50 percent suspension) of Iowa gum-botil 528-8 with addition of $\text{Ca}(\text{OH})_2$.

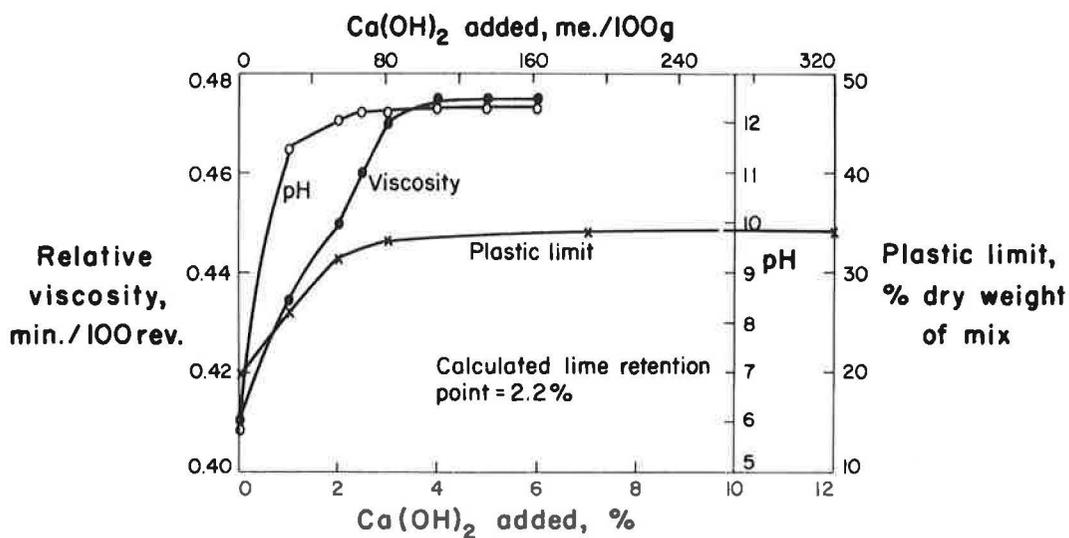


Figure 14. Changes in plastic limit and viscosity (50 percent suspension) of Iowa plastic loess 528-4 with addition of $\text{Ca}(\text{OH})_2$.

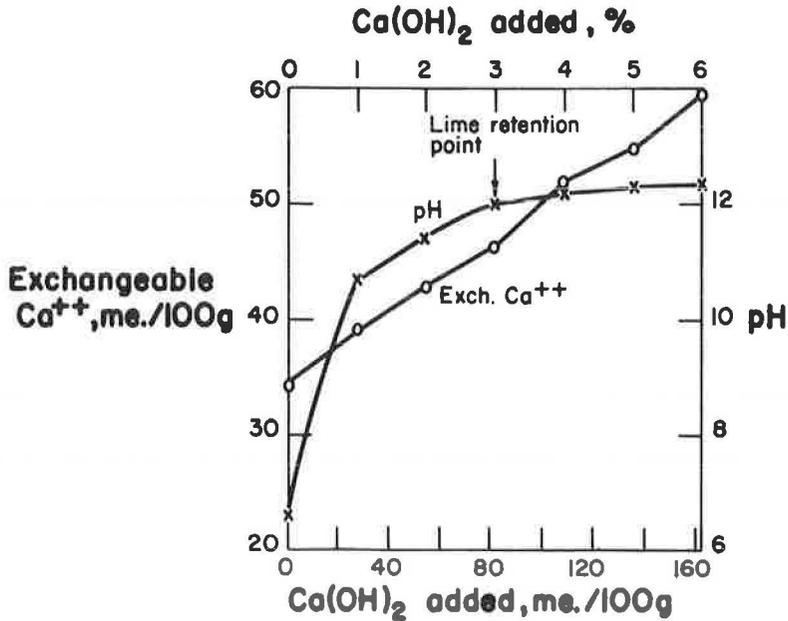


Figure 15. Relationships between $\text{Ca}(\text{OH})_2$ added, pH, and exchangeable Ca^{++} in Iowa gumbotil 528-8.

The viscosity curve of the gumbotil was similar to that of the pure clay systems, forming a maximum at the lime retention point. However, the changes in viscosity in the loess sample were gradual. Although no maximum at the lime retention point could be obtained, the inflection point coincides with that of the plastic limit curve. Furthermore, the viscosity changes with $\text{Ca}(\text{OH})_2$ in loess were small compared with those obtained with pure clays and gumbotil. These differences probably were caused by the lower percentage of clay fraction (33 percent) in the loess.

Exchangeable Ca^{++} data for lime-treated gumbotil indicate that exchangeable Ca^{++} increases with $\text{Ca}(\text{OH})_2$ added and with corresponding increase in pH (Fig. 15). This is in good agreement with data obtained with Ca-bentonite.

ANALYSIS

The cation exchange capacity of clays is ordinarily measured at pH 7.0. At higher pH's, more cations are adsorbed, perhaps because of increasing dissociation of weakly acidic SiOH - groups exposed on clay crystal edges (10, 11, 12). Below pH 5 the cation exchange capacity is constant (10, 11), usually attributed to isomorphous substitutions in the clay mineral structure.

The pH dependence of the lime retention reaction suggests a close kinship with cation exchange phenomena. In this respect it differs from cation fixation reactions or a "cation crowding" action, and the term "lime retention" is to be preferred. The increased adsorption of Ca^{++} to clay surfaces at high pH may be the cause of better bonding between particles, evidenced by bigger flocs, higher suspension viscosity, and higher plastic limit.

The lime-montmorillonite reactions are divided into three levels (Fig. 16). In the first, the total cation exchange is ordinarily measured at pH 7. In the second, the additional lime retention is dependent on an alkaline pH. In the third, after Ca^{++} -holding capacities are fulfilled, pozzolanic reactions with additional lime can proceed.

CONCLUSIONS

Previous work has shown that the reduction in plasticity, especially the increase in

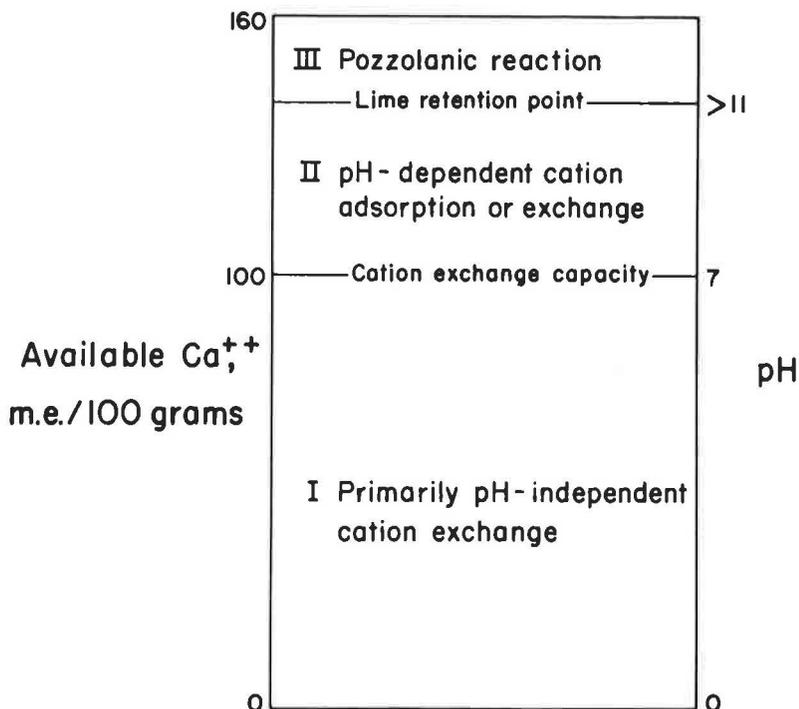


Figure 16. Levels of $\text{Ca}(\text{OH})_2$ treatment of a montmorillonitic clay.

plastic limit, of a montmorillonitic soil reaches a maximum at a certain percentage of lime, called the lime retention point. Other characteristics of this phenomenon are as follows:

1. The viscosity of slurries and the floc size also reach maxima at the lime retention point.
2. The difference between the lime retention percentages for sodium saturated and calcium saturated montmorillonites equals the cation exchange capacity; i. e., lime first converts the Na-clay to Ca-clay before additional lime is adsorbed to reach the lime retention point.
3. The amount of lime retention increases with setting time, suggesting removal of excess lime from the system by pozzolanic reaction.
4. Lime retention requires an alkaline pH, little or no retention occurs below pH 11.0, and retention is a maximum at about 11.75. $\text{Ca}(\text{OH})_2$ provides a near optimum ratio of Ca^{++} and OH^- ions, Mg^{++} and Na^+ ions are relatively ineffective.
5. Lime added below the lime retention point does not reduce autoclaved clay mineral X-ray and DTA peaks; lime in excess of this percentage does. Thus, lime in amounts less than the lime retention point does not engage in pozzolanic reaction. This agrees with earlier compressive strength results.
6. Lime used in the lime retention reaction no longer gives an indication of $\text{Ca}(\text{OH})_2$ on DTA curves, suggesting use of $\text{Ca}(\text{OH})_2$ as ions. Argument is made for considering lime retention as a pH-dependent cation adsorption reaction.

ACKNOWLEDGMENTS

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Appendix A

RELATIVE VISCOSITY WITH THE STORMER PADDLE-TYPE VISCOSIMETER

1. Satisfactory sample sizes for preparation of slurries were found to be as follows:

Na-bentonite	2-3 g
Ca-bentonite	30 g
Soils (33-50 percent Ca-montmorillonite)	50 g

2. Samples plus various measured amounts of reagent grade $\text{Ca}(\text{OH})_2$ and 100 ml of de-ionized water were placed in 250-ml Erlenmyer flasks, stoppered, and shaken thoroughly by hand or, in the case of Na-bentonite, with an electric stirrer. After 1-hr equilibration with occasional shaking, a suspension is shaken and poured into a 250-ml beaker for viscosity measurement.

3. The relative viscosity was determined with a Stormer viscosimeter using 25-g weight, by counting the exact time required for 100 revolutions for the rotar. The pH reading for each corresponding sample was taken immediately after the viscosity measurement, using a Beckman pH meter.

Appendix B

EXCHANGEABLE Ca^{++} AND Mg^{++}

A sample containing from 1 to 5 meq of exchangeable Ca^{++} or $\text{Ca}^{++} + \text{Mg}^{++}$ is weighed into a Buchner funnel (2 in. in o. d.) fitted with a moist Whatman No. 44 filter paper,

and leached with 80 ml of N KCl (in 10-ml increments). The leachate was made up to 100 ml with de-ionized water, and an aliquot was withdrawn for titration with standard EDTA.

In the Ca(OH)_2 -treated samples, free Ca(OH)_2 was first removed by mechanical shaking for 2 hr with excess water which was calculated from the solubility of Ca(OH)_2 at room temperature and the known quantities of Ca(OH)_2 added. After samples were filtered and washed with water, the determination of exchangeable Ca^{++} can be proceeded as that previously described. However, the time of leaching should be limited to around 15 to 20 min if possible, because prolonged leaching might lead to gradual dissolution of CaCO_3 .

