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SOIL STABILIZATION WITH LIME FLY ASH

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by D. T. Davidson and Associates

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> Iowa State University Ames, Iowa

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Soil Stabilization with Lime Fly

by

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and

Associates

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PREFACE

The sixth in a series, this bulletin further compiles the reports on completed research done for the Iowa State Highway Research Board under its Project HR-1, The loess and glacial till materials of Iowa; an investigation of their physical and chemical properties and techniques for processing them to increase their all-weather stability for road construction. The research, started in 1950, has been conducted by the Iowa Engineering Experiment Station at Iowa State University under its Project 283-S.

The other bulletins in the series, all by Dr. D. T. Davidson and associates, are the following:

Bulletin 20. Geologic and engineering properties of Pleistocene materials in Iowa.

Bulletin 21. Methods for testing engineering soils.

Bulletin 22. Soil stabilization with chemicals.

Bulletin 23. Soil stabilization with cement.

Bulletin 25. Soil stabilization with lime.

Bulletin 26. Soil stabilization with lime fly ash.

As in the other publications, most of the nine papers in this bulletin were prepared originally as graduate theses. Each was rewritten with the assistance of other project workers and Dr. D. T. Davidson, the project leader. The revised paper then was submitted as a report to the Iowa Highway Research Board.

Most of the papers herein have been published previously. The title heading for each identifies all authors and gives the place and date of first publication. None has been revised nor updated, nor has the original data been changed; hence the reader may find some contradictions. However, the facts and conclusions presented are those of the authors at the time the paper was presented. Much repetition of material has been eliminated, and the papers have been arranged by subject matter.

In the REFERENCES list at the end of each manuscript, those indicated as theses in the Iowa State University Library are so shown because only there are all the data given.

J.H.B.

EVALUATION OF LIME AND FLY ASH STABILIZATION OF SOILS BY COMPRESSIVE STRENGTH TESTS

by

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(Progress Report, 1955. A slightly condensed version in Highway Research Board Bull. 108:102-112, 1955.)

INTRODUCTION

The enormous increase in traffic on highways has highlighted the importance of the base course and subbase in modern highway construction. Stable, durable road bases are vital to good roads. The construction of such bases presents few problems in localities having satisfactory supplies of such base materials as gravel and crushed stone. In localities not so favored there is a great need for means of converting natural soils and soil materials economically into satisfactory base material. Methods of doing this are commonly termed *soil stabilization*.

The use of admixtures for soil stabilization has been a subject of great interest to highway and airfield engineers in recent years. Various organic and inorganic materials have been investigated for possible use as stabilizing agents in the construction of subbase, base, or surface courses. Lime and fly ash mixtures are among those that have shown promise^{11, 28, 42, 43,} ^{44, 50}

Lime and fly ash stabilized soils are used mainly in the construction of base and subbase courses for city streets, highways, and airfields^{47, 58}. The lime and fly ash stabilized base course materially reduces the thickness of wearing surface needed. Lime and fly ash also can be used as admixtures to stabilize subgrade materials.

The success of lime and fly ash stabilization depends on the addition of the proper proportions of lime and fly ash, the addition of the proper amount of water, and compacting to a proper uniform density, all of which produce the necessary strength and stability for the mixture. Laboratory tests are commonly used in the design and evaluation of lime, fly ash, and soil mixtures.

Laboratory studies were made of lime fly ash stabilization of a variety of soils sampled in different parts of the United States (table I). The chief purpose of the studies was to develop a test method for the preliminary evaluation of lime fly ash stabilized soils. Another was to make a

				TABLE I.	THE SULL SA	MPLES.		,	
	Soil	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8
	Source	Texas	Virginia	Iowa	Kentucky	Kansas	New Jersey	Iowa	Iowa
N	Geological origin	Coastal plain deposit, largely deltaic (Beaumont clay)	Residual soil on diorite	Friable loess from near Missouri River floodplain	Natural levee deposit from Ohio River	Recent Eolian sand	Alluvial gravel over marine sand (Bridgeton formation)	Glacio- fluvial gravel (Kansan outwash)	Glacial till (Cary till)
	Soil series	Lake Charles	Davidson	Hamburg	Melvin*	Pratt	Not	Not	Webster
	Horizon	C	В	С	С	С	Relevant	Relevant	A
	Engineering classification (AASHO)	A-7-6(20)	A-7-5 (18)	A-4(8)	A-6(8)	A-2-4(0)	A-2-4(0)	A-1-b(0)	A-7-5(15)

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"There is some question as to whether the soil should be classified in Melvin or Lindside series. The sample is probably from the C horizon.

REVIEW OF PREVIOUS WORK

Development of lime, fly ash, and soil stabilization

Fly ash is a very fine, grey, dust-like ash which results from burning powdered coal⁴⁶. The fly ash is largely made up of tiny spheres of silica and alumina glass. As the finely pulverized coal is burned, particles of fly ash are suspended in the gas stream that reaches the boiler. As the hot gases pass off into the atmosphere, the fly ash is collected on the plates of electrostatic precipitators located within the heating system.

Of the nearly 100 million tons of coal consumed annually in the United States, approximately 70 percent is burned as powdered coal. About 10 percent of the powdered coal burned is non-combustible, and of this, about 95 percent is fly ash. This amounts to about 7 million tons of fly ash produced annually, and most of it is collected. The annual production predicted by 1963 will be 17 million tons⁵⁶. Obviously, the disposal of collected fly ash is a major problem. Groups of utilities and government agencies are doing a great deal of work to promote the utilization of fly ash.

Fly ash is a pozzolanic material^{10, 19, 27}, similar to the volcanic ash used in early Roman construction^{11, 26, 56}. The use of fly ash in concrete has been investigated since the early 1930's. Laboratory tests and construction experience have proved that fly ash of acceptable chemical and physical composition can be used as a partial replacement, up to 50 percent, for Portland cement to make a better and less costly concrete. Fly ash in concrete gives higher ultimate strengths, inhibits alkali aggregate reactions and efflorescence, lowers the heat of hydration and improves the workability^{4, 9, 18, 20, 21, 41, 49, 53, 54}. Because of its effect on heat of hydration, fly ash has been used extensively in concrete dam construction^{6, 8}.

In road construction, fly ash has long been used as an ingredient in Portland cement concrete and bituminous concrete road pavements^{12, 13, 37}. Fly ash has also been used for choking voids between the lumps of slag or crushed stone in a bituminous road base. It was found that the fly ash used in the choking operation ran into the voids without the hand work that is necessary to brush in fine slag by conventional practice, and that the surface of sheet asphalt containing fly ash could be handled easier than the standard mix. Several demonstration roads which have been built have performed satisfactorily^{5, 7, 8}.

Though hydrated lime has been used successfully as a stabilizing agent for different soils in the past thirty years^{22, 31, 34,38, 48}, the use of combinations of lime and fly ash as admixtures in soil stabilization is fairly recent. A patent was granted in 1951 on the use of lime and fly ash with fine aggregates²⁸; in 1954, another patent covered the stabilization of soils²⁹.

Some laboratory test procedures were developed for use with New Jersey and Maryland soils in designing and evaluating the use of lime and fly ash for stabilizing soils⁴⁵. The optimum quantity of lime and fly ash with coarse grained soils and boiler slags was determined by using a maximum density criterion. With fine grained soils, the optimum mixture was determined by examining the results of unconfined compression tests, sonic beam tests, or group velocity measurements. All the Proctor size specimens were:

(1) Compacted in three layers with 25 blows per layer of a 10 pound hammer dropped through 18 inches,

(2) cured in air tight containers for a period of 7 days at 140°F., and

(3) oven dried at 110° F. at the end of curing for one day before being tested.

Marked changes in engineering properties of the lime and fly ash treated soils were obtained immediately; compressive strength and resistance to successive cycles of freezing and thawing and wetting and drying were considerably improved. Field tests of mix designs studied in the laboratory showed them to be performing satisfactorily.

The use of lime and fly ash for stabilizing a silty soil has been studied as well as the effect of different amounts and combinations of lime and fly ash on the moisture density relation and compressive strength⁵¹. Final evaluation of selected combinations of lime, fly ash, and the soil was made by use of the standard wet-dry and freeze-thaw tests for soil cement (ASTM Designations: D559-44 and D560-44) and the criteria of the Portland Cement Association for soil cement.

Stretches of primary and secondary roads in Maryland, shoulders and interchanges on the New Jersey Turnpike, and airport runways in Pennsylvania, New Jersey, and Missouri were constructed with lime and fly ash during 1952 and 1953. The amounts and proportions of lime and fly ash used on these projects ranged from 4 to 8 percent hydrated lime and 8 to 20 percent fly ash. The lime is expressed as percent by weight of the fly ash plus oven dry soil; the fly ash is expressed as percent by weight of the mixture of fly ash and oven dry soil. Also, some access roads and parking lots in industrial plants and business areas have been successfully stabilized with lime and fly ash^{11, 43, 48, 59}.

Besides improving the stability and other properties of soils, an important incentive to the use of lime and fly ash in soil stabilization is their low cost in many parts of the U. S. The cost of lime and fly ash stabilized bases is said to be not more than one-half that of most bases. Another advantage claimed is that the slow rate of pozzolanic reaction allows construction to be interrupted for extended periods without harm to the final product⁴⁴. An added advantage for airport runways and aprons is the great thermal resistance of lime and fly ash stabilized bases to the hot exhaust gases from jet planes and rocket missiles. Stabilized lime, fly ash, and soil or aggregate combinations have much greater thermal resistance than other types of known paving materials, including Portland cement concrete.

Lime and fly ash stabilized materials are reported to have resisted temperatures as high as 2400°F.⁵².

Mechanism of lime and fly ash stabilization

A pozzolan is a siliceous or aluminous material, which in itself possesses little or no cementitious value but in finely divided form with moisture, it will react chemically with calcium hydroxide (lime) at ordinary temperatures to form compounds possessing cementitious properties⁴. Fly ash is one type of artificial pozzolan. The cementitious material created by the pozzolanic reaction may be regarded as a calcium silicate, but since good pozzolans ordinarily contain small amounts of the alkalies, sodium and potassium, it is likely that some other important complex compounds are also formed^{19, 50}. On the basis of free lime determinations in hydrated Portland cement and fly ash mixtures, some believe that lime acts only as a catalyst to hydrate the fly ash³⁷.

The reaction between lime and fly ash is utilized to stabilize soils. Theoretically, the lime, fly ash, and soil mixture should be compacted to make a maximum number of points of contact among the soil particles or aggregations of particles available for cementing. Such a maximum compaction for any given compactive effort can be obtained at an optimum moisture content which may differ from the moisture content required for a complete lime and fly ash reaction.

LABORATORY INVESTIGATIONS

The stability of lime, fly ash, and soil mixtures is affected by many variables. The variables given (figure 1) are considered most impor-



Fig. 1. Factors affecting the stability of lime, fly ash, and soil mixtures.

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-	•	TABLE	II. PROP	ERTIES OF SC	IL SAMPLE	s.			
	Soil	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8
Textural composition,* percent by weight	Gravel Sand Silt Clay Colloidal	0 7.7 48.2 44.1 36.8	$0\\3.4\\12.0\\84.6\\72.9$	$0\\0.778.321.015.8$	$0 \\ 7.7 \\ 55.7 \\ 36.6 \\ 19.4$	$0\\86.4\\4.0\\9.6\\8.6$	$50.8 \\ 36.6 \\ 6.2 \\ 6.4 \\ 5.2$	$15.7 \\ 74.9 \\ 4.2 \\ 5.2 \\ 3.0$	$0\\18.9\\37.1\\44.0\\28.0$
Textural class	ification†	Clay	Clay	Silty clay loam	Silty clay	Sand	Gravel	Gravelly sand	Clay
Physical properties	L.L., % P.L., % P.I., % S.L., % C.M.E., % F.M.E., % Sp. Gr.,‡ 25°C./4°C.	$57 \\ 20 \\ 37 \\ 14.4 \\ 21.2 \\ 21.2 \\ 2.67$	75 51 24 27.3 29.5 47.2 2.91	$32 \\ 25 \\ 7 \\ 25.2 \\ 15.2 \\ 26.4 \\ 2.68$	$33 \\ 22 \\ 11 \\ 22.9 \\ 21.4 \\ 25.5 \\ 2.69$	NP 17.5 5.1 20.9 2.67	$20 \\ 12 \\ 8 \\ 14.0 \\ 6.4 \\ 19.2 \\ 2.69$	NP 21.2 7.8 26.2 2.74	$54 \\ 34 \\ 20 \\ 20.6 \\ 25.7 \\ 46.3 \\ 2.52$
Predominant c	lay minerals§	M//	H#	M//	M// or illite	Illite	Illite	Illite	M//
Chemical properties	Cat. Ex. Cap.** m.e./100 pH‡ Carbonates,** % Organic matter,** %	$25.5 \\ 5.9 \\ 2.7 \\ 0.6$	$11.3 \\ 4.1 \\ 1.0 \\ 0.5$	13.4 7.8 10.2 0.2	$11.4 \\ 4.5 \\ 1.2 \\ 1.0$	$7.3 \\ 5.6 \\ 1.5 \\ 0.4$	$7.8 \\ 6.4 \\ 0.6 \\ 1.2$	$7.3 \\ 6.1 \\ 1.0 \\ 0.2$	$42.0 \\ 7.4 \\ 4.8 \\ 10.2$

*Gravel—above 2.0 mm., sand—2.0 to 0.074 mm., silt—0.074 to 0.005 mm., clay—below 0.005 mm., colloidal—below 0.001 mm. †Textural classification are based on the U. S. Bureau of Public Roads system except that 0.074 mm. was used as the lower limit of the sand fraction. ‡Determined on the fraction passing No. 10 sieve. §Estimated from differential thermal analysis of the fraction passing No. 200 sieve. //Abbreviation for montmorillonite. #Abbreviation for hallyosite. **Determined on the fraction passing No. 40 sieve.

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tant in affecting the stability of a processed soil in which lime and fly ash are the only additives. The use of a third additive for improving the effectiveness of lime fly ash stabilization will introduce still other variables. As many of these variables as possible are to be evaluated in the Iowa Engineering Experiment Station lime fly ash stabilization research of which the work presented herein is a part. The experiments in this report are primarily concerned with the effect of the following variables on the stability of lime, fly ash, and soil mixtures:

1. The amount of lime and fly ash.

- 2. The ratio of lime to fly ash.
- 3, The moisture content during mixing and compaction.
- 4. The length of curing.
- 5. The condition during curing.

Properties of soils and admixtures

Properties of soils used. The main object of the long-range lime, fly ash, and soil stabilization investigation is to evaluate lime and fly ash stabilization with a wide variety of soils from different parts of the United States. Eight soil samples were used in the part of this investigation being reported (tables I, II). According to reports of previous investigations^{44, 51}, soil no. 6 from eastern United States and soil no. 4 from central United States have reacted satisfactorily with lime fly ash admixtures.

Properties of lime and fly ash used. The lime used is a hydrated lime consisting essentially of calcium hydroxide. The fly ash used is representative of the low carbon content varieties available in the central United States (table III). It has been found that the fineness and carbon content of fly ash will affect its effectiveness as a stabilizing agent^s.

	Properties	lime*	ash [†]
Specific gravity	·		2.67
Fineness	Material passing No. 325 sieve, percent Specific surface area, sq. cm. per gm. (Based on the specific gravity of 2.67)	99.0	94.3 3470
Chemical analysis	Silicon dioxide, percent Aluminum oxide, percent Iron and aluminum oxide, percent Magnesium oxide, percent Sulfur trioxide, percent Calcium carbonate, percent Total calcium hydroxide, percent Available calcium hydroxide, percent Loss on ignition	0.80	$ \begin{array}{c} 38.90 \\ 22.92 \\ \hline 0.52 \\ 2.00 \\ 8.36 \\ \hline 2.10 \end{array} $

TABLE III. PROPERTIES OF THE HYDRATED LIME AND FLY ASH.

*The hydrated lime and the test data were furnished by the Linwood Stone Products Co., Inc., Buffalo, Iowa. †The fly ash was obtained from the Paddy's Run Station, Louisville Gas and Electric Co., Louisville, Kentucky. The tests on fly ash were made by the Robert W. Hunt Co., Chicago, Illinois.

Method of testing

There is no standard method for evaluating the stability of lime, fly ash, and soil mixtures. Therefore, a simple method of test is needed to provide data for determining the benefits to the stability of the soils processed and for selecting the more promising combinations of lime, fly ash, and soil for further studies. Other features desired in the test are:

The use of small test specimens molded to near standard Proctor density.
 The use of curing conditions comparable with those obtainable in the field.

3. The testing of specimens after immersion in water to simulate one of the more severe field conditions.

4. The attainment of a fairly high degree of reproductibility of test results. Test procedures for fine grained soils, utilizing the unconfined compres-

sion test and 2 by 2 inch specimens, were developed to meet these requirements. Soils no. 1, no. 2, no. 3, and no. 4 were used in the development of the test method. The preliminary evaluation of lime and fly ash stabilized soils no. 5, no. 6, no. 7, and no. 8 was by the developed method, except for the method of molding and size of specimens of the two coarse grained soils, no. 6 and no. 7.

The experiments made in developing the test method deal with many of the previously mentioned variables affecting the stability of lime, fly ash, and soil mixtures and are discussed in detail in the Appendix.

Preparation of lime, fly ash, and soil mixtures. The fine grained soil samples were air dried and pulverized and screened through a no. 10 sieve. The pulverization was done so as not to reduce the size of the individual soil particles. The entire sample of most of the fine grained soils passed through the no. 10 sieve. The two coarse grained soils were air dried and total soil samples were used.

The soil was dry mixed by hand with a predetermined amount of lime and fly ash. The proper amount of distilled water was then mixed with the three materials to obtain a uniform mixture at optimum moisture content for standard Proctor density. The mixing was done first by hand and then was completed by a mechanical mixer at moderate speed. The determination of the moisture content is discussed in the Appendix.

Two mechanical mixers were used in the experiments. One is a Model C-20 kitchen mixer, and the other is a Model K4-B kitchen aid mixer. Most of the mixing in the experiment was done by the Model C-20 mixer.

A series of studies of the mixing time of lime, fly ash, and soil mixtures was performed. The results showed that the effect of various mixing times (from 3 to 7 minutes) on the compressive strength of the mixture was very small. Arbitrary mixing times of 3 minutes for silty and gravelly soils, 5 minutes for clayey soils, and 7 minutes for sandy soils were selected.

Molding of specimens. In the lime and fly ash stabilization studied with fine grained soils, 2 by 2 inch specimens were molded to near standard Proctor density (figure 2). The lime, fly ash, and soil mixture was then



Fig. 2. Apparatus for molding 2 inch diameter by 2 inch high test specimens. (a) Drop hammer and molding cylinder in place. (b) Drop hammer and molding cylinder shown separately.

placed in the molding cylinder in one layer and compacted by a 5 pound hammer dropping from a height of 12 inches. Experiments with a number of fine grained raw soils and stabilized soils showed that five blows of the hammer on each side of the specimen were needed to obtain a density near standard Proctor density. The double plunger action of the apparatus resulted in comparatively uniform density throughout the specimen.

Immediately after being molded, all specimens were weighed to the nearest 0.1 gram, and their height was measured to nearest 0.001 inch. This information permitted determination of the density of the specimen.

In the studies with coarse grained soils standard Proctor size specimens, 4 inch diameter by 4.6 inch high, were molded. The molding was done according to the procedure of ASTM Designation: D698-42T.

Curing of specimens. After being molded, specimens were cured in a

moist cabinet where a temperature of $70\pm3^{\circ}$ F. and a relative humidity of not less than 90 percent were maintained. The purpose of the moist curing is to allow the lime, fly ash, and soil moisture to develop adequate stability. Seven day and 28 day curing periods, as commonly used in the testing of Portland cement concrete, were used.

Testing of specimens. After the specimens were cured, they were completely immersed in distilled water at about 70° F. for 24 hours, and then were tested for compressive strength by the unconfined compression test. The rate of deformation during the compressive strength test was 0.05 inch of height of specimens per minute. On the basis of experiments conducted in the Iowa Engineering Experiment Station, this rate of deformation appeared to be satisfactory. The maximum load causing failure of the specimen was taken as its compressive strength (figure 3). All specimens were weighed and measured both before and after immersion to determine water absorption and volume change.

Presentation and discussion of test results

Preliminary evaluations of the lime and fly ash with each of the eight soils were made by using the test procedures just described. The lime fly ash content in the mixture is expressed in percent by dry weight of the total mixture. The proportion of lime and fly ash in the mixture is expressed as a ratio by weight of lime to fly ash. Compressive strength values reported are the average of three test specimens.

The test results are presented in two parts. The first part includes the fine grained soils, no. 1, no. 2, no. 3, no. 4, no. 5, and no. 8; the second part deals with the two coarse grained soils, no. 6 and no. 7. All the test data



Fig. 3. Apparatus used for determining compressive strength of 2 inch diameter by 2 inch high specimens.

for the fine grained soils were obtained with 2 by 2 inch specimens, and all data for the coarse grained soils with standard Proctor size specimens.

Experiments with fine grained soils. Lime and fly ash admixtures affect the moisture density relationship of the soil. Usually the maximum dry density was decreased and the optimum moisture content was slightly increased when the lime and fly ash were added (table IV). The decrease in density may not be very important since the stability of lime and fly ash stabilized soils also depends on many other factors (figure 4).

The addition of lime and fly ash to the soils materially improved their stability, as indicated by the compressive strength of 24 hour immersed specimens. All untreated specimens failed during the immersion period. The compressive strengths of stabilized fine grained soil specimens ranged from about 200 pounds for organic soil to over 1100 pounds for sandy soil after 7 day moist curing at near 70° F., and increased to as much as 1660 pounds for sandy soil after 28 day curing (table V). Criteria for judging the adequacy of these strength values is not at present available and must be determined by additional studies.

The data indicate that the texture of silty and clayey soils may not be the controlling factor in their response to lime and fly ash treatments. Test results for soils no. 1, no. 2, and no. 3 show that about the same degree of stabilization can be obtained with silty or clayey soils. Sufficient data is not yet available to explain the comparatively low compressive strength obtained with soil no. 4. The difference in compressive strength of soils no. 3 and no. 4 may be partly due to the difference in carbonate contents. The difference in the amount and type of clay minerals may be

Soil	Lime and fly ash admixtures, percent	Ratio of lime to fly ash	Maximum dry density, lb./per cu. ft.	Optimum moisture content, percent	
No. 1	33 No admixture	1:1	$97.8\\109.5$	$\begin{array}{c} 22.5\\ 18.3 \end{array}$	
No. 2	33 No admixture	1:1	$87.8 \\ 85.6$	$\begin{array}{c} 32.2\\ 37.1 \end{array}$	
No. 3	33 No admixture	1:1	$\begin{array}{c} 97.7 \\ 109.9 \end{array}$	$\begin{array}{c} 20.7 \\ 18.2 \end{array}$	
No. 4	33 No admixture	1:1	$97.3 \\ 108.6$	$\begin{array}{c} 22.6 \\ 17.7 \end{array}$	
No. 5	25 No admixture	1:2	$\begin{array}{c} 114.1 \\ 119.3 \end{array}$	$\begin{array}{c} 12.7\\ 11.8\end{array}$	
No. 8	33 No admixture	1:1	$\begin{array}{c} 86.6\\ 86.9\end{array}$	$\begin{array}{c} 29.0\\ 27.9\end{array}$	

TABLE IV. DATA ILLUSTRATING THE EFFECT OF LIME AND FLY ASH ADMIXTURES ON MOISTURE, DENSITY VALUES OF FINE GRAINED SOILS.*

*Determined with the molding apparatus shown in figure 3. The molding procedure was correlated to give moisture, density values closely approximating those obtainable by the standard Proctor density test (ASTM Designation: D698-42T).



Fig. 4. Comparison of moisture density curves of a fine grained soil and a mixture of lime, fly ash, and the soil.

TABLE V.	COMPRESSIVE STRENGT	AS OF THE	SIX FINE GRAINED	SOILS STABILIZED
	WITH LIME A	ND FLY AS	H ADMIXTURES.*	

a	Lime and fly ash admixtures,	Ratio of lime to	Compress	ive strength, lbs.
Soil	percent	fly ash	7 day	28 day
No. 1	$\begin{array}{c} 15\\ 25\\ 25\end{array}$	1:2 1:2 1:9	$640 \\ 740 \\ 715$	805 1130 1175
No. 2	$\begin{array}{c} 15\\ 25\\ 25\end{array}$	$1:2 \\ 1:2 \\ 1:1.5$	230 525 520	$350 \\ 1005 \\ 1110$
No. 3	15 25 25	$1:2 \\ 1:2 \\ 1:9$	485 640 600	$735 \\1045 \\1130$
No. 4	$\begin{array}{c} 15\\ 25\\ 25\end{array}$	$1:2 \\ 1:2 \\ 1:2.3$	225 350 350	$255 \\ 430 \\ 445$
No. 5	$\begin{array}{c} 15\\ 25\\ 25\end{array}$	$1:2 \\ 1:2.3 \\ 1:4$	$\begin{array}{r} 410 \\ 1075 \\ 1160 \end{array}$	$605 \\ 1540 \\ 1660$
No. 8	20 25 25	1:2 1:2.3 1:19	280 265 370	415 435 460

*Compressive strength is the total load causing failure of the 2 inch diameter by 2 inch high speciman.





the reason for the difference in compressive strength of soils no. 1 and no. 2. More experimental data are needed to determine the effect of these variables on lime and fly ash stabilization. It is believed that the texture of the sandy soil, no. 5, and the high organic matter content in soil no. 8 are possible explanations for the comparatively high and low compressive strengths of these two soil respectively.

Variations in the ratio of lime to fly ash will affect the compressive strength of lime, fly ash, and soil mixtures. The effect may not be great for some soils when the variation is within a certain range (figure 5). The variation in compressive strength for most of the soils is fairly small when the lime and fly ash ratio is within the range of 1:1 to 1:9. The same gen-



Fig. 6. Effect of variations in the amount of lime and fly ash on the compressive strengths of lime and fly ash stabilized fine grained soils.

eral relationship was found for lime and fly ash mixtures without soil.

For a given ratio of lime to fly ash, the compressive strength of the lime, fly ash, and soil mixture increased with an increase in the amount of lime and fly ash used (table V). The mixtures containing 25 percent lime and fly ash have higher compressive strength than those containing 15 percent (figure 6).

Experiments with coarse grained soils. As has been mentioned, the evaluation of the lime and fly ash with the two coarse grained soils was similar to that for the fine grained soils except that standard Proctor size specimens were used instead of 2 by 2 inch specimens. Results obtained with mixtures of the two coarse grained soils and various amounts of lime and fly ash are given (tables VI, VII, VIII).

As with the fine grained soils, the usual effect of lime and fly ash on the moisture-density relation was to cause a decrease in the maximum dry density and an increase of the optimum moisture content.

The better compressive strength results obtained with soil no. 7 stabilized with lime and fly ash are very likely due to its sandy texture (table VII). A more complete explanation cannot be offered at this time due to lack of data. The data shown do indicate the effect on compressive strength of varying the ratio of lime to fly ash in the mixture. If both 7 day and 28 day strength data are considered, the ratio 1:2 gave best results with soil no. 6 and the ratio 1:9 would probably be considered best for soil no. 7.

In a given ratio of lime to fly ash, an increase in the amount of lime and fly ash resulted in a marked increase in 28 day unconfined compressive strength (table VIII). The strength increase trend of the 7 day data is less conclusive.

Sufficient data is not available at this time to compare the amount and proportion of lime and fly ash needed in fine grained soils with that in

TABLE VI. EFFECT OF LIME AND FLY ASH ADMIXTURES ON MOISTURE DENSITY VALUES OF THE TWO GRAINED SOILS.*

Śoil	Lime and fly ash admixtures, percent	Ratio of lime to fly ash	Maximum dry density, lb./per cu. ft.	Optimum moisture content, percent
No. 6	25 25 No admixture	$1:1 \\ 1:2$	$112.9 \\ 116.9 \\ 136.4$	$13.2 \\ 12.1 \\ 7.6$
No. 7	25 25 No admixture	$1:1 \\ 1:2$	111.4 115.3 119.9	$14.3 \\ 13.2 \\ 13.5$

Determined by the standard Proctor density test (ASTM Designation: D698-42T).

TABLE VII. EFFECT OF VARIATIONS IN THE RATIO OF LIME TO FLY ASH ON THE COMPRESSIVE STRENGTHS OF LIME AND FLY ASH STABILIZED COARSE GRAINED SOILS.*

9-11	Lime and fly ash admixtures,	Ratio of lime to fly osh	Compressiv lt	ve strength, os. 28 day
5011	percent	ny asn	Tuay	20 day
No. 6	25 25 25	1:1 1:2 1:9	1270 1240 960	$1965 \\ 2400 \\ 2210$
No. 7	25 25 25	$1:1 \\ 1:2 \\ 1:9$	$1510 \\ 2220 \\ 1800$	$\begin{array}{r} 4080 \\ 4890 \\ 6240 \end{array}$

Compressive strength is the total load causing failure of the standard Proctor size specimen.

TABLE VIII. EFFECT OF DIFFERENT AMOUNTS OF LIME AND FLY ASH ON COMPRESSIVE STRENGTHS OF THE TWO COARSE GRAINED SOILS.*

	Lime and fly ash admixtures.†	Compressi II	ve strength, os.
Soil	percent	7 day	28 day
No. 6	$\begin{array}{c} 15\\ 25\\ 35\end{array}$	$1275 \\ 1240 \\ 1370$	$1740 \\ 2400 \\ 3240$
No. 7	$\begin{array}{c} 15\\ 25\\ 35\end{array}$	$1480 \\ 2220 \\ 1595$	$3275 \\ 4890 \\ 6005$

*Compressive strength is the total load causing failure of the standard Proctor size specimen. †Ratio of lime to fly ash is 1:2 by weight. coarse grained soils. No general statement could be arrived at concerning the higher lime and lower fly ash requirements for fine grained soils⁴⁵.

SUMMARY AND CONCLUSIONS

As part of a research project in the Iowa Engineering Experiment Station for the evaluation of lime and fly ash as an admixture for soil stabilization, preliminary studies were made with eight soils from different parts of the United States. The soils represent a wide variety of textures and properties. The primary objectives of the work reported were:

1. To develop a test method for the preliminary evaluation of lime and fly ash stabilized soils.

2. To make a preliminary evaluation of the merits of lime and fly ash stabilization with the soils sampled.

Laboratory experiments were made to compare different ways of preparing, curing, and testing lime and fly ash stabilized soil specimens. Results of these studies with four silty and clayey soils were found helpful in formulating a test method.

Preliminary evaluations of various lime, fly ash, and soil mixtures were made by means of the test method. Lime and fly ash was found to be a promising admixture for stabilization purposes in most of the soils investigated.

The findings of the investigation are the following:

1. The incorporation of lime and fly ash in the soils materially improves their stability.

2. The maximum dry density usually decreases and the optimum moisture content usually increases when lime and fly ash are added to the soil.

3. The compressive strength of a lime and fly ash stabilized soil varies greatly with the moisture content at which the stabilized soil is compacted. With the soils tested, the highest compressive strength is obtained with a moisture content equal to or slightly lower than the optimum moisture for maximum dry density.

4. It is usually believed that curing lime and fly ash stabilized soils at near 100 percent relative humidity is desirable. On the contrary, experimental results in some cases indicate that the strength of specimens cured at lower humidities is higher than that of specimens cured at higher humidities.

5. The compressive strength of lime and fly ash stabilized soils is greatly affected by the temperature at which the stabilized soils are cured. Experimental data indicate that the practice of curing specimens at 140°F. for 7 days as commonly used in testing lime and fly ash stabilized soils gives a strength which may not be obtainable by conventional curing methods in the field.

6. The strength of lime and fly ash stabilized soils depends on the ratio of

lime to fly ash. Similarly the strength of lime and fly ash mixtures without soil varies with the proportion of these two materials.

7. The compressive strength of lime and fly ash stabilized soils, with a constant ratio of lime to fly ash in the mixture, increases with an increase in the amount of lime and fly ash.

8. The stability of lime and fly ash stabilized soils is improved by aging in the presence of moisture, due to the slow pozzolanic activity between the fly ash and hydrated lime.

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APPENDIX

DEVELOPMENT OF TESTING METHOD FOR PRELIMINARY EVALUATION

In developing the method of testing, a series of experiments were performed to determine the most desirable ways of preparing, curing, and testing lime and fly ash stabilized soil specimens. The properties of the lime, the fly ash, and the four silty and clayey soils used in the experiments are given (tables I, II, and III). Lime and fly ash admixtures are expressed in percent by weight of the mixture of lime, fly ash, and soil; ratios of lime to fly ash are by-weight. Most of the data reported are the average of results of three test specimens.

Preparation of specimens

The moisture content of a lime, fly ash, and soil mixture during compaction, and the method and degree of compaction have an important effect on the stability of the compacted mixture.

Molding of specimens. In the lime fly ash stabilization studies with silty and clayey soils, 2 inch diameter by 2 inch high specimens were molded to near standard Proctor density at optimum moisture content (figure 2). The lime, fly ash, and soil mixture was placed in the molding cylinder in one layer and compacted by a 5 pound hammer dropping from a height of 12 inches. Experiments with a number of fine grained raw soils and stabilized soils showed that five blows of the hammer on each side of the specimen were needed to compact the soil or stabilized mixture to near standard Proctor density (figure 7).





Moisture content of mixture

The moisture density relation and the moisture compressive strength relation were studied with mixtures of lime, fly ash, and each of the four soils. Specimens used for determining compressive strengths were cured at 70 ± 3 °F. in a moist cabinet for seven days and then were tested after 24 hour complete immersion in distilled water. Results of the experiments indicate that the amount of moisture required for maximum dry density for most of the mixtures is more than that required for the highest 7 day compressive strength (table IX). However, the difference between the two moisture requirements, if there is any, is not great. Since the moisture content giving maximum dry density can be determined with less effort and in a much shorter time, it seems desirable to use this moisture content for preparing specimens of lime, fly ash, and soil (figures 8 and 9).

In general, the data on the effect on compressive strength of varying the moisture content of the mixture show that better results were obtained with optimum moisture content than with moisture contents either above or below optimum (figures 10, 11). The optimum moisture contents were the optimum moisture contents for maximum dry densities. All test specimens used for determining compressive strengths were cured at 70 ± 3 °F. for various lengths of time.

	Lime and fly ash	Moisture co For maximum	ntent, percent _ For highest
Soil	admixtures,* percent	dry density	7 day compres- sive strength
No. 1	25 33	$\begin{array}{c} 21.6 \\ 22.5 \end{array}$	20.8 21.5
No. 2	33	32.2	32.2
No. 3	25 33	$\begin{array}{c} 20.3\\ 20.7\end{array}$	$\begin{array}{c} 16.8\\ 18.8\end{array}$
No. 4	33	22.5	19.0

TABLE IX. COMPARISON OF THE MOISTURE CONTENT GIVING MAXIMUM DRY DENSITY WITH THAT GIVING HIGHEST COMPRESSIVE STRENGTH OF LIME, FLY ASH, AND SOIL MIXTURES.

*Ratio of lime to fly ash is 1:1 by weight.





Estimation of optimum moisture content of lime, fly ash, and soil mixture

The optimum moisture content for preparing lime, fly ash, and soil specimens can be estimated, either by computation or by means of a triangular chart, if three moisture density relation tests are run, one on the raw soil, a second on a mixture containing 50 percent of soil and 50 percent of lime, and a third on a mixture containing 50 percent of soil and 50 percent of fly ash.

Calculation method. From the results of the three moisture tests, the variation in the values of optimum moisture and maximum dry density of the raw soil due to the replacement of one percent of soil by one percent of either lime or fly ash can be calculated and used to determine the opti-



Fig. 9. Comparison of the moisture-density relation and the moisture-compressive strength relation of a lime and fly ash stabilized silty soil.



Fig. 10. Effect of different moisture contents on the compressive strength of a lime and fly ash stabilized clayey soil cured for various periods.





mum moisture and maximum density values for any mixture of lime, fly ash, and soil. For example:

	Max. dry density	Opt. moisture
Raw soil No. 4	108.6 lb./cu. ft.	17.7%
50% soil $+$ $50%$ lime	85.5 lb./eu. ft.	29.2%
50% soil $+$ $50%$ fly ash	106.2 lb./cu. ft.	17.9%
Variation in optimum moisture for 1%	= 11.5/50	
= 0.230% increase		
Variation in optimum moisture for 1%	$f_{0} = 0.2/50$	
= 0.004 % increase	-	

Variation in maximum density for 1% lime = 23.1/50

= 0.462 lb./cu. ft. decrease

Variation in maximum density for 1% fly ash = 2.4/50= 0.048 lb./cu. ft. decrease

For a mixture containing 12.5% lime and 12.5% fly ash,

Estimated optimum moisture = $17.7 + 0.230 \times 12.5 + 0.004 \times 12.5 = 20.6\%$

Estimated maximum density = $108.6 - 0.462 \times 12.5 - 0.048 \times 12.5$

= 102.2 lb./cu. ft.

Triangular chart method. The triangular chart for estimation of optimum moisture is an equilateral triangle (figure 12). The percent of soil is shown along the base of the triangle, and the grid lines extend upward and to the right. The percent of lime is shown along the left side of the triangle, and the grid lines extend downward and to the right. The percent of fly ash is shown along the right side of the triangle, and the grid lines



Fig. 12. Triangular chart for estimation of optimum moisture content of mixtures of lime, fly ash, and soil No. 4.

extend horizontally across the chart. The equal moisture lines are then plotted on the chart on the basis of the optimum moisture of the three experimental mixtures at the three apexes of the triangle. From this chart the optimum moisture for any mixture of lime, fly ash, and soil can be easily determined. A similar chart can be made for maximum dry densities. This graphical method is especially useful for research work when many different combinations of lime, fly ash, and soil will be used.

The difference between the estimated and the experimental optimum moisture content is very small (table X).

Curing of specimens

The stability of lime, fly ash, and soil mixtures depends on the curing time and on such curing conditions as the temperature and relative humidity during curing. On the basis of the following curing experiments, it seems desirable to cure specimens in a moist cabinet with a temperature of 70 ± 3 °F. and a relative humidity of not less than 90 percent for periods of 7 and 28 days. In these experiments, the compressive strength was

	Lime and fly ash admixtures,*	Optin moisture perce	num content, ent	Maximum dry density, lb. per cu. ft.				
Soil	percent	Experimental	Estimated	Experimental	Estimated			
No. 1	25 33	$\begin{array}{c} 21.6 \\ 22.5 \end{array}$	$\begin{array}{c} 20.8\\ 21.5\end{array}$	$\begin{array}{c} 100.5\\97.8\end{array}$	$102.9 \\ 100.7$			
No. 2	33	32.2	32.9	87.8	87.9			
No. 3	25 33	$\begin{array}{c} 20.5\\ 20.7\end{array}$	$\begin{array}{c} 20.1 \\ 20.6 \end{array}$	97.5 97.7	$\begin{array}{c} 101.1\\98.9\end{array}$			
No. 4	33	22.5	21.6	97.4	100.1			

TABLE X. COMPARISON OF ESTIMATED AND EXPERIMENTAL OPTIMUM MOISTURE CONTENTS AND MAXIMUM DRY DENSITIES FOR LIME, FLY ASH, AND SOIL MIXTURES.

*Ratio of lime to fly ash is 1:1 by weight.

determined by using 24 hour immersed specimens as discussed under Testing of Specimens.

Temperature during curing. The data for 7 day curing of specimens indicate that the use of elevated temperature $(140\pm2^{\circ}F. \text{ and } 110\pm2^{\circ}F.)$ results in a compressive strength much higher than obtained by curing at $70\pm3^{\circ}F.$ (table XI). The compressive strengths of many of the 7 day specimens cured at near $140^{\circ}F.$ are even much higher than the compressive strengths of 180 day specimens cured at near $70^{\circ}F.$ It is concluded that curing at near $140^{\circ}F.$ as done by others^{24, 45, 51} gives results which may not be attainable in the field by conventional methods of curing (figures 13, 14). The rapid increase in compressive strength of elevated temperature specimens early in the curing period is especially noteworthy.

Relative humidity during curing. In many of the mixtures tested, the highest compressive strength was obtained with a relative humidity lower than 100 percent (table XII). Since no uniform trend of variation in compressive strength is indicated by the test data, it is not possible to select a relative humidity which will result in the highest compressive strength for all mixtures. Until further studies can be made, it seems advisable to cure specimens at a relative humidity of not lower than 90 percent.

Effect of carbon dioxide in the air. Carbon dioxide in the air during curing affected the compressive strength of mixtures of lime, fly ash, and each of the four soils (table XIII). Figure 15 shows for two of the soils a comparison of compressive strengths of lime, fly ash, and soil specimens having and not having direct contact with carbon dioxide in the air. In this experiment, test specimens were cured at 70 ± 3 °F. and a relative humidity of not less than 90 percent. The test data indicate that the effect of carbon dioxide in the air on the compressive strength of specimens during curing is not great. Therefore, it appears unnecessary to control the amount of carbon dioxide present during the curing period.



Fig. 13. Relationship between compressive strength and length of curing at various temperatures of specimens of a lime and fly ash stabilized clayey soil.



Fig. 14. Relationship between compressive strength and length of curing at various temperatures of specimens of a lime and fly ash stabilized silty soil.

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				Com	pressive sti	strength, lbs.		
Soil	Lime and fly ash admixtures, percent	Ratio of lime to fly ash	Cured at 140±2°F.* for 7 days	Cured at 110±2°F.* for 7 days	7 day	Cured at 28 day	70±3°F.† 60 day	180 day
No. 1	25	1:2	3600	1850	740	1130	1580	2530
No. 2	$15 \\ 25 \\ 25$	$1:2 \\ 1:2 \\ 1:4$	430 1590 765	$315 \\ 1200 \\ 590$	$230 \\ 525 \\ 310$	$350 \\ 1005 \\ 575$	$320 \\ 1235 \\ 725$	ND‡ ND‡ ND‡
No. 3	25	$1\!:\!2$	7040	ND‡	640	1045	1550	3180
No. 4	$\begin{array}{c} 15\\ 25\\ 25\end{array}$	1:2 1:2 1:4	$1575 \\ 3125 \\ 2330$	265 550 335	$225 \\ 350 \\ 300$	$255 \\ 430 \\ 380$	$310 \\ 505 \\ 425$	$420 \\ 610 \\ 575$
Lime and mixture	d fly ash without soil	1:2	4660	4220	630	3240	4680	ND‡

TABLE XI. DATA ILLUSTRATING THE EFFECT OF VARIATIONS IN TEMPERATURE DURING CURING ON THE COMPRESSIVE STRENGTH OF LIME, FLY ASH, AND SOIL MIXTURES.

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*Specimens were kept in air-tight containers to prevent evaporation during curing. †Specimens were cured in a moist cabinet capable of maintaining a relative humidity of not less than 90 percent. ‡Not determined.

TABLE XII. DATA ILLUSTRATING THE EFFECT OF VARIATIONS IN RELATIVE HUMIDITY DURING CURING ON THE COMPRESSIVE STRENGTH OF LIME, FLY ASH, AND SOIL MIXTURES.

	fly ash admixtures.*	Compressive strength, lbs. 7 day curing at $70\pm3^{\circ}$ F. 28 day curing at $70\pm3^{\circ}$ F.						
Soil	percent	32% R.H.†	65% R.H.	100% R.H.	32% R.H.	65% R.H.	100% R.H.	
No. 1	25	530	480	540	765	770	770	
No. 2	25	360	405	380	940	870	860	
No. 3	25	810	735	590	1170	1120	980	
No. 4	25	370	355	330	575	425	380	
Lime and without se	fly ash mixture oil (ratio 1:2)	880	1110	740	880	2475	2885	

*Ratio of lime to fly ash is 1:2 by weight. †Relative humidity.

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various factors affecting the rate of increase in compressive strength, the most important are the kind of soil stabilized and an Lengthof curing. The compressive strength of lime, fly ash, and soil tion of lime and fly ash used. Curves illustrating the influence of these variables on the rate of increase in compressive strength are given (figures 16, 17). The data shown were obtained by using specimens cured for varying periods at 70 ± 3 °F. and a relative humidity of not less than 90 percent. Noteworthy is the rapid increase in compressive strength of the lime and fly ash mixture without soil during the 60 day curing period studied. These data and the data of related experiments suggest that the relationship between the compressive strength of a lime, fly ash, and soil mixture, and the amount of lime and fly ash used, is as follows: If the ratio of lime to fly ash is maintained constant, the greater the amount of lime and fly ash, and soil mixture.

It is apparent from all the test data that a fairly long curing period is desirable in evaluating the stability of lime, fly ash, and soil mixtures. It is probable that a curing period longer than one month may not be practical, especially when results are urgently needed in planning seasonal construction projects. A solution is to use the 7 day and 28 day curing periods commonly used for testing soil cement and Portland cement concrete.

Testing of specimens

The compressive strength of lime, fly ash, and soil mixtures is materially









a	Lime and fly ash admixtures,*	Compressive strength, lbs. 7 day 28 day 60 day					
Soll	percent	Wrapped†	Unwrapped†	Wrapped	Unwrapped	Wrapped	Unwrapped
No. 1	25	- 700	740	1030	1130	1500	1580
No. 2	25	500	525	1020	1005	1400	1235
No. 3	25	615	640	1010	1045	1700	1550
No. 4	25	305	. 350	390	430	405	505
Lime and t without so	fly ash mixture il (ratio 1:2)	605	630	3755	3240	5350	4680

TABLE XIII. DATA ILLUSTRATING THE EFFECT OF CARBON DIOXIDE IN THE AIR. ON THE COMPRESSIVE STRENGTH OF LIME, FLY ASH, AND SOIL MIXTURES.

*Ratio of lime to fly ash is 1:2. †Unwrapped or wrapped specimens represent the condition that the mixture has or has not direct contact with carbon dioxide in the air.

TABLE XIV. DATA ILLUSTRATING THE EFFECT OF IMMERSION OF SPECIMENS BEFORE TESTING ON THE COMPRESSIVE STRENGTH OF LIME, FLY ASH, AND SOIL MIXTURES.

	Lime and fly ash	Ratio of	7 d	lay	Compressive 28	strength, lbs. dav	60 dav	
Soil	admixtures, percent	lime to fly ash	24 hour immersion	Without immersion	24 hour immersion	Without immersion	24 hour immersion	Without immersion
No. 2	$\begin{array}{c} 15\\ 25\\ 25\end{array}$	1:2 1:2 1:4	$230 \\ 525 \\ 310$	1010 1560 1200	$350 \\ 1005 \\ 575$	$\frac{1560}{2670}\\2230$	$320 \\ 1235 \\ 725$	$1675 \\ 2785 \\ 2300$
No. 4	25	1:2	350	615	430	1180	505	_
Lime and f without soi	ly as mixture l (ratio 1:2)	1:2	630	845	3240	4530	4680	5725



Fig. 17. Curves comparing the rate of increase in compressive strength of lime, fly ash, and soil mixtures containing varying proportions of lime and fly ash.

affected by the treatment of the specimens before testing. A comparison of the compressive strengths of specimens tested with and without 24 hour immersion in distilled water after curing is shown (table XIV). Curves illustrating the influence of this factor on the rate of increase in compressive strength were plotted (figure 18). The data shown were obtained by using specimens cured at 70 ± 3 °F. and a relative humidity of not less than 90 percent. The compressive strengths of 7 day cured specimens from most of the mixtures were tested without immersion after curing. The results are even higher than that of the 60 day cured specimens tested after 24 hour complete immersion. It is believed that the immersion treatment of specimens before testing more closely simulates conditions in the field and for this reason, it was used in the evaluation experiments. The compressive strength after immersion was determined by the unconfined compression test.

As previously shown, elevated temperature has a beneficial effect on the compressive strength of lime, fly ash, and soil mixtures, and the practice of oven drying specimens to constant weight at 110° F. at the end of curing period, as recommended by some investigators^{43, 45, 51}, may give test results which are not attainable in the field.

The water absorption and volume change of a specimen during immersion can be determined by weighing and measuring the height and diam-


Fg. 18. Curves illustrating the effect of treatment of specimens before testing on the compressive strength of lime, fly ash, and soil mixtures.

eter of the specimen before and after immersion. Test data obtained in the experiments indicate that the compressive strength values are usually sufficient for preliminary evaluation purposes. The compressive strength of a specimen may be expressed in terms of the total load causing failure of the specimen during the unconfined compressive strength test, or it may be expressed as a unit stress (in lb. per sq. in.) computed from total load causing failure. The first alternative is used in this report.

STABILIZATION OF FINE AND COARSE GRAINED SOILS WITH LIME AND FLY ASH ADMIXTURES

by

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ABSTRACT

As part of a research project for the evaluation of lime fly ash as an admixture for soil stabilization, laboratory studies were made of eight fine and coarse grained soils from Iowa, Kentucky, New Jersey, Texas, and Virginia. The objectives of this research were to study the effect of several important variables on the unconfined compressive strength of lime fly ash stabilized soils; to study the effect of lime fly ash admixtures on the standard Proctor moisture density relationship, the consistency limits, and the pH of soils; and to evaluate the resistance of lime fly ash stabilized soils to freezing and thawing and wetting and drying.

Results of this investigation indicate that:

1. Maximum density decreases and optimum moisture content increases with the addition of lime and fly ash, and maximum strength is obtained at a moisture content equal to or slightly less than the optimum;

2. Strength increases with the thoroughness of mixing;

3. Strength increases with an increase in the curing time;

4. Elevated temperature curing greatly increases strength;

5. No consistent relationship was found between strength and relative humidity during curing;

6. Specimens sealed from contact with the air gave greater strength after six months or more curing than did unsealed specimens;

7. A combination of moist curing and immersed curing gave increased strength for silty and clayey stabilized soils;

8. The optimum ratio of lime to fly ash and the amount of lime fly ash additive needed varies with the properties of the soils used;

9. Small percentages of calcium chloride additive gave substantial increases in the strength of stabilized clayey and sandy soils;

10. Addition of lime fly ash improved the consistency limits and shrinkage properties of soils;

11. If compacted to near standard Proctor density and moist cured at near 100 percent relative humidity and 70°F. for 7 and 28 days, lime fly ash stabilized soils do not meet present durability criteria for soil-cement; however, further work needs to be done on the development of a more realistic test for resistance to the effects of freezing and thawing and of wetting and drying;

12. By compacting lime fly ash stabilized soil to a density greater than standard Proctor density and by curing at temperatures near 140°F., the resistance to freezing and thawing and to wetting and drying can be greatly improved.

INTRODUCTION

The principle involved in lime fly ash stabilization, that is, the cementation of soil particles or aggregations with cementing compounds resulting from the chemical or colloidal reaction of lime and reactive siliceous or aluminous materials in the presence of moisture, dates back at least to the Roman Empire. The Romans used a cement comprised essentially of lime and a volcanic ash called *pozzolan*. Today a pozzolan is usually defined as any siliceous material, either natural or artificial, processed or unprocessed, which in the presence of lime and water, at ordinary temperatures, will develop cementitious properties. Fly ash is an artificial pozzolan, and its use in combination with lime and soil stabilization is fairly recent^{3, 4, 6, 7, 8, 11}.

Being a waste product of the power industry, fly ash is not a quality controlled material. Lime is only partially quality controlled; its properties vary significantly from one source to another. Consequently, a large number of variables affect the stabilization of soils with lime and fly ash. These variables have been discussed in a previous report¹, and a study of them is the primary purpose of the Iowa Engineering Experiment Station investigation of lime fly ash stabilization. This paper presents laboratory findings of the investigation to date with respect to the following:

1. The effect of lime fly ash admixtures on the moisture-density relationship of soils;

2. The effect of many variables on the unconfined compressive strength of lime fly ash stabilized soils;

3. The effect of lime fly ash admixtures on the consistency limits and pH of stabilized soils;

4. An evaluation of the resistance of lime fly ash stabilized soils to freezing and thawing, and wetting and drying.

MATERIALS USED IN STABILIZATION STUDIES

The findings discussed in this paper are based on experiments with various soils and lime and fly ash.

Soils

Eight samples of both fine and coarse grained soils were used (tables I, II). According to reports of previous investigators, soils no. 4 and no. 6 have reacted satisfactorily with lime fly ash admixtures^{6, 9}.

Lime and fly ash

Properties of the different types of lime and fly ash used are given (table III). Hydrated lime no. L1 and fly ash no. FA2 were used in most of the experiments.

METHODS OF TEST

Preparation of mixtures

Fine grained soils were air dried, and the portion passing the no. 10 sieve was used; the whole soil was finer in most than this sieve size. Coarse grained soils were air dried, and the whole soil was used. A representative

			Тле	LE I. BRIEF D	ESCRIPTION OF	SOILS USED.			
	Soil	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8
	Source	Texas	Virginia	Iowa	Kentucky	Kansas	New Jersey	Iowa	Iowa
ວາ	Geological origin	Coastal plain deposit largely deltaic (Beaumont clay)	Residual soil on diorite	Friable loess from near Missouri River floodplain	Natural levee deposit from Ohio River	Recent eolian sand	Alluvial gravel over marine sand (Bridgeton formation)	Glacio- fluvial gravel (Kansan outwash)	Glacial till (Cary till)
	Soil series	Lake Charles	Davidson	Hamburg	Melvin*	Pratt	Not relevant	Not relevant	Webster
	Horizon	С.	В	С	С	С		,	Α
	Engineering classification (AASHO)	A-7-6(20)	A-7-5(18)	A-4(8)	A-6(8)	Á-2-4(0)	A-2-4(0)	A-1-b(0)	A-7-5(15)

*There is some question as to whether the soil should be classified in the Melvin or Lindside series. The sample is probably from the C horizon.

sample of the soil to be tested was first dry mixed with a predetermined amount of lime and fly ash. Then the desired amount of distilled water

			TABL	E II. PRO	PERTIES OF	SOILS USED.				
		Soil	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8
	Textural composition*, percent by weight	Gravel Sand Silt Clay Colloidal	$0\\7.7\\48.2\\44.1\\36.8$	$0\\3.4\\12.0\\84.6\\72.9$	0 0.7 78.3 21.0 15.8	$0 \\ 7.7 \\ 55.7 \\ 36.6 \\ 19.4$	$0\\86.4\\4.0\\9.6\\8.6$	$50.8 \\ 36.6 \\ 6.2 \\ 6.4 \\ 5.2$	$15.7 \\ 74.9 \\ 4.2 \\ 5.2 \\ 3.0$	$0\\18.9\\37.1\\44.0\\28.0$
36	Textural classi	fication†	Clay	Clay	Silty clay loam	Silty clay	Sand	Gravel	Gravelly sand	Clay
36	Physical properties	L.L. P.L. P.I. S.L. C.M.E. F.M.E. Sp. G.‡ 25°C./4°C.	$57 \\ 20 \\ 37 \\ 14.4 \\ 21.2 \\ 21.2 \\ 2.67$	$75 \\ 51 \\ 24 \\ 27.3 \\ 29.5 \\ 47.2 \\ 2.91$	$32 \\ 25 \\ 7 \\ 25.2 \\ 15.2 \\ 26.4 \\ 2.68$	$33 \\ 22 \\ 11 \\ 22.9 \\ 21.4 \\ 25.5 \\ 2.69$	NP 17.5 5.1 20.9 2.67	$20 \\ 12 \\ 8 \\ 14.0 \\ 6.4 \\ 19.2 \\ 2.69$	NP 21.2 7.8 26.2 2.74	$54\\34\\20\\20.6\\25.7\\46.3\\2.52$
	Predominant clay mineral§		M//	H#	M//	M// or illite	Illite	Illite	Illite	M//
	Chemical properties	Cat. Ex. Cap.** m.e./100 pH‡ Carbonates**, % Organic matter**, %	$25.5 \\ 5.9 \\ 2.7 \\ 0.6$	$11.3 \\ 4.1 \\ 1.0 \\ 0.5$	$13.4 \\ 7.8 \\ 10.2 \\ 0.2$	$11.4 \\ 4.5 \\ 1.2 \\ 1.0$	$7.3 \\ 5.6 \\ 1.5 \\ 0.4$	$7.8 \\ 6.4 \\ 0.6 \\ 1.2$	$7.3 \\ 6.1 \\ 1.0 \\ 0.2$	42.0 7.4 4.8 10.2

*Gravel—above 2.0 mm., sand—2.0 to 0.074 mm., silt—0.074 to 0.005 mm., clay—below 0.005 mm., colloidal—below 0.001 mm. †Textural classification is based on the U. S. Bureau of Public Roads system except that 0.074 mm. was used as the lower limit of the sand fraction. ‡Determined on the fraction passing the No. 10 sieve. \$Determined by differential thermal analysis on the fraction passing the No. 200 sieve. //Abbreviation for montmorillonite. #Abbreviation for halloysite. **Determined on the fraction passing the No. 40 sieve.

was added, and the mixing was continued to completion. The mixing was done in a mechanical mixer at a constant moderate speed, supplemented

		Fly	rash	Hydrated lime			Quicklime
		FAI	FA4	1/1	112	10	QЦL
Specific gravity		2.56	2.67	2.29^{*}			3.09*
Fineness	Material passing no. 325	93.10	94.3	99.00	98.75	87.0†	100.00‡
	sq. cm. per gm.	3479§	3470//				
Chemical analysis	Silicon dioxide, % Aluminum oxide, %	$\begin{array}{c} 43.40\\ 20.10\end{array}$	$38.90 \\ 22.92$	0.80	$0.25 \\ 0.23$	1.4	1.97
	Iron and aluminum oxide, %			0.82	0.03#	0.6	1.17
	Magnesium oxide, % Sulfur trioxide, %	$\begin{array}{c} 0.43\\ 3.04\end{array}$	$\begin{array}{c} 0.52 \\ 2.00 \end{array}$	0.49	$\begin{array}{c} 0.17\\ 0.01 \end{array}$	33.8	00.84
	Calcium carbonate, %		8.36	0.77			7.94
	CO2, % Total calcium hydroxide, % Available calcium hydroxide, 9			97.82	0.84	0.8	
		ł		97.38	94.85		
	Calcium oxide, %	7.30			74.53^{**}	47.8	7.94 96.02 92.52
,	Available calcium oxide, $\%$				71.79		92.52
	Free water, %	0.30	$0.17 \\ 2.10$	24 56	98 1544	0.5	1 15
	Mechanical moisture, %	0.20	2.10	21.00	0.69	15.0	4.40
*Determined by A.S.T.M †Passing the No. 200 size ‡Passing the No. 50 siev §Based on a specific gra //Based on a specific gra #Ferric oxide. **Total calcium figured a ††Does not include carbox	I. Désignation: C188-44. eve. ve. vity of 2.56 A.S.T.M. Designation: C204- vity of 2.67. us calcium oxide. n dioxide and mechanical moisture.	-46T.					

TABLE III. PROPERTIES OF LIME AND FLY ASH USED.

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weight of the total mixture. The proportion of lime and fly ash is expressed as a ratio by weight of lime to fly ash.

Moisture density relationship

With fine grained soils, the moisture density relationship of both raw soils and lime, fly ash, and soil mixtures was determined with the molding apparatus used for preparing 2 by 2 inch specimens¹. The moist soil or mixture is placed in the molding cylinder in one layer and compacted by a 5 pound hammer dropping from a height of 12 inches. Experiments with a number of fine grained soils showed that five blows of the drop hammer on each side of the specimen results in a density close to standard Proctor density (figure 1). The standard Proctor test (A.S.T.M. Designation: D698-42T) was used for determining moisture density relations of the coarse grained soils. When the optimum moisture contents of mixtures of several different proportions of lime and fly ash with a soil are to be determined, either of the estimation methods given in the Appendix to the preceeding paper can be used to advantage.