Appendix C

MICRODISTILLATION METHOD FOR MEASUREMENT OF CATION EXCHANGE CAPACITY

Cation exchange capacities were determined by ammonium acetate method using Bremner's microdistillation apparatus:

1. A sample containing about 0.2-meq CEC was accurately weighed into a small Gooch crucible fitted with a moist Whatman No. 44 filter paper disc. The sample was leached slowly with 20-ml NH_4AC solution for 20 to 30 min. Excess NH_4AC was then removed by washing three or four times with 75 percent methyl alcohol.

2. The NH_4 -saturated sample was transferred to a 100-ml distillation flask and steam-distilled for 4 min with about 0.3 g of heavy MgO (ignited at 800 C overnight) at a distillation rate of 7.5 ml per min. The distillate was collected in a 50-ml Erlenmeyer flask containing 5-ml boric acid-indicator solution.

3. Solutions in the Erlenmeyers are titrated with standardized 0.01N H_2SO_4 .

The boric acid-indicator solution is very sensitive with regard to the change in end point and must be carefully prepared as follows: Dissolve 20 g of reagent grade boric acid in de-ionized water in a 1-1 volumetric flask. Dissolve 0.0066 g of methyl red and 0.0132 g of bromocresol green in 225 ml of 95 percent alcohol. Combine the boric acid and the indicator solutions and make approximately 998 ml with de-ionized water. Adjust this mixture with 0.1N NaOH (drop by drop) to such an extent that 1 ml of de-ionized water will change 1 ml of the boric acid indicator solution from purplish red to grayish green. Excess addition of NaOH must be avoided. Glass- or teflon-stoppered containers should be used because rubber or Tygon tubing will change the color of the solution.

For standardizing the 0.01N H_2SO_4 , THAM (i. e., Tris (hydroxy methyl) amino methene) is recommended as the primary standard, using the mixed indicator described.

Lime-Clay Mineral Reaction Products

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Separate slurries of high calcium hydrated lime or dolomitic monohydrate or dihydrate lime were made with pure bentonite, kaolinite, pulverized muscovite, quartz, or vermiculite mineral and various amounts of water. The mixes were sealed in plastic containers to prevent loss of water or entry of carbon dioxide, and cured at 70 F for two years, after which X-ray diffraction and differential thermal analysis showed varying proportions of conversion of minerals and lime into pozzolanic reaction products.

Reaction products were identified on the basis of diffractometer d-spacings. Single crystals of several of the products were isolated and unit cell parameters obtained by use of a diffractometer single crystal orienter. Debye-Scherrer powder diffraction data from crushed single crystals are presented in ASTM format and related to the diffractometer data.

Among the reaction products are the calcium silicate hydrate 10-Å tobermorite, 8.2- and 7.9-Å polymorphs of tetra-calcium aluminate hydrates, and several compounds of unknown composition.

•HYDRATED LIME added to clayey soils causes two beneficial modifications: (a) there occurs a rapid depression of the plasticity index, usually attributed to cation exchange and ion adsorption phenomena (1); and (b) lime added in excess of the lime fixation capacity causes a long-term cementation attributed to the chemical reactions between lime and silicious minerals or glasses. The latter reactions are termed pozzolanic reactions.

REVIEW

Pozzolanic Reactions

In investigations of pozzolanic reaction products, Leonard and Davidson (2) report new X-ray diffraction peaks at 17.3, 12.6, and 3.08 Å after reaction of $\text{Ca}(\text{OH})_2$ and fly ash in a slurry for one year at room temperature, and report no new peaks for mixes cured at 40, 60, and 80 C. They conclude from lime depletion data that the initial product in either case is non-crystalline, after which there is slow crystallization to calcium silicate hydrate I, similar to the mineral tobermorite.

Minnick (3) reports new diffraction peaks at 3.31, 3.05, 2.97, and 2.77 Å for $\text{Ca}(\text{OH})_2$ -fly ash samples either cured at 140 C or autoclaved, and essentially the same peaks with dolomitic lime, $\text{Ca}(\text{OH})_2 + \text{MgO}$. He also notes that MgO goes to $\text{Mg}(\text{OH})_2$.

From pozzolanic reactions with natural clay minerals, Eades and Grim (4) report new diffraction lines for $\text{Ca}(\text{OH})_2$ -kaolinite cured at 60 C, the lines being at 5.09, 3.04, 2.8, and 1.8 Å. New lines were weaker for lime-illite or lime-montmorillonite cured at the same temperature. Hilt and Davidson (5) report new diffraction lines at 8.11

and 7.59 Å for $\text{Ca}(\text{OH})_2$ -montmorillonite reacted for 30 days at room temperature. The 7.59-Å compound was isolated and found to be rhombohedral, with lattice constants in the hexagonal system $a_0 = 5.7550 \text{ \AA}$ and $c_0 = 46.654 \text{ \AA}$. The compound was tentatively identified as a hydrated calcium aluminate. Despite its sharpness, the 8.11-Å peak was assigned to montmorillonite. In mixes with variable water content the 8.11-Å peak was stronger in the water mixes. Other peaks appear on diffractometer traces at about 10 and 7.1 Å. Glenn and Handy (6) isolated a 7.94-Å mineral formed from reaction of montmorillonite and dolomitic lime [$\text{Ca}(\text{OH})_2 + \text{MgO}$] and indexed it in the triclinic system.

Silicate Hydrates

In the field of portland cement chemistry, hydration of tricalcium silicate six days at room temperature in a ball mill resulted in a product identified as the calcium silicate hydrate afwillite, $\text{C}_3\text{S}_2\text{H}_3$, with a faint suggestion of tobermorite structure with the same formula (7). The strong afwillite lines are at 6.61, 3.18, 2.83 (strongest), and 2.73 Å. Hydration for 21 to 30 months in a paste gave only poorly crystallized 11-Å hydrate tobermorite, with a very broad line at 3.03 Å and weaker lines at 11, 2.83, and 1.82 Å.

Extensive Italian work on portland-pozzolan cements indicates formation of a tobermorite gel, a hexagonal calcium aluminate hydrate, C_4AH_{13} , and possibility of C_2SAH_x (Stratling's compound) (8). The latter was first reported from reaction of $\text{Ca}(\text{OH})_2$ and calcined kaolinite.

Aluminate Hydrates

Hydrated calcium aluminates crystallize in a variety of stable and metastable hexagonal and cubic structures. Recent work indicates existence of a family of hexagonal, platey aluminates, sulfoaluminates (9), and carboaluminates (8). Most pertinent to the present work are α and β polymorphs of C_4AH_{13} , with 8.2 and 7.9 Å maximum d-spacings, respectively, which may dehydrate to C_2AH_8 ($d_{\text{max}} = 10.6 \text{ \AA}$) or CAH_{10} ($d_{\text{max}} = 14.6 \text{ \AA}$). (There is now evidence that the 8.2-Å form may be a hemicarbonat.)

A summary of strongest d-spacings for pertinent compounds is given in Table 1.

METHODS

Difficulties inherent in the powder X-ray method have limited positive interpretations in both cement and pozzolan chemistry. Fortunately, slow pozzolanic reaction at room temperature yields crystals as large as 200 μ in diameter, large enough to be picked from the matrix and examined by single crystal X-ray techniques (5, 6). The objective of the present investigation is to gain further single crystal data and relate to powder diagrams and to known compounds. A General Electric XRD-5 single crystal orienter was used. Most of the single crystals examined were also crushed and photographed in a Siemens Debye-Scherrer camera to give accurate powder identification data. Studies of chemical compositions are planned by means of an electron-microprobe.

Slurries of clay minerals with lime and variable amounts of water were prepared, sealed in plastic containers, and allowed to cure at $70 \pm 2 \text{ F}$ for up to two years. The clay minerals are as follows:

1. Otay, Calif., bentonite, AAPG reference clay mineral 24 (10).
2. Lewiston, Mont., kaolinite, AAPG reference clay mineral 17 (10).
3. Montana vermiculite, Lincoln County, Mont.
4. Muscovite.

Three types of lime were used each with clay mineral:

1. High calcium hydrated lime, $\text{Ca}(\text{OH})_2$
2. Dolomitic monohydrated lime, $\text{Ca}(\text{OH})_2 + \text{MgO}$
3. Dolomitic dihydrated lime, $\text{Ca}(\text{OH})_2 + \text{Mg}(\text{OH})_2$

TABLE 1
MEDIUM AND STRONG POWDER DIFFRACTION LINES FOR SOME HYDRATED CALCIUM SILICATES AND ALUMINATES¹

Compound	Cryst. System	Ref. No.	d	I	hkl	Compound	Cryst. System	Ref. No.	d	I	hkl
Tobermorite group (CSH I)	Ortho	15	9.14	vs	002	CAH ₁₀ (Cont.)			2.26	6	
			3.08	vs	220				2.18	6	
			2.81	s	400				2.11	4	
			1.83	s	040				2.06	4	
			1.67	m	620				1.94	5	
			1.54	w	440				1.83	3	
10-Å tobermorite CSH	Ortho	15	1.40	m	800	1.79	4				
			10	vs	002	1.71	3				
			3.05	vs	220	1.64	5				
			2.93	s	206	1.60	6				
			2.80	s	400	1.38	4				
			1.83	s	040						
Afwillite C ₃ S ₂ H ₅	Monoclinic	15	6.46	s	202	C ₃ AH ₆	Cubic	16	5.13	s	211
			5.74	s	200				4.45	s	220
			5.08	mw	110				3.36	s	321
			4.73	s	002				3.14	s	400
			2.84	vs	314				2.814	vs	420
			2.74	vs	512				2.47	m	510
			2.35	m	004				2.30	vs	521
			2.145	s	406, 424, 510				2.04	vs	611
			1.989	m	222				1.679	s	642
			1.949	s	806, 623, 624				1.595	m	
			1.862	m	625				1.572	m	
			1.805	s	024				1.372	m	
			1.776	s	204, 602				1.342	m	
			1.704	m	916				1.199	m	
α C ₂ AH ₁₂	Hex	16	1.683	m	330, 135	C ₂ ASH ₆ (Stralling's compound)	Hex	18	12.7	vs	001
			1.630	m	334, 10, 06				6.3	s	002
			1.604	s	532, 826				4.20	bs	111
			1.589	m	512				2.89	m	210
			8.2	vs	301				2.62	m	212
			4.1	m	002				2.51	m	301
			3.9	m	111				2.37	m	302
			2.9	m	210				1.90	m	313
			2.45	m	301				1.83	m	402
			2.24	m	203				1.67	mw	411
β C ₄ AH ₁₂	Hex	17	1.67	m	410	C ₃ AH ₁₁ · CaCO ₃	Hex	19	7.567	100	001, 100
			7.92	vvs	001				3.784	44	002, 111, 200
			3.98	s	002				2.858	30	112, 210
			2.87	s	110				2.725	14	202?
			2.70	m br	114				2.524	18	003, 300
			2.46	s br	114, 024, 204				2.488	18	
			2.24	m	114				2.441	18	103?
			2.05	su	104, 014				2.419	24	301?
			1.857	ms	300				2.339	22	-
			10.6	vs	001				1.943	10	-
C ₂ AH ₆	Hex	16	5.73	s	002	Pozz. reac. prod. Hex (C ₂ AH ₆ · 12H ₂ O)	5		1.824	12	-
			3.55	m	201				1.661	12	402
			2.87	s	120				1.652	16	321
			2.48	m	301				1.642	12	410
			1.667	m	410				7.59	10	006
			1.443	m	420				3.85	7	008
CAH ₁₀	Hex	19	14.3	10					3.42	4	10, 10
			7.16	10					2.87	9	110, 113
			5.39	4					2.52	6	10, 16, 11, 9
			4.75	4					2.33	6	11, 12
			4.52	3					2.30	8	208
			4.16	3					2.20	4	02, 10
			3.72	5					2.11	5	01, 20
			3.56	7					1.93	3	11, 19, 02, 16
			3.26	6					1.86	3	21, 4, 12, 5
			3.10	5					1.72	4	20, 20
			2.88	6					1.66	5	300
			2.69	5					1.63	4	306
			2.55	7					1.53	3	30, 12
			2.47	5					1.475	4	12, 20
2.36	6										

¹ a₀ = 5,7550 Å; C₀ = 46,65 Å.