Unconfined compressive strength

In the unconfined compressive strength studies with fine grained soils, the previously mentioned 2 by 2 inch specimens were used. With coarse grained soils, standard Proctor size specimens, 4 in. diameter by about 4.6 in. high, were used. Unless otherwise specified, specimens were compacted to near standard Proctor density.



Fig. 1. Moisture-density curves of soil No. 4 obtained by the standard Proctor method and by the method of giving various number of blows to each side of a 2 inch by 2 inch specimen. Five blows give results closest to those obtained by the standard Proctor method.

After being molded, specimens were usually cured unwrapped in a moist cabinet capable of maintaining a temperature of 70 ± 3 °F. and a relative humidity of not less than 90 percent. The length of curing time was varied but was commonly 7 and 28 days. In some of the experiments, the temperature, relative humidity, and other conditions affecting curing were varied.

Cured specimens were completely immersed in distilled water at about 70°F. for 24 hours before being tested for unconfined compressive strength; so unless mentioned otherwise, the strength values reported represent 24 hour immersed unconfined compressive strength. The rate of deformation during the test was 0.05 per inch of height of specimen per minute. Trial experiments indicated this rate to be satisfactory for both raw soils and lime fly ash stabilized soils. The maximum load causing failure is reported as the compressive strength. Because of this, the size of the specimen tested is important in any comparison of test data. Compressive strength values reported are the average of at least two specimens and usually of three specimens.

Durability

Resistance of lime fly ash stabilized soils to freezing and thawing and to wetting and drying was evaluated by the standard test methods for compacted soil cement (A.S.T.M. Designations: D560-44 and D559-44) and by the Portland Cement Association short-cut procedure⁵. The standard size specimens were used for both fine and coarse grained stabilized soils.

FINDINGS OF THE INVESTIGATION

The effect of admixtures of lime and fly ash on certain properties and the stability of fine and coarse grained soils, and some of the many variables involved are discussed.

Moisture density relationship

As shown by the illustrative data (table IV), lime and fly ash admixtures in soils change the standard Proctor moisture density relationship for maximum dry density. With the percentages of lime and fly ash used in the experiments, the change was usually not great; the maximum dry density was slightly decreased and the optimum moisture content was increased, sometimes by as much as 5 percent of the dry mixture weight. The New Jersey gravel, soil no. 6, was most affected; its maximum dry density was greatly reduced by the larger additions of lime and fly ash. Results obtained with the Virginia clay, soil no. 2, also tended to deviate from the usual trend.

Unconfined compressive strength

The unconfined compressive strength of compacted and cured specimens after 24 hours complete immersion in water was used as a criterion of relative stability in studies of some of the variables affecting lime fly ash stabilization. The immersed strength values have not yet been correlated

Soil	Lime fly ash admixture, percent	Ratio of lime to fly ash	Maximum dry density, lbs. per cu. ft.	Optimum moisture content, percent
No. 1	33 No admixture	1:1	97.8 109.5	$\begin{array}{c} 22.5 \\ 18.3 \end{array}$
No. 2	33 No admixture	1:1	$\begin{array}{c} 87.8\\ 85.6\end{array}$	$\begin{array}{c} 32.2\\ 37.1 \end{array}$
No. 3	33 No admixture	1:1	$\begin{array}{c} 97.7 \\ 109.9 \end{array}$	$\begin{array}{c} 20.7 \\ 18.2 \end{array}$
No. 4	33 No admixture	1:1	$\begin{array}{c} 97.3 \\ 108.6 \end{array}$	$\begin{array}{c} 22.6 \\ 17.7 \end{array}$
No. 5	25 No admixture	1:2	$114.1 \\ 119.3$	$\begin{array}{c} 12.7\\11.8\end{array}$
No. 8	33 No admixture	1:1	$\begin{array}{c} 86.6\\ 86.9\end{array}$	$\begin{array}{c} 29.0\\ 27.9 \end{array}$

TABLE IV. DATA ILLUSTRATING THE EFFECT OF LIME FLY ASH ADMIXTURES ON MOISTURE DENSITY VALUES OF FINE GRAINED SOILS.*

*Determined with the molding apparatus used for preparing 2 in. diameter by 2 in. high specimens. The molding procedure was correlated to give moisture density values closely approximating those obtainable by the standard Proctor density test (ASTM Designation: D698-42T).

with the field performance of lime fly ash stabilized soils, and therefore should be regarded only as indicators of stability, assuming, of course, that high compressive strength is a desirable characteristic.

Moisture content for compaction. The unconfined compressive strength of lime fly ash stabilized soils is affected considerably by the moisture content of the mixture at the time of compaction, using standard Proctor compactive effort. With the mixtures studied, maximum compressive strength was obtained with a moisture content equal to or slightly less than optimum moisture for maximum dry density. Test results indicate that it is better to compact on the dry side of optimum moisture content than on the wet side (figures 2, 3).

Time of mixing. Increasing the time of mixing in a mechanical mixer at constant speed gave increased unconfined compressive strength. It seems logical that an intimate mix of the water and solids is necessary for maximum utilization of the cementing properties of lime and fly ash. Since the lime and fly ash reaction is slow, prolonged mixing is not objectionable. The rate of increase in strength was nearly constant as the mixing time was increased (figure 4).

Curing: time. Because of the slow rate of reaction between lime and fly ash, increased time of curing improves the strength of lime fly ash stabilized soils under normal curing conditions. Test specimens over a year old are still gaining in strength (figure 5).

Curing: temperature. Elevated temperatures during curing greatly increase the compressive strength of lime fly ash stabilized soils and give strengths that may not attainable under field curing conditions. Lime fly

ash stabilized roads should be constructed during the hot season of the year (figure 6). Evidence also indicates that long periods of elevated temperature curing may be harmful and may reduce the strength of stabilized soils. In laboratory testing for design purposes it is recommended that realistic curing temperatures be used.



Fig. 2. Comparison of the moisture-density relation and the moisture-compressive strength relation of a lime, fly ash, and clayey soil mixture. The 2 inch diameter by 2 inch high specimens for unconfined compressive strength determinations were cured at near 100 percent relative humidity and 70°F.



Fig. 3. Effect of compaction at different moisture contents on the compressive strength of a lime fly ash stabilized silty soil. Two inch by 2 inch specimens used were cured at near 100 percent relative humidity and 70°F.

Curing: relative humidity. It has generally been assumed that moist curing of compacted lime fly ash stabilized soils at near 100 percent relative humidity is desirable. To investigate this, specimens of stabilized soils were cured in atmospheres of from 0 to 100 percent relative humidity at near 70°F. No consistent relationship was found between unconfined compressive strength and relative humidity during curing (figure 7). Maintaining 100 percent relative humidity may not be too important, and higher strengths might be obtained with curing at some lower relative humidity. However, until more is known about the effect of relative humidity, it seems advisable to follow the moist curing procedures commonly used for Portland cement concrete and soil cement; that is, keep the relative humidity as near 100 percent as possible.

Curing: wrapped vs. unwrapped. Specimens of stabilized soils sealed from air were cured in a moist cabinet (near 100 percent relative humidity and $70 \,^{\circ}$ F.). The specimens were wrapped in Saran-wrap and aluminum foil and sealed with cellophane tape and paraffin wax. The wrapped specimens were found to maintain a slightly higher moisture content than duplicate unwrapped specimens. There was little difference between the strength of wrapped and unwrapped specimens after two months of curing, but after six months ane one year of curing, the unconfined compressive strength of the wrapped specimens was greater than that of the unwrapped specimens













and/or to the exclusion of carbon dioxide, which reacts with the lime to form calcium carbonate. In the early stages of curing, calcium carbonate may form an important cementing compound and may contribute to the strength of stabilized soil. However, calcium carbonate being relatively insoluble in water reduces the amount of calcium available for reacting with fly ash and, as the experimental data indicate, this deficiency may cause a lower ultimate strength.

Curing: complete immersion. Curing by a combination of moist curing followed by complete immersion in water was found to be beneficial to the

Soil*	Curing condition†	7 day	Compre 28 day	essive streng 2 mo.	rth, lbs. 6 mo.	1 yr.
Texas clay	wrapped unwrapped	$\begin{array}{c} 700 \\ 735 \end{array}$	$1030 \\ 1125$	$\begin{array}{c} 1500 \\ 1575 \end{array}$	$\begin{array}{c} 3135\\ 2530 \end{array}$	
Virginia clay	wrapped unwrapped	$\begin{array}{c} 500\\ 440 \end{array}$	$\begin{array}{c} 1020 \\ 1005 \end{array}$	$\begin{array}{c} 1400 \\ 1435 \end{array}$		
Iowa silt	wrapped unwrapped	615 640	$\begin{array}{c} 1010 \\ 1045 \end{array}$	$\begin{array}{c} 1700 \\ 1550 \end{array}$	6390 3180	$\begin{array}{c} 8010\\ 3245\end{array}$
Kentucky silt	wrapped unwrapped	$\begin{array}{c} 305\\ 340 \end{array}$	390 430	$\begin{array}{c} 405\\ 505 \end{array}$	$\begin{array}{c} 640 \\ 610 \end{array}$	960 795

TABLE V. DATA ILLUSTRATING THE EFFECT OF WRAPPED AND UNWRAPPED CURING ON COMPRESSIVE STRENGTH OF FOUR FINE GRAINED SOILS.

*Lime fly ash admixture constituted 25 percent of the total mix in a ratio of 1:2. †The 2 in. by 2 in. specimens used were cured in a moist cabinet at near 100 percent relative humidity and 70°F.







Fig. 8. Comparison of moist curing and combined moist and immersed curing of a lime and fly ash stabilized silty soil. The 2 inch by 2 inch specimens used were moist cured at near 100 percent rela-tive humidity and 70°F. The moist cured only specimens were immersed in distilled water at near 70°F. for 24 hours before testing.

strength of lime fly ash stabilized silty and clayey textured soils, giving in some cases up to 50 percent increase in compressive strength over that obtainable by moist curing at near 100 percent relative humidity and 70°F. for the same length of curing time. The strength gain was greatest for specimens that were moist cured the longest before immersed curing began (figures 8, 9). Experiments to date with coarser textured soils indicate that complete immersion is not beneficial. For example, specimens of stabilized Kansas sand, soil no. 5, had lower strengths after immersed curing.

Ratio of lime to fly ash. There does not seem to be a well defined optimum ratio of lime to fly ash. In the experiments, maximum compressive strength was obtained with a given proportion of lime; and then as the proportion of lime was increased the strength was not greatly changed, although some reduction in strength usually occurred. The ratio of lime to fly ash giving highest strength varied with the properties of the soil; for lime and fly ash mixtures without soil, this ratio was about 1:2; with the soils the ratio varied from about 1:9 to 1:1.5 (figure 10, table VI). Undoubtedly the properties of the lime and the fly ash will also affect the ratio, and this is being investigated. On the basis of what is now known, it would seem that a ratio of 1:2 might be close to the optimum ratio; and if testing facilities are not available, it might be the best choice. However, laboratory testing is recommended to determine the best and the most economical ratio of lime to fly ash.

2500 Soil no.1 75% Lime 10% 2000 Fly ash 15% 7day moist cure + immersion 1500 day moist cure + immersion Immersed day moist cure + immersion compressive strength, lbs. 1000 Moist cure only 500 ٥ċ 5 10 15 20 30 25 35 40 Length of curing, days

Amount of lime fly ash. Unconfined compressive strength will increase with an increase in the amount of lime fly ash in the mixture for a satis-



factory ratio of lime to fly ash. However, because some soils provide active material which enters into the lime and fly ash reaction, the rate of increase is variable, depending on the properties of the soil involved (figure



Ratio by weight of lime to fly ash

Fig. 10. Effect of variations in the ratio of lime to fly ash on the 28 day compressive strength of lime and fly ash stabilized soils. The 2 inch by 2 inch specimens used were cured at near 100 percent relative humidity and 70°F.

TABLE VI. EFFECT OF VARIATIONS IN THE RATIO OF LIME TO FLY ASH ON THE COMPRESSIVE STRENGTH OF LIME FLY ASH STABILIZED COARSE GRAINED SOILS.

	Lime fly ash admixture,	Ratio of lime to	Compressi [.] lb	ve strength, s.*
Soil	percent	fly ash	7 day	$28 \mathrm{day}$
No. 6	25 25 25	$1:1 \\ 1:2 \\ 1:9$	$1270 \\ 1240 \\ 960$	$1965 \\ 2400 \\ 2210$
No. 7	25 25 25	$1:1 \\ 1:2 \\ 1:9$	$1510 \\ 2220 \\ 1800$	$4080 \\ 4890 \\ 6240$

*Proctor size specimens used were cured at near 100 percent relative humidity and 70°F.

11, table VII). The exceptional strength gain of the Kansas sand, soil no. 5, and the slow strength gain of Iowa topsoil, soil no. 8, illustrate the importance of the soil variable in lime fly ash stabilization. Actually, the strength of some lime, fly ash, and soil mixtures were greater than that of lime and fly ash without soil.

In general, the amount of lime and fly ash used should be the smallest amount required for a satisfactory degree of stability; at least 25 percent will be required with many soils. A comparison in table VIII shows the effect of 25 percent lime fly ash, in the optimum ratio for each soil, on the

	Lime fly ash admixture,*	Compressive lb	ve strength, s. †
Soil	percent	$7 \mathrm{day}$	28 day
No. 6	15 25 35	$1275 \\ 1240 \\ 1370$	$1740 \\ 2400 \\ 3240$
No. 7	$\begin{array}{c} 15\\ 25\\ 35\end{array}$	$1480 \\ 2220 \\ 1595$	$3275 \\ 4890 \\ 6005$

TABLE VII. EFFECT OF DIFFERENT AMOUNTS OF LIME FLY ASH ON THE COMPRESSIVE STRENGTH OF THE TWO COARSE GRAINED SOILS.

*Ratio of lime to fly ash is 1:2 by weight. \dagger Proctor size specimens used were cured at near 100 percent relative humidity and 70°F.



Fig. 11. Effect of variations in the amount of lime and fly ash on the 28 day compressive strength of lime and fly ash stabilized fine grained soils. The 2 inch by 2 inch specimens used were cured at near 100 percent relative humidity and 70°F.

7 and 28 day compressive strength of the eight soils used in this investigation. The beneficial effect of the longer curing period is especially noticeable.

Calcium chloride additive. Data illustrating the beneficial effect of calcium chloride admixtures on the 7 and 28 day unconfined compressive strength of five lime fly ash stabilized soils are given in table IX. The strengths of all of the stabilized soils were increased, but some considerably more than others, and the optimum amount of calcium chloride varied for the different soils. The clayey soils, no. 1 and no. 2, were benefited most, the increase in strength being over 100 percent. The sandy soil, no. 5, was also considerably benefited. The strength of the silty soils, no. 3

TABLE VIII. COMPRESSIVE STRENGTHS OF THE EIGHT SOILS EVALUATED, STABILIZED WITH THE OPTIMUM RATIO OF LIME TO FLY ASH.

	Lime fly ash	Ratio of lime to	Compressiv	ve strength,
Soil	percent	fly ash	7 day	28 day
No. 1	25	1:9	715	1175
No. 2	25	4:6	520	1110
No. 3	25	1:9	600	1130
No. 4	25	$1\!:\!2$	350	430
No. 5	25	1:9	1510	2210
No. 6†	25	$1\!:\!2$	1240	2400
No. 7†	25	1:9	3745	6240
No. 8	25	1:19	370	460

*Specimens were cured at near 100 percent relative humidity and 70°F. †Proctor size specimens were used for soils No. 6 and No. 7; all other specimens were 2 in. in diameter by 2 in. high.

TABLE IX. EFFECT OF CALCIUM CHLORIDE ON THE UNCONFINED COMPRESSIVE STRENGTH OF FIVE FINE GRAINED SOILS STABILIZED WITH LIME FLY ASH.

	Curing		Compre	ssive streng	th, lbs.†	
Soil*	time	0‡	1/2‡	1‡	$2\frac{1}{2}$ ‡	5‡
No. 1 (1:2)	7 day 28 day	$\begin{array}{c} 740 \\ 1130 \end{array}$	$\begin{array}{c} 1330\\ 1985 \end{array}$	$\begin{array}{c} 1600 \\ 2000 \end{array}$	$\begin{array}{c} 1855\\ 2630 \end{array}$	$\begin{array}{c} 1175 \\ 2430 \end{array}$
No. 2 (4:6)	7 day 28 day	520 1110	$\frac{1480}{2440}$	$\begin{array}{c} 1030 \\ 1845 \end{array}$	$\begin{array}{c} 850\\ 1340\end{array}$	_
No. 3 (1:9)	7 day 28 day	600 1130	$570\\1025$	$\begin{array}{c} 610\\1250\end{array}$	$\begin{array}{c} 775\\1250\end{array}$	$\begin{array}{c} 855\\ 1315\end{array}$
No. 4 (4:6)	7 day 28 day	$\begin{array}{c} 325\\ 395\end{array}$	310 540	$\begin{array}{c} 330 \\ 1020 \end{array}$	340 910	$\begin{array}{c} 195 \\ 510 \end{array}$
No. 5 (1:9)	7 day 28 day	$\begin{array}{c} 1510 \\ 2210 \end{array}$	$\begin{array}{c} 1370 \\ 2620 \end{array}$	$2300 \\ 3395$	$\begin{array}{c} 1165\\ 1890 \end{array}$	$\begin{array}{c} 330\\1125\end{array}$

*Lime fly ash admixtures constituted 25 percent of the mix, and the ratio of lime to fly ash is indicated in *Dime by ash admitted to be provided to provide a provided to provide a provided to provide a provided to provide a format and the provided to provided to provide a format and the provided to provide

and no. 4, were improved the least. Further studies of calcium chloride and other additives to improve the lime, fly ash, and soil reaction are in progress.

Kind of lime and fly ash. Since both lime and fly ash have variable properties, it seems likely that the kind of lime and fly ash used will be important variables in fly ash stabilization of soils. Only a beginning has been made on this study; the 7 and 28 day strength results of experiments with two kinds of fly ash, three kinds of hydrated lime, and three soils are summarized (table X). The properties of the limes and fly ashes have been presented (table III). All of the soils are different in the compressive strength obtainable; but these differences are, with a few exceptions, not great. There is an indication that the dolomitic lime, no. L3, gives higher strength than the two calcitic limes, and that the fly ash with the higher silica content, no. FA1, gives highest strength.

With the two silty soils investigated, the use of quicklime gave equal or higher strength than hydrated lime after 7 day curing, but after 28 day curing, hydrated lime gave slightly higher strength. With quicklime, maximum strength occurred at comparatively low ratios of lime to fly ash (table XI, figure 12). In the experiments with quicklime, it was found that when the amount of quicklime exceeded 5 percent of the dry weight of the total mixture, excessive expansion and hair cracking of the stabilized specimens occurred during curing.

EFFECT OF DIFFERENT TYPES OF LIME AND FLY ASH ON THE UNCONFINED TABLE X. COMPRESSIVE STRENGTH OF A CLAYEY, A SILTY, AND A SANDY STABILIZED SOIL.

Soil*	Kind of	7 da	y	28 da	ay
	lime and	Ratio range of	Compressive	Ratio range of	Compressive
	fly ash†	lime to fly ash‡	strength, lbs.	lime to fly ash‡	strength, lbs.
No. 1	A	1:9 to 5:5	720	1:9 to 3:7	$1175 \\ 1440 \\ 1405$
No. 1	- B	2:8	1120	2:8 to 5:5	
No. 1	- C	1:9 to 3:7	945	2:8 to 3:7	
No. 3 No. 3 No. 3 No. 3 No. 3 No. 3	A B C D E F	1:9 to 2:8 1:9 to 4:6 2:8 to 3:7 1:9 2:8 to 3:7 3:7 to 4:6	670 880 805 755 950 885	$\begin{array}{c} 1:9 \text{ to } 2:8\\ 1:9 \text{ to } 3:7\\ 3:7 \text{ to } 5:5\\ 1:9\\ 3:7 \text{ to } 4:6\\ 3:7\end{array}$	$1090 \\ 1295 \\ 1025 \\ 1185 \\ 1425 \\ 1440$
No. 5	A	1:9	915	1:92:8 to 5:51:9 to 2:81:9 to 2:82:8 to 5:51:9 to 3:7	1240
No. 5	B	1:9 to 3:7	955		1330
No. 5	C	1:9	1025		1350
No. 5	D	1:9 to 2:8	1090		1505
No. 5	E	1:9 to 5:5	1115		2070
No. 5	F	1:9 to 3:7	1110		1830

*Lime fly ash admixture constituted 25 percent of the mix. Specimens were cured at near 100 percent relative humidity and near 70°F.

-L1 & FA2 -L1 & FA1 -L2 & FA1 -L2 & FA2

L3 & FA1 L3 & FA2

range was taken where values were within 10 percent of maximum.

Modification of consistency limits and pH

The effect of lime fly ash admixtures on the consistency limits and the pH of six of the soils is shown in table XII. In general the addition of lime fly ash to soils reduces the plasticity and shrinkage properties, and these properties are affected most in clayey soils. An increase of the shrinkage limit denotes a decrease in shrinkage properties. The pH of lime, fly ash, and soil mixtures is highly alkaline.

To study the effect of curing time on the consistency limits and pH of stabilized soils, 2 by 2 inch specimens were prepared and moist cured for 7 and 28 days and ground up at the end of these periods for testing. The changes of the consistency limits with time, especially the shrinkage limit,

TABLE XI. COMPARISON OF THE EFFECT OF QUICKLIME AND HYDRATED LIME ON THE UNCONFINED COMPRESSIVE STRENGTH OF TWO SILTY SOILS.

		7	day	28	8 day		
Soil	Kind of lime	Ratio of lime to fly ash	Compressive strength, lbs.*	Ratio of lime to fly ash	Compressive strength, lbs.*		
No. 3	hydrated†	1:9	670	1:9	1090		
No. 3	quick‡	1:9	800	1:9	950		
No. 4	hydrated†	3:7	345	3:7	425		
No. 4	quick‡	1:9	345	1:9	370		

*Lime fly ash admixture constituted 25 percent of the total mixture. Specimens were cured near 100 percent relative humidity and near 70°F.

†L1 & FA2. ‡QL1 & FA2.





were not very consistent, but the general trend of the data indicates either little change or some further improvement of the plasticity and shrinkage properties. The slight decrease in alkalinity of some of the stabilized soils may indicate that some of the free bases were entering into a chemical reaction.

Durability

The durability of standard Proctor size specimens of selected mixtures of lime fly ash and each of the eight soils was evaluated by wetting and drying and freezing and thawing tests. The choice of amounts and proportions of lime and fly ash was based on the results of the unconfined compressive strength studies. The standard ASTM methods were used in evaluating stabilized soil no. 2 after moist curing periods of 7 and 28 days. The stabilized soil failed to meet the criteria for soil cement¹⁰, but it was found that the durability was substantially improved by longer curing (figure 13). The P.C.A. short-cut method was used in testing the freeze thaw and wet dry resistance of the other stabilized soils after 28 days moist curing.

TABLE XII.	IMMEDIA	rei an	D SH	ORT-TIME	CURING	EFFECT	OF LIME	FLY	ASH
ADMIXTURI	ES ON TH	s pH	AND	ATTERBE	RG LIMIT	S OF ST	ABILIZED	SOIL	S.

Soil	Optimum ratio of lime to fly ash*	Curing period, days†	$_{ m pH}$	L.L.	P.I.	S.L.
No. 1	Raw soil‡	0	$5.9 \\ 12.5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\ 5 \\ 11 \\$	$57\\43$	3710	$\begin{array}{c} 14.4\\ 34.4\\ \end{array}$
	1.9	28	11.5 11.4	37 38	$\frac{4}{5}$	$\begin{array}{c} 36.5 \\ 27.7 \end{array}$
No. 2	Raw soil‡		4.1	75	24	27.3
	4:6	7 28	12.4 12.2 12.1	65 55	16 8	$ \begin{array}{r} 34.3 \\ 34.3 \\ 41.2 \\ \end{array} $
No. 3	Raw soil‡		7.8 12 1	32 30	7	25.2
	1:9	7 28	12.0 11.4	35 35	4 4	$15.2 \\ 17.5 \\ 30.4$
No. 4	Raw soil‡	<u> </u>	$\begin{array}{c} 4.5\\ 12.3\end{array}$	33 39	11 10	22.9 15 7
	4:6	$\frac{7}{28}$	$\begin{array}{c} 12.3 \\ 12.3 \end{array}$	40 36	9 6	27.9 29.2
No. 5	Raw soil‡	0	$5.6 \\ 12.0$	19	$\frac{NP}{2}$	17.5
	1:9	7 28	11.5 12.1	·	NP NP	$25.2 \\ 22.4$
No. 8	Raw soil‡	0	7.4 12.0	54	20 11	20.6
	1:9	$\overline{\overline{7}}$ 28.	$\frac{10.9}{10.7}$	$\begin{array}{c} 46\\ 46\end{array}$	8 10	27.6 29.6

*Lime fly ash admixture constituted 25 percent of the material proportioned as a ratio of lime to fly ash. L1 & FA2. *Compacted specimens were cured at near 100 percent relative humidity and near 70°F. *Contains no admixture.

ure.

The results of the testing (figures 14, 15, and 16) indicate quite clearly that none of the mixtures studied would meet P.C.A. criteria for soil cement when compacted to standard Proctor density and cured for 28 days at near 100 percent relative humidity and 70° F. These findings should not be construed to mean that lime fly ash stabilized soils will not withstand freezing and thawing and wetting and drying in the base course of a road.



Fig. 13. Effect of curing time on volume change of a lime and fly ash stabilized clayey soil during wetting and drying tests, A.S.T.M. Designation: D559-44.



Fig. 14. Soil loss during freezing and thawing tests of eight soils stabilized with lime and fly ash. The P.C.A. short-cut procedure was used for all soils except No. 2.

The severity and unrealistic conditions of the standard tests, even for soil cement, are well recognized, and there is need for improved methods of evaluating the durability of stabilized soils.







Fig. 16. Comparison of soil loss during freezing and thawing tests on a sandy soil with varying percentages of lime and fly ash admixture. P.C.A. short-cut procedure was used.

Some investigators who have reported favorably on the resistance of lime fly ash stabilized soils to freezing and thawing and to wetting and drying have used a compactive effort greater than standard Proctor in molding test specimens and have cured the specimens at a higher temperature than 70°F. The recommended molding and curing procedure is: Compact the Proctor size specimen at optimum moisture content with 25 blows of a 10 pound hammer dropped through 18 inches on each of three equal layers of the mixture, and then moist cure in a sealed container for 7 days at 140°F.². Using this procedure, durability tests were made with soil from the same sampling location as soil no. 4. A compacted and cured mixture of 85 percent soil, 15 percent fly ash, and 7 percent lime met the P.C.A. criteria, except for permissible volume change⁹.

As a check on the effect of increased density and curing temperature on durability, experiments were performed with one mixture each of lime, fly ash, and soil no. 3 and lime, fly ash, and soil no. 4. The results indicate the degree of improvement obtained by using the compactive effort or 140°F. temperature or both (table XIII, figure 17). With the lime, fly ash, and soil no. 3 mixture, either the increased temperature alone or the combination of increased compactive effort and curing temperature produced 7 day specimens that could satisfactorily meet P.C.A. criteria for permissible soil loss and maximum moisture content. Increased temperature alone was not sufficient with the lime, fly ash, and soil no. 4 mixture, but the combination of increased compactive effort and temperature enabled 7 day specimens to satisfy the two criteria. No evaluation was made of volume change in these tests. Further studies of the effect of density on lime fly ash stabilization are in progress.

Soil	Compaction method	Curing time* and temperature	Freezing an Soil loss, percent	nd thawing Moisture content, percent†	Wetting and drying Soil loss, percent
No. 3#	Std. Proctor Std. Proctor Corson§	28 days @ 70°F. 7 days @ 140°F. 7 days @ 140°F.	$\begin{array}{c} 44.6\\7.3\\6.0\end{array}$	30.6/25.2 23.4/25.0 21.0/21.3	$N.D.\ddagger \\ 7.4 \\ 5.0$
No. 4**	Std. Proctor Std. Proctor	28 days @ 70°F. 7 days @ 140°F.	2nd// 7th//		$\substack{\mathrm{N.D.}\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ $

7 days @ 140°F.

7 days @ 140°F.

TABLE XIII. EFFECT OF COMPACTIVE EFFORT AND ELEVATED TEMPERATURE CURING ON THE DURABILITY OF TWO LIME FLY ASH STABILIZED SILTY SOILS.

*Cured at near 100% relative humidity.

Corson

Moisture content is given as maximum moisture content over allowable according to PCA criteria for soil cement. Not determined.

6.5

2.9

(Not determined.)
 (Prototor size specimen compacted in three layers with 25 blows of a ten pound hammer dropped through 18 inches for each layer.
 (/Cycle at failure.)
 #Soil 75%, lime 2.5%, and fly ash 22.5%.
 **ASoil 75%, lime 10%, and fly ash 15%.

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Number of cycles of drying and thawing

Fig. 17. Effect of increased density and elevated temperature curing on the resistance to freezing and thawing and wetting and drying of two lime and fly ash stabilized soils. The P.C.A. short-cut procedure was used. Standard and Proctor refer to the compaction system used. Proctor size specimens were cured for 7 days at near 100 percent relative humidity and 140°F. prior to testing.

Corson, Inc., Plymouth Meeting, Pennsylvania, for furnishing samples of lime.