Limes were synthesized from reagent-grade laboratory chemicals. In soil stabilization the dolomitic monohydrate lime has been shown to be most effective for pozzolanic strength (11).

Powder Diffractometer Method

Reacted slurries were X-rayed under conditions approaching 100 percent r. h. with a CO₂-free atmosphere (Fig. 1).

Initially the reacted slurries were hand-packed into flat bakelite holders. Later it was found that the denser packing obtained from use of a circular brass ring mount and 1,000-psi pressure gave much higher reaction product intensities, and this proce-

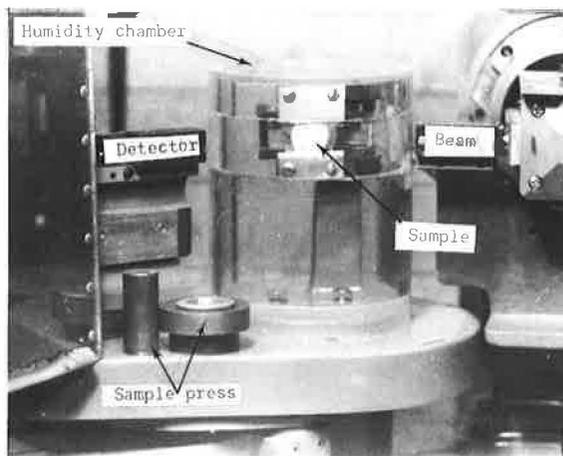


Figure 1. X-raying under 100 percent r. h. in CO_2 -free atmosphere. Before entering chamber, air is passed through CaCl_2 , ascarite, and warm water. Window for passage of X-rays is Mylar plastic.

ture was employed for most curves. (Sample holders are nominal $\frac{1}{2}$ -in. brass washers, 1.25 in. o.d. and 0.091 in. thick, with the center drilled out to give a $\frac{1}{2}$ -sq in. hole area.)

Occurrence and intensity of new peaks relate to amount of water in the mix as well as kind of mineral and kind of lime, suggesting solubility as an important factor. Slurries with the largest new peaks were selected for detailed study.

Selection of Single Crystals

Sections were sliced from cured mixtures, broken up manually, and placed in a flat transparent dish illuminated on a dark background. A binocular microscope having about 90X provided sufficient magnification to permit picking of crystals with a glass fiber. The crystals were placed in a container such that most surficial material could be picked away manually under the microscope.

Pairs of similar crystals were then chosen and cemented to glass fibers using Canada balsam so that the long dimension of the crystal was perpendicular to the fiber. Preliminary rotating single crystal films were made to assure that the crystals were of similar internal structure. A Siemens 70-mm by 50.8-mm diameter camera and chromium $K\alpha$ radiation were used for these explorations. Final selection was made of the pairs of crystals that gave a strong d-spacing previously shown as a reaction product in diffractometer traces of the slurries. One of the crystals was then demounted, placed on a glass plate and crushed for a powder photograph using the Siemens camera and $\text{CrK}\alpha$ radiation. A very fine glass fiber dipped in a thin solution of rubber cement was rolled in the powder and Debye-Scherrer film was made. Films were measured directly to 0.01 mm with a Kirem coincidence scale. Four or five of the strongest lines were measured to determine θ angles and expedite the oriented single crystal studies.

Single Crystal Orienter

The remaining single crystal was mounted, optically oriented on the single crystal orienter, and the unit was mounted on an XRD-5 diffractometer. A search for a reflection was made utilizing $\text{CuK}\alpha$ radiation, first setting 2θ for one of the strongest lines observed in the powder film. This search was made with the approximate ab (basal) plane horizontal ($X = 0^\circ$), rotating the crystal about the polar or ϕ axis (the near-vertical glass fiber) in the X-ray beam. If no reflection was found in a 360° rotation, 2θ settings for other strong film lines were searched in a similar manner until a reflection was found. The crystal orientation was then adjusted by means of the

goniometer head until the intensity of the diffracted X-ray beam was balanced for this and its $180^\circ \phi$ related reflection (12).

A second reflection was sought and found, preferably with the crystal rotated about the ϕ -axis between 60° and 90° from the first reflection. Usually the most difficult reflection to find, this is required for plotting the reciprocal lattice of the basal plane. When the second reflection was obtained and balanced under fine conditions in conjunction with the first, the two were plotted on polar coordinate paper and predictions of other reflections were made by extension of the two-dimensional reciprocal lattice plane. Reflections were checked and their intensities recorded. From this, polar planes were identified, and searches were extended to the third dimension by varying the inclination of the polar axis (θ angle) so as to identify the principal crystallographic axes of the crystal.

Indexing Reflections

The reciprocal space plots of all reflections obtained in the basal and polar planes were indexed based on the assignment of crystallographic axes and choice of the unit cell. Unit cell constants and d-spacings were then calculated using Buerger's procedure (13).

Powder Data

After determination of crystal parameters the single crystal was demounted and crushed to obtain further powder diffraction data on the pure compound. A Debye-Scherrer camera was used. The powder method enables quick measurement of d-spacings and intensities which would require much time to find by single crystal methods.

Differential Thermal Analysis

Differential thermal analysis of reacted clay-lime mixtures was run using an apparatus similar to that of Kerr et al. (10). The heating rate was 10 C per min, with an air atmosphere.

RESULTS

X-Ray Diffraction of Cured Mixtures

Kaolinite.—X-ray diffraction shows a reduction in kaolinite peak intensities after reaction with lime (Fig. 2). However, much unreacted $\text{Ca}(\text{OH})_2$ remains in the calcitic lime mix after two years. Small amounts of $\text{Ca}(\text{OH})_2$ and much $\text{Mg}(\text{OH})_2$ remain in the dolomitic lime mixes. New diffraction peaks are given in Table 2, and include evidences of 12.6-Å material from reaction with dolomitic limes, and a 7.6-Å product which appears strongest from reaction with calcitic lime but is also present in the other mixes.

Vermiculite.—The raw, wet vermiculite sample shows, in addition to vermiculite, considerable regular interlayering of the 15.2-Å Ca-vermiculite with 10-Å biotite to give strong 27-, 12.6-, and 8.7-Å spacings (Fig. 2). This interlayer is sometimes called rectorite (14). On treatment with any of the three kinds of lime, vermiculite and rectorite spacings shift to 15.0 and 25 Å, respectively, and higher orders of these, perhaps indicating additional adsorption of Ca^{++} as exchangeable ion, similar to the reaction of lime with montmorillonite (1). Assignment of peaks on the diffractometer charts is aided by this shift of basal spacing multiples.

Although the mineral peaks are reduced in the lime mixes compared with the raw sample, few new peaks were found, and much lime remained unreacted after two years. The mix with calcitic lime showed moderate peaks at 8.1 and 4.59 Å.

Muscovite.—In the muscovite mixes, only that with $\text{Ca}(\text{OH})_2$ showed any crystalline reaction product, the peak being at 7.5 Å (Fig. 2). All mixes contained appreciable unreacted lime.

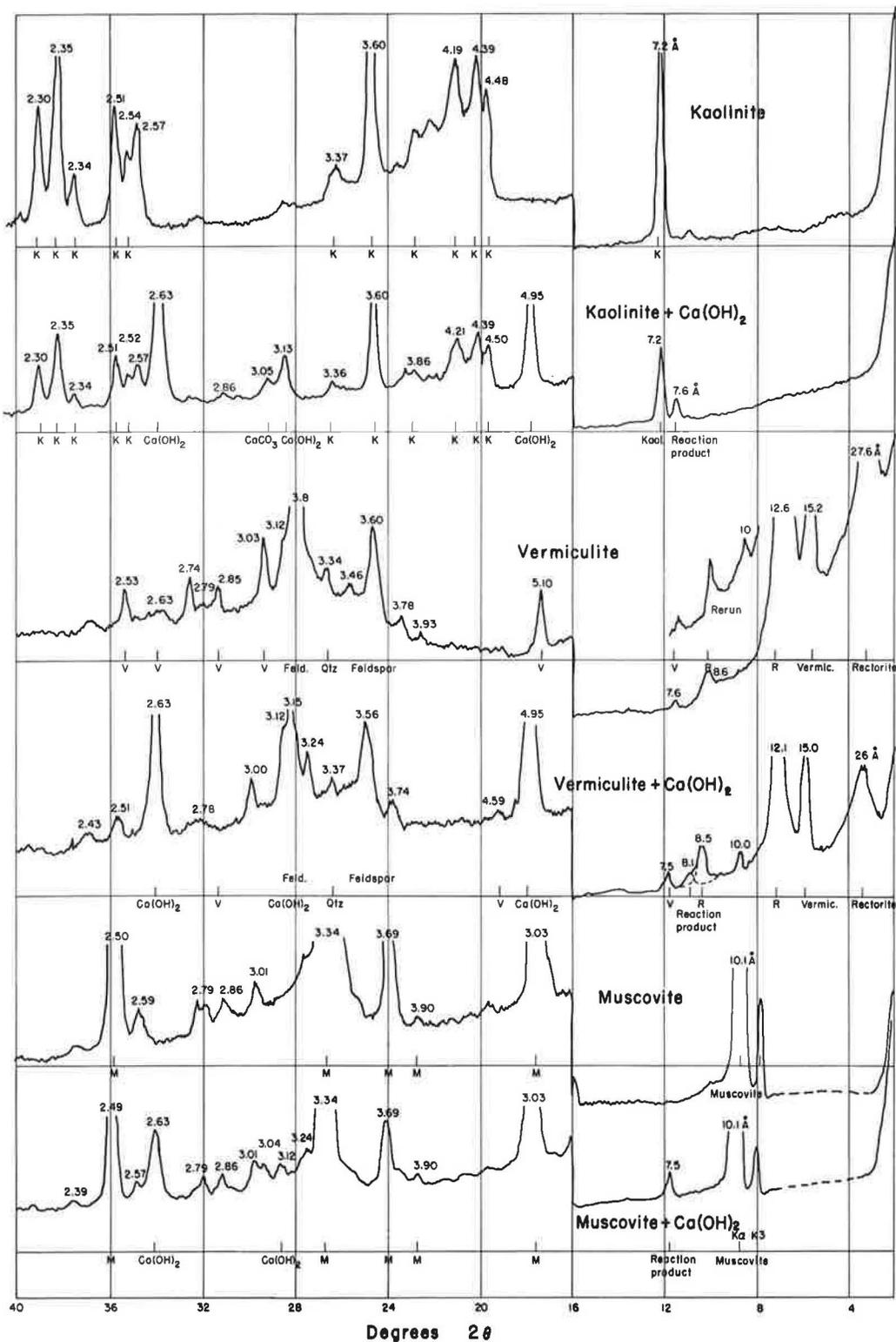


Figure 2. X-ray diffractometer curves for kaolinite, vermiculite-rectorite, and muscovite, with and without reaction with lime.