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EFFECT OF DENSITY ON STRENGTH OF LIME FLY ASH STABILIZED SOIL

by

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ABSTRACT

The strength of an artificially cemented soil mass such as soil cement or soil, lime, and fly ash is highly dependent on the intimacy of grain to grain contact. The controlling factor here should be degree of compaction. With this in mind, various soil, lime, and fly ash mixes were compacted at four different controlled densities and the specimens were moist cured at normal temperature and tested. Three soils were used: an Iowa silt (loess), a Kansas dune sand, and a Texas coastal plain clay. The lime was calcitic (high calcium) hydrated lime. Mixes were prepared with 25 percent lime fly ash and with different ratios of lime to fly ash. Specimens were soaked in water and tested after 7 and 28 days.

Evaluation of the compressive strength data shows that density is a highly important variable. Compaction to above standard Proctor density increased 7 day strengths on the average 100 percent and 28 day strengths 70 percent. A higher compaction to modified Proctor density raised the average to 120 and 110 percent. Compaction to a super-modified Proctor increased the averages to 150 and 130 percent over strengths previously realized at standard Proctor density. It is concluded that density is not only important, but that it may be an economical consideration in design. The silt showed influence from overcompaction, but the influence vanished on 28 day curing. The clay gave the best response to increased compaction, and strengths with modified Proctor density were approximately three times those obtained at standard Proctor. With modified density all soils showed 28 day strengths of 600 to 1000 psi with ordinary room temperature moist curing.

Attendant with this investigation was an evaluation of an optimum lime fly ash ratio. With most soils the ratio was not critical, but highest strengths were realized with a lime fly ash ratio of 1:9 or 2:8. A ratio of 1:9 was nearly optimum for all three soils regardless of compactive effort.

INTRODUCTION

Objectives of this research were to study the effect of degree of compaction on the strength of lime, fly ash, and soil mixtures. Four compactive efforts were chosen: one to give densities equivalent to standard

Proctor, one to give densities between standard and modified Proctor, one to duplicate modified Proctor, and one to give densities greater than modified Proctor (table I). A second objective was to determine the effect of a variable compactive effort on the selection of an optimum lime fly ash ratio.

MATERIALS

Soils .

Three soils were selected for this study: a sand, a silt and a clay. The sand is from a stable dune area associated with the Arkansas River in south central Kansas. The silt is a friable, calcareous loess from the deep loess area in western Iowa. The clay is a deltaic deposit from the Coastal Plain region in Texas; it was sampled a few miles south of Houston (tables II, III). A.S.T.M. procedures were followed for laboratory testing except where otherwise noted.

Lime and fly ash

The hydrated lime is a calcitic lime from the Linwood Stone Products Co., Buffalo, Iowa. A laboratory analysis was furnished by the manufacturer (table IV). The fly ash is a fine ash with low loss on ignition; it is from Paddy's Run Station, Louisville Gas and Electric Co., Louisville, Kentucky. Data is from the Robert W. Hunt Co., Chicago (table IV).

METHODOLOGY

Correlation study

Because of the advantage of small specimen size for rapid molding and testing, the 2 by 2 inch size was used in this study. The 2 inch height gives the advantage of molding in one layer; the compactive effort is ap-

TABLE	I.	DESIGNATIONS	OF	COMPACTIVE	EFFORT.
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Compaction	Density obtained
A	Standard Proctor density
B	Between standard and modified
C	Modified Proctor density
D	Above modified

TABLE II. FIELD INFORMATION ON SOIL SAMPLES.

Sample:	Kansas sand	Iowa silt	Texas clay
Geological origin:	Recent dune sand from the Great Bend tract	Wisconsin age loess from near Missouri River	Deltaic (Beaumont) clay from coastal plain
Soil series:	Pratt	Hamburg	Lake Charles
Horizon:	C .	С	С
Location:	28 mi. S. of Great Bend	In the town of Missouri Valley	South of Houston
Sampling depth, ft.:	1 1/2 - 3 1/2	49-50	3¼-12 (composite)

plied at both ends. Specimens were molded with a drop hammer molding apparatus, and extensive correlation work was done to determine the proper hammer weights and numbers of blows for standard and modified Proctor densities (figure 1). The modified Proctor density was approximated by 10 blows on each end of the 2 by 2 inch specimen, and the modified Proctor density was duplicated with different soils (table V). These results were obtained with 20 blows (10 on each end) with a 10 lb. hammer dropping a distance of 1 foot with the holding apparatus mounted on a concrete pedestal. Other compactive efforts used are B (table I) obtained by 10 blows with the same arrangement and D obtained with 30 blows. Standard Proctor density (Density A) was duplicated by 10 blows from a 5 lb. hammer falling 1 foot with the apparatus resting on a wooden bench.

Constants and variables

To reduce the number of variables, a constant percentage of lime fly ash was used in all tests, the lime plus fly ash making up 25 percent of the dry weight of the mixtures. Previous work has shown that 25 percent is both a satisfactory and an economical content¹.

The moisture contents were adjusted to the optimums for each mixture and for each compactive effort. The optimum moisture contents of mix-

Sample	Kansas sand	Iowa silt	Texas clay
Textural composition, $\%^*$ Gravel (> 2 mm.) Sand (2-0.074 mm.) Silt (74-5 μ) Clay (< 5 μ) 'Colloids (< 1 μ)	0 86.4 4.0 9.6 8.6	0 0.7 78.3 21.0 15.8	0 7.7 48.2 44.1 36.8
Predominant clay mineral‡	Mont- morillonite	Ca mont- morillonite	Ca mont- morillonite
Specific gravity 25C./4C.	2.67	2.68	2.67
Chemical properties: Cat. ex. cap., m.e./100 gm.‡ Carbonates, %§ pH Organic matter, %‡	7.3 0 5.6 0.4	$13.4 \\ 10.5 \\ 7.8 \\ 0.2$	$25.5 \\ 0 \\ 5.9 \\ 0.6$
Physical properties: Liquid limit, % Plastic limit, % Plasticity index Shrinkage limit, % Centrifuge moist. equiv., % Field moist. equiv., %	— NP 18 5 21	$32 \\ 25 \\ 7 \\ 25 \\ 15 \\ 26$	57 20 37 14 21 21
Classification: Textural Engineering (AASHO)	Sand $A_{-2-4}(0)$	Silty clay loam $A_{-4}(8)$	Clay A-7-6 (20)

TABLE III. PROPERTIES OF SOIL SAMPLES.

*Dispersed by air-jet with sodium metaphosphate dispersing agent. †From differential thermal analysis of fraction passing no. 200 sieve.

Fraction passing no. 40 sieve. From differential thermal analysis.

tures with different ratios of lime to fly ash were read from a triangular chart in which optimum moisture contents of soil, of 75/25 soil fly ash, and of 75/25 soil lime are plotted at corners of the triangle and intermediate values are found by interpolation^{1, p. 81}.

The second major variable is ratio of lime to fly ash. Testing was continued at each of the four compactive efforts to show any change in optimum ratio. The previously found optimums with these soils has been between 1:9 and 2:8 by weight of lime to fly ash. In the present study, specimens were molded with ratios 0:10, 1:9, 2:8, 3:7, 4:6, and 5:5.

A third variable was age. Strengths were measured after 7 and after 28 days moist curing.

Curing and testing

Curing was done at 70 ± 3 °F. and with a relative humidity near 90 percent. Specimens were not wrapped, as is sometimes done to exclude carbon dioxide from the air. After curing the specified time, specimens were immersed in distilled water at 70°F. for 24 hours, then removed and tested for unconfined compressive strength. The rate of strain was 0.05 inch per minute per inch of specimen height. Results are expressed in pounds; if

Material	Linwood hydrated lime	Louisville fly ash
Specific gravity	2.29	2.67
Fineness % passing No. 325 sieve Specific surface, sq. cm./gm.	99.00	94.30 3470
Chemical analysis, % Total Ca (OH) ₂ Available Ca (OH) ₂ MgO CaCO ₃ Fe and Al oxides SiO ₂ Al ₂ O ₃ SO ₃ Free water Loss on ignition	97.8297.380.490.770.820.8024.56	0.52 8.36 Not determined 38.90 22.92 2.0 0.17 2.10

TABLE IV. PROPERTIES OF LIME AND FLY ASH.

TABLE V. COMPARISON OF MODIFIED PROCTOR DENSITIES FROM 2" X 2" TEST WITH THOSE FROM THE FULL SIZE A.S.T.M. TEST.

Sample:	Kansas sand	Iowa silt	Texas clay
A.S.T.M. test: Max. dry dens., pcf. Opt. moist. cont., %	128.1 9.2	$\begin{array}{c} 121.8\\ 13.2 \end{array}$	$118.8\\13.8$
2" x 2" test: Max. dry dens., pcf. Opt. moist. cont., %	$\begin{array}{c} 128.9\\ 9.3\end{array}$	122.0 13.3	$118.9\\13.7$

the height to diameter ratio is neglected, results can be converted to pounds per square inch by dividing by 3.14.

Other measurements include absorption and volume change during curing and immersion.

RESULTS

In the plotted results, most of the curves are displaced upward by increased compactive effort, and 7 day strengths were on the average about 100 percent higher with compactive effort B than at standard Proctor density A (figures 2, 3, 4). Compaction to modified Proctor density raised this to 120 percent, and compaction to beyond modified Proctor gave on



Fig. 1. Typical moisture-density relationships from 2 inch by 2 inch specimens. Ten blows with a 10 lb. drop hammer give a maximum density and optimum moisture content close to modified Proctor.



Fig. 2. Effect of compactive effort on strength of Kansas sand stabilized with 25 percent lime and fly ash in varying ratios.



Fig. 3. Effect of compactive effort on strength of lowa silt (loess) stabilized with 25 percent lime and fly ash in varying ratios.

the average a 150 percent increase in 7 day strength. Twenty-eight day strengths reflect the same trends.

Density and percent solids

Density of course depends on compactive effort, but density also depends on lime to fly ash ratio. Density is decreased by higher contents of lime because of two factors: the lime itself is less dense than soil or fly ash, and lime causes aggregation of clay. The first factor is calculable and can be corrected by converting measured densities to percent solids by volume (figures 5, 6, and 7). Compressive strength has been plotted against percent solids, regardless of the lime to fly ash ratio. The fact that smooth curves are usually obtained indicates that the lime to fly ash ratio is not critical.

In the Texas clay (figure 7) the influence of clay aggregation on density is found to be a maximum. As an example, points labelled 1, 2, 3, 4, 5 under compactive effort A are with lime to fly ash ratios of 1:10, 2:8, 3:7, 4:6 and 5:5. A higher lime content decreases the volume percent solids. With higher compactive efforts the same trend is found, but it will be





noted that the range in percent solids shown at the top of the graph is less for B, C and D, indicating that higher effort may break down the clay aggregates and better their compaction. This tendency is particularly pronounced with the silt (figure 6) in which with effort A there is a wide range in percent solids depending on the lime content. The range is progressively smaller with efforts B, C and D. It is believed that the silty soil aggregates may have less strength than those formed in the clay soil and are thus easier to break down.

The sand (figure 5) offers a direct contrast to this. With low compactive effort, addition of more lime has practically no effect on the percent solids, as shown by the narrow horizontal range in points under A. With higher compactive efforts the range is greater, as in B and C, but the range is greatly reduced with D, the highest compactive effort. While the reason for this is not known, it is suspected that lower lime and higher fly ash contents improve the gradation of the sand for compaction.



Volume percent solids

 \cdot Fig. 5. Relation of strength to percent solids in lime and fly ash stabilized Kansas sand.

Unconfined compressive strength for evaluation of stabilized soils

Unconfined compressive strength is primarily influenced by cementation and does not give a true measure of the frictional strength developed in a confined state. Therefore a stabilized granular material with relatively low unconfined compressive strength may show satisfactory stability. It is known that the C.B.R. values for a given stabilized soil are directly proportional to unconfined compressive strength²; and it has been found that, for example, a lime fly ash stabilized sand with an unconfined compressive strength of 138 psi. has a C.B.R. of 213, while a stabilized clay must have an unconfined compressive strength of 705 psi. to develop the same C.B.R.³.

Strength vs. percent solids

Points have been plotted to show the relationships between strength and percent solids without regard to lime fly ash ratio (figures 5, 6, 7). The striking feature is that most of the points fall on or very close to the curves. The exceptions are numbered to indicate their ratios, which are either very low (1:10 or 2:8) or very high (5:5).

Curves for the different soils show a similarity in that strength is approximately proportional to percent solids, and the proportionality factor indicated by the slope of the curve is much the same. An exception is sand, where the strength gain between efforts A and B is not nearly in accord



Volume percent solids

Fig. 6. Relation of strength to percent solids in lime and fly ash stabilized lowa silt.

with the increase in percent solids. Apparently cementation of the sand is not greatly improved until compaction reaches above a critical percent solids, about 75 percent. For some reason a critical degree of packing is necessary before grain contact and cementation are improved.

Overcompaction and tendency to heal

In one curve (figure 6) strength loss from overcompaction is evident. This is the 7 day strength curve for the silt. The same trend can be seen in figure 3, where the 7 day curves in B, C and D are progressively lower even though density is increased and absorption and volume change are reduced by the greater compaction. It is therefore believed that strength loss may be due to shearing displacements in the specimen causing intrinsic planes of weakness. Particularly significant is that at 28 days the strength curve follows a normal pattern, and the shear planes, if any, have apparently healed. Such a tendency for healing of overcompaction failure planes could be of considerable importance in field construction. Presum-



Fig. 7. Relation of strength to percent solids in lime and fly ash stabilized Texas clay.
ambly continued intimate contact would be necessary for failure plane healing.

Optimum lime to fly ash ratio

Previous work has shown that for highest strength with standard Proctor compaction the optimum lime to fly ash ratio is usually in the range 1:9 to 2:8¹, though a halloysitic clay was an exception and required more lime. In the present study, increased compaction does not greatly or consistently change the optimum ratio (figures 2, 3, 4). From an economic standpoint a low ratio is desirable, since the cost of fly ash is usually a fraction of the cost of lime. A limit is imposed by difficulties in mixing and securing uniform distribution of very small percentages of lime. However, ratios of 1:9 and 2:8 can be successfully handled in construction with a wide variety of soil textural types.

SUMMARY

A summary of the relation between compaction and compressive strength is illustrated in figure 8. The curve represents an average for all three soils; the scatter of points is greater than in figures 5, 6 and 7 because of disregard of other variables such as soil type and the dependence of density on percent lime. The average increase in compressive strength is $43.5 \ p$, where p is the percent increase in density over standard Proctor. That is,

S = So + 43.5 p,

where So is the strength in psi. at standard Proctor density. On the aver-



Fig. 8. Average effect of increase in density on compressive strengths of a sand, a silt, and a clay.

age a 10 percent increase in density will about double the unconfined compressive strength. This density is approximately equivalent to modified Proctor for the sand and the clay; because of poor gradation it is not readily obtainable with the silt.

CONCLUSIONS

1. Strengths of lime fly ash stabilized soil after 7 and 28 days are greatly increased by increased density and compaction, but the optimum lime to fly ash ratio is little influenced. The optimum ratio for these soils remained 1:9 or 2:8.

2. Increasing the additions of lime to the clay and silt soils results in a decreasing percent solids with the same compactive effort, probably because of clay aggregation by lime. The resulting decrease in strength is approximately proportional to the decreasing percent solids. This relationship was not found in the case of the sand.

3. Lowered strengths of stabilized silt due to overcompaction were evident after 7 days curing, but at 28 days the influence had vanished. It is concluded that overcompaction shear planes in lime, fly ash, and soil tend to heal on long curing.

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DURABILITY OF SOIL LIME AND FLY ASH MIXES COMPACTED ABOVE STANDARD PROCTOR DENSITY

by

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ABSTRACT

Lime fly ash stabilized Kansas dune sand, Iowa silt (loess) and Texas coastal plain clay show a definite increase in durability when compacted to densities above standard Proctor. In fact, silt and clay mixes gained strength more rapidly through artificial weathering than after sustained moist curing.

Two by two inch cylindrical specimens prepared with 25 percent lime, fly ash and optimum ratios of lime to fly ash (1:9 for sand and silt, and 2:8 for the clay) were cured for 14 days at near 100 percent relative humidity and 70°F. prior to being subjected to cycles of freezing and thawing or wetting and drying. Moisture absorption, swelling and unconfined compressive strength of the specimens, after various cycles of freeze-thaw and wet-dry were used as a means of analyzing the durability of the stabilized soils.

The increases in strength during wet dry and freeze thaw tests over normal moist curing are attributed to improved intimacy of contact between lime and fly ash grains following dissolution and reprecipitation of the lime.

INTRODUCTION

Earlier studies have indicated that compaction to a density greater than standard Proctor greatly improved the resistance of a soil, lime, and fly ash mixture to wetting and drying or freezing and thawing³. The present study was undertaken to check the resistance of lime fly ash stabilized soils compacted to densities above standard Proctor and within the capabilities of present day compaction equipment.

Three compactive efforts were used (table I):

(1) between standard and modified Proctor density,

(2) equivalent to modified Proctor and

(3) above modified Proctor.

The criteria used to evaluate the effects of increased density on durability of the specimens after various cycles of wetting and drying or freezing and thawing were unconfined compressive strength, moisture absorption and average increase in height of specimens, the latter being an indication of volume change or swelling.

MATERIALS

Soils

Soils selected for this study are the same as those used previously⁵. The silt is a friable, calcareous loess from western Iowa; the clay is a deltaic deposit from the coastal plain region of Texas; and the sand is from a stable dune area associated with the Arkansas River in south central Kansas (tables II and III).

Lime and fly ash

The lime used in this study is a hydrated calcitic (high calcium) lime from the Linwood Stone Products Co., Buffalo, Iowa. A laboratory analysis was furnished by the manufacturer (table IV). The fly ash used is from Paddy's Run Station, Louisville Gas and Electric Co., Louisville, Kentucky. A chemical anlysis of the fly ash was obtained from the Robert W. Hunt Co., Chicago, Illinois (table IV).

METHOD OF TEST

Mixing

The soils were air dried, pulverized and screened through a No. 10 sieve. Each soil was dry mixed by hand with the various amounts of lime and fly ash. Predetermined amounts of distilled water were then hand mixed into the blend and mixing was completed with a Hobart, Model C100, mixer at moderate speed for three minutes.

Molding

Two by two inch specimens were molded at each density using a drop hammer molding apparatus⁵. Compaction of the 2 by 2 inch specimens to standard and modified Proctor densities with this apparatus has been correlated very closely with recognized laboratory compactive procedures^{1, 5}.

	TABLE I. I	DESIGNATIONS	OF COMPACTIVE EFFOR	r.5
	Compaction	tion Density obtained		
	A B C D	Standar Between Modified Above n	rd Proctor density n standard and modified l Proctor density nodified density	ed density
	TABLE II. I Kan	FIELD INFORM	ation on soil sampl Iowa silt	ES. ⁵ Texas clay
Geological origin	Recent from the	dune sand Great Bend tract	Wisconsin age loess from near Missouri River	Deltaic (Beaumont) clay from coastal plain
Soil series	J	Pratt	Hamburg	Lake Charles
Horizon		С	С	С
Location	28 1 Gre	ni. S. of at Bend	In the town of Missouri Valley	South of Houston
Sampling depth,	ft. 1	1/2-31/2	49-50	3¼-12 (composite)

Curing

Curing was in a humidity cabinet at approximately 70°F. and near 100 percent relative humidity. After designated lengths of curing the samples were measured for height and weight.

TABLE III. PI	ROPERTIES OF	SOIL SAMPLES. 5	
	Kansas sand	Iowa silt	Texas clay
Textural composition, % Gravel (> 2 mm.) Sand (2 — 0.074 mm.) Silt (74 — 5μ) Clay (< 5μ) Colloids (< 1μ)	$0\\86.4\\4.0\\9.6\\8.6$	$\begin{array}{c} 0 \\ 0.7 \\ 78.3 \\ 21.0 \\ 15.8 \end{array}$	$\begin{array}{c} 0 \\ 7.7 \\ 48.2 \\ 44.1 \\ 36.8 \end{array}$
Predominant clay mineral*	Mont- morillonite	Ca mort- morillonite	Ca mont- morillonite
Specific gravity 25C./4C.	2.67	2.68	2.67
Chemical properties Cat. ex. cap., m.e./100 gm.† Carbonates, %‡ pH Organic matter, %†	$7.3 \\ 0 \\ 5.6 \\ 0.4$	$13.4 \\ 10.5 \\ 7.8 \\ 0.2$	$egin{array}{ccc} 5.5 & 0 \ 5.9 & 0.6 \end{array}$
Physical properties Liquid limit, % Plastic limit, % Plasticity index, % Shrinkage limit, % Centrifuge Moist. Equiv., % Field Moist. Equiv., %		$32 \\ 25 \\ 7 \\ 25 \\ 15 \\ 26$	57 20 37 14 21 21
Classification Textural Engineering (AASHO)	Sand A-2-4(0)	Silty clay loam A-4 (8)	Clay A-7-6(20)

*From X-ray and differential thermal analysis of whole soil. †Fraction passing No. 40 sieve. ‡From differential thermal analysis.

5

	Linwood hydrated lime	Louisville fly ash
Specific gravity	2.29	2.67
Fineness % passing No. 325 sieve Specific surface, sq. cm./gm.	99.00	94.30 3470
Chemical analysis, % Total Ca(OH) ₂ Available Ca(OH) ₂	97.82 97.38	
MgO CaCO ₃	$\begin{array}{c} 0.49 \\ 0.77 \end{array}$	$\begin{array}{c} 0.52 \\ 8.36 \end{array}$
Fe and Al oxides SiO AloOo	0.82 0.80	38.90
SO ₃ Free water		22.92 2.00 0.17
Loss on ignition	24.56	, 2.10

Wet dry testing

The method of wet dry test adopted was as follows:

1. Specimens were prepared at the designated density and optimum moisture content, then moist cured for fourteen days.

2. Specimens were air dried for 24 hours at room temperature and then were completely immersed in distilled water for 24 hours. This completed one cycle of wetting and drying. Further cycles were a repetition of this step.

3. After designated cycles of wetting and drying, specimens were wiped with a towel to a surface dry condition, measured for height and weight, and tested for unconfined compressive strength.

Freeze thaw testing

The method of freeze thaw test adopted was as follows:

1. Specimens were prepared at the designated density and optimum moisture content. After moist curing for fourteen days, specimens were placed on one-half inch thick felt pads set in approximately one-fourth inch of water.

2. Specimens on moist felt pads were placed in a freezer at -10° F. for 24 hours.

3. After removal from the freezer, specimens were allowed to thaw in open air at room temperature for two hours.

4. Specimens were placed in a humidity cabinet at approximately 70° F. and 100 percent relative humidity for 22 hours. This completed one cycle of freezing and thawing. Further cycles were a repetition of steps 2, 3, and 4.

5. The specimens to be tested were measured for height and weight and tested for unconfined compressive strength.

Compressive test

After completion of curing and/or various cycles of wet dry or freeze thaw, all specimens were tested for unconfined compressive strength. The rate of deformation of the testing machine was held constant at 0.05 inch per minute per inch of specimen height.

Absorption and volume change

The percentage of moisture absorbed during the wet dry and freeze thaw test was determined by subtracting the weight of the specimen after molding from the weight after immersion and dividing by the oven dry weight of the specimen. Though the actual volume change was not measured, an easily determinable indicator of volume change was used—the average increase in height of specimens. This was determined by subtracting the height of the specimen after molding from the height after various cycles and dividing by the height after molding.

EVALUATION OF TEST RESULTS

Selection of mixes

Previous research indicates that increasing the density of lime fly ash stabilized soils greatly increases strengths (figures 1, 2, 3). As lime content increases, the density and immersed compressive strength at each compactive effort in general tend to decrease, probably because of increased clay aggregation by the lime⁵.

Optimum ratios of lime to fly ash at densities greater than standard Proctor are 1:9 for the sand and silt, and 2:8 for the clay. These ratios were used in the wet-dry and freeze-thaw testing described in this paper. On the basis of previous studies, a mix with 25 percent lime fly ash was chosen as being satisfactory and economical. All test points were run in duplicate or in triplicate.

Wet dry tests

Silt. Wet dry test results with silt are shown in figure 4, along with freeze thaw results with the same soil. The wet dry cycles apparently



Fig. 1. Effect of variations in lime to fly ash ratio, compactive effort, and curing on compressive strength and density of lime, and fly ash stabilized sand.



Fig. 2. Effect of variations in lime to fly ash ratio, compactive effort, and curing on compressive strength and density of lime, and fly ash stabilized silt.

cause a general increase rather than a decrease in strength. Similar trends have been reported elsewhere for lime and lime fly ash stabilized soils^{2, 4}. Strength curves (figure 4) for different compactive efforts tend to diverge after 12 cycles, but this was accompanied by a similar divergence in the data for each point, indicating greater statistical error. No particular significance is therefore attached to the upturn or downturn of the curves after 12 cycles. On a strength basis alone there is an advantage to compacting the silt to modified Proctor density (effort C), but the wet dry tests show little benefit from compacting beyond this.

During early cycles all specimens absorbed water and expanded. Increased compaction reduced expansion but tended to increase the absorption of water, perhaps due to improved capillarity. After five cycles the absorption by specimens molded to compactive effort D is drastically reduced, which could be due either to reduced permeability or increased cementation tending to hold the specimen together. Since the latter effect does not appear in the compressive strength, one can conclude that pozzolanic reaction products may be plugging the pores or at least rendering them impermeable to water.

Clay and sand soils. The clay and sand were tested after compaction to modified Proctor density (effort C, figure 5). Both clay and sand show a uniform increase in strength through the wet-dry cycles, and moisture absorption and expansion both are reduced.





Comparison with normal moist curing. Compressive strengths after normal moist curing are plotted for the silt and for the sand and the clay (figures 6, 7). The silt and the clay are considerably benefited by wet-dry cycles, whereas the sand is not. Similar data show that clay and silt were benefited by prolonged soaking, but the sand was not³.

Freeze thaw tests

Silt. All silt specimens gained strength through the first 10 or 12 freeze thaw cycles, then took the more logical trend downward (figure 4). A satisfactory resistance is indicated for all three compactive efforts, since the strengths after weathering are higher than those before weathering started. Moisture absorption trends upward, indicating progressive failure



during freezing and thawing. Expansion remains small, but the silt compacted with effort D shows a gradual increase, suggesting overcompaction.

Fig. 4. Effect of compactive effort, wetting and drying, and freezing and thawing on compressive strength, moisture absorption and expansion of lime, and fly ash stabilized silt.



Fig. 5. Effect of wetting and drying, and freezing and thawing on compressive strength, moisture absorption, and expansion of lime, and fly ash stabilized clay and sand compacted to modified Proctor density.

Overcompaction was previously noted for effort D with this soil, but had disappeared after 28 days normal curing^{5, conclusion 3}.

Clay and sand soils. Strengths of clay and of sand specimens during cycles of freeze thaw correlate well with the moisture absorption (figure 5). The clay shows a drastic increase in absorption up to five cycles, after which the specimens slowly lose water. Strengths drop about 50 percent from the first to the fifth cycle, after which there is a slow gain. A slight volume expansion also takes place after the fifth cycle.

Curves for the sand are somewhat reversed to those for clay, but show the same relationships. Moisture absorption increases on the ninth cycle, and coincident with this the strengths go down. A sharp increase in volume is noted after the eighth cycle.

Comparison with normal moist curing. Freeze thaw cycles benefit the

strengths of the silt and are somewhat deleterious to the strengths of the sand (figures 6, 7). The same results were found with wetting and drying. The clay is benefited by freezing and thawing for one cycle. After this



Fig. 6. Relationship of effect of compactive effort, curing, wetting and drying, and freezing and thawing on compressive strength of lime, and fly ash stabilized silt.



Fig. 7. Relationship of effect of curing, wetting and drying, and freezing and thawing on compressive strength of lime, and fly ash stabilized clay and sand compacted to modified Proctor density.

the strength progressively decreases until it approximates that obtained during normal moist curing, then strengths start back up.

DISCUSSION AND CONCLUSIONS

The obvious conclusion is that high density does improve durability of soil, lime, and fly ash mixtures, to the extent that after an initial moist cure, clay and silt soils gain strength even more rapidly during wet-dry or freeze-thaw cycles than they do in a continued moist cure. The sand soil gave comparable strength gains in either weathering cycles or moist cure. The comparatively high durabilities were realized by compacting to modified Proctor density. Previous studies showed the durability of soil, lime, and fly ash to be questionable after compaction to standard Proctor³.

The uniqueness of a strength gain during a supposedly destructive testing program deserves more than a passing remark. Wetting and drying could result in periodic dissolution and redistribution of part of the lime, giving greater intimacy of contact and promoting the reactions with fly ash. Prolonged soaking in water can apparently give the same mobility, as strengths are then high also³. Similar results from lime stabilization indicate the importance of contact between lime and soil grains.

Beneficial effects of freeze thaw cycles are more problematical and have not been noted before. The extreme was that of the clay, where strength was doubled by one cycle. After this, destruction started, but the trend again reversed after the fifth cycle. Benefits were less marked with the silt and nil for the sand, further emphasizing the importance of surface reactions not only with fly ash, but also with soil. In dolomitic lime, the solubility of MgO and Mg(OH)₂ increases with a rising temperature, and the solubility of Ca(OH)₂ decreases. Therefore a critical redistribution of lime may result from a single freeze-thaw cycle. The redistribution is apparently of lesser importance with coarse-grained soils, which have lower surface area for cementation reactions.

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REACTIVITY OF FOUR TYPES OF FLY ASH WITH LIME

by

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ABSTRACT

The pozzolanic reactivity of four fly ashes having different properties is evaluated using unconfined compressive strength of lime fly ash mortars as the criterion of reactivity. Test results indicate that the carbon content of fly ash, as determined by loss on ignition, has an important effect on mortar strength and appears to be a reliable indicator of fly ash reactivity.

INTRODUCTION

A pozzolan is defined as a siliceous or as a siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties⁷. Although the principal products of the reaction between a pozzolan and calcium hydroxide are considered to be calcium silicates and aluminates, there is some evidence that more complex compounds are formed.

The Romans utilized the pozzolanic action of volcanic ash with calcined limestone in the construction of such historic landmarks as the Appian Way, the Colosseum and the Pantheon. Since then various other natural substances and some artificially produced materials have been found to possess various degrees of pozzolanic activity⁴.

Fly ash is an artificial pozzolan which results from burning pulverized coal. The coal, of which about 80 percent passes a No. 200 sieve, is blown into a furnace with primary air, and the combustion of the organic material in the suspended particles occurs almost instantly. The unburned inorganic materials form minute molten globules at a temperature of approximately 2800°F. These globules congeal into spherical particles about 75 microns in diameter as they leave the zone of high temperature^{4, 5}. Some partially burned organic particles result and are of a more irregular shape and are somewhat larger. These particles are considered to be mostly carbon. After passing through the super heater, economizer and preheater, the ash (containing both incompletely burned and unburned particles) is separated from the exhaust gas stream by various methods. Collection of fly ash in stack gas is usually by the use of electrical precipitators or mechanical collectors⁸.

The design of power plant boilers generally falls into three basic categories: dry bottom, wet bottom, and cyclone. The total ash produced in the operation of dry bottom boilers is approximately 90 percent fly ash; the remaining 10 percent is larger particles (bottom ash) which fall out by gravity. Wet bottom boilers produce about 50 percent of total ash as fly ash, and cyclone equipment produces only about 15 percent fly ash⁸.

Electrical precipitators are more efficient than mechanical collectors and usually remove a higher percentage of the fly ash from the flu gases. Fly ash collected by electrical precipitators contains a high percentage of fine particles, and therefore has a high specific surface which is considered conducive to high pozzolanic activity^{2, 3, 9}.

Each power plant produces fly ash of a relatively different character, that is, it varies in particle size and chemical composition. These variations are due to the type of coal, treatment prior to combustion, method of combustion, amount of recirculation and method of collection. Studies of the use of fly ash in Portland cement mortar and concrete have indicated that fineness and carbon content are possible criteria for differentiating fly ashes. Analyses of fly ashes include the term "loss on ignition" which is expressed as a percentage of the total fly ash and approximately represents the carbon content. The loss on ignition is determined by oxidation at high temperatures of the organic material in the fly ash. There are several methods of determining fineness; one of the more common is sieving the fly ash through a No. 325 sieve to determine the percent passing.

An extensive investigation of the chemical properties of fly ashes was conducted in 1944°. Chemical analyses of different size fractions of fly ash indicated that the non-combustible SiO_2 , Al_2O_3 and Fe_2O_3 tend to be concentrated in the finer fractions. The residual carbon, as determined by loss on ignition tests, predominates in the coarser particles. Photomicrographs showed that the carbon in fly ash is in irregular, porous, coke-like particles. The non-combustible particles generally have a characteristic spherical shape, although a small portion of these particles are thin walled polyhedrons called cenospheres.

Little information has been published about the effects of fly ash properties on its reactivity with lime. In this study, four fly ashes having different properties were used to investigate these effects. The unconfined compressive strengths of lime fly ash mortars were used to evaluate reactivity, the assumption being that strength is a positive function of reactivity.

MATERIALS

Fly ashes

The sources and properties of each fly ash are tabulated and the fly ashes have been assigned arbitrary numbers by which they will be identified

(tables I, II). X100 photomicrographs of fly ashes nos. 10, 11, 12 and 15 (figures 1, 2, 3, 4) tend to corroborate the earlier findings⁹. Comparison of the photomicrographs of no. 10 and no. 11 fly ashes is particularly interesting, since these fly ashes contain the least and the most carbon respectively. Notice the greater degree of fineness and the relative absence of carbon in fly ash no. 10, and the particles in fly ash no. 11 appear to be somewhat aggregated and coated by more abundant carbon.

TABLE I. SOURCE AND COLLECTION DATA OF FOUR FLY ASHES.¹⁴

Fly ash	Type boiler	Coal	Collecting	efficiency, percent
No. 10	Dry bottom	Western Kentucky	Electrical	98*
No. 11	Wet bottom	50 percent eastern Kansas and 50 percent petroleum coke	Electrical	Less than 90†
No. 12	Dry bottom	Western Kentucky and southern Illinois	Mechanical	Less than 70‡
No. 15	Dry bottom	Southern Illinois	Electrical	95§

*Electric precipitators were used and the combustion chamber and coal pulverizing equipment were designed for extremely efficient burning. The precipitators are oversize to obtain high collection efficiency. 'This unit has electric precipitators but for the last year has burned a 50-50 blend of coal and petroleum coke. The coke does not fully burn in the short time it is in the combustion chamber; therefore the loss due to ignition (carbon content) is increased. 'Low efficiency here is due to the use of mechanical precipitators. The loss on ignition is from 8 to 12 per-cent. Most of this loss was evident in the material retained on the No. 200 sieve while the relatively finer material retained on the No. 325 sieve was very low in loss on ignition is fairly high. This is due to over-loading of the boilers (approximately 110 percent of rated capacity) resulting in incomplete combustion of the coal.

TABLE II. PROPERTIES OF FLY ASHES.

		Fly ash			
		No. 10	No. 11	No. 12	No. 15
Specific gravity		2,56	unknown	2.30	2.24
Fineness	Residue passing a No. 325 sieve, percent by weight	93.1*	60.3†	81.0*	82.3*
Chemical	Silicon dioxide	43.40	39.19	41.16	35.94
analysis, percent by weight	(S1O ₂) Aluminum oxide (AloO2)	20.10	13.23	18.39	18.19
	Ferric oxide (Fe ₂ O ₃)	19.00	13.41	21.23	19.63
	Calcium oxide (CaO)	7.30	2.52	5.54	6.89
	Magnesium oxide	0.43	1.16	0.77	0.85
	Sulphur trioxide	3.04	0.41	1.47	1.86
	Loss on ignition	3.20	27.67	10.18	15.59

*Method of test: ASTM Designation C204-46T. \uparrow Method of test: Material was screened until the percent passing the No. 325 sieve was less than 0.5 percent after five minutes in a mechanical sieve shaker.



Fig. 1. Photomicrograph of No. 10 fly ash: x 100. Fig. 2. Photomicrograph of No. 11 fly ash: x 100.



Fig. 3. Photomicrograph of No. 12 fly ash: x 100. Fig. 4. Photomicrograph of No. 15 fly ash: x 100.

Lime

The lime used was laboratory reagent powdered calcium hydroxide. The manufacturer has listed the maximum limit of impurities as follows:

Insoluble in HCl	0.03 %
Chloride	0.005%
Sulfate	0.10 %
Heavy metals such as Pb	0.003%
Iron	0.05 %
Substances not precipitated by ammonium oxalate	1.0 %

SAMPLE PREPARATION AND TESTING

Preparation of mixtures

The amounts of lime added to each fly ash were 2, 4, 6 and 8 percent based on the dry weight of the fly ash. The amount of distilled water in each was sufficient to produce the maximum dry density for standard Proctor compactive effort.

In the moisture-density curves for four mortar compositions, using fly ash no. 10, several definite points of maximum density were found in the lower moisture range; but extension of the moisture-density curve into the upper reaches of moisture content revealed a true maximum density (figure 5). Similar results were obtained with the other fly ashes. The true maximum density occurred in all cases slightly below the moisture content at which the mortar began to act as a viscous liquid. The optimum moisture contents and corresponding true maximum densities are presented in table III.

Optimum moisture content tends to decrease with the fineness of the fly ash. Similar results were found in an investigation of fly ash as an additive to Portland cement³. Optimum moisture content and loss on ignition were correlated (figure 6). The increase of moisture requirement with increased carbon content is probably due to the porous nature of the carbon. A similar trend was found in the water requirement of Portland cement fly ash mortars¹.



Fig. 5. Moisture-density relationship of No. 10 fly ash illustrating relative maxima at low moisture contents and the absolute maximum for the 8 percent lime content. These curves are typical for the other three fly ashes.



TABLE III. OPTIMUM MOISTURE CONTENTS AND CORRESPONDING TRUE MAXIMUM DRY DENSITIES OF THE LIME, FLY ASH MORTARS PREPARED WITH THE FOUR FLY ASHES.



Mixing and molding

Lime fly ash mixtures were proportioned and mixed dry. Optimum water was added and the materials were machine mixed for four minutes. Two inch by two inch specimens for unconfined compressive strength tests were prepared at approximate standard Proctor density with a double plunger drop-hammer molding apparatus.

Curing

Specimens were cured for various times at two different constant temperatures, to study the rate and duration of the pozzolanic reaction. Curing times were 0, 7, 14, 28 and 45 days; curing temperatures were 20° C. and 60° C. Each specimen was first wrapped in either Saran Wrap or wax paper, then in an aluminum foil wrapping sealed with Scotch tape. The difference in the inner wrapping should be noted, since this inconsistency accounts for a variation in the test results. Specimens cured at 20° C. were stored in an atmosphere with a relative humidity of approximately 90 percent. Specimens cured at 60° C. were kept in an oven having a non-humid atmosphere; the sealing of each specimen was assumed to be sufficient to prevent any loss of moisture by evaporation.

Testing

At the end of the curing periods, specimens were unwrapped, weighed to determine moisture loss during curing, and then tested in unconfined compression using a load travel rate of 0.05 inches per minute. The results reported are the average of three test samples and represent the load at failure, uncorrected for height to diameter ratio.

DISCUSSION OF RESULTS

The order of the strength time curves of the lime fly ash mortars fluctuated somewhat during the early stages of curing at 20°C. (figure 7). However, the curves appear to have reached their proper positioning in relation to each other after 45 days curing. The 45 day strength values show that increased lime contents are directly responsible for higher strengths.

Time and temperatures are two very significant factors responsible for some of the variations. The slopes of the strength time curves at 20°C. are still definitely positive after 45 days curing, indicating that the pozzolanic reaction has not yet reached completion. The lone exception is shown by the curve for No. 10 fly ash with 2 percent lime. Here the reaction appears to be nearly complete after 28 days curing. These data support the validity of the assumption that compressive strength is a criterion for studying the progress of the pozzolanic reaction. Apparently strength develops at a rate that parallels the rate of the reaction. As the lime combines with the fly ash and the amount of free lime decreases, the rate of strength increase gradually slows, and the curve tends to become horizontal. This is best shown by the mortars containing 2 percent lime.

Samples cured at 60° C. showed a decidedly higher rate of strength development during the first few days than those cured at 20° C. The increase in temperature caused an increase in reaction rate during the first 7 days in all cases. Acceleration of the reaction was anticipated because it has long been known that many chemical reactions may double or treble their velocity with a 10° rise in the temperature of the reactants.

Curing of all but a few specimens beyond 7 days at 60°C. caused the



Fig. 7. Effect of variation in the amount of lime, curing time and curing temperature on the compressive strength of 2 inch by 2 inch specimens prepared from the four fly ashes.

strength time curves to flatten out and then to decrease (figure 7). The loss of strength is due apparently to an excessive loss of moisture caused by improper choice of interior wrapping material (wax paper). Specimens (No. 10 fly ash with 2 percent lime and No. 11 fly ash with 2, 4 and 8 percent lime) wrapped with Saran Wrap did not show a decrease in strength or a significant loss of moisture during curing. A comparison of the moisture losses after curing showed an extreme loss of moisture at 28 days for all specimens wrapped in wax paper, for example, the moisture loss in 45 days for a Saran wrapped specimen was 13 grams, and the moisture loss in 45 days for a wax paper wrapped specimen was 30 grams.

Other investigators have suggested the use of strength after 7 days curing at 60°C. for predicting 28 day strengths of room temperature cured specimens. Comparison of these values shows that there is no simple relationship between them. However, the strength values after 7 days curing at 60°C. place the fly ashes in their correct order of reactivity. This suggests the possibility of using accelerated curing for rating fly ash reactivity when time does not permit the longer periods of curing required at room temperatures.

Regrouping the curves in figure 7, so that the curves of identically proportioned mortars made from different fly ashes are grouped together, reveals the results shown in figure 8. The highest strength was attained by No. 10 fly ash mortars, which indicates that this fly ash is by far the most reactive of those tested.

There are two apparent reasons for the superior performance of No. 10 fly ash. Comparison of the photomicrographs of the four fly ashes (figures 1, 2, 3, 4) shows that No. 10 fly ash is much finer and contains less carbon than the others. Table II also verifies this.

Correlations of various mortar properties with the amount of fly ash passing the No. 325 sieve were attempted. These correlations were not good, but they indicated a rough relationship of density, of optimum moisture, and of strength to fly ash fineness. However, by using loss on ignition as the independent variable, better correlations were established. Figure 9 shows the relation between maximum dry density of lime fly ash mortars and loss on ignition. The decrease in density with increase in carbon content cannot be charged to the difference in specific gravity between carbon and the Al_2O_3 and SiO_2 replaced by the carbon. A material balance comparison of the highest and lowest densities shows that specific gravity differences account for a density change of only about 2 pounds per cubic foot, and the true density difference is about 31 pounds per cubic foot. The difference is thought to be due to aggregating and porosity effects of the carbon.

The unconfined compressive strength after 45 days curing at 20°C. has a significant relation to loss on ignition (figure 10). The strength of lime fly ash mortars drops rapidly with increasing carbon content up to about



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Fig. 8. Comparison of compressive strengths of 2 inch by 2 inch specimens prepared from the four fly ashes and the indicated percentages of lime.





10 percent carbon, here the curve begins to level off. It is interesting to note that a fly ash with a carbon content near 30 percent probably would show little or no pozzolanic activity. Apparently fly ashes containing less than about 10 percent carbon are needed to produce lime fly ash mortars having high compressive strength. The advantage of using low carbon content fly ashes is evident, but additional work with more fly ashes is needed to establish an upper specification limit of carbon content.

Carbon in fly ash appears to harm pozzolanic reactivity and strength of lime fly ash mortars because of its adverse effects on reactive surface area and mortar density. Microscopic examinations of the fly ashes showed that the carbon tends to adhere to and partially cover the reactive surfaces, reducing the interfacial area available for pozzolanic reactions with lime. In addition to reducing reactive surface area of individual particles, carbon coatings also act as links between adjacent particles to produce a



Fig. 10. Unconfined compressive strength of lime, and fly ash mortars plotted as a function of fly ash loss on ignition. The lime contents of 2, 4, 6 and 8 percent are shown at the left of each curve.

porous aggregated structure. This structure, in addition to further reducing the available active surface area, reduces the compacted density attainable; the decreased density results in fewer and less intimate contacts between cementitious particles.

CONCLUSIONS

1. Carbon content as determined by loss on ignition seems to be a reliable indicator of the pozzolanic reactivity of fly ashes with lime. The upper limit of carbon content for good pozzolanic cementation appears to be less than 10 percent. Additional work with more fly ashes is necessary to establish a specific upper limit.

2. The amount of fly ash passing a No. 325 sieve decreases as carbon content increases and is, to a lesser extent, also an indicator of the pozzolanic reactivity of fly ash. Evaluation from this criterion was not as reliable as from loss on ignition.

3. The use of lime fly ash mortar strength tests for evaluating fly ash reactivity appears to give valid results. The results at both room temperature (20° C.) and at 60° C. are consistent. Curing at the higher temperature has the advantage of less time requirement for reactivity evaluation.

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POZZOLANIC REACTIVITY STUDY OF FLY ASH

by

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ABSTRACT

This paper presents some basic research on the nature of the pozzolanic reaction between lime and fly ash. Six fly ashes of various physical and chemical properties were investigated. The reaction between the fly ashes and calcium hydroxide was followed by means of an electroconductivity procedure. The reaction was studied at several temperatures and for several concentrations. The reaction products were investigated by means of X-ray diffraction and differential thermal analysis techniques.

Calcium hydroxide absorption on fly ashes, using electroconductivity procedures, gave essentially the same evaluation results as those obtained from compressive strength results. The influence of temperature on the reaction was most significant in the 20° to 60° C. range. A crystalline reaction product was detected, but this may be a secondary product.

An explanation is offered for the mechanism of the pozzolanic reaction. The rate limiting step of the reaction is explained on the basis of diffusion.

INTRODUCTION

Due to the interest in pozzolans for soil stabilization and other purposes, many investigations of certain materials showing pozzolanic activity have been made. (The term *pozzolanic activity* is used to denote the ease with which lime reacts at ordinary temperature with any natural or artificial siliceous or aluminous material in the presence of water to form stable, insoluble compounds with cementing properties.) For largely economic reasons fly ash has received much attention and study as a pozzolan. However, most of the investigations have dealt only with the strength contributing properties of the fly ashes and not with the fundamental chemical aspects of the pozzolanic reaction.

Basic studies of the pozzolanic activity of fly ashes and other pozzolanic materials are necessary for both practical and theoretical reasons. Explanations for the behavior of a given fly ash under given field conditions, such as density and temperature, are of practical importance. Theoretically, if the mechanism and the nature of the reaction products were better known, it might be possible to improve the reaction.

A few basic studies have been made of pozzolans^{9, 10, 13, 14}. Many of the basic concepts of pozzolanic activity postulated over twenty years ago have not been improved upon. One theory is that the reaction was a surface reaction between the dissolved lime and the solid resultant product and that equilibrium would not be reached except after long periods of time.

Many of the factors that influence the pozzolanic reaction have been recognized, but they have not been investigated thoroughly. Some factors of the pozzolanic reaction are: temperature, nature of the pozzolan, surface area, carbon content, alkali and sulfate content, carbon dioxide, hydrogen ion concentration, lime variables, moisture and time.

The identification of the reaction products of the pozzolanic reaction has been difficult, and the problem has not been completely solved. The problem is analogous to the identification of the hydration products of Portland cement. Because of the minute size of particles, the difficulty in isolating them, and the scarcity of necessary identification equipment, very few of the reaction products have been determined. Complex hydrated solid solutions of CaO, SiO_2 and Al_2O_3 are believed to be the major constituents.

PROPERTIES OF THE MATERIALS USED

Six samples of fly ash were selected for this study. Though these samples did not represent all of the property variations found with this material, the major factors were represented (tables I and II).

It will be noted that the mean diameter values are based on hydrometer and sieve analyses. The difficulty in determining the particle size distribution of fly ashes has been discussed⁷; on the basis of these conclusions, the mean diameters are probably close enough for the comparisons made in this study. Ignited samples were used because the combustible portion of fly ash has a specific gravity of one or less.

In addition to the physical and chemical analysis, a sodium hydroxide

	IABLE I.	I HISICAL FR	OFENTIES OF FD	ASIIES.	
Fly ash	Total* specific gravity	Ignited† specific gravity	Percent passing* 325 sieve	[`] Mean‡ diam., mm.	Specific§ surface, cm.²/qm.
10	2.56	2.65	93.1	.0305	3479
11	2.25	2.53	48.2	.0380	N.D.
12	2.42	2.55	81.0	.0310	3370
13	2.93	3.21	65.1	.0320	N.D.
14	2.67	2.96	94.3	.0265	3470
15	2.43	2.54	82.3	.0333	3649

DRODEDWIES OF FLV ASHES

Total sample as received was used.

"Total sample ignited in a muffel at 600°C. was used. Total sample ignited in a muffel at 600°C. was used. IBased on hydrometer and sieve analysis of ignited samples. \$Supplied by the Robert W. Hunt Co., of Chicago, Illinois. Based on a gas adsorption method, total samused.

Chemical analysis*			Fly a	ish no.		
reported as	10	11	12	13	14	15
C	3.20	27.67	10.18	2.90	2.10	15.59
SO_3	3.04	0.41	1.47	2.93	2.00	1.86
CaO	7.30	2.52	5.34	11.22	8.36	6.89
MgO	0.43	1.16	0.77	2.65	0.52	0.85
$\rm Fe_2O_3$	19.30	13.41	21.23	10.75	21.20	19.63
Al_2O_3	20.10	13.23	18.39	29.25	22.92	18.19
${ m SiO}_2$	43.40	39.18	41.16	39.20	38.90	35.94
H_2O	0.30	N.D.	N.D.	N.D.	0.17	N.D.
pH†	11.50	12.30	12.10	11.80	11.60	11.90

TABLE II. CHEMICAL PROPERTIES OF FLY ASHES USED.

*Reported as percent by weight. †Based on 10 gm. of total sample in 25 ml. of water.

solubility test similar to that used by the Bureau of Reclamation was performed on the fly ashes (table III).

Because of the impurities in commercial limes, reagent grade calcium hydroxide was used to make up all of the lime solutions used.

TEST PROCEDURE

Absorption methods were explored to determine the rate of reaction between the lime and the fly ashes. An electroconductivity method was used to determine the rate of calcium absorption. This method enabled the reaction rate of a single sample to be traced without altering the solution or the material being tested. At certain periods the absorption of calcium was checked by titration procedures.

The test procedure was to place a known amount of material along with 90 ml. of lime solution of known concentration in a 10×1 inch glass test tube. Each test tube was sealed with a two-holed rubber stopper equipped with two glass tubes. One tube reached about 7 inches into the test tube so that the lime solution could be withdrawn, tested, and returned to the test tube. The other tube was rather short and acted as a vent while the lime

TABLE III. PERCENT REDUCTION IN ALKALINITY OF THE FLY ASHES.

Fly ash no.	Percent reduction in alkalinity, R_a
10	62.80
11	39.36
12	50.60
13	61.34
14	59.79
15	56.17

solution was being withdrawn and returned to the test tube. Both tubes extended about one-half inch above the top of the stopper. A piece of rubber tubing connecting the upper ends of the glass tubes served to keep the test tubes air tight during the reaction period. During testing periods the rubber tube served as a connector between the test tube and the conductivity pipettes. The vent tube was connected to a calcium chloride tube containing barium hydroxide to prevent the formation of calcium carbonate in the solution (figure 1).

Because of the decrease in solubility of $Ca(OH)_2$ with increased temperature and alkalinity, the concentration of $Ca(OH)_2$ solutions used were kept well below the maximum solubility values. This was done to insure against the precipitation of $Ca(OH)_2$ in the test tube during the reaction period. Concentrations of about 1.1, 1.0, 0.9 and 0.8 gm. per liter were used for reaction temperatures of 20°, 40°, 60° and 80°C. respectively. At first deionized water was used to make up the solutions, but later it was found that more satisfactory solutions could be made by double filtering $Ca(OH)_2$ solutions made with distilled water. Filtering was necessary to remove small amounts of $CaCO_3$ which was formed by calcium combining with small amounts of CO_2 in the distilled water. After being filtered, the concentrations of the $Ca(OH)_2$ solutions were checked by titration methods.

Test tubes were exposed to concentrated $Ca(OH)_2$ solutions at the specified temperature before being used. A check of the reactions by electroconductivity methods showed very little reaction between the pre-treated glass and $Ca(OH)_2$.







The samples were placed in constant temperature baths and ovens. Constant temperature baths were used to maintain reaction temperatures of $20\pm1^{\circ}$ C. and $40\pm1^{\circ}$ C. Ovens were used to maintain the higher temperatures of $60\pm1^{\circ}$ C. and $80\pm1^{\circ}$ C.

Cementation of particles of the reactive material had to be prevented so that the complete sample would remain exposed. To prevent cementation the samples were rodded and shaken frequently during the testing period.

Conductivity readings were taken at specified intervals with a pipette type conductivity cell (Cenco 700-72). The cell was partially encased in a small water tight plastic box (figure 1). Water from the constant temperature bath was pumped through the box to insure a constant temperature during the conductivity reading operation. Samples in the constant temperature baths were tested at their respective temperatures, and samples from the ovens were placed in the $40\pm1^{\circ}$ C. bath and tested at this temperature.

Conductivity readings were taken using a null point conductivity bridge (Industrial Instruments Type RC 16B). Readings were taken at 1000 cycles per second to prevent reactions at the electrodes. All air coming in contact with the solution passed through calcium chloride tubes containing barium hydroxide to prevent the formation of calcium carbonate during the testing period. Immediately after testing, the 60° and 80° C. samples were placed in their respective ovens. About two hours was required for the oven samples to reach the bath temperature and be tested and returned to the oven.

Cell constants of the conductivity pipettes were determined using KCI solutions of known specific conductance. Curves giving the relationship between measured resistances and $Ca(OH)_2$ concentrations were prepared for each testing temperature. Specific conductivity data for $Ca(OH)_2$ solutions were utilized for these curves¹⁰.

For a given reaction temperature, three test tubes of each material were prepared. These samples were used to obtain a set of average readings for the initial reaction period and to provide samples for testing the solutions at given time intervals, usually 15, 30 and 45 days after being exposed to the solid material. At the end of the time intervals, a sample was removed and filtered to separate the solution from the solid material. The lime concentration of each filtered solution was determined by means of Versenate titrations. The solid material was oven dried at 60°C. in contact with barium hydroxide to prevent the formation of calcium carbonate.

DISCUSSION OF RESULTS

Three gram samples of each fly ash were allowed to react with $Ca(OH)_2$ solutions at 20°C. for a period of one year. The absorption of lime was followed by electroconductivity methods and checked by Versenate titra-

tions at 90, 180 and 365 days. The values obtained by these two methods checked to within 5 percent (figures 2, 3).

Fly ash 10 absorbed the most lime over the 365 day test period. The differences between the fly ashes were less apparent at the end of the test period than in the initial stages. The absorption by fly ash 11, the high carbon fly ash, was particularly slow during the initial period, but at the end of 365 days it had absorbed approximately the same amount of lime as samples 12, 13, 14, and 15. The major differences between the fly ashes at



Fig. 2. Absorption of calcium by fly ash samples 10, 11 and 12 at 20°C. over a one year period.



Fig. 3. Absorption of calcium by fly ash samples 13, 14 and 15 at 20°C. over a one year period.

this temperature were shown most readily during the initial lime absorption testing period of about 45 days.

The negative values of lime absorption which were observed can be explained by the solubility of compounds in the fly ash and perhaps by cation exchange. Sodium, magnesium and calcium compounds reported in fly ashes are soluble in partially saturated $Ca(OH)_2$ solutions. Alkali solutions cannot exist in equilibrium with saturated lime solutions¹². Fly ash 13 has the largest amount of calcium and magnesium compounds, 13.87 percent (reported as oxides); the greatest amount of negative absorption is observed with sample 13 (table II). Sample 11 has the smallest amount of calcium and magnesium oxides, 3.68 percent, and only a slight amount of negative absorption was observed for sample 11. Samples 10, 12, 14 and 15 had approximately the same amounts of calcium and magnesium compounds, 7.73, 6.11, 8.88, and 7.45 percent respectively, and these have intermediate negative absorption values.

A comparison of the absorption results with the compressive strength results shows that the initial 45 day absorption results seem to agree with



Fig. 4. Unconfined compressive strengths of four fly ash mortars with 8 percent $Ca(OH)_2$ cured at 20°C.⁵ Strengths are for 2 inch by 2 inch specimens at near standard Proctor density which were moist cured at 20°C. for indicated times, then immersed in water 24 hours before testing.

the compressive strength results (figure 4). The compressive strength and absorption curves of some are very similar in shape. It is evident that fly ash 10 is the best pozzolan on the basis of compressive strength and lime absorption. The second best pozzolan appears to be sample 12; this conclusion is reached from both the absorption and the compressive strength results. Sample 15 had a rather flat and constant slope in both absorption and compressive strength curves. Sample 11 did not achieve an apparent equilibrium until after about 300 days. The very shallow constant slope of the absorption curve for sample 11 agrees very well with the slope of the strength curve for this fly ash.

From the compressive strength and absorption data it appears that there is a quantitative or semi-quantitative relationship between calcium absorption by fly ash and its compressive strength in lime fly ash mixtures. By comparing the maximum slope of the compressive strength curves with the maximum slope of the absorption curves in the figures previously discussed, a relationship was obtained (figure 5). The relationship is significant in that it indicates a correlation between absorption and strength; however, this relationship is likely to vary with temperature and concentration factors. If a relationship is to be established, it should be on the basis of standardized tests.



Fig. 5. Maximum rate of compressive strength development versus maximum rate of $Ca(OH)_2$ absorption.

INFLUENCE OF TEMPERATURE

Each sample of fly ash was tested by the lime absorption method at 20° , 40° , 60° and 80° C. (figures 6 through 13).

Although 20°C. absorption curves for the six fly ashes have been discussed previously, the curves presented in figures 6 and 7 represent another set of test data. One day readings are shown in these curves. It will be noted that there was an apparent initial absorption of calcium before the negative absorption that was evident in figures 2 and 3. There are two plausible explanations for the initial up-take of calcium. Possibly it is due to an ion exchange reaction, such as calcium ions replacing other ions in the fly ash. Another possibility is that it is due to charge deficiencies in the fly ash as a consequence of its non-crystalline, glassy nature.



Fig. 6. Absorption of calcium at 20°C. by fly ash samples 10, 11 and 12 (3 gm. samples).



Fig. 7. Absorption of calcium at 20°C. by fly ash samples 13, 14 and 15 (3 gm. samples).



The influence of temperature on the rate of absorption of $Ca(OH)_2$ by the fly ashes was very pronounced with all fly ashes. After initial reac-

Fig. 10. Absorption of calcium at 60°C. by fly ash samples 10, 11 and 12 (3 gm. samples).
tions, the rate of absorption increased with temperature in all cases. The pozzolanic reaction or reactions observed were endothermic reactions, that is, fly ash $Ca(OH)_2$, and H_2O pozzalonic reaction products. Increasing the



Fig. 11. Absorption of calcium at 60°C. by fly ash samples 13, 14 and 15 (3 gm. samples).



Fig. 12. Absorption of calcium at 80°C. by fly ash samples 10, 11 and 12 (3 gm. samples).



Fig. 13. Absorption of calcium at 80°C. by fly ash samples 13, 14 and 15 (3 gm. samples).

amount of heat drives the reaction faster to the right. This increase in reaction rate due to an increase in temperature has been termed the Arrhenius effect⁶.