TABLE 2
REACTION PRODUCT d-SPACINGS FROM SELECTED MIXES

Mineral	Lime	New Peaks (Å)
Kaolinite	Ca(OH) ₂	7.6m, 3.8w, 3.05m, 2.8w
	Ca(OH) ₂ + MgO	12.6m vb, 7.6vw, 3.05m, 2.10vw
	Ca(OH) ₂ + Mg(OH) ₂	12.6m vb, 7.6vw, 3.80vw, 3.05s, 2.85vw, 2.10w
Vermiculite	Ca(OH) ₂	8.1m, 4.59w
	Ca(OH) ₂ + MgO	3.03w?
	Ca(OH) ₂ + Mg(OH) ₂	None
Muscovite	Ca(OH) ₂	7.5m
	Ca(OH) ₂ + MgO	None
	Ca(OH) ₂ + Mg(OH) ₂	None

Quartz.—No reactions were observed in the quartz-lime mixtures except hydration of MgO in the monohydrate lime.

Montmorillonite.—The bentonite mixtures showed appreciable crystalline pozzolanic reaction products (Fig. 3; Table 3). In the Ca(OH)₂ mix, 14 new peaks were found which could not be assigned to the clay, lime, or carbonates. Seven of these coincide with reflections from 10-Å tobermorite, CSH (15). In the dolomitic monohydrate lime mixes, some of the same and some different peaks appear, with seven of those present being characteristic of the tobermorite group. In the dihydrate lime mixes fewer peaks occur, all being characteristic of tobermorite.

If 10-Å tobermorite is assumed as a reaction product in all three montmorillonite mixes, unexplained peaks remaining in the Ca(OH)₂ mix are at 8.1, 7.9, 7.6, 4.05, 3.94, 3.79, 2.89, and 2.54 Å. Four of these coincide with the first four peaks listed for αC₄AH₁₃ (Table 1). This leaves 7.9-, 7.6-, 3.79-, and 2.54-Å peaks unexplained. The 7.9-Å lime may indicate presence of βC₄AH₁₃ (Table 1), and the line at 7.6 Å could be C₃AH₁₁ · CaCO₃ or the compound reported by Hilt and Davidson (5). The former is reported to further carbonate and decompose on exposure to air, and may have been found in reacted lime-fly ash (5).

In the monohydrate dolomitic lime, peaks not accounted for by tobermorite and βC₄AH₁₃ are at 8.8 (vw), 3.30 (s), 4.31 (w), 1.74 (w), and 1.54 (w) Å.

In the dihydrate lime mix, all reaction product peaks may be ascribed to tobermorite.

Differential Thermal Analysis of Cured Mixtures

Differential thermal analysis shows that montmorillonite endotherms at 650 and 850 C have disappeared from bentonite reacted with any of the three types of lime (curves 1 to 4, Fig. 4), suggesting weakening of the octahedral layer. X-ray shows the basal and (060) dimensions to be unchanged, but the peaks are smaller. Small endotherms from 200 to 400 C (curves 2 to 4) may be reaction products.

The DTA curves for the kaolinite mixture (curve 6) show considerable reduction of the kaolinite 600 C endotherm, but unreacted Ca(OH)₂ and kaolinite are still present after two years.

DTA of the vermiculite-Ca(OH)₂ slurry shows only unreacted mineral and lime (curve 7), whereas the curve for muscovite-Ca(OH)₂ indicates depletion of the lime and new peaks at 240 and 470 C. The latter suggests a degraded muscovite structure rather than a pozzolanic reaction product.

Single Crystal Data

7.95-Å Hexagonal Platey Product.—Single crystal data for 7.95-Å crystal picked

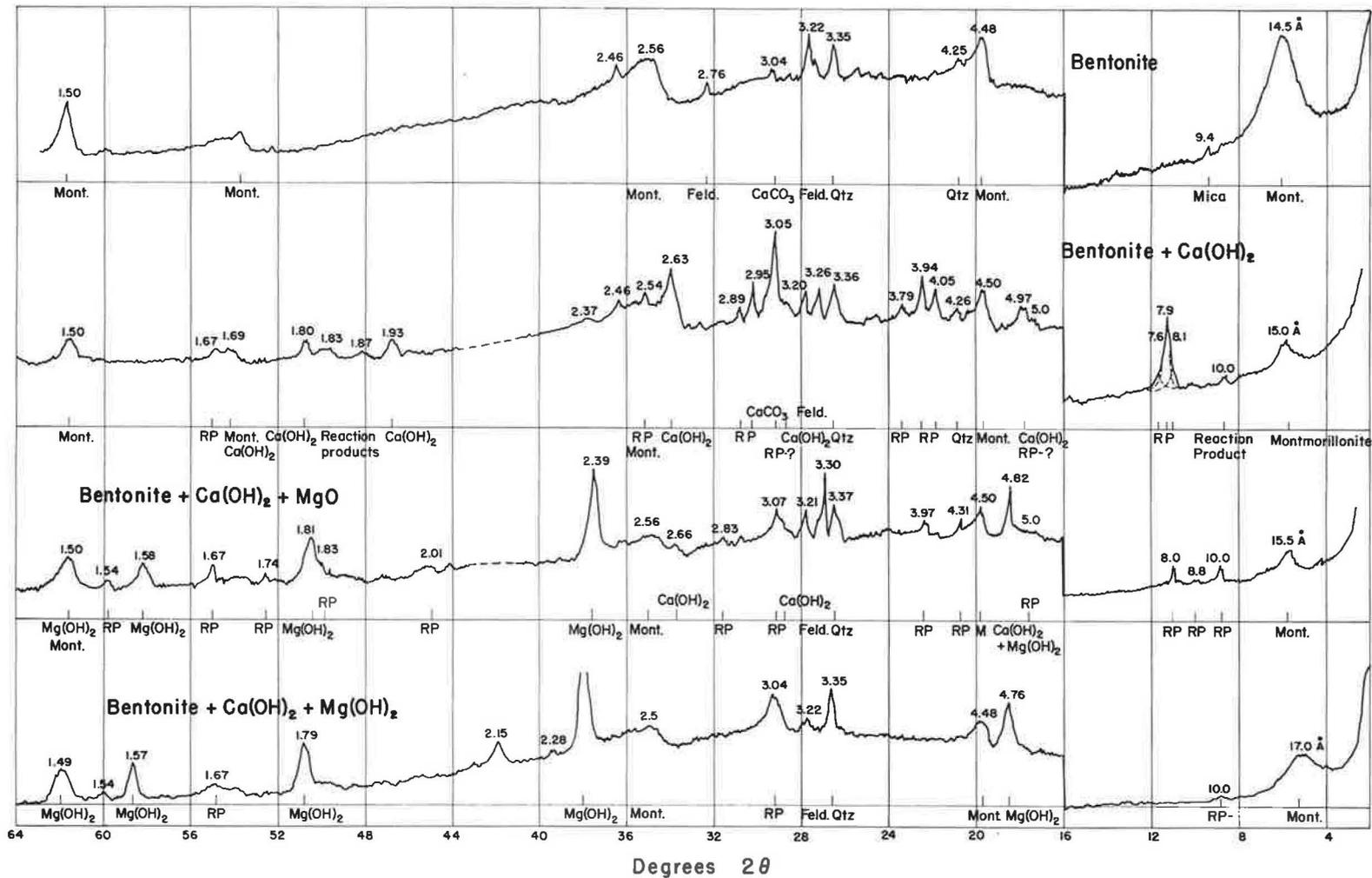


Figure 3. X-ray diffractometer curves for montmorillonite slurries with various kinds of lime.

TABLE 3
REACTION PRODUCT d-SPACINGS FROM
CURED LIME-BENTONITE MIXTURES

Ca(OH) ₂		Ca(OH) ₂ +MgO		Ca(OH) ₂ +Mg(OH) ₂	
d	I	d	I	d	I
10	w	10	m	10	vw
		8.8	vw		
8.1	m				
7.9	vs	7.95	m		
7.6	m				
5.0	m vb	5.0	m vb		
		4.31	w		
4.05	m				
3.9	s	3.97	w		
3.79	w				
		3.30?	s		
3.05	vs	3.07	vs b	3.04	s b
2.95	s				
2.89	m				
		2.83	w b		
2.54	w				
		2.01	w b		
1.83	m vb	1.83	m vb		
		1.74	w		
1.67	mb	1.67	m	1.67	m b
		1.54	w	1.54	w
		1.43	w	1.43	w

from a cured Ca(OH)₂-bentonite mix are given in Table 4. The powder data show a close resemblance to βC₄AH₁₃ (Table 1), but Miller indexes differ, primarily because of discovery of a (11·2) reflection and its second, third, and fourth, and fifth orders. Representative single crystal data and the powder data from a crushed single crystal are also given in Table 4. The refractive index of the ordinary ray was determined by the immersion method to be 1.548 ± 0.004. The crystal is uniaxial negative.

A previously described 7.94-Å triclinic crystal (unit cell parameters are a₀ = 8.49; b₀ = 12.62; c₀ = 8.31; α = 109.42°; β = 110.29°; γ = 95.33°) from the monohydrate dolomitic lime mix (6) gives only a weak 7.94-Å reflection on a Debye-Scherrer X-ray film of the crushed crystal. The three strongest lines were 3.22, 1.80, and 3.76 Å, which do not correspond with strong lines observed in the mixes. Therefore, the strong 7.95-Å peaks seen in Figure 2 are believed to relate to the hexagonal product.

8.1-Å Product.—An 8.1-Å peak occurs in Ca(OH)₂ reacted with either montmorillonite or vermiculite. As previously mentioned, this and other peaks occurring in the montmorillonite mix coincide with those of αC₄AH₁₃. Single crystals have not been isolated.