It will be noted that the periods of initial and negative absorption decrease as the temperature increases. At 60° C. very short periods of negative absorption were observed. Neither initial reactions nor negative absorptions were evident during the 80° C. test period. All curves at 80° C. have the same shape, and the maximum ordinates are essentially the same. High temperature results are of little use for the selection of desirable pozzolans because of the rapidity of the reaction.

The curves shown in figure 14 illustrate the over-all effect of the temperature range used on the six fly ashes studied. Because of the initial reactions and solubility effects observed, the times given for one-half of the original $Ca(OH)_2$ concentrations to be depleted are not true half-lives. It is possible to estimate the effect of temperature on the pozzolanic reaction for the fly ashes beyond the range of temperature used in this study. Relatively speaking, little was gained by raising the reaction temperature above 50°C. However, between 20°C. and 50°C. the influence of temperature on the reactions was quite noticeable. Only at temperatures less than 50°C. was it possible to distinguish between the fly ashes from their absorption rates. The curves shown in figure 14 asymptotically approach time infinity as the temperature decreases below 20°C. For practical purposes, at temperatures below 20°C. most of the fly ashes can be assumed to be almost non-reactive.

Compressive strengths of lime fly ash mortars at 60° C. using four of the six fly ashes are shown in figure 15. The strength differences between the fly ashes at this temperature are more pronounced than at 20° C. (figure 4).



Fig. 14. Effect of temperature on time required for one-half of Ca(OH)2 to be absorbed.

From the figure it is noted that samples 10 and 11 are respectively the most satisfactory and the most unsatisfactory pozzolans; this is also true when the mortars were cured at 20° C. Unlike the compressive strengths at the 20° C. curing temperature, all samples reached about 90 percent of their strengths in about seven days. From figures 10 and 11, it can be seen that the major portion of the lime absorption occurred within the first ten days. The rates of initial strength gains correspond with the rates of initial calcium absorption for each fly ash. Though the maximum ordinates of the absorption curves are nearly the same, the maximum ordinates of the compressive strength curves are considerably different. It is therefore evident that there is a relationship between the absorption of lime and the compressive strength, but the total strength development of lime pozzolan mortars depends on other factors in addition to the pozzolanic reaction.



INFLUENCE OF PARTICLE SIZE ON THE REACTION

The influence of particle size on the reaction was studied by using mate-

Fig. 15. Unconfined compressive strengths of four fly ash mortars with 8 percent Ca(OH)₂ cured at 60°C.⁵ Strengths are for 2 inch by 2 inch specimens at near standard Proctor density which were moist cured at 60°C. for indicated times, then immersed in water 24 hours before testing.

rial passing the number 200 sieve (.074 mm.) and the number 325 sieve (.044 mm.) from fly ashes 10 and 11. Fly ash 10 was used because all tests indicated it was the most reactive fly ash of the six tested, and fly ash 11 was used because all tests indicated it was unsuitable as a pozzolan. $Ca(OH)_2$ solutions used at each temperature were of equal concentrations. Three gram samples were used.

Figures 16 and 17 are two sets of curves given to illustrate the influence of particle size on $Ca(OH)_2$ absorption at a given temperature. Practically speaking, there was little difference between the rates of absorption and the maximum amounts of absorption for the different fractions at a given temperature.

An increase in reaction rate with increasing surface area was not evident in these tests; this may be explained on the basis of another property of the fly ashes. Most of the larger particles in fly ashes are of unburned coal



Fig. 16. Influence of grain size of fly ash sample 10 on calcium absorption at 40°C.



Fig. 17. Influence of grain size of fly ash sample 11 on calcium absorption at 40°C.

and coke, which is reported as loss-on-ignition in table IV. From this table the decrease in organic content with grain size is very evident. The larger fly ash particles are not very active. Also, the larger particles have low specific gravity, less than 1.0. Hence, there was little change in the amount of active surface area present when the particle size samples used were compared on the basis of weight. Although the presence of organic mateterial had little effect on the chemical activity of the fly ashes, there is reason to believe that it influences the compressive strength results.

INFLUENCE OF THE LIME TO FLY ASH RATIO

The influence of the lime to fly ash ratio on the reaction was studied by decreasing sample weights one-half while keeping $Ca(OH)_2$ concentrations constant. The curves obtained had essentially the same characteristics as the 3 gram sample curves. However, at any time interval the amount of $Ca(OH)_2$ absorbed per gram by the 1½ gram samples was nearly twice that absorbed by the 3 gram samples. The influence of concentration on the reaction will be discussed on the basis of diffusion later.

Percent increases in compressive strengths due to doubling the amount of $Ca(OH)_2$ at two temperatures are shown (tables V and VI). At 20°C. negative values were obtained for some of the 7 and 14 day test periods. This probably indicates that cementation had not taken place to a significant extent during the test period and that the $Ca(OH)_2$ decreased the shear strength by acting as a lubricant during the initial period. Although all of the 45 day percentage increases in compressive strengths are positive, they are erratic and do not correspond to the increases in absorption. The compressive strengths of the lime fly ash mixtures increased with increas-

TABLE I	v.	INFLUENCE	\mathbf{OF}	PARTICLE	SIZE	0'N	LOSS	0N	IGNITION.

Size fraction finer than	Percent loss Fly ash 10	on ignition Fly ash 11
0.420 mm.	3.20	27.67
$0.074 \mathrm{~mm}$.	2.34	19.55
0.044 mm.	2.11	10.32

Table V. Percent increase in compressive strength at the end of various test periods due to increasing Ca $(OH)_2$ content from 2 to 4 percent at 20° and 60°C.⁵

Fly ash			Per 20°	cent inci C.	rease in co	mpressive s	trength 60	°C.	
no.		· 7	Test peri 14	od, days 28	45	7	Test per 14	iod, days 28	45 ·
10		-31.1	24.7	41.6	62.0	41.6	54.4	56.7	19.8
11		0.8	2.7	2.2	15.1	43.3	38.0	45.2	36.4
12		-51.2	0.2	6.2	47.2	44.1	49.1	36.5	46.1
15		4.3	7.5	6.3	25.8	15.7	6.3	23.3	15.5

TABLE VI. PERCENT INCREASE IN COMPRESSIVE STRENGTH AT THE END OF VARIOUS TEST PERIODS DUE TO INCREASING CA $(OH)_2$ content from 4 to 8 percent at 20° and 60°C.⁵

		Per	rcent incr	ease in cor	npressive st	trength		
Fly ash sample	#	20° Test peri	C. od, days	4 E .	- 7	60 Test per	°C. iod, days	45
no.	1	.14	28	40	7	14	48	49
10	-3.7	-10.8	6.0	11.0	6.9	17.6	19.0	36.6
11	-4.5	9.8	15.8	30.0	11.7	21.2	17.7	27.3
12	8.6	34.4	12.5	18.2	25.4	34.4	78.0	86.0
15	60.8	32.0	33.4	40.3	45.9	39.9	39.6	73.1

ing amounts of $Ca(OH)_2$, but the amount of increase depended on other factors in addition to absorption.

REACTION PRODUCTS

After fly ash samples had been tested with the absorption test procedure, they were removed from the solutions. The solid material was filtered from the solutions and dried in the presence of barium hydroxide to prevent carbonation. After being dried, the fly ash samples were subjected to optical, differential thermal analysis and X-ray diffraction procedures to determine the nature of the reaction products formed.

No differences between treated and untreated samples could be ascertained by the petrographic microscope. Previous research in the field of cement chemistry has also indicated that petrographic techniques are of little value for this type of work because of the small amounts of reaction products and their minute colloidal size.

For the most part, the differential thermal analysis results were not as satisfactory as the X-ray diffraction results. The unreacted fly ash in the absorption samples partially masked the peaks of the reaction products. Also, the lack of established data about the reaction products as determined by this method tended to limit the applications of the apparatus.

X-ray diffraction patterns of the $Ca(OH)_2$ treated fly ash samples were compared with patterns for untreated samples. All samples were treated with ethylene glycol before X-ray examination.

Fly ash samples that had been exposed to $Ca(OH)_2$ in solution for 365 days at 20°C. yielded X-ray diffraction patterns that indicated a reaction product. Significant peaks were obtained for the six fly ashes tested (figure 18). Dotted lines indicate the X-ray diffraction pattern obtained from untreated samples of the same fly ash. A reaction product was formed and it is crystalline, as shown by the definite peaks indicating an orderly arrangement of atoms. Two distinct spacings are show (figure 15), one at 12.6Å and another at 12.3Å; these spacings may be transitional as evidenced by the broad peaks obtained from samples 11 and 12. In addition to the peaks in figure 18, faint peaks were obtained for samples 10 and 14 at 29°. This corresponds to a spacing of about 3.08Å. Carbonate peaks that were obtained from the untreated samples were not obtained from the treated samples. $Ca(OH)_2$ peaks were not obtained from the X-ray diffraction of the treated samples.





The X-ray diffraction pattern obtained for the 365 day samples is similar to the two major peaks of calcium silicate hydrate I (table VII). Calcium silicate hydrate I has been studied and reported by several investigators in connection with Portland cement hydration products^{2, 11}. It is frequently observed at temperatures below 100°C. with various molar ratios of CaO to SiO_2^2 . The diffuse scattering of X-rays by calcium silicate hydrate I has been attributed to water. Previous research indicates that it is a layer type mineral similar in structure to montmorillonite and other swelling clay minerals². The basal reflections vary with the amount of water between the layers. Treatment of the samples with ethylene glycol permits a shift in the *C* dimension of the mineral due to the absorption of the organic molecules between the individual layers. This usually results in sharper X-ray diffraction peaks.

Examination of X-ray diffraction patterns for the fly ash samples from the higher temperature absorption tests did not reveal a crystalline reaction product. Also, reaction product peaks were not observed in the diffraction patterns for 20°C. absorption samples that were removed from the $Ca(OH)_{2}$ solutions at 45 days and less. All absorption test results indicated that the reaction period for most samples was over at the end of 45 days. Normally under hydrothermal conditions, crystals grow in thickness with time; and the largest crystals grow, though more slowly, at ordinary temperatures. Hence, it appears that a crystalline product did not form as the reaction proceeded and that the crystalline product developed from a non-crystalline reaction product. This intermediate product is probably a gel. Aside from the regular atomic order in the calcium silicate hydrate crystallites, there probably is little difference between the crystallites and the amorphous gels. The size of the particles that make up some classical gels are well within the range of the calculated dimensions of calcium silicate hydrate I².

TABLE V	ΊΙ.	Powder	X-ray	DIFFRACTION	PEAKS	FOR	CALCIUM	SILICATE	HYDRATE	I.11
---------	-----	--------	-------	-------------	-------	-----	---------	----------	---------	------

d spacings in Å	Relative intensity
9-14 (highly variable)	. 10
3.06	10
2.81	. 8
1.83	8
1.67	4
1.53	2
1.40	4
1.17	1
1.11	2
1.07	1

FACTORS AND MECHANISM OF THE REACTION

The presence of cementitious reaction products was proved indirectly by the compressive strength tests and the absorption tests. Apparently the compressive strength of lime fly ash mortars depends on the amount and number of contacts of the cementitious material. Coke and other organic, non-reacting materials prevent contacts of the cementitious material that forms on the surface of the pozzolan.

If the desired cementitious product forms on the surface, it follows that for any more reaction product to be formed, the necessary calcium must pass through the reacted layer to react with the enclosed pozzolan. Thus, the mechanism of the pozzolanic reaction is one of simultaneous diffusion and chemical reaction of the calcium. The activity of the reacting material is limited by the rate of diffusion of calcium through the reaction product.

The mathematical theory of diffusion in isotropic substances^{1, 4} is based on the hypothesis that the rate of diffusion through a unit area of section is proportional to the concentration gradient normal to the section, that is,

$$\frac{\mathrm{d}\mathbf{Q}}{\mathrm{dt}} = -\mathrm{D}\,\frac{\mathbf{c}}{\mathrm{x}}$$

where $\frac{\mathrm{dQ}}{\mathrm{dt}}$ is the rate of transfer per unit area of section, c is the concentration of the diffusing substance, x is the space coordinate measured

normal to the section, and D is called the diffusion coefficient.

It is assumed that the rate limiting step is diffusion of calcium through the reaction zone. The reacting material is assumed to be in the form of a sphere. It is also assumed that the thickness of the reaction zone is negligible in comparison with the diameter of the reacting material; so the surface can be considered a plane, and the following mathematical development can be made.

Let $\mathbf{x} = \text{thickness}$ of reacting zone

Q = amount of Ca (OH) $_2$ which has reacted/unit area

V = molar volume of the product

C =concentration of Ca(OH)₂ at any time

 $C_{\circ} = initial concentration of Ca(OH)_2$

 $R = total amount of Ca (OH)_2$ reacting

S = specific surface of solid

If $\mathbf{x} = \mathbf{Q} \nabla$

then $\frac{\mathrm{d}\mathbf{Q}}{\mathrm{d}\mathbf{t}} = \frac{\mathrm{D}\mathbf{C}}{\mathbf{x}} = \frac{\mathrm{D}\mathbf{C}}{\mathbf{Q}\mathbf{V}}$

The amount removed by reaction will be V ($C_o - C$). By reaction stoichiometry

 $V (C_o - C) = a Q S$

where a is a "ratio of small whole numbers." Hence

$$\mathbf{C} = \mathbf{C}_{o} - \frac{\alpha \mathbf{Q} \mathbf{S}}{\mathbf{V}} = \mathbf{C}_{o} - \beta \mathbf{Q}^{+}$$

where $\beta = a \frac{S}{\nabla}$ hence $\frac{dQ}{dt} = \frac{D(C_o - \beta Q)}{Q\nabla}$

thus $\frac{\text{Dt}}{\nabla} = \frac{-Q}{\beta} \frac{-C_{\circ}}{\beta^2} \frac{\ln (1 - \frac{\beta Q}{C_{\circ}})}{\ln C_{\circ}}$. Expanding the equation by MacLaurin's power series the equation becomes

$$\frac{\mathrm{Dt}}{\mathrm{\nabla}} = \frac{\mathrm{Q}^2}{\mathrm{C_o}} \left[0 + \frac{1}{2} \left(\frac{\beta}{\mathrm{C_o}} \right)^{\mathrm{Q}}_{\mathrm{3}} + \cdots + \left(\frac{\beta}{\mathrm{C_o}} \right)^{\mathrm{n-2}} \frac{\mathrm{Q}^{\mathrm{n-2}}}{\mathrm{n}} + \cdots \right]^{\mathrm{r}}$$

If $\frac{\beta Q}{C_{\circ}}$ is less than 0.1 then all terms greater than the third term are negligible. Then

$$rac{ ext{Dt}}{ extsf{V}}\!=\!rac{ ext{Q}^2}{2 ext{C_o}}$$

and

$$Q = \left(\frac{2C_{o}D}{V}\right)^{\frac{1}{2}} (t)^{\frac{1}{2}}$$

 \mathbf{If}

and

 $\mathrm{K=}\left(rac{2\mathrm{C_{o}D}}{\mathrm{V}}
ight)^{rac{1}{2}} \mathrm{R=QS}.$

The following relationship is obtained

$$\mathbf{R} = \mathbf{KS} \ (\mathbf{t})^{\frac{1}{2}}.$$

Assuming that the reacting particles are spherical in shape and heterogeneous, the following equation may be used for the specific surface.

$$\mathbf{S} = \frac{4 \pi \mathbf{r}^2}{\frac{4}{3} \pi \mathbf{r}^2 \lambda} = \frac{3}{\lambda \mathbf{r}}$$

where λ and r are, respectively, the specific gravity of the material and the mean radius of the reacting matter. Hence, the following relationship can be obtained

$$R = \frac{3 K (t)^{\frac{1}{2}}}{\lambda r}$$

This equation is valid only for cases where the concentration is high. The K values given in this report are for the initial portion of the reaction. The relationship for the lower concentrations can be developed using the adjustable parameters β and D instead of K.

Absorption data for the fly ashes in $Ca(OH)_2$ solutions satisfied the relationship given previously since linear plots of R versus \sqrt{t} were obtained until the concentration of $Ca(OH)_2$ dropped to about 0.1 gm./liter. From the slopes of these plots, average values of K were calculated for the fly ash samples (table VIII).

The K values indicated are not diffusion coefficients, but they do indicate the trend of the diffusion coefficients. It will be noted that the rate of diffusion increases with temperature. The relationship

$$\log \mathrm{KC_o}^{\frac{1}{2}} = \mathrm{C_1} + \frac{\mathrm{C_2}}{\mathrm{T}}$$

where T is the absolute temperature, may be used to express the influence of temperature on the rate of diffusion. Secondly, the rate of diffusion was increased by decreasing the amount of reacting sample while keeping the concentration of the Ca(OH)₂ constant.

It will be further noted that the better fly ashes, as determined by the other tests, also give the higher K values. Probably this results from the fact that there is normally more reactive material in these samples and they have a higher specific surface area.

CONCLUSIONS

The rate of compressive strength development by lime fly ash mortars is directly related to the rate of lime absorption of the fly ash. The greater the rate of lime absorption, the greater the rate of compressive strength development. The rate of lime absorption is limited by the rate of diffusion of the calcium through the reaction product. The rate of diffusion, which is increased by increasing the temperature and the lime concentration.

TABLE VIII. K CONS	TANTS FOR	FLY ASI	I SAMPLES.
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Fly ash sample	Solution temperature,	Constan cm.²/day	t K x 10 ⁷ /gm./liter
no.	°C.	3 gm. sample	$1\frac{1}{2}$ gm. sample
10	20	5.88	16.52
	40	7.37	21.50
	60	9.80	37.40
	80	14.15	55.40
11	20	1.43	3.25
	40	3.02	9.60
	60	6.10	20.60
	80	8.53	27.60
12	20	3.43	7.64
	40	4.66	16.00
	60	6.40	20.90
	80	9.10	25.60
13	20	0.00	0.00
	40	4.91	19.58
	60 .	9.98	24.90
	80	12.00	31.00
14	20	3.48	14 45
	40	518	18 20
	60	8.05	32 50
	80	11.25	49.80
15	20	3.95	14,10
	40	.7.02	18 45
	60	9.98	29.80
	80	14.03	4910

varies with the type of fly ash used. At temperatures less than 20°C. most fly ashes may be considered non-reactive.

The source of the strength of lime fly ash mortars is the reaction products that form as the result of the pozzolanic reaction. From X-ray diffraction studies, it seems that a crystalline product does not form at first, but develops from a non-crystalline reaction product. The initial product is probably a gel. The final crystalline product is believed to be calcium silicate hydrate I, a reaction product that has been found in set Portland cement pastes.

Unburned organic material that is found in most fly ashes is non-reactive with calcium hydroxide, and its presence seems to indicate a fly ash of coarse grain size in both the organic and inorganic phases. Since the total mortar strength developed depends on the number of contacts of the cementitious reaction products, the organic material breaks the continuity of this system and thus decreases the total strength.

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DISCUSSION

Z. C. Moh, *Massachusetts Institute of Technology*.—In the past few years considerable increase in the interest in using pozzolanic materials, both natural and synthetic, has led to the recognition of the importance of understanding the nature of the pozzolanic activity and the reaction prod-

ucts. The authors have made an important contribution in this respect. However, some of the conclusions described in the paper seem to be somewhat inconsistent with the data presented. The writer wishes to discuss these inconsistencies.

The authors interpreted the products of the reaction between lime and fly ash after one year as calcium silicate hydrate I (CSH-I) based on their X-ray diffraction pattern. However, the evidence, as shown in figure 18, is not conclusive. The X-ray patterns reveal only the presence of one line, the 12.6 Å line, which is only one of the strong lines of CSH-I (table IX). The two strongest lines of CSH-I, 3.06 Å and 2.80 Å^{15, 16, 19}, are not present in the authors' data. Furthermore, the 17.3 Å of fly ash No. 10 does not correspond with any calcium silicate hydrate pattern reported in the literature. Moreover, CSH-I has a very large exothermic peak at about 830° to 900°C. on the DTA pattern¹⁷. The authors did not indicate such.

TABLE IX.	X-RAY DIFFRACTION PATTERNS	OF CALCIUM SILICATE I.
References	d spacing in Å	Relative intensity
Taylor ¹⁵	10.0 - 11.5 3.03 2.80 1.81	Strong Very strong Medium strong Medium strong
Bogue ¹⁹	13.72 3.06 2.80	- 100 100 100
Bernal ^{16*}	10 - 15 3.1 2.8	$\begin{array}{c} 7\\10\\9\end{array}$

*Interpreted from line diagram.

According to Bogue¹⁶, Taylor¹⁹, and others, the calcium silicate hydrates formed in a lime solution of concentration well below saturation are low in lime. If one accepts the authors' interpretation of the end products as CSH-I, this naturally would be the low lime hydrous silicate, that is, CaO·SiO₂ ag. The maximum lime absorption after one year at 20°C., as reported in the paper, was 2.5 gm. $Ca(OH)_2/gm$. of fly ash x 10² (fly ash No. 10 in figure 2). This value is equivalent to 0.0189 gm. CaO/gm. of fly ash. With CaO-to-SiO₂ ratio of one, this means only 0.0435 gm./gm. of the fly ash was reacted (fly ash No. 10 has 43.4 percent SiO₂); that is, only 4 percent of the pozzolan participated in the reaction in a period of one year. Furthermore, in the authors' experiments the fly ash was prevented from cementation. The total surface area exposed to the reaction would undoubtedly be much greater than in a compacted lime, fly ash mortar. In other words, if the experiments described really represent the type of reaction postulated by the authors in a lime, fly ash mixture, it is inconceivable that such a small percentage of reactants was able to bind all the fine particles providing fairly high compressive strength. (Although there are no strength data of mixtures with 2 to 4 percent lime presented in the

paper, however, it can be deduced from figure 4 and from tables V and VI that the one year strength of such mixtures would be fairly high.)

Furthermore, the decreases in strength $(20^{\circ}C.)$ at 7 and 14 days by increasing the calcium hydroxide contents in the mixture (tables V and VI) contradict the absorption data discussed. The increase in absorption by lime at all curing ages would indicate to the writer an increase in the total amount of reaction product at all curing ages. Hence, higher strength would be expected—as stated by the authors: "The strength of lime, fly ash mortars is dependent on the amount and number of contacts of the cementitious materials." Therefore, it seems likely that the absorption test is not adequate to account for the proposed mechanism.

The authors explained the reaction rate on the basis of diffusion. It is clearly stated by the authors that rate of diffusion is a function of $Ca(OH)_2$ concentration. Therefore, at higher lime content the rate of calcium ion diffusion is faster; the reaction rate between lime and fly ash should then be faster. In other words, the strength development should also be faster at high lime content than at low lime content, but the data in tables V and VI do not agree with this explanation.

The negative absorption and also the initial absorption at temperatures above 20° C. cannot be satisfactorily explained by exchange reaction or due to soluble components of the fly ash as stated by the authors. Otherwise, the conductivity measurement would not represent the lime concentration in solution and definitely would not agree with titration data if titrations were carried out at those ages. The reverse absorption phenomena may be explained by the view of uneven distribution of ions on the fly ash particles as described for cements¹⁸.

The majority of large particles in fly ashes are composed of unburned coal and coke which generally have little effect on the chemical reactivity of the pozzolans, as stated by the authors. Yet, as shown in figure 6, sample No. 10 has a pronounced negative absorption by using the entire sample, while the No. 200 and No. 325 fractions exhibited positive absorption (the sample has 93 percent passing No. 325). It would seem that a more plausible explanation for this difference of 8 gm. of Ca(OH)₂ absorption per gm. of fly ash x 10³ is due to the small percentage of the coarse fraction.

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DISCUSSION

R. J. Leonard and D. T. Davidson, *Closure*.—Although Mr. Moh's discussion does not make known any new information about the lime fly ash pozzolanic reaction, it is appreciated for it offers the authors an opportunity to clarify and discuss further some of the major points of their study.

Although X-ray spectrometer curves were obtained for the absorption test fly ashes for a range of 2θ of 2 to 100° , only that portion showing the interesting peaks at 12.6 Å and 17.3 Å were shown in figure 14. The other significant peaks that were observed at 3.08 Å and 2.81 Å were reported in the text of the paper. The data for CSH(I) reported in table VII of the paper are more recent than that presented by Mr. Moh, and it may be significant to give Dr. Lea's addendum to these data, "This is the data for the poorly crystalline material. The long spacing can vary considerably and may also be undetected." It should be noted that the peaks appear to be transitional; this is particularly evident with sample 11. Sample pre-treatment and the small amount of reaction product available in any particular sample probably play major roles in the location of the first peak. On the basis of the evidence of the 12.6 Å to 17.3 Å peaks and the presence of the second peak, it seems reasonable to identify one of the reaction products as CSH(I).

The size of the DTA exothermic peak at about 830 to 900°C. reported by Kalousek²² for CSH(I) is a function of the mass of this reaction product. Since only a small amount of reaction product was available for each fly ash sample this affected the DTA results. Peaks were observed at 830 to 900°C, but they were not very significant. Also, in the large amounts of unburned carbonaceous materials some of the fly ashes caused some drift despite the fact that a nitrogen atmosphere was used in the furnace. In addition to Kalousek's²² work, Greenberg²¹ and others have done DTA work on CSH(I); however, most researchers prefer the use of X-ray diffraction procedures for the examination of crystalline minerals of this type. None of these reports include information on the reaction of lime with fly ash and the resulting products; also most of the reports dealt with pure mixtures of Ca(OH)₂ with silica gel or minerals that had been exposed to solutions of high concentrations of Ca(OH)₂ for long periods of time.

At the 38th Annual Meeting of the Highway Research Board, Benton²⁰ of the Bureau of Reclamation, presented some information on the reaction products of pozzolans. Mr. Benton indicated that there were two reaction products of the lime, fly ash reaction; one product is CSH (I) and the other is a calcium alumino hydrate. The latter product may or may not contribute to the strength of the lime and fly ash mixture.

The lime absorption reported in figure 2 should not be misconstrued to be the maximum amount of $Ca(OH)_2$ that these fly ashes can possibly absorb in that length of time and at that temperature. The curves shown in figures 2 and 3 represent the amounts absorbed from an original

 $Ca(OH)_2$ solution of slightly less than saturated concentration. At 20°C. a saturated $Ca(OH)_2$ solution is 0.165 gm. per 100 gm. of water²³. The concentrations of the solutions used were kept below the saturted concentration because of the presence of soluble constituents in the fly ashes. Any precipitation of calcium in the solution would make the absorption test meaningless.

Since the pozzolanic reaction is between calcium and the reactive constituents that occur on the surface of the fly ash particles, total exposure of the fly ash surface area is necessary for the absorption test results to be significant. Although No. 10 fly ash has 43.4 percent SiO_2 not all of this SiO_2 will be available for immediate reaction. The amount reacted for a particular pozzolan will be dependent on the surface area of the fly ash, the rate of diffusion of calcium through the reaction product that forms on the surface of the particles and the availability of calcium in the system. Lea²⁴, among others, has estimated that under normal conditions a pozzolan will not react in a year with more than about 20 percent of its weight of lime.

Due to concentration restrictions only a small amount of the fly ash was reacted in the absorption tests; nevertheless, there was enough calcium present to cause cementation of the particles. The samples were stirred to prevent this cementation from forming aggregates. Some of the fly ashes have enough calcium to provide significant compressive strength without the addition of lime.

Mr. Moh has confused the term *mechanism*; it is not clear whether he means chemical or mechanical mechanism. The authors sought to explain the chemical mechanism of the pozzolanic reaction. By the use of absorption tests and X-ray spectrometer methods the chemical reactions were investigated. If there were no reactions that produced the cementitious material, there would be no strength development; hence the importance of the chemical mechanism should be obvious.

There are various physical factors in addition to the chemical factors that affect the development of compressive strength. The strength of lime, fly ash mortars is dependent on the amount and number of contacts of the cementitious material; therefore, any thing that affects either the amount or number of contacts will affect the strength. Physical effects such as the depletion of lime at certain points in the mortar, or an excess of lime may even prevent the cementitious material from making contact; these factors may arise from inadequate mixing of the mortars. Non-reactive material may prevent contact of the cementitious material. Density is another physical factor that may affect the strength²⁶; increasing the density of the mortar increases its compressive strength. However, despite the physical effects it seems reasonable there must be a relationship between the chemical reactions and the strength development of the lime, fly ash mortars.

The rate of compressive strength development of lime, fly ash mortars at ordinary temperatures generally is low initially, then the strength development is rapid, and finally the rate decreases significantly, but the strength continues to increase; this was shown in figure 4 of this paper. The rate of absorption of fly ashes in Ca(OH)₂ solutions proceeds in the same manner. The rapid increase in strength is due to the cementitious material produced by the lime, fly ash reaction, and the decrease in compressive strength is due to the local depletion of lime and limitation due to the diffusion of calcium through the reaction products. By increasing the concentration of the lime the rate of strength development will be increased after the initial period; but the rate of strength development will not be exactly the same as the rate of absorption. This is due to the physical factors that are not involved in the absorption tests. It was noted in the paper that fly ashes having a high rate of calcium absorption also have a high rate of compressive strength development. Therefore, the correlation between the rate of absorption and the rate of strength development must be made on the basis of standardized tests if it is to have interpretation meaning.

The negative absorption can be satisfactorily explained by the presence of soluble components in the fly ashes. The presence of calcium compounds in the fly ashes will be noted in the chemical analysis of the fly ashes given in table II of the paper. Some of the fly ashes have enough soluble calcium to be weakly cementitious without the addition of lime. The solubility of these fly ash compounds is of little importance to the pozzolanic chemical mechanism. The inconsequential initial absorption values may be due to charge deficiencies that are associated with glasses, or other phenomena; they do not enter into any of the calculations. Lea²⁴ chooses to ignore the initial reactions in his studies.

From the physical data given in table IV and the discussion given in the paper it should be obvious that the minor differences are due to the coarse fraction; the major role of the coarse fraction, containing the majority of the unburned coal and coke, is not the chemical reactivity of the pozzolans, but is the physical factor of preventing the contact of cementitious material. The presence of unburned coal and coke also generally indicates a coarser fly ash and hence a lower specific surface.

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ACTIVATION OF THE LIME FLY ASH REACTION BY TRACE CHEMICALS

by

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(Highway Research Board Bulletin 231, 1959.)

ABSTRACT

The results of an investigation of the effect of small amounts of fortyseven different chemicals on the strenth of Ottawa sand, lime, and fly ash mixtures are presented. An explanation is offered for the mechanism of strength improved by the different groups of chemicals studied.

INTRODUCTION

Road bases stabilized with lime and fly ash may not gain sufficient strength in 7 or 28 days to carry heavy traffic satisfactorily or to withstand repeated freeze-thaw cycles. This prolongs the period that the road must be closed to traffic or reduces the construction season in northern climates. An economical method of speeding up the rate of strength gain of lime and fly ash stabilized soil would extend its usefulness in road construction.

Heat is known to be a good activator for the lime, fly ash (pozzolanic) reaction; 7 day strengths of over 1000 psi. may result from curing compacted specimens of soil, lime, and fly ash at 140°F. in sealed containers. But since high temperature curing of road bases is not economical with presently available fuels, a more practical alternative for activation of the lime, fly ash reaction would be with trace chemical additives.

MATERIALS

Soil

Natural monomineralic silica sand from Ottawa, Illinois, was used as the soil component of mixtures to eliminate variables due to the complex mineral composition of natural soil. The gradation of the sand met the requirements (ASTM Designation: C 109-56) for graded standard sand:

SIEVE SIZE %	PASSING
No. 16 (1190 micron)	100
No. 30 (590 micron)	98±2
No. 50 (297 micron)	28 ± 5
No. 100 (149 micron)	2 ± 2

Fly ash

The fly ash was from the Detroit Edison Company St. Clair Power Plant, St. Clair, Michigan. The composition and physical properties of the sample used are:

Silicon dioxide (SiO ₂), $\%$	41.9
Magnesium oxide (MgO), %	1.0
Calcium oxide CaO), %	2.7
Aluminum oxide (Al_2O_3) , %	22.5
Iron oxide (Fe ₂ O ₃), $\%$	25.8
Sulphur trioxide (SO ₃), %	0.8
Available alkalies (as Na_2O), %	0.3
Loss on ignition, %	3.6
Moisture content, %	0.2
Specific gravity	2.61
Fineness (% passing a No. 325 sieve)	88.7
Specific surface (sq. cm./g.)	2720

Lime

The lime was calcium hydroxide, reagent grade, from the Allied Chemical and Dye Corporation.

Chemicals

Forty-seven chemicals were evaluated. The selection included chemicals known or suspected to improve the pozzolanic reaction as well as chemicals whose effect on the reaction was unknown^{1, 2, 3, 4, 5, 6}. The chemicals were reagent, technical or purified grade. They are grouped (table I) primarily on the basis of whether their reactions were basic, neutral or acid. Bases and basic salts, also known as alkalies and alkaline salts, produce hydroxyl ions in water solution to varying extents. Acid salts produce hydrogen ions in water solution to varying extents. Neutral salts in water solution do not upset the natural balance of hydrogen and hydroxyl ions. Unclassified chemicals are in the miscellaneous group.

METHODS

Mix proportions

Lime fly ash mortars were composed of 75 percent Ottawa sand and 25 percent lime and fly ash, with the ratio of lime to fly ash either 1:9 or 1:4. (75:2.5:22.5 or 75:5:20 Ottawa sand, lime, and fly ash.) The trace chemical additive was 0.5 percent and/or 1 percent, based on the dry weight of the mortar. Chemicals were added to the dry mortar as a powder or as a component of the mix water which was close to standard Proctor optimum moisture (ASTM Designation: D698).

Mixing and molding

Mixing of batches for preparing test specimens was done in a Hobart kitchen mixer, model C-100, at low speed. The dry ingredients were mixed 25 seconds; then the mix water was added, and mixing was continued for 4 minutes.

Molding was started immediately after a batch was mixed. A double

plunger drop-hammer apparatus was used to mold 2 inch diameter by 2 inch high specimens to approximate standard Proctor density.

Curing

Specimens of each batch were moist cured at near 70°F. and 100 percent relative humidity for 7 days, 28 days and 4 months. To preserve moisture better and to reduce entry of carbon dioxide from the air, all specimens were wrapped in wax paper sealed with cellophane tape.

Strength testing

After each curing period, specimens were unwrapped and immersed in distilled water for one day. Then they were tested for unconfined compressive strength using a load travel rate of 0.10 inch per minute. Tests were run in triplicate and the average strengths are reported.

Freeze-thaw testing

Twenty-eight day cured 2 inch by 2 inch specimens of selected mixtures were subjected to cycles of freezing and thawing. Specimens on water saturated felt pads were frozen at -10° F. for 23 hours; then they were removed from the freezer and allowed to thaw in open air for 2 hours and in a moisture (near 100 percent R. H.) room for 23 hours. This was one cycle. Specimens were tested for unconfined compressive strength after 0, 1, 4, 5, 7, 9 and 12 cycles. Strengths reported are the average for three specimens.

GENERAL COMPARISON OF CHEMICALS

The first study of the investigation was a general comparison of the chemicals to determine their relative effects on 7 day, 28 day and 4 month strengths. The amount of each chemical treatment was 0.5 percent, based on the dry weight of the mortar of 75 percent Ottawa sand, 2.5 percent lime, and 22.5 percent fly ash. The chemicals were added dissolved or suspended in the mix water.

Test results are shown by bar graphs (figure 1). Starting at the top of the figure, chemicals are listed in order of decreasing benefit to 7 day strength.

7 day strength

The 7 day strength of a soil, lime, and fly ash road base is of much practical significance. Seven days is about as long as newly constructed roads can be kept closed to traffic. To avoid rutting, base course strength, as measured by the unconfined compression test, may have to reach 100 to 300 psi., depending on soil type stabilized, road usage, and thickness of bituminous surfacing. Since the strength gain of soil, lime, and fly ash road bases is greatly reduced or even halted when the temperature drops to near freezing, 7 days of curing in northern climates may represent the maximum obtainable in late season construction. For adequate freeze-thaw resistance, soil, lime, and fly ash bases may need a strength of 300 to 500 psi., depending on soil type stabilized, thickness of bituminous surfacing, and severity of the climate.

The 7 day strength, after 24 hours immersion, of untreated Ottawa sand, lime, and fly ash specimens averaged only 9 psi. (figure 1), illustrating the slowness of the pozzolanic reaction under normal curing conditions. Many of the chemicals greatly increased 7 day strength; these may be classed as



Unconfined compressive strength after one day immersion, psi

Fig. 1. Immersed strengths after 7 day, 28 day, and 4 month normal moist curing of 2 inch by 2 inch specimens prepared at standard Proctor density from 75:2.5:22.5 Ottawa sand, lime, and fly ash mortar treated with 0.5 percent of each chemical listed. Chemicals are listed from top to bottom in decreasing order of benefit to 7 day strength. All chemicals were added to the mortar dissolved or suspended in the mix water.

accelerators for the lime fly ash reaction. The primary purpose of this investigation was to discover such chemicals.

Best strength acceleration was with lithium carbonate which gave an average 7 day strength of 426 psi., over 47 times the strength of the untreated reference specimens. Potassium hydroxide, sodium hydroxide, sodium permanganate and Quadrafos gave 7 day strengths near or above 300 psi., Sodium carbonate, sodium sulfite, potassium permanganate, potassium bicarbonate, lithium sulfate, potassium carbonate, sodium bicarbonate, lithium fluoride, and lithium nitrate also are worthy of special mention, all giving 7 day strengths near or over 200 psi.

A look at the classification of chemicals (table I) shows that the two bases were good activators for the pozzolanic reaction. The basic salts, with the exception of sodium borate, gave 7 day strengths over 150 psi. The neutral salts produced variable results; best were the strong oxidizing agents, potassium permanganate and sodium permanganate, both giving strengths over 200 psi. The acid salts did not appreciably improve 7 day strength. Of the chemicals in the miscellaneous group, only Quadrafos gave good 7 day strength.

Group and chemical	Formula	Manufacturer
BASES Potassium hydroxide Sodium hydroxide	KOH NaOH	J. T. Baker Chemical Co. J. T. Baker Chemical Co.
BASIC SALTS Lithium carbonate Potassium bicarbonate Potassium carbonate Sodium bicarbonate Sodium borate Sodium carbonate Sodium sulfite	Li ₂ CO ₃ KHCO ₃ K ₂ CO ₃ Na ₂ B ₄ O ₇ •10H ₂ O Na ₂ CO ₃ Na ₂ SO ₃ •7H ₂ O	Fisher Scientific Co. Mallinckrodt Chemical Works J. T. Baker Chemical Co. J. T. Baker Chemical Co. Fisher Scientific Co. J. T. Baker Chemical Co. J. T. Baker Chemical Co.
NEUTRAL SALTS Lithium chloride Lithium fluoride Lithium nitrate Lithium sulfate Potassium chlorate* Potassium chloride Potassium dichromate* Sodium chloride Sodium chloride Sodium hypochlorite* Sodium nitrate Sodium permanganate* Sodium permanganate*	LiCl LiF LiNO ₃ Li ₂ SO ₄ •H ₂ O KClO ₃ KCl K ₂ Cr ₂ O ₇ KMnO ₄ NaCl Na ₂ Cr ₂ •2H ₂ O NaOCl \dagger NaMnO ₄ •3H ₂ O Na ₂ SO ₄	J. T. Baker Chemical Co. Fisher Scientific Co. Fisher Scientific Co. Fisher Scientific Co. J. T. Baker Chemical Co. J. T. Baker Chemical Co. Fisher Scientific Co. J. T. Baker Chemical Co.
ACID SALTS Aluminum chloride Ammonium chloride Calcium chloride Calcium hypochlorite*	AlCl ₃ •6H ₂ O NH ₄ Cl CaCl ₂ Ca (OCl) ₂	J. T. Baker Chemical Co. Allied Chemical & Dye Corp. J. T. Baker Chemical Co. Fisher Scientific Co.
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TABLE I. CHEMICALS E	EVALUATED AS	ADDITIVES.
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	TABLE I, Continued	
Group and chemical	Formula	Manufacturer
Calcium sulfate Chromic chloride Cobaltous chloride Cupric chloride Ferric sulfate Ferric sulfate Ferrous sulfate Magnesium chloride Manganous chloride Nickel chloride Stannous chloride Titanium tetrachloride Zinc chloride	$\begin{array}{c} CaSO_4 {\scriptstyle \bullet} 2H_2O\\ CrCl_3 {\scriptstyle \bullet} yH_2O\\ CoCl_2 {\scriptstyle \bullet} 6H_2O\\ CuCl_2\\ FeCl_3\\ Fe_2(SO_4)_3 {\scriptstyle \bullet} xH_2O\\ FeCl_2 {\scriptstyle \bullet} 4H_2O\\ FeSO_4 {\scriptstyle \bullet} 7H_2O\\ MgCl_2 {\scriptstyle \bullet} 6H_2O\\ MnCl_2 {\scriptstyle \bullet} 4H_2O\\ NiCl_2 {\scriptstyle \bullet} 6H_2O\\ SnCl_2 {\scriptstyle \bullet} 2H_2O\\ TiCl_4\\ ZnCl_2 \end{array}$	J. T. Baker Chemical Co. Fisher Scientific Co. Fisher Scientific Co. Fisher Scientific Co. Fisher Scientific Co. Allied Chemical & Dye Corp. Fisher Scientific Co. J. T. Baker Chemical Co. Allied Chemical & Dye Corp. Fisher Scientific Co. Fisher Scientific Co. Fisher Scientific Co. Fisher Scientific Co. Fisher Scientific Co. Fisher Scientific Co.
MISCELLANEOUS Ethylene glycol Magnesium oxide Portland cement Potassium biphthalate Quadrafos‡ Vanadyl dichloride	$\begin{array}{c} \mathrm{CH}_{2}\mathrm{OHCH}_{2}\mathrm{OH}\\ \mathrm{MgO}\\ \hline \overrightarrow{\mathrm{K}} \mathrm{HC}_{8}\mathrm{H}_{4}\mathrm{O}_{4}\\ \hline \hline \mathrm{VO} \mathrm{Cl}_{2} \end{array}$	Wilkens-Anderson Co. Allied Chemical & Dye Corp. Hawkeye Portland Cement Co. J. T. Baker Chemical Co. Rumford Chemical Co. Fisher Scientific Co.

*Strong oxidizing agent. †In solution with 5 to 6% available chlorine. ‡Trade-mark for sodium tetraphosphate (P₂O₅ 63.5%; Na₂O 36.0%; H₂O 0.5%).

28 day strength

Twenty-eight days of curing can usually be counted on in late summer or early fall construction and, ideally, the soil, lime, and fly ash mixture should reach adequate strength before the first freeze. The untreated lime fly ash mortar specimens did not gain adequate strength in 28 days (figure 1), the average being only 34 psi.

The chemicals cited as most beneficial to 7 day strength also greatly improved 28 day strength, but it will be noted that the order of merit is somewhat changed. Also, a number of chemicals which did not look promising on the basis of 7 day strength improvement, showed up well in the 28 day tests. Chemicals which gave 28 day strengths near or above 600 psi. (about 18 times the 28 day untreated strength) are: potassium hydroxide, sodium hydroxide, potassium permanganate, potassium carbonate, sodium carbonate, calcium chloride, sodium bicarbonate, and potassium bicarbonate, listed in order of decreasing merit. Many of the other chemicals gave 28 day strengths of 400 to 500 psi. or higher. Among the most promising of this group are: lithium carbonate, sodium permanganate, sodium sulfite, sodium chloride, and the 50:50 combination of ammonium chloride and sodium chloride.

The bases and the basic salts, except sodium borate, gave best results. The neutral salts, except sodium hypochlorite, gave 28 day strengths over 300 psi. Calcium chloride, ammonium chloride, magnesium chloride, and manganous chloride of the acid salts gave strengths over 300 psi., but as a whole the chemicals in this group rated low. Quadrafos and the 50:50

combination of ammonium chloride and sodium chloride were the most promising in the miscellaneous group.

4 month strength

The average 4 month strength of the untreated Ottawa sand, lime, and fly ash specimens was 589 psi., which is very adequate, and demonstrates that the long-term strength of lime fly ash stabilized soil is not a problem. In northern climates, spring or early summer construction is necessary to obtain 4 months of curing. But even when this is possible, sufficient early strength to carry traffic is necessary.

Most of the chemicals cited as being very beneficial to 7 and/or 28 day strengths caused little or no improvement of 4 month strength, or even decreased it (figure 1). The greatest benefit to 4 month strength was obtained with calcium chloride (855 psi) and manganous chloride (852 psi). Both of these chemicals were beneficial to 28 day strength, particularly calcium chloride, but they rated low in 7 day strength improvement. Calcium hypochlorite gave a 4 month strength of 789 psi, but 7 and 28 day strengths with this chemical were very low. Potassium permanganate might be rated highest in strength improvement at all three ages. Other chemicals giving 4 month strengths above 700 psi, and which were also noteworthy for 7 and 28 day strength improvements, were sodium permanganate, lithium fluoride, and lithium nitrate. Potassium dichromate and sodium dichromate gave 4 month strengths over 700 psi and also gave good 28 day strengths, but both rated low at 7 days.

The bases and the basic salts did not contribute much to the 4 month mortar strength, indicating that the chemicals in these groups act mainly as activators of the lime fly ash reaction. The three chemicals which gave the highest 4 month strengths are acid salts, but several neutral salts also were beneficial; other chemicals in these two groups reduced 4 month strength. Several of the better neutral and acid salts are strong oxidizing agents (table I). In the miscellaneous group, vanadyl dichloride and the 50:50 combination of ammonium chloride and sodium chloride were slightly beneficial.

Density variations

Some of the chemical additives influenced the compaction characteristics of the 75:2.5:22.5 Ottawa sand, lime, and fly ash mortar, as evidenced by variations in the dry density of test specimens. The maximum variation from the compacted density of the untreated mortar was plus 6.4 or minus 2.6 pcf. However, the variations in density do not correlate with the improvements in strength.

pН

Determinations of pH were made on material from specimens tested for unconfined compressive strength. (The electrometric method was used, employing 15 grams of sample in 30 milliliters of distilled water.) The object was to find a relationship between strength and pH after each curing period. A significant correlation was not evident from the data (figure 2), although mixtures containing the bases and basic salts maintained a relatively constant and high pH during the three curing periods.

All pH values were in the alkaline range due to the presence of calcium hydroxide. The amount of lime present in the mixtures was greater than the maximum solubility (1.2 grams per liter) of calcium hydroxide at 25°C. Enough lime was present to counter-balance the influence of the acid salts used as additives. Decreases of the pH of mixtures with time is presumably due to the lime being used up in the pozzolanic reaction.



Fig. 2. Relationship between strength and pH of 75:2.5:22.5 Ottawa sand, lime, and fly ash mortar treated with 0.5 percent of the different chemicals.

POWDERED VS. LIQUID APPLICATION OF CHEMICALS

Since some of the chemicals could be used in powdered form, and dry mixed with the lime fly ash mortar prior to adding the mix water, a check was made to find out what effect powdered application would have on mortar strengths. Test results show that most of the chemicals gave better results when mixed as a powder (figure 3). Most noteworthy is powdered sodium carbonate, which produced 7 day, 28 day and 4 month strengths that are 71 percent, 28 percent and 18 percent higher than the comparable strengths produced by liquid application of sodium carbonate.



AMOUNT OF CHEMICAL

The use of more chemical additive than 1 percent may not be economical; 0.5 percent represents a more desirable treatment level. From a comparison of strength benefits from 1 percent and 0.5 percent chemical treatments of 75:2.5:22.5 and 75:5:20 mortars there seems to be little advantage and perhaps a disadvantage in using more than 0.5 percent chemical, particularly of the more promising additives such as sodium carbonate, sodium hydroxide, potassium hydroxide, calcium chloride, aluminum chloride, potassium permanganate and magnesium oxide (figures 4, 5).



INFLUENCE OF LIME TO FLY ASH RATIO

The ratio of lime to fly ash may be important in affecting the strength attained by soil, lime, and fly ash mixtures. In the present investigation the influence of the ratio was studied by comparing the strength gains of chemically treated 75:2.5:22.5 (ratio 1:9) and 75:5:20 (ratio 2:8) Ottawa sand, lime, and fly ash mortars (figures 6, 7).

7 day strength

The 7 day strengths, with a few exceptions, were similar for both ratios. The exceptions are of interest because they involve three of the most promising activators: potassium hydroxide, sodium hydroxide and sodium carbonate. One percent powdered sodium carbonate gave highest strength with the 2:8 ratio mortar, the increase being 144 psi; with 0.5 percent sodium carbonate the 7 day strength difference between the two mortars was negligible. One-half percent sodium hydroxide gave best results with the 1:9 ratio mortar by about 100 psi, but little strength difference was observed for the 1 percent treatment. Potassium hydroxide likewise favored the 1:9 ratio mortar, the strength increase being about 100 psi. for the 0.5 percent treatment.

28 day strength

It is difficult to conclude which lime to fly ash ratio gave the best 28 day strength. With the better chemicals previously cited, good strengths were



Fig. 3. Comparison of powder vs. mix water application of chemical additives. The amount of chemical was 0.5 percent; the mortar was 75:2.5:22.5 Ottawa sand, lime, and fly ash.

obtained with both ratios. Examples of chemical treatments most sensitive to the ratio are: 0.5 percent and 1 percent calcium chloride, which gave best results with the 1:9 ratio mortar by about 150 to 230 psi; 1 percent potassium permanganate, which was best with the 2:8 ratio mortar by about 260 psi; and 0.5 percent and 1 percent sodium carbonate, which produced best strengths with the 2:8 ratio mortar by about 250 to 390 psi.

4 month strength

The best 4 month strengths without exception were obtained using the 2:8 lime to fly ash ratio; in some cases the strength was two or more times that obtained when the ratio was 1:9. The following chemicals produced 4 month strengths above 1,000 psi: aluminum chloride, calcium chloride,



Fig. 4. Effect of amount of chemical additive on strength of 75:2.5:22.5 Ottawa sand, lime, and fly ash mortar.







Immersed unconfined compressive strength,psi Fig. 6. Effect of lime to fly ash ratio on strength benefits from 1.0 percent chemical treatments. The

Fig. 6. Effect of lime to thy ash ratio on strength benefits from 1.0 percent chemical freatments. The mortars were composed of 75 percent Ottawa sand and 25 percent lime and fly ash.



Fig. 7. Effect of lime to fly ash ratio on strength benefits from 0.5 percent chemical treatments. The mortars were composed of 75 percent Ottawa sand and 25 percent lime and fly ash.

potassium permanganate, sodium carbonate and magnesium oxide. Of these, only magnesium oxide was appreciably less effective when the amount used was 0.5 percent.

FREEZE-THAW RESISTANCE

The strength retention through 12 cycles of alternate freezing and thawing of 28 day cured 2 inch by 2 inch specimens provides an indication of relative durability. The effects of 0.5 percent of any one of four chemicals on the durability of the 75:2.5:22.5 mortar are shown (figure 8).

The mortar specimens with no chemical additive showed high strength retention, but initial strength was less than 200 psi. Specimens containing sodium carbonate (added as a powder) had a marked decrease in strength, but after 12 cycles the strength retained was more than 600 psi. Potassium permanganate treated specimens, after a decrease in strength through 4 cycles, showed an abrupt increase to over 600 psi, which was retained with little reduction through the last 8 cycles. Specimens containing calcium chloride and sodium chloride failed after the fifth cycle.

SUMMARY DISCUSSION

Evaluation of chemicals by groups

Classification of the chemical additives as in table I permits some generalized statements concerning the effects of each group on the lime fly ash



Fig. 8. Effect of freezing and thawing on strength of 28 day cured 2 inch by 2 inch specimens of 75:2.5:22.5 Ottawa sand, lime, and fly ash mortar treated with 0.5 percent each of sodium carbonate, potassium permanganate, calcium chloride and sodium chloride. Sodium carbonate was added to the mortar in powdered form; the other chemicals were added in the mix water.

(pozzolanic) reaction. The *bases* and the *basic salts*, except sodium borate, greatly improved early strength, but did not improve long-term strength to a proportionate degree. Thus most chemicals in these two groups appear to be good activators (accelerators) of the pozzolanic reaction. One of the basic salts, sodium carbonate (soda ash), is considered the most promising trace chemical evaluated.

The *neutral salts* produced very good 28 day strengths, which were further improved after 4 months curing. With only two exceptions, 4 month strengths were close to or higher than the untreated mortar strength. Seven day strengths with neutral salt additives were higher than the corresponding untreated mortar strength, but the improvement

was not always exceptional. The most promising activators in this group are potassium permanganate, sodium permanganate, lithium sulfate, lithium fluoride, lithium nitrate, sodium nitrate and sodium chloride (because it is cheap).

The *acid salts* did not appreciably improve 7 day strength, but some gave marked improvement to 28 day and 4 month strengths. The most promising chemicals in this group are the chlorides, particularly calcium chloride and manganous chloride. Calcium hypochlorite gave very good 4 month strength, but 7 and 28 day strengths were low with this chemical.

In the *miscellaneous* group of chemicals, Quadrafos was most beneficial to 7 day strength; magnesium oxide was beneficial to 28 day and 4 month strengths.

Mechanism of benefits

An explanation of the mechanism of the beneficial effects obtained with the different chemicals is difficult because each chemical or narrow range of chemicals may act in a completely different way. A chemical may act as a catalyst, as an inhibitor, or as a component of the pozzolanic reaction, and also may go into side reactions other than the pozzolanic reaction and produce either cementing or inert materials.

Bases and basic salts. Alkaline additives increase the amount of available hydroxyl ions in the moistened Ottawa sand, lime, and fly ash system, and as a result the pozzolanic reaction may be accelerated by the increased solubility of the siliceous material⁴. For example, sodium hydroxide may act as a catalyst in which:

(1) it first reacts with the siliceous material to produce intermediate sodium silicates,

(2) the overall reaction goes to completion when the intermediate sodium silicates subsequently react with lime (calcium hydroxide) to form sodium hydroxide and cementitious insoluble calcium silicates,

(3) the sodium hydroxide is then free for further reaction with unreacted siliceous material.

The most promising of the alkaline additives, sodium carbonate, very likely reacts with lime in the moist Ottawa sand, lime, and fly ash mixture to form calcium carbonate and sodium hydroxide. The precipitated calcium carbonate contributes cementation to the system, and, as hypothesized in the preceding paragraph, the sodium hydroxide acts as a catalyst. The formation of calcium carbonate from the lime obviously decreases the amount of lime available for the pozzolanic reaction. The hypothesis is apparently consistent with the experimental data: with a 1:9 ratio of lime to fly ash better strength results were obtained with 0.5 percent sodium carbonate than with 1 percent, whereas the opposite results were obtained with a 2:8 ratio. This indicates the possibility of an optimum amount of sodium carbonate for a given amount of lime.

Acid salts. Acid salts undergo a hydrolysis reaction with the precipitation of weak bases (hydroxides). This may be summarized as follows:

 $R_n X_m + nm H OH \rightleftharpoons n R (OH)_m + m H_n X_2$

With calcium hydroxide this reaction proceeds as follows:

 $2R_n X_m + nm Ca (OH)_2 \rightleftharpoons 2n R (OH)_m + m Ca_n X_2.$

with aluminum chloride as the acid salt, a weak base is precipitated and an equivalent amount of lime is withdrawn from the reaction,

 $2 \operatorname{AlCl}_3 + 3 \operatorname{Ca}(OH)_2 \rightarrow 2\operatorname{Al}(OH)_3 + 3 \operatorname{CaCl}_2.$

weak base

The removal of lime results in a reduction of the lime to fly ash ratio. Thus acid salts may impede the development of strength when the ratio of lime to fly ash is 1:9. For example, when aluminum chloride was used in mixtures having a 1:9 ratio of lime to fly ash, strengths were much lower than when the ratio was 2:8. In mixtures with a 1:9 ratio, 1 percent aluminum chloride gave lower strengths than 0.5 percent chloride.

Some of the weak bases formed, such as Al $(OH)_3$ and $Fe(OH)_3$, have some cementing and water-proofing properties which may be beneficial. Such weak bases may also affect the long-term formation of hydrated calcium silicates and thus increase long-term strength.

Although calcium chloride is an acid salt, the principal long-term strength benefits obtained with this chemical are thought due to a different type of chemical mechanism than discussed above. Since calcium chloride is highly hygroscopic and deliquescent, a relatively high concentration of calcium ions is insured over a long period of time by providing moisture for a solution. Since lime has a lower ionization constant than calcium chloride, the concentration of calcium ions from lime is lower than that from calcium chloride. Also lime is subject to conversion to calcium carbonate during long curing periods; when this takes place, pozzolanic action ceases. The experimental data tend to support this line of reasoning: calcium chloride was found to be very beneficial to 4 month strength, but it only slightly improved 7 day strength.

Sodium chloride may act similarly to calcium chloride, but there appears to be less benefit to long-term strength, perhaps because sodium chloride is less hygroscopic and deliquescent than calcium chloride. Another difference is that some sodium hydroxide is probably formed, and thus a small amount of catalysis would be expected. This may explain why sodium chloride gave slightly higher 7 day strength than calcium chloride.

Neutral salts. The reactions of neutral salts with the Ottawa sand, lime, and fly ash mixtures are somewhat more complicated than those of the other groups. The most promising neutral salts, potassium permanganate and sodium permanganate, are strong oxidizing agents. It is believed that these chemicals oxidize the carbon in the fly ash with the consequent production potassium carbonate or sodium carbonate, and the precipitation of manganese dioxide. These carbonates, as discussed previously, then give rise to further reactions which are beneficial to strength. It is also believed that the permanganates, and other strong oxidizing agents, benefit strength by reacting with grains of fly ash, cleaning the surfaces and making them more available for chemical reactions with lime. As already mentioned, several of the better neutral and acid salts were strong oxidizing agents.

Miscellaneous chemicals. Quadrafos (sodium tetraphosphate), the only chemical in the miscellaneous group that greatly benefited early strength, may react with lime to produce complex phosphate cementation products which supplement those produced by the pozzolanic reaction. The availability of sodium ions to form sodium hydroxide may improve the pozzolanic reaction as previously discussed under bases and basic salts.

The beneficial effect of magnesium oxide, another miscellaneous chemical, is in agreement with the findings of previous research that dolomitic monohydrate (Type N) lime gives greater strengths than high-calcium lime⁶. The data show that magnesium oxide was most effective when added as a powder, when the amount was 1 percent, and when the fly ash ratio was 2:8. The mechanism of the benefit can only be guessed at. Cementation by calcium magnesium silicates may be better than by calcium silicates.

Powdered vs. liquid application of chemicals. All chemicals tried gave best results when used in powdered form, rather than when added as a solution or suspension in mix water. This may be due to consumption of the chemical by side reactions that take place more rapidly in solution or suspension than in a semi-dry system. Another possibility is that lesser amounts of chemicals than were studied are needed for optimum benefits when the chemicals are added in water.

The greatest benefit from use of a powered chemical was with sodium carbonate. The reaction of this chemical with lime in the mortar is responsible for the previously discussed precipitation of calcium carbonate cement. Calcium carbonate is thought to be more effective as a cement when precipitated after the mortar has been compacted, because then the carbonate crystals are formed on the sand (and fly ash) grains. This is more apt to occur when sodium carbonate is added in powdered form. When added in the mix water, the calcium carbonate may be prematurely precipitated before compaction is completed. Perhaps another advantage in using powdered sodium carbonate is that a slower production of sodium hydroxide may be more favorable to the sustained formation of pozzolanic cementing products.