10-Å Product.—Although powder data tend to confirm existence of 10-Å tobermorite in the cured montmorillonite mixes, 10-Å crystals that were not tobermorite were

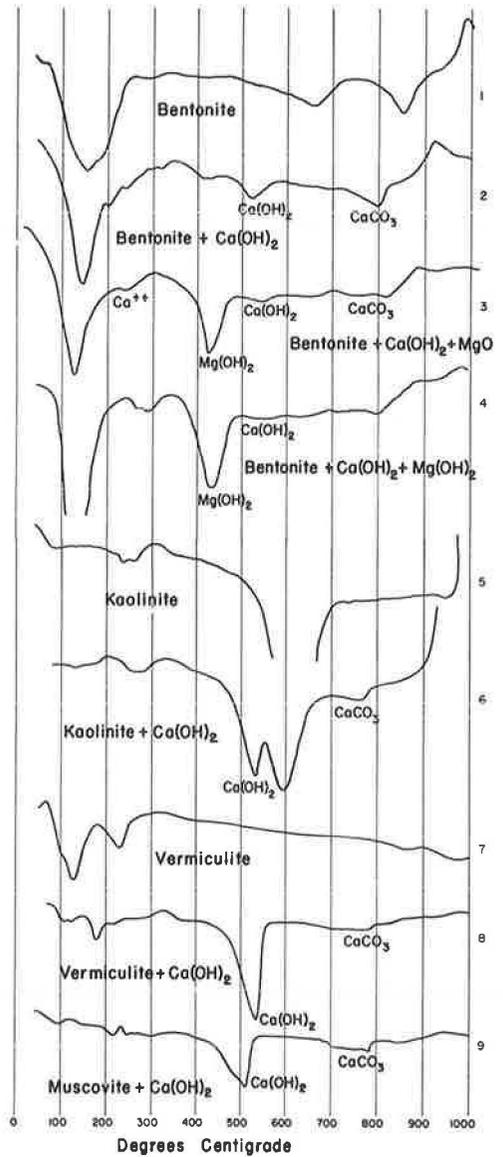


Figure 4. DTA curves for various clay-lime mixes.

TABLE 4

X-RAY DIFFRACTION DATA FOR A HEXAGONAL 7.95-Å REACTION PRODUCT ISOLATED FROM CURED MONTMORILLONITE+ Ca(OH)₂-WATER MIXTURE

Single Crystal ^a			Index (hexagonal) (hk.l)	Powder ^b of Crushed Crystal (d-spacing)	
Observed	Calc.	Intensity		Observed	Intensity
7.95	7.95	10	00.6	<u>7.92</u>	10
4.90	4.89	~ 1	11.2	<u>3.95</u>	7
3.91	3.98	7	00.12	<u>3.73</u>	5
				3.37	1
				2.98	1
				<u>2.85</u>	8
2.88	2.88	~ 1	30.0		
2.85	2.84	~ 1	30.3		
2.72	2.71	~ 1	30.6	2.67	2
2.60	2.65	~ 1	00.18		
2.54	2.53	2	30.9	2.51	7
2.45	2.44	3	22.4	2.43	6
2.34	2.33	1	30.12	2.32	5 ^c
				2.19	2
2.13	2.14	~ 1	30.15	2.11	5 ^c
1.96	1.99		00.24	1.99	1
1.95	1.95		30.18	1.96	1
				1.90	1
				1.86	1
				1.70	2 ^c
1.67	1.67	5	33.0	1.66	7
1.63	1.63	1	33.6	1.63	4
1.56	1.59		00.30		
1.54	1.54	~ 1	33.12	1.53	1
1.44	1.44	2	60.0	1.47	1
1.43	1.43	2	60.3	1.45	1 ^c
1.42	1.42	~ 1	60.6	1.41	1 ^c
				1.38	1 ^c
1.36	1.35	1	60.12	1.35	1 ^c
				1.31	1 ^c
1.27	1.27	~ 1	60.18	1.28	1 ^c
				1.26	1 ^c
				1.25	1 ^c
1.22	1.22	~ 1	44.8	1.23	1 ^c
				1.21	~1 ^c
0.98	0.98	~1	55.10		
0.83	0.83	5	66.0		

^aIncomplete data; unit cell parameters: $a_0 = 9.98 \text{ \AA}$; $c_0 = 47.4 \text{ \AA}$.

^bDebye-Scherrer film.

^cBroad.

picked from the monohydrate dolomitic lime mix. The crystal is in the monoclinic system, with strongest powder diffraction peaks at 10, 4.5, 3.73, 3.20, 2.54, 2.38, and 1.51 Å. The diffraction patterns and β -angle are very similar to synthetic 1M muscovite (14), except for much weaker basal spacing peaks. Refractive indexes and analysis for potassium may indicate whether this is a micaceous contaminant or a true reaction product. Single crystal and powder data are given in Table 5.

TABLE 5

X-RAY DIFFRACTION DATA FOR A MONOCLINIC 10-Å PRODUCT ISOLATED FROM A MONTMORILLONITE-Ca(OH)₂-MgO-WATER MIXTURE

Single Crystal ^a				Powder ^b of Crushed Crystal (d-spacing)	
d-Spacing			Index (monoclinic) (hk.l)	Powder ^b of Crushed Crystal (d-spacing)	
Observed	Calc.	Intensity		Observed	Intensity
10.03	10.03	2	002	<u>10.06</u>	10 ^c
5.01	5.02	1	004		
4.53	4.53	2	020		
4.53	4.52	3	$\bar{1}11$	<u>4.50</u>	9
4.47	4.48	6	110		
				4.30	
4.11	4.13	~1	022	4.13	
3.88	3.87		112	3.89	
				3.73	6
3.49	3.48		113	3.52	
				3.45	
				3.38	
3.35	3.36		024		
3.34	3.34	9	006		
				3.29	
				3.20	7
				3.10	
				2.98	
				2.90	
				2.84	
				2.79	
				2.62	
2.60	2.60	6	130		
2.57	2.58	7	200		
				<u>2.54</u>	10
2.50	2.51	1	008		
				2.45	8
				2.38	
2.26	2.26	1	040		
2.25	2.24		220, $\bar{2}20$	2.25	
2.21	2.21	~1	040	2.21	
2.19	2.19	~1	221	2.21	
2.16	2.14	2	204	2.14	
2.05	2.06	~1	044		
				2.00	
1.89	1.89	~1	206		
				1.71	
1.67	1.66	~1	208	1.66	
				1.64	
				1.61	
				1.56	
				1.53	
1.51	1.51	10	060, 330	1.51	8
1.14	1.13	~1	080		

^aIncomplete data; unit cell parameters: $a_0 = 5.235 \text{ \AA}$; $b_0 = 9.054 \text{ \AA}$; $c_0 = 20.386 \text{ \AA}$; $\beta = 100.20^\circ$.

^bDebye-Scherrer film.

^cSpotty.

12.6-Å Product. — A very broad 12.6-Å peak occurs only in kaolinite-dolomitic lime mixes. Broadness of the peak indicates that the material is very finely or poorly crystalline, preventing use of the single crystal technique. Identification and composition of this compound remain highly speculative.

7.6-Å Product. — The 7.6-Å product previously isolated from Ca(OH)_2 -bentonite and given in Table 1 is absent from bentonite mixes with dolomitic lime, but occurs in kaolinite reacted with any of the three limes. A similarity of basal plane dimensions to those of the aluminates has been noted (5), and d-spacings somewhat correspond to C_3AH_8 -12 (20) as well as $\text{C}_3\text{AH}_{11} \cdot \text{CaCO}_3$ (Table 1). The former composition has been discredited and variously assigned to the carbo-aluminate (21) or a solid solution of C_2A and C_4A hydrates (9).

CONCLUSIONS

1. Powder diffraction and single crystal data indicate that room temperature reactions between various minerals, lime, and water yield the crystalline products given in Table 6.

TABLE 6
CRYSTALLINE PRODUCTS

Mineral	Crystalline Product		
	Ca(OH)_2	$\text{Ca(OH)}_2 + \text{MgO}$	$\text{Ca(OH)}_2 + \text{Mg(OH)}_2$
Montmorillonite	10-Å tobermorite $\alpha\text{C}_4\text{AH}_{13}$, $\beta\text{C}_4\text{AH}_{13}$, 7.6-Å aluminate? 3.79 Å 2.54 Å	10-Å tobermorite $\beta\text{C}_4\text{AH}_{13}$, 8.8 Å unknown	10-Å tobermorite
Kaolinite	7.6-Å aluminate? 3.05-Å silicate?	12.6 Å unknown 7.6-Å aluminate? 3.05-Å silicate?	12.6 Å unknown 7.6-Å aluminate? 3.05-Å silicate?
Vermiculite	8.1 Å, 4.59 Å unknown	3.03 Å?	None
Muscovite	7.5 Å unknown	None	None
Quartz	None		

2. X-ray and DTA show unreacted lime remaining in the mixes after two years, and unreacted mineral in all but montmorillonite mixes. DTA of the montmorillonite and muscovite mixes indicates weakening of the octahedral layer structure.

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Measuring Thermal Expansion of Lime-Fly Ash-Aggregate Compositions Using SR-4 Strain Gages

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This report presents the results of a laboratory investigation on use of SR-4 strain gages for evaluation of the thermal expansion of lime-fly ash-aggregate compositions. The study was initially concerned with the selection of the most suitable gages, their application, waterproofing, and the interpretation of the results. Beams (12 in. long, 3- × 3-in. section) were prepared with the same aggregate but with different percentages of lime and/or fly ash. Other beams were prepared with the same percentages of lime and fly ash but using aggregates with different amounts of fines. These beams were subjected to a temperature change of approximately 200 F (from -25 F to 175 F). Six different types of strain gages were applied to the beams and the expansion caused by the temperature change was measured. A steel beam of known thermal properties was used for a thermal expansion reference in an active gage-dummy gage system of temperature compensation.

The influence of dry density, percent of lime and/or fly ash, and percent of fines in the aggregate parameters on the coefficient of thermal expansion is also discussed. It has been found for example that the coefficient of expansion increases with an increase in the percent of lime, other variables remaining constant.

•THE USE of cement and other hydraulic binders, such as a combination of lime and fly ash, has been increasingly developed in the field of highway construction. The problems involved are numerous and have been the subject of a great deal of laboratory and field research work in the United States and elsewhere. The problem of thermal expansion of stabilized subgrades is, indeed, a complex one.

In many jobs involving stabilized subgrades of a semi-rigid nature, various cracks and fractures have been observed which frequently cannot be attributed to normal degradation. In these stabilized subgrade layers, no joints are in use and a significant thermal expansion could have very well induced sufficient stresses to cause cracking, swelling and/or dislocation.

In a preliminary investigation the methods of measuring linear expansion—such as, mechanical calipers or gages, optical means (interference fringes) displacement of a fluid, and SR-4 electrical strain gages—were evaluated. SR-4 strain gages were finally chosen for accuracy, ease of reading, adaptability and consistency of results.

Applying SR-4 strain gages to a fairly homogeneous material such as the lime-fly ash-gravel combination was, in itself, a problem. It was also necessary for the gages to produce accurate and reproducible results at various temperatures ranging from -10 to 170 F.

Some of the various parameters (dry density, percent of lime and/or fly ash, percent of fines in aggregate) influencing the variation of the thermal coefficient of expansion were also investigated.

EVALUATION OF VARIOUS SR-4 STRAIN GAGES

As a preliminary step to the evaluation of the thermal coefficient of expansion it was necessary to determine the type of strain gages to be used. The gages were to be applied to a lime-fly ash-gravel composition similar to concrete in appearance. No large aggregate was to be used in the molding of the samples and the average size of the larger particles at the surface, where the gage was to be bonded, was of the order of 0.5 mm. Due to the only fairly homogeneous quality of the material, the gages used were to be of a sufficient gage length to average out the stress or expansion difference over the many grains of the aggregate. Possible effects of a protruding piece of aggregate, or of small holes on the surface, were minimized by special preparation of the surface consisting of an application of a thin layer of cement.

A successful strain gage application is a combination of many different factors that must be studied, compared, and weighed against each other before a choice can be made. Static strain measurements over a period of alternate loading and unloading conditions at changing temperatures impose the greatest demands on strain gage performance. The selection of the strain gage and associated accessories (bonding cement, waterproofing materials, and lead wires) must be made for each application in view of the limitations of each and the effect on the overall installation performance.

Types of Gages Evaluated

Six different types of gages were used, 3 in each of the two major types: the bonded wire type—paper base or bakelite base—and the etched foil type—epoxy base (Table 1).

Operating Temperature Ranges for Gages and Adhesives

The operational temperature limit of a strain gage depends on the individual components of the gage. The generally accepted temperature limits, for static strain measurements (Table 1) are based on ideal conditions during short-term testing where slight environmental changes do not create inaccuracies beyond the normally accepted limits for experimental stress analysis. Very conservative limits for this investigation were applied because the test periods were long and environmental conditions were constantly changing.

Bonding Adhesives

The nitrocellulose (Duco, SR-4 cement) adhesive, a solvent-release type, is specified for use only with paper-type and wire and foil gages from freezing temperatures to 180 F. The main advantages are low cost, easy storage and use, and rapid curing at room temperature.

A maximum temperature of 170 F was used to test the samples. Despite this, cracking, burning, bond slipping and important changes in the strain readings were noticed over the testing period (2 weeks of alternate heating and cooling), and this type of adhesive was rejected.

The epoxy elevated-temperature curing type (EPY-400) adhesive is designed for use

TABLE 1
DESCRIPTION OF SR-4 GAGES

Gage	Description	Bonding Cement	Max. Operating Temp. (°F)	Resistance (ohms)	Gage Factor	Length (in.)	Width (in.)	Price per Unit (\$)
A ¹	Constantan wire	Duco	180	300	2.10	6	$\frac{1}{32}$	2.00
B ¹	Constantan wire	Duco	180	120	2.10	1	$\frac{1}{32}$	1.60
D ²	Constantan foil	EPY-400	300	180	2.06	$\frac{1}{2}$	$\frac{1}{32}$	3.50
C ²	Constantan foil	EPY-400	300	180	2.05	1	$\frac{1}{4}$	4.20
E ²	Constantan foil	EPY-400	250	750	2.11	6	0.74	9.00
F ³	Constantan wire	EPY-400	250	350	2.08	$\frac{1}{8}$	$\frac{1}{32}$	2.20

¹Paper base. ²Epoxy back. ³Bakelite back.

with foil and wire, bakelite and epoxy type gages at more than 400 F. The cement has an extremely high resistance per unit volume allowing maximum resistance to ground and heat distortion of all epoxies tested. It requires a bonding pressure of 10 to 15 psi with any one of the following time-temperature cures: 1 hour at 400 F, 2 hours at 350 F, 3 hours at 300 F, or 6 hours at 250 F. The 6 hours at 250 F cycle was used. The low temperature cure was employed to protect the gage from high desensitization. The length of the curing time was not a serious problem in this study; however, disadvantages of this adhesive are the short life of the cement after it has been mixed (24 hours unless stored at -30 F) and excessive cost (approximately \$24.00 for 3 packs of 20 g each).

As an average, one 20-g pack provided 100 sq in. of bonded area (first layer of epoxy plus bonding of gage), representing roughly the preparation of 10 samples, carrying 3 gages each (gages C, D and E).

This adhesive performed satisfactorily; no cracking or noticeable slipping was noted. Some gages were retested 6 months after their initial installation and gave readings similar to the initial readings (correct to the nearest 10 to 20 μ in./in.). The EPY-400 was then used to bond all gages of a similar type throughout the test.

Selection of Gages

The final selection of the most suitable gage was based on the following criteria:

1. Greatest sensitivity to longitudinal strain (gage factor);
2. Minimum sensitivity to lateral strain (transverse sensitivity);
3. Lowest sensitivity to temperature (resistivity change coefficient);
4. Maximum electrical and dimensional stability;
5. Ease of application; and
6. Cost of installation.

Two gage types (D and F) were selected, and were both used on each sample tested. An E gage was used jointly with D and F gages on samples where determinations of the tensile and compressive stress-strain curves were to be made after the thermal expansion coefficient had been determined.

Types A and B Gages.—The two paper back gages, used only up to 160 F, were severely damaged. Cracking of the cement was observed, the stability was poor, and the readings were not consistent. Despite their low nominal and installation costs these gages were abandoned.

Type C Gage.—The C gage gave good results but was of comparatively higher cost.

Type D Gage.—The D gage is one of two gages which appeared to be most acceptable. It provided excellent stability and consistency in the results, as did type F. However, values obtained through this gage were always 0.1 to 0.2 μ in./in./ $^{\circ}$ F higher than those obtained through the use of type F. The explanation for this consistent difference is the comparatively higher transverse sensitivity of type D which results in a greater resistance change. Thermal expansion is the same along any direction, and more specifically, the strains created by temperature change along two perpendicular axes are equal.

Type E Gage.—Type E is the same epoxy-backed, etched foil type as gage D but with a 12 times longer active gage length. It gave excellent results also, but was not used when only thermal expansion evaluation was required because of its 3 times higher total cost of installation compared with gages D and F. The same remark about a consistent difference in result with gage F is still valid.

Because of their long gage length (6 in.), these gages gave excellent results in the establishment of a stress-strain curve for the determination of a modulus of elasticity. Gages D and F, on the other hand, gave poor results in this last evaluation for two reasons: (a) they cover a small area where the stress is not representative, and (b) the failure zone which is the location where it is desired to average the strain was not covered by these short length gages.

Type F Gage.—Of the 6 gages that were evaluated this bakelite backed, constantan wire gage, 1 in. long, appears to be the most suitable. Its stability and consistency were excellent. Epoxy provided a strong carrier for the wire, which is thus less sen-

sitive and less affected by a protruding grain of sand or irregularities in the surface on which the gage is to be bonded. J. Taylor of Lehigh University experimented on concrete with a similar gage (BLH AB-3 gage length $1\frac{3}{16}$ in.) and reported excellent results.

Compensating Materials or Standards

The coefficient of expansion of the standard had to be of the order of magnitude of the one for the compositions studied, which was assumed to be 6μ in./in./ $^{\circ}$ F. A 4340 alloy grade steel with a linear coefficient of expansion between 0 and 200 F of 6.3×10^{-6} per $^{\circ}$ F, within an accuracy of 2 percent was used. The size of the standard was $1 \times 2.5 \times 12$ in. (Fig. 1). To verify the theory of the evaluation of thermal expansion through the uses of SR-4 gages, two other standards were used and checked against the alloy steel.

1. A $\frac{1}{4}$ - \times 3- \times 12-in. aluminum beam with a given coefficient of expansion of 12.72μ in./in./ $^{\circ}$ F showed an expansion of 12.84μ in./in./ $^{\circ}$ F through the use of the SR-4 gages.

2. A $\frac{3}{8}$ - \times 3- \times 12-in. medium grade carbon steel of unknown thermal expansion coefficient was tested. A coefficient of expansion of 6.42μ in./in./ $^{\circ}$ F was found which compares with a handbook average value of 6.7.

Throughout the remaining evaluation of the thermal expansion of the lime-fly ash-aggregate compositions only the alloy steel compensating beam was used as the reference standard.

THERMAL COEFFICIENT OF EXPANSION OF LIME-FLY ASH-AGGREGATE COMPOSITIONS

In all, 30 beams or cylindrical samples were used for the purpose of evaluating the influence of the dry density, percent of lime and/or fly ash, and percent of fines (material passing through No. 200 sieve) on the thermal expansion coefficient.

Materials for Molding Test Specimens

Aggregate. — The aggregate used in making the beam-type specimens was a uniform sandy gravel with less than 5 percent fines, $\frac{3}{4}$ -in. maximum size particle.

Lime and Fly Ash. — A standard raw Eddystone fly ash and a commercially available hydrated lime were used.

Manufactured Aggregates. — "Manufactured gravels" that were prepared for a previous investigation (1) were used in the study portion concerned with the effect of fines on thermal expansion. The properties and grain-size distributions of these gravels

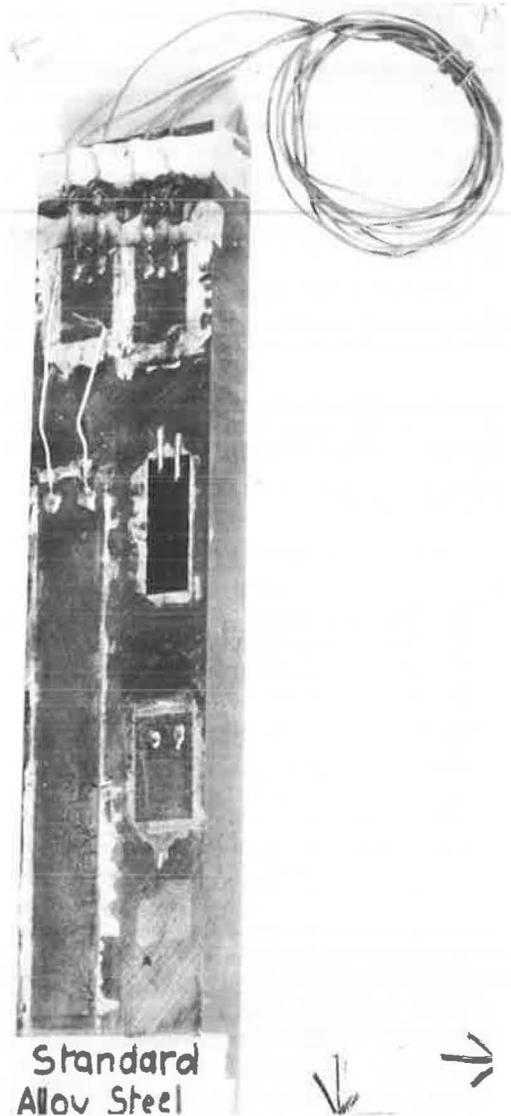


Figure 1.

were previously reported (1); however, Figure 2 is repeated herein to show the grain size curves.

Preparation of Test Specimens

Mixing of Materials.—The materials were mixed both by hand and/or with a mechanical mixer. The air-dried materials (lime, fly ash, gravel) were first blended into a uniform mixture; the water was then incorporated into the mix and evenly dis-

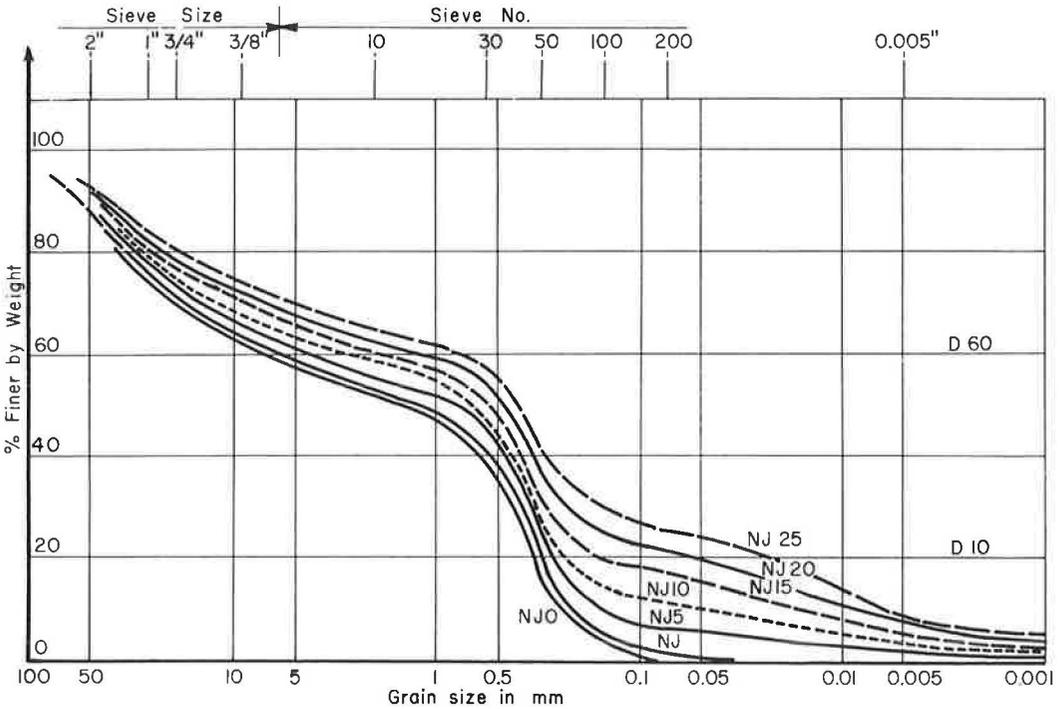


Figure 2. Grain size curves for natural and manufactured gravels.

TABLE 2
DATA FOR 3- BY 3- BY 12-IN. BEAMS

Purpose	Beam	Mix (%)			Accel. Curing (days)	Comp. Strength (psi)	Dry Density	Expansion (μ in./in./ $^{\circ}$ F)	
		Lime	Fly Ash	Gravel				As Tested	Corrected
Effect of dry density change	4, 10, 91, 1	4	10	90	7	920	117	5.45	—
	.2	4	10	90	7	920	122	5.62	—
	.3	4	10	90	7	920	130	5.93	—
	.4	4	10	90	7	920	134.5	6.32	—
Effect of lime and fly ash content	2, 10, 90	2	10	90	7	640	131.5	5.34	5.2
	4, 10, 90	4	10	90	7	920	130.5	5.85	5.82
	6, 10, 90	6	10	90	7	1,050	129.5	6.02	6.06
	2, 15, 85	2	15	85	7	840	127.5	5.10	5.35
	4, 15, 85	4	15	85	7	1,080	129.5	5.93	6
	6, 15, 85	6	15	85	7	1,160	125.5	6.16	6.3
	2, 20, 80	2	20	80	7	—	116.7	5.56	5.9
	4, 20, 80	4	20	80	7	—	112.5	5.56	6.17
	6, 20, 80	6	20	80	7	—	112	5.75	6.42
	8, 20, 80	8	20	80	7	—	112	6.0	6.68
Comparison	Soil cement concrete	7.2% cement			7 ^a	1,400	128.6	6.5	—
		3,000 lb			28 ^a	3,000	140	5.7	—

^aMoist cured.



Figure 3. Sample beams and testing setup.

tributed. The amount of water added to each mixture was that required to produce the maximum dry density using Standard AASHO compaction (5.5-lb rammer).

Composition of Specimens.—A series of 4 beams, 12 in. long and 3- × 3-in. cross-section, was prepared to study the dry density effect. Beams were molded using a 4.10.90 mix (4% lime, 10% fly ash, 90% natural gravel), but with different compactions (Table 2).

Three series of beams, 12 in. long and 3- × 3-in. cross-section, were prepared with 10, 15 and 20 percent fly ash, respectively, to study the effect of percent of lime and/

or fly ash. In each series, beams with 2, 4 and 6 percent lime were molded (a beam with 8% lime was added to the 20.80 series). Natural gravel was used in all mixes (Fig. 3 and Table 2).

Seven series of two specimens each were molded to study the effect of percent of fines. Specimens were cylindrical in shape, 4-in. diameter and 4.6 in. high. A 4.12.88 mixture was used for all specimens, but for each series the mix was prepared using a different gravel (Fig. 4 and Table 3).

Curing of the Specimen.—All beams and cylinders were cured using an accelerated procedure, consisting of 7 days in a sealed container at 130 F. The specimens were then submerged for 24 hours in water. Before applying the gage, the samples were dried for two days in an oven at 250 F. After the gages were cemented on, all beams and cylinders were submitted to four 24-hr cycles of successive heating and cooling

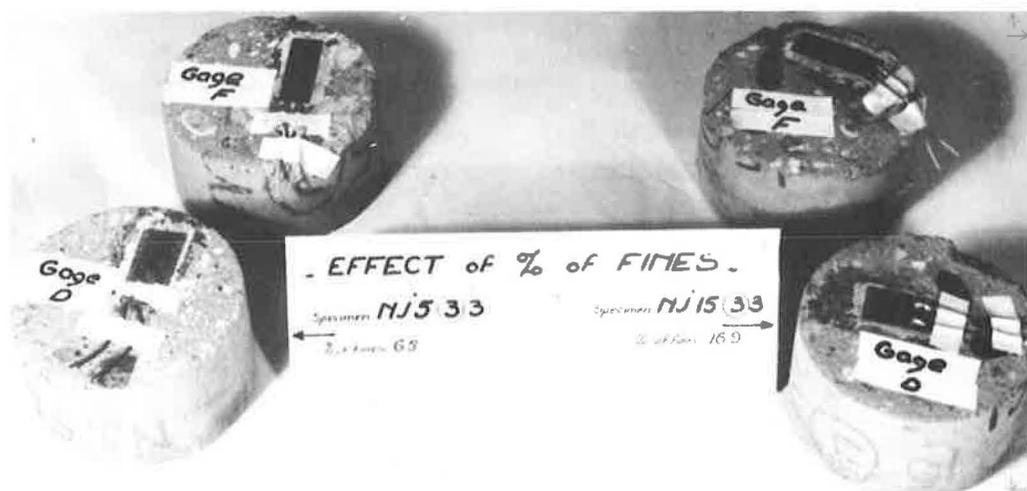


Figure 4. Cylindrical specimens.

TABLE 3
EFFECT OF PERCENTAGE OF FINES

Beam	Fines Passing No. 200 Sieve (%)	Compressive Strength (psi)	Dry Density (pcf)	Expansion (μ in./in./ $^{\circ}$ F)	
				As Tested	Corrected
Nj. 0	0	1,000	125.8	5.95	6.05
				123.5	6.32
Nj	1.4	1,340	129.5	5.85	5.80
			128	6.05	6.01
Nj. 5	6.5	1,100	131	6.06	5.92
			130.3	6.0	5.90
Nj. 10	11.7	1,050	125.1	6.0	6.10
			124	6.12	6.27
Nj. 15	16.9	950	125.8	6.05	6.15
			125.1	6.20	6.30
Nj. 20	22.1	905	126	6.12	6.20
			125.8	6.16	6.26
Nj. 25	27.0	820	122	6.12	6.33
			121	6.22	6.47

between 200 and 0 F. All these curings were necessary to insure stability and consistency in the readings.

Application of Strain Gages to the Specimens

On the beams, gages D and F were bonded side by side. The cylindrical specimens were first sawed cross-sectionally. A fairly homogeneous area, without any particles over 3 mm, was selected on the exposed face of the cylinder. One of the gages was bonded to this area, and the other gage was bonded on its exact counterpart on the other half of the cylinder. Both were bonded with their strain sensitive axes lying in the same general direction. In addition, gage E was bonded on certain rectangular beams wherever necessary for further study.

Preparation of the Bonding Area.—The selected area of the specimen was cleaned with cotton soaked in carbon tetrachloride, until the cotton remained clean. Cleaning was continued using toluol and finally acetone. On the cleaned area, which was slightly larger than the gage used, a $\frac{1}{64}$ -in. layer of epoxy was applied with a clean spatula and cured at 300 F for 4 hr. When the specimen had cooled, the epoxy layer was thinned down by using acetone applied with cloth pads. The thinning was continued until the specimen surface reappeared clean and smooth and without protuberances.

Bonding of the Gage.—The following procedure was used to apply the gages to the specimens:

1. Cleaning with acetone.
2. Application of a liberal coat of epoxy.
3. Application of the gage (squeezing out excess epoxy, and all trapped bubbles of air).
4. Taping on the top of the gage a teflon sheet and then a rubber pad slightly larger than the gage.
5. Curing for 6 hours at 250 F under a 10- to 15-psi pressure.
6. After cooling, checking resistance to ground and the gage.
7. Soldering of the lead wires.
8. Waterproofing.

The specimens were tested in a series of 4 or 5 at a time. All common lead wires were one foot long and soldered together. A common lead wire was then connected from the juncture to the strainometer. All other lead wires were 3.5 ft long.

Testing Procedure

A 215 F range of temperatures (-15 F to 200 F) was used. The specimen and compensating standard were first placed in an oven. The temperature was raised slowly to 200 F, maintained for 24 hours, and then readings of the strain were made. For the next 24 hours, the specimen was cooled to room temperature. This 48-hr cycle was then repeated 3 to 4 times; readings at the maximum temperature were taken in each cycle. At the end of this 3- or 4-cycle period, zero readings at room temperature were also taken. Zero readings at room temperature were made only for reference, and to verify roughly the linearity of the coefficient of expansion over the temperature range.

The specimens were then placed in a freezer where the preceding cycles were repeated identically but from room temperature to -15 F. Zero readings at room temperature were also taken at the end of these cycles.

The difference in expansion between the standard and the specimen over the 215 F range was then computed using the average of the readings. The coefficient of expansion was then computed. Table 4 gives data for specimen NJ 20.5.1.

Results

For all the series of specimens except those used to study the effect of dry density, the beams and cylindrical samples were compacted identically to eliminate dry density as a variable. Furthermore, to correlate all the values a standard dry density was

TABLE 4
TYPICAL DATA FOR STRAIN GAGE MEASUREMENTS

Gage Type	Cycle No.	Test Reading			Room Temperature Readings		Results			
		T° C	Readings	Average	T° C	Reading	Δ T° C	Δ Reading	Δ Expansion	Expansion
F	1	+94	-2,605	-2,575	+33	-2,545	120	-45	-0.38	10.92
	2	+94	-2,560							
	3	+97	-2,570							
K = 2.08	1	-26	-2,530	-2,530	+33	-2,550	120	-45	-0.38	10.92
	2	-26	-2,525							
D	1	+94	-2,460	-2,470	+33	-2,455	120	-50	-0.43	10.87
	2	+94	-2,460							
	3	+97	-2,470							
K = 2.06	1	-26	-2,420	-2,420	+33	-2,450	120	-50	-0.43	10.87
	2	-26	-2,420							

Note: Compensating material: alloy steel standard, 11.3×10^{-6} ; sample NJ 20-5-1 (March 21, 1962). Readings are in μ in./in./°C.

assumed. On this basis and using the dry density curve, a correction was then applied to each coefficient of expansion obtained through testing. The resulting value was called the corrected value.

When studying the effect of percent of lime and/or fly ash, the standard dry density assumed was 130 pcf. When studying the effect of percent of fines the value taken was 128 pcf. This was assuming that all mixes tested for dry density effect would give a family of curves parallel to the curve in Figure 5. Using this curve, the thermal expansion for a 128-pcf dry density is 5.80×10^{-6} in./in./° F, and for a 126-pcf dry density is 5.72×10^{-6} in./in./° F, a difference of 0.08. If a sample with a 126-pcf dry density is tested and a value for the thermal expansion of 6.12×10^{-6} in./in./° F is obtained, the corrected value is $6.12 + 0.08 = 6.20 \times 10^{-6}$ in./in./° F. This was the case for sample NJ 20.5.2.

The results of the investigation are as follows:

1. Dry Density Curve (Fig. 5).—The thermal expansion increases with an increase in dry density. The 5th point on the curve was provided by the 4-10-90 beam of the 10-90 series.
2. Thermal Expansion vs Percent of Fines (Fig. 6 and Table 3).—It would appear that for a certain optimum value of the percent of fines present in the mix (value around 5%) that the thermal expansion is a minimum. On both sides of this value the thermal expansion increases. When the percent of fines increases, the curve is flattening, indicating a possible maximum value.
3. Thermal Expansion vs Percent of Lime (Fig. 7).—Three curves were drawn showing again an increase in the expansion with an increase in the percent of lime, all other variables remaining constant.
4. Thermal Expansion vs Percent of Fly Ash (Fig. 8).—Three curves were obtained corresponding, respectively, to 2, 4, and 6 percent lime. There is an increase of the expansion with an increase of the percent fly ash, all other variables remaining constant. Apparently, the 3 curves would, if extended, converge toward the same value.
5. For the purpose of comparison, a soil-cement beam stabilized with 7.2 percent cement and a 3,000-lb concrete beam were molded and tested (Table 2 and Fig. 5).

This study was a part of a larger investigation of the thermal expansion of the lime-fly ash-gravel composition and its parameters. Its primary objective was to determine the value of the SR-4 strain gage in such an investigation. Parameters such as the physical properties of the gravel particles and gravel aggregate, water content, and effect of subfreezing temperatures on a moistened sample, were not considered. It was concluded that in such a study SR-4 gages would be of great value. The epoxy insured complete waterproofing of the gage.

This investigation showed that variation in the mix itself and in the compaction do affect the thermal expansion of lime-fly ash-gravel compositions. Although these results should be verified by additional data, the present study indicated the following:

1. The thermal effect increased substantially with an increase in dry density, in

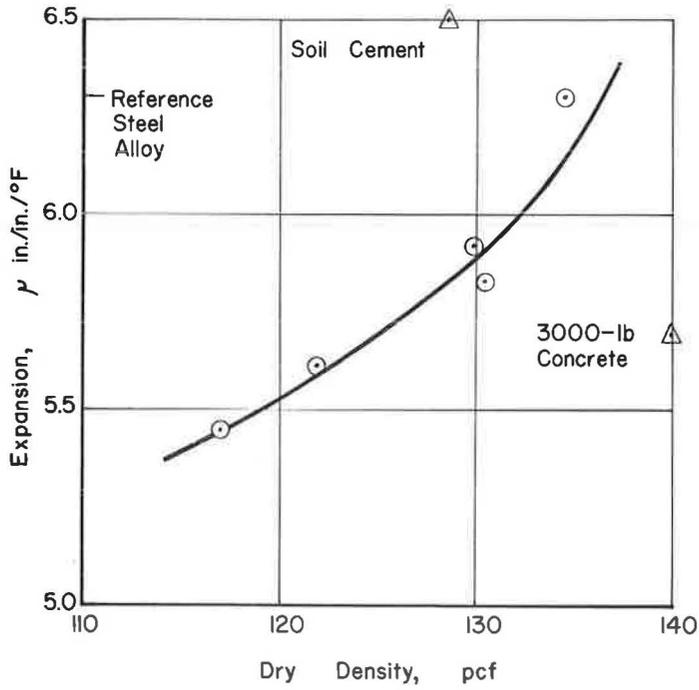


Figure 5. Thermal expansion vs dry density for a 4.10.90 mix.

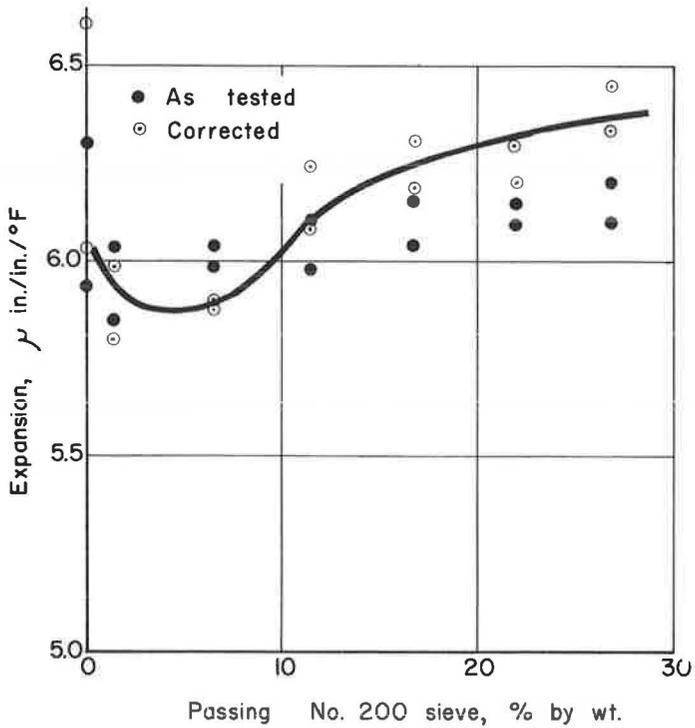


Figure 6. Thermal expansion vs percent of fines for a 4.12.88 mix.

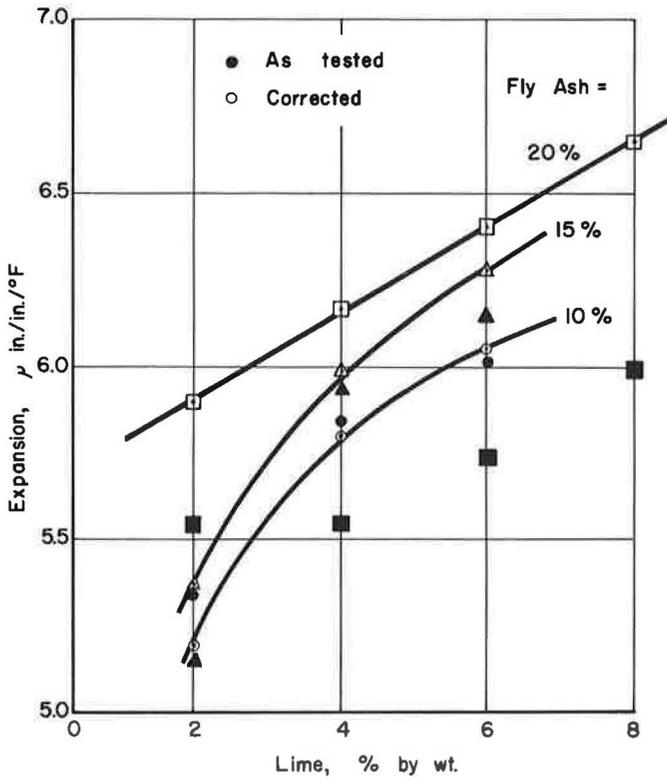


Figure 7. Thermal expansion vs percent of lime, percent fly ash constant.

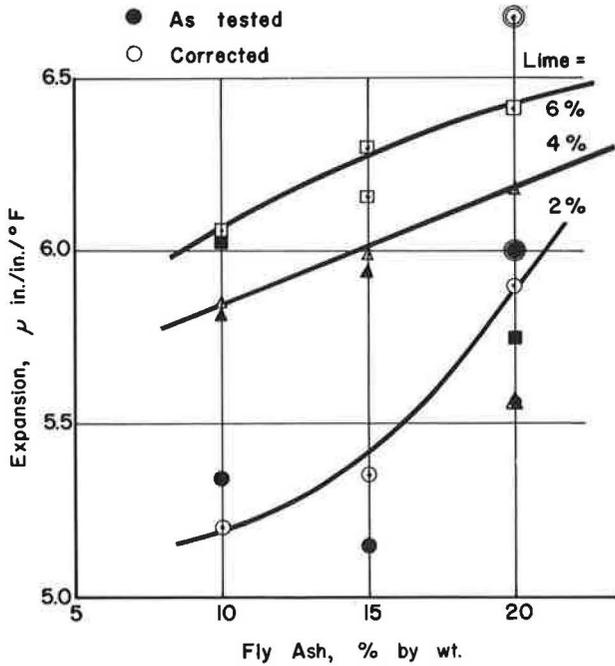


Figure 8. Thermal expansion vs percent of fly ash, percent lime constant.

percent of lime and/or fly ash—the other two variables remaining constant.

2. Lime-fly ash-gravel compositions expanded at a rate comparable to that of steel (6×10^{-6} per °F).

3. There is an optimum value of the percent of fines in the gravel for which the coefficient of expansion will be minimum.

4. After proper selection, careful installation and use, and intelligent interpretation of the data obtained, strain gages can be used successfully for a laboratory investigation of strain in lime-fly ash-gravel compositions and similar aggregates.

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