CONCLUSION

Several of the forty-seven chemicals evaluated in lime fly ash mortars greatly increase early strength. Other chemicals benefit long-term strength more than early strength; calcium chloride is one of the most promising chemicals in this group.
All factors considered, sodium carbonate (soda ash) is considered the most promising trace chemical activator investigated. Best results are obtained when it is mixed in powdered form. The use of 0.5 percent powdered sodium carbonate in a mixture of 75 percent Ottawa sand: 5 percent lime: 20 percent fly ash increased 7 day strength about sixty times, and 28 day and 4 month strengths about two times.

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VARIATION IN POZZOLANIC BEHAVIOR OF FLY ASHES

by

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INTRODUCTION

Fly ash belongs to a class of material known as pozzolans. These are any siliceous or siliceous and aluminous material which in the presence of lime and water will become cementitious¹. Pozzolans may be subdivided into two categories: natural and artificial. Natural pozzolans include some volcanic ashes, diatomaceous earth, and bauxite. Artificial pozzolans include heat treated clay and shale, blast furnace slag, and fly ash.

Fly ash, produced at power plants which burn pulverized coal for fuel, is collected from stacks either by electrical or mechanical precipitators. Because the primary purpose for collecting fly ash is to prevent air pollution of neighboring communities, fly ash is a by-product. The production of fly ash in the United States reaches 10 million tons annually. With such a great quantity produced, there is naturally much interest in finding commercial uses for it. Some of the promising and established uses for fly ash are: as a grouting component, as a mild abrasive, as a filtering medium, in structural and mass concrete, and for stabilizing soil².

The use of fly ash in soil stabilization for roads and other kinds of pavements has been prompted by its low cost and the shortage of good natural base course materials in many areas. Soil stabilization with lime fly ash mixtures is still in the experimental stage, but many streets and highways have been so constructed, and recent reports indicate that most of the projects are satisfactory¹³. Roads have been built with lime fly ash stabilized soils in Pennsylvania, Maryland, New Jersey, and Iowa.

The Soil Research Laboratory of the Engineering Experiment Station of Iowa State University of Science and Technology has been investigating the use of lime and fly ash for soil stabilization with the assistance of the Iowa State Highway Commission and several private companies. In 1957 test sections of primary road totaling approximately 3,000 feet of lime fly ash stabilized base and 1,000 feet of lime fly ash stabilized sub-base were constructed near Colfax, Jasper County, Iowa. After four winters, these experimental sections are in excellent condition. This field trial is still under investigation and a final report will be made soon⁷. In investigations conducted to correlate strength of lime fly ash mixtures with chemical or physical properties of pozzolans, only general indications were obtained^{8, 12}. However on the basis of these and similar investigations, tentative specifications for use of pozzolans in concrete, based mostly on chemical and physical properties, have been established^{1, 11}.

More information is required on the lime fly ash reaction, particularly about products formed, and the other effects of chemical and physical properties, crystalline structure, and heat of reaction on strength of lime fly ash mixtures.

The aim of this investigation was to determine the relative effectiveness of different fly ashes for soil stabilization, and also to attempt to correlate the unconfined compressive strength of compacted lime fly ash mixtures with fly ash properties by isolating groups of fly ashes having several similar characteristics. The pozzolanic reaction of lime and fly ash was investigated with the use of unconfined compression tests, as this is a satisfactory criterion of fly ash reactivity with lime⁹. To reduce variables no soil was used, and all test specimens were of commercial hydrated lime (calcitic or dolomitic), fly ash, and distilled water.

MATERIALS

Fly ashes

The samples selected represent a good coverage of the major production factors which are believed to influence the properties and behavior of fly ashes. These major factors are: type of coal, the method of pretreatment of the coal, particle size of the coal, the type of burner, and the method of collection (tables I and II).

Fly ash sources and production

Sample No. 1 This sample was collected by multiple cyclone and electrical precipitators. The coal is from districts 8 and 3 in Ohio and northern West Virginia. It is processed through pulverizing mills so that 70 percent passes a No. 200 mesh. The sample was sent from the Conners Creek Power Plant of the Detroit Edison Company, Detroit, Michigan.

Sample No. 2 This sample was collected by electrical precipitators. The coal is also from districts 8 and 3 in Ohio and northern West Virginia, and is processed through pulverizing mills so that 70 percent passes the No. 200 mesh. The sample was sent from the Marysville Power Plant of the Detroit Edison Company.

Sample No. 3 This sample was collected by multiple cyclone and electrical precipitators. The coal is also from districts 8 and 3 in Ohio and northern West Virginia, and is processed through pulverizing mills so that 70 percent passes the No. 200 mesh. The sample was sent from the River Rouge Power Plant of the Detroit Edison Company.

Sample No. 4 This sample was collected by multiple cyclone and elec-

Fly ash No.	% passing No. 100 sieve	% passing No. 200 sieve	% passing No. 325 sieve	Specific gravity*	Sq. cm./g. Specific surface*	% Hygroscopic moisture content
$1 \\ 2 \\ 3 \\ 4 \\ 5$	99.0 93.6 98.1 97.2 98.2	93.9 86.9 93.2 94.1 90.1	73.672.568.275.770.8	2.39^{+} 2.28^{+} 2.61^{+} 2.61^{+} 2.26^{+}	2950† 4550† 3070† 2720† 4490†	.03 .04 .03 .03 .02
	98.2 94.3 93.5 92.1 89.8	$91.6 \\ 80.9 \\ 86.8 \\ 85.7 \\ 64.3$	$80.2 \\ 54.9 \\ 49.8 \\ 51.3 \\ 22.6$	2.52 2.37 2.39 2.04 3.43	$2336 \\ 4550 \\ 2663 \\ 3112 \\ 576$.02 .08 .03 .07 .01
$11\\12\\13\\14\\15$	98.2 90.0 53.0 95.1 93.1	$91.6 \\72.5 \\24.7 \\82.3 \\75.4$	$\begin{array}{c} 31.8 \\ 54.9 \\ 14.0 \\ 64.8 \\ 57.4 \end{array}$	2.82 2.34 2.15 2.69 2.33	$1460 \\ 4240 \\ 1903 \\ 2048 \\ 1730$	$.01 \\ .11 \\ .06 \\ .05 \\ .02$
$16 \\ 17 \\ 18 \\ 19 \\ 20$	98.6 99.0 99.4 93.9 91.6	$86.7 \\ 94.5 \\ 94.4 \\ 81.3 \\ 73.7$	$57.6 \\ 82.7 \\ 86.1 \\ 66.6 \\ 47.0$	$2.44 \\ 2.40 \\ 2.60 \\ 2.21 \\ 2.43$	$\begin{array}{c} 2109 \\ 3315 \\ 3226 \\ 2248 \\ 3649 \end{array}$.03 .03 .04 .03 .08
$\begin{array}{c} 21 \\ 22 \end{array}$	$\begin{array}{c} 96.9\\90.0\end{array}$	$\begin{array}{c} 84.5 \\ 70.8 \end{array}$	$80.7 \\ 53.9$	$\begin{array}{c} 2.34\\ 2.22 \end{array}$	$\begin{array}{c} 2539 \\ 1990 \end{array}$.02

TABLE I. PHYSICAL PROPERTIES OF FLY ASHES.

*Tests by Robert W. Hunt Co. of Chicago, Illinois. †Data supplied by Detroit Edison Company, Detroit, Michigan.

TABLE II. CHEMICAL COMPOSITION OF FLY ASHES.

Fly			, Chemical	analysis ren	orted as*		
No.	SiO_2	$\rm Fe_2O_3$	Al_2O_3	CaO	MgÕ	SO_3	С
$1 \\ 2 \\ 3 \\ 4 \\ 5$	$\begin{array}{c} 41.60 \\ 40.50 \\ 47.00 \\ 41.90 \\ 57.50 \end{array}$	$25.07 \\ 22.30 \\ 18.40 \\ 25.84 \\ 6.0$	$24.90 \\ 18.40 \\ 24.00 \\ 22.48 \\ 22.2$	$2.21 \\ 2.20 \\ 1.65 \\ 2.68 \\ 1.20$	$\begin{array}{c} 2.08 \\ 0.87 \\ 0.84 \\ 0.98 \\ 0.80 \end{array}$	$\begin{array}{c} 0.69 \\ 0.97 \\ 0.55 \\ 0.79 \\ 0.45 \end{array}$	$6.0 \\ 8.0 \\ 3.5 \\ 4.5 \\ 4.5 \\ 4.5$
6 7 8 9 10	$\begin{array}{r} 42.4 \\ 36.20 \\ 36.68 \\ 43.72 \\ 11.26 \end{array}$	$\begin{array}{c} 21.9 \\ 16.68 \\ 24.33 \\ 18.37 \\ 68.40 \end{array}$	$21.1 \\ 15.80 \\ 21.29 \\ 16.87 \\ 0.90$	$5.5 \\ 8.3 \\ 3.45 \\ 6.04 \\ 12.27$	$\begin{array}{c} 0.7 \\ 0.92 \\ 0.98 \\ 1.32 \\ 0.25 \end{array}$	$\begin{array}{c} 3.4 \\ 1.47 \\ 2.02 \\ 1.09 \\ 3.19 \end{array}$	$3.4 \\ 18.55 \\ 7.20 \\ 8.70 \\ 0.70 \end{cases}$
$11 \\ 12 \\ 13 \\ 14 \\ 15$	$\begin{array}{r} 40.09 \\ 38.50 \\ 28.26 \\ 35.32 \\ 39.16 \end{array}$	$36.65 \\ 16.20 \\ 22.16 \\ 43.35 \\ 30.22$	$13.13 \\ 18.08 \\ 9.88 \\ 7.75 \\ 11.92$	$5.80 \\ 3.25 \\ 8.35 \\ 5.33 \\ 11.60$	$\begin{array}{c} 0.30 \\ 0.17 \\ 0.66 \\ 0.88 \\ 0.77 \end{array}$	$2.36 \\ 1.05 \\ 1.09 \\ 1.37 \\ 1.86$	$\begin{array}{r} 0.15 \\ 13.89 \\ 28.06 \\ 3.75 \\ 2.85 \end{array}$
$16 \\ 17 \\ 18 \\ 19 \\ 20$	$\begin{array}{r} 40.50\\ 45.6\\ 42.5\\ 41.6\\ 35.94\end{array}$	20.84 17.2 20.0 11.9 19.63	$12.40 \\ 21.4 \\ 23.4 \\ 34.7 \\ 18.19$	$10.56 \\ 6.9 \\ 5.7 \\ 2.1 \\ 6.89$	$\begin{array}{c} 0.33 \\ 1.1 \\ 0.8 \\ 1.3 \\ 0.85 \end{array}$	1.98 2.3 2.3 0.8 1.86	$7.80 \\ 4.7 \\ 2.6 \\ 6.9 \\ 15.59$
$\frac{21}{22}$	$\begin{array}{c} 51.2\\ 43.48 \end{array}$	$\begin{array}{c} 20.2 \\ 21.35 \end{array}$	$\begin{array}{c} 10.0\\ 19.77 \end{array}$	$\begin{array}{c} 6.3 \\ 5.48 \end{array}$	$\begin{array}{c} 1.6 \\ 0.67 \end{array}$	$\begin{array}{c} 1.7 \\ 0.79 \end{array}$	1.0 6.40

*Reported as percent by weight.

trical precipitators. The coal is also from districts 8 and 3 in Ohio and northern West Virginia, and is processed through pulverizing mills so that 70 percent passes a No. 200 mesh. The sample was sent from the St. Clair Power Plant of the Detroit Edison Company.

Sample No. 5 This sample was collected by electrical precipitators. The coal is from district 8, east Kentucky and West Virginia, and is processed through pulverizing mills so that 70 percent passes a No. 200 mesh. The sample was sent from the Trenton Channel Power Plant of the Detroit Edison Company.

Sample No. 6 This sample was collected by mechanical equipment. The coal is from western Kentucky and is crushed in bowl type crushers. The sample was sent from the Du Pont Neoprene Plant (adjoining Paddy's Run Station) south of Louisville, Kentucky.

Sample No. 7 This sample was collected by mechanical precipitators. The coal is from northern Illinois and is burned in a Springfield boiler. This sample from the Sixth Street Station in Cedar Rapids, Iowa, was sent by the Iowa Electric Light and Power Company.

Sample No. 8 This sample was collected by mechanical equipment. The coal is from northern Illinois and is burned in a B and W boiler. This sample from the Sixth Street Station in Cedar Rapids, Iowa, was sent by the Iowa Electric Light and Power Company.

Sample No. 9 This sample was collected by mechanical equipment. The fuel is a mixture of northern Illinois coal (65 percent) and residue from furfural production (35 percent) and is burned in a B and W boiler. This sample from the Sixth Street Station, Cedar Rapids, Iowa, was sent by the Iowa Electric Light and Power Company.

Sample No. 10 This sample was collected by mechanical (centrifugal) precipitators. The coal is from Illinois and is pulverized in a ball mill prior to burning. The sample from the Riverside Station Power Plant at Davenport, Iowa, was sent by the Iowa-Illinois Gas and Electric Company.

Sample No. 11 This sample was collected by mechanical precipitators (multicone dust collector). The coal is from Iowa (Monroe, Polk, Marion and Mahaska Counties); it is unwashed steam coal which is pulverized and tangential fired. The sample was from the Des Moines Power Plant of the Iowa Power and Light Company.

Sample No. 12 This sample was collected by mechanical equipment (VGR multicone). The coal (from southern Illinois) is washed, dried, and pulverized with Riley mills. The sample was sent from the Waterloo Power Plant by the Iowa Public Service Company.

Sample No. 13 This sample was collected by mechanical precipitators. The coal is from Iowa and burned on spreader stokers without being pulverized. The sample was from Bridgeport Station Power Plant, Eddyville, Iowa, of the Iowa Southern Utilities Company.

Sample No. 14 This sample was collected by mechanical precipitators (cyclone type). The coal is from Missouri and Kansas mines. The coal is pulverized and burned in suspension in Combustion Engineering boilers. The sample was sent from the Hawthorne Station Power Plant of the Kansas City Power and Light Company, Missouri.

Sample No. 15 This sample was collected by mechanical precipitators (cyclone type). The coal is from Missouri and is pulverized and burned in suspension in Combustion Engineering boilers. The sample was from Montrose Station Power Plant of the Kansas City Power and Light Company.

Sample No. 16 This sample was sent by the Kansas City Power and Light Company, Missouri. No data are available on the source.

Sample No. 17 This sample was collected by electrical precipitators. The coal is from western Kentucky and is crushed in ball mills. The sample from the Cane Run Power Plant south of Louisville, Kentucky, was sent by the Louisville Gas and Electric Company.

Sample No. 18 This sample was collected by electrical precipitators from a dry bottom type of boiler using unwashed coal from western Kentucky. The sample was from Paddy's Run Power Station at Louisville, Kentucky, of the Louisville Gas and Electric Company.

Sample No. 19 This sample was collected by mechanical collectors. The coal is from the Birmingham, Alabama, area and is crushed in bowl crushers. The sample was from the Barry Power Plant at Mobile, Alabama.

Sample No. 20 Sample was collected on electrical precipitators from a dry bottom type of boiler using southern Illinois coal. For economic reasons, the boilers are operated at 110 percent of rated capacity, with a lowering of combustion efficiency. This sample was from the Venice Station Power Plant near St. Louis, Missouri, of the Union Electric Company.

Sample No. 21 This sample was collected by electrical precipitators. The coal is from southern Illinois and is crushed in a bowl crusher. The sample was from the Meramec Station Power Plant south of St. Louis, Missouri (near Jefferson Barracks), of the Union Electric Company.

Sample No. 22 This sample was from the Oak Street Power Station by the Wisconsin Electric Power Company. No data are available on the source of coal or method of fly ash collection.

Limes

The two commercial grade limes used in this investigation were furnished by the U. S. Gypsum Company. One is hydrated calcitic lime, $Ca(OH)_2$, brand name *Kemikal*, and the other is type N monohydrate dolomitic lime, $Ca(OH)_2 + M_gO$, brand name *Kemidol*. The calcitic lime was produced at New Braunfels, Texas, and the dolomitic lime was produced at Genoa, Ohio. The typical properties of these limes are shown in table III. Commercial type limes were used because the primary purpose of this investigation was to compare relative strengths obtained from mixtures with the different fly ashes, to facilitate selection of fly ashes for future field and laboratory tests of soil, lime, and fly ash stabilization.

Water

Throughout the laboratory work the water used in mixtures and for specimen immersion was distilled water obtained from a Barnstead Automatic water still, extra duty type ELH-2 with a floatless low water cut-off. Distilled water was used so that results could be reproduceable and so that impurities contained in tap water would not affect the lime and fly ash reaction.

LABORATORY INVESTIGATION

Analyses

The chemical analyses of most of the fly ashes were performed by the Robert W. Hunt Company of Chicago, Illinois. The sieve analyses of fly ashes for determining the percentage passing the No. 100, No. 200, and No. 325 mesh sizers were performed on two separate samples of each fly ash. One hundred grams of fly ash were sieved through the No. 100 and No. 200 sieves to determine the percent passing through each. After shaking five minutes with a mechanical shaker, the sieves were washed and the residue on each sieve was dried in an oven at 105° C. The procedure for determining the percentage passing the No. 325 sieve was the same except that the No. 325 was submitted for the No. 200 sieve and only 50 grams of fly ash were used. Sieving of fly ash through the three sieves in one operation was attempted, but proved impractical as fly ash clogged both the No. 200 and No. 325 sieves at the same time. The weights of 100 grams and 50 grams were chosen for ease of computations and ease in sieving.

Proportioning and mixing

Two mix proportions by weight of lime and fly ash were used with each fly ash for both types of lime. These ratios of lime to fly ash were 1:4 and 1:9. The moisture contents of the mixes were computed as percentages

TABLE]	III.	PROPERTIES	\mathbf{OF}	LIMES.*
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Chemical analysis,	Hydrate	d limes
percent by weight	Dolomitic	Calcitic
Silicon dioxide	0.4	0.28
Iron and aluminum oxide	0.3	0.6
Magnesium oxide	31.8	0.59
Sulfur trioxide	1.1	0.25
Carbon dioxide	1.0	ND
Total calcium oxide Available calcium oxide Loss on ignition Combined H ₂ O	$\begin{array}{c} 48.8 \\ 47.1 \\ 17.1 \\ 18.0 \end{array}$	73.82 70.3 24.1 ND

*Supplied by the U. S. Gypsum Company.

of the total dry weight of ingredients. Sufficient materials were added to make four samples of each mixture for preliminary work and 10 samples for the final work. The materials to be tested were first dry mixed for 30 seconds in a Hobart mixer (model C-100) at low speed and then hand scraped. The calculated volume of water was added and mixed for two minutes. This mix was hand scraped and then mixed for two more minutes.

Molding

The strength test specimens molded were cylinders 2 ± 0.05 inches high and 2 inches in diameter. The wet lime and fly ash mixture was placed in the molding cylinder in one layer and compacted five times on each end by a five pound hammer dropping from a height of 12 inches^{4, 6}. The double plunger action of the apparatus resulted in uniform density throughout the specimen. The resulting density was approximately equal to standard Proctor density⁶. After molding, the specimen was extruded, weighed to the nearest 0.1 gram, and then measured to the nearest 0.001 inch. During molding, a wet cloth was kept over the bowl to prevent drying of the mixture.

Curing

Preliminary investigation. Specimens to be moist cured were wrapped in waxed paper, sealed with cellophane tape, and placed in a humidity room in which the temperature was $70 \pm 5^{\circ}$ F. and the humidity was above 80 percent. Specimens were cured 7 days before being totally immersed in water for 1 day.

Specimens to be oven cured were wrapped in Saran wrap (or two layers of waxed paper) and sealed with cellophane tape, and were placed in an oven at 100°F. for 6 days before 1 day immersion in water.

Final investigation. All specimens were wrapped in waxed paper, sealed with cellophane tape, and placed in a humidity room in which the temperature was $70 \pm 5^{\circ}$ F. and the humidity was over 80 percent. Specimens were allowed to cure for periods of 7 days, 28 days or 4 months before 1 day immersion.

Testing

All specimens were tested for unconfined compressive strength; the rate of load travel was 0.1 inch per minute. This testing procedure was selected because of its simplicity. Three specimens were tested for each condition, and average strengths were recorded. This is in accordance with ASTM specification designation C-109-58 which requires a minimum of three specimens for each set of curing conditions¹. A series of three observations is generally sufficient to detect any readings which deviate excessively. Specimens that differed by more than 10 percent from the average value of test specimens made from the same mix and tested at the same age were not considered in determining compressive strength. If two specimens were rejected, new specimens were prepared.

Procedure

Preliminary investigation. The work done during the preliminary study was to determine the moisture-strength and moisture-density relationships. Three specimens were molded at a given moisture content, weighed and measured $(2 \pm 0.05 \text{ inch})$ then retained for strength tests after 7 days curing and 1 day immersion. This procedure was continued by increasing moisture content 4-5 percent each time. Moisture-density and moisture-strength curves were then plotted to determine the optimum moisture contents for density and strength.

Final investigation. The specimens for final work were molded at the moisture contents found in the preliminary work. When the optimum moisture contents for strength and density did not coincide, a compromise was made by selecting a moisture content which minimized the total percentage deviation from both maximum strength and maximum density. An example of this procedure is given in Appendix A. Nine specimens of each mix were molded at the selected moisture content, wrapped and placed in the humidity room. Specimens were cured as follows, before 1 day immersion and strength testing:

Specimen Nos. 1, 4, 7 Specimen Nos. 2, 5, 8 Specimen Nos. 3, 6, 9 4 months.

DISCUSSION

Fly ashes from different sources vary considerably in both chemical and physical properties due to differences between the coals from which they are derived and the conditions of burning and collection. Consequently, it is to be expected that their behavior in concrete or stabilized soils will not be uniform. This investigation was conducted primarily to study the pozzolanic behavior of fly ash as shown by its effect on the strength of cured specimens of lime fly ash mixtures. The strengths produced by the mixtures were used to study the relations between pozzolanic activity and the various chemical and physical properties of fly ash.

The preliminary study included analysis of the physical and chemical properties of fly ash and experiments to determine optimum molding moisture contents of lime fly ash mixtures for maximum unconfined compressive strength after 7 days curing and 1 day immersion and for maximum dry density. The final study included strength tests to determine relative immersed strengths produced by the mixtures after 7, 28 days and 4 months curing and to correlate these strengths with physical and chemical properties of the fly ash.

Preliminary investigation

Fly ash analyses. The results of physical and chemical analyses of the

fly ashes used were compared to determine if there was any good correlation between physical properties and chemical composition. The percent carbon and the percent $SiO_2 + R_2O_3$ ($R_2O_3 = Al_2O_3 + Fe_2O_3$) in each fly ash were compared with the percent of the total sample passing the No. 200 and No. 325 sieves (figure 1). The data show that sieve analyses give indications of both the percent carbon and the percent reactive materials ($SiO_2 + R_2O_3$) in fly ash.

The various fly ash collection methods were compared to determine the



Fig. 1. Comparison between fineness and chemical composition of fly ashes.

influence of this variable on physical properties and chemical composition of the fly ashes used (table IV). Generally the fly ashes collected by electrical methods are finer and hence contain more reactive material and less

~



Decementer	Floatminel	Machanical	Electrical- mechanical
roperty	meetillai	meenannear	
No. 100	96.45	90.11	98.1
2.00 200	(91.6-99.4)	(53.0-98.2)	(97.2-99.0)
No. 200	87.35	76.10	93.73
	(73.7-94.5)	(24.7-91.6)	(93.2-94.1)
No. 325	73.3	49.85	72.5
	(47.0-86.1)	(14.0-80.2)	(68.2 - 75.7)
S.G.	2.39	2.48	2.54
	(2.26-2.60)	(2.04 - 3.43)	(2.39 - 2.61)
Sp. Surf.	3628	2442	2913
	(2539 - 4550)	(576-4550)	(2720-3070)
SiO_2	45.54	35.(4	43.0
	(35.94 - 57.50)	(11.26-43.74)	(41.0-47.0)
Fe_2O_3	17.56	28.20	40.10 (10.40.95.94)
	(6.0-22.30)	(11.9-08.4)	(10.40-20.04)
Al_2O_3	- 19.68	10.08	40.18 (99.48-94.90)
a' a	(10.0-23.4)	(0.90-54.7)	918
CaO	4.81	(0.04 (011097)	(1.65-2.68)
N ((1.2-0.9)	(2.1-12.21)	1 30
MgO	1.00	(0.17 1.32)	(0.84-2.08)
90	(0.80-1.0)	1 70	0.68
SO_3	(0.45.2.2)	(0.8-3.4)	(0.55 - 0.79)
C	(0.40-2.0) 6 07	8 56	4.67
U	(1.0-15.59)	(0.15-28.06)	(3.5-6.0)
	(1.0-10.00)	(0.10 10.00)	(0.0 0.0)

TABLE IV. COMPARISON OF PHYSICAL PROPERTIES AND CHEMICAL COMPOSITION OF FLY ASHES ACCORDING TO METHOD OF COLLECTION.*

*Upper figures are averages. Lower figures indicate maximum and minimum.

carbon that the fly ashes collected by mechanical methods. The three fly ashes collected by combination electrical-mechanical methods appear to have physical properties and chemical properties similar to the fly ashes collected by electrical methods. However, since only three fly ashes were collected by electrical mechanical methods, no definite conclusions should be drawn.

The specific surface of each fly ash sample was compared with the percent carbon and the percent passing No. 325 sieve as shown in figure 2, but the correlation was not good. The fly ashes which varied considerably from the others were those which were exceptionally coarse (fly ash samples 10 and 13) or had exceptionally high carbon content (fly ash samples 7 and 13). It appears that specific surface is a function of fineness and carbon content. It is believed that the specific surface is influenced by both the external and internal surfaces of particles of carbon. Consequently, increase in the carbon content of fly ash may be accompanied by an increase in total surface area even though the external surface area and the actual fineness of the fly ash decreases³. Therefore, specific surface was not used in further correlation studies.

Since the correlation studies also were to be made on the basis of chemical composition of fly ashes used, a table of mole fractions was prepared. Mole fraction is the number of moles of one component divided by the total number of moles of all substances present¹⁴. For this study, the percent

by weight of unknown chemical components (approximately 5 percent) was neglected. The percent by weight of each component was divided by the formula weight (table V) and the mole fraction was obtained. The mole fractions of chemical components is shown in table VI. The procedure used to obtain mole fractions is shown in Appendix B.

Moisture density and moisture strength studies. Initially, moisture density and moisture strength studies were made for all fly ashes with calcitic hydrated lime at ratios of 1:4 and 1:9. Mixtures for which all specimens failed during immersion after moist curing for 7 days at 70° F. were re-evaluated after 6 days at 100° F.

Five fly ashes, samples 2, 6, 7, 11 and 19, were selected for studies with dolomitic lime in a ratio of 1:4. They were selected as representative of

TABLE V. FORMULA WEIGHTS OF CHEMICAL COMPONENTS OF FLY ASHES USED.14

Components	Formula weight*
$egin{array}{c} { m SiO}_2 \ { m Fe}_2{ m O}_3 \ { m Al}_2{ m O}_3 \ { m CaO} \end{array}$	60 160 102 56
$egin{array}{c} MgO\ SO_3\ C \end{array}$	40 80 12

*Based on molecular weights of elements in each component.

TABLE VI. MOLE FRACTIONS OF CHEMICAL COMPOSITION OF FLY ASHES USED.*

гц							
ash			Chem	ical compon	ents †		
no.	SiO_2	Fe_2O_3	Al_2O_3	CaO	МgО	SO_3	С
1	40.9	9.3	14.4	2.3	3.1	.5	29.5
2	38.0	7.9	12.6	2.2	1.2	.07	27 A
3	52.9	7.8	15.8	$\bar{2.0}$	1.4		10.6
. 4	45.5	10.6	14.3	3.1	16	.0 R	94.9
5	58.6	2.3	13.3	1.3	1.2	.0 .4	$24.3 \\ 22.9$
6	47.4	9.2	13.8	6.6	1.2	2.9	18.9
7	23.3	4.0	6.0	5.7	.9	7	59 /
8	36.2	9.1	12.4	3.7	15	15	25.6
9	38.5	6.1	8.7	5.7	17		28 6
10	19.7	45.4	.9	23.1	.6	4.2	6.1
11	56.6	19.5	10.9	8.8	.7	25	1.0
12	29.8	4.7	8.2	2.7	.2	2.0	53.8
13	14.6	4.3^{-1}	3.0	4.6	.5	.0	72.6
14	42.6	19.7	5.5	6.9	1.6	1.2	22.5
15	45.1	13.1	8.1	14.4	1.3	1.6	16.4
16	37.3	7.3	6.8	10.5	.4	1.4	36.3
17	46.1	6.5	12.7	7.5	1.7	18	237
18	49.6	8.8	16.0	7.1	1.4	2:0	15 1
19	39.3	4.2	19.3	2.2	1.9	6	30.5
20	25.4	5.2	7.5	5.2	.9	1.0	54.8
21	63.9	9.5	7.3	8.5	3.0	16	62
22	42.4	7.9	11.3	5.7	1.0	6	21 1

*Based on determined composition only. †By percent of assumed total moles present.

the other fly ashes tested. A comparison of the optimum moisture contents for dolomitic lime fly ash mixtures and calcitic lime fly ash mixtures shows that they were approximately the same. Since the optimum moisture contents were so close, verifying previous findings by other investigators¹⁵, it was decided to use the same moisture contents for both limes. It was observed at this time that mixtures with dolomitic lime gave higher densities and strengths than mixtures with calcitic lime.

It was observed that the moisture content for maximum strength was generally below the moisture content for maximum dry density (figure 3). It should be pointed out that with fly ash sample 9 the moisture content for maximum dry density was not obtained, because the specimens were too plastic and slumped slightly, which made measurements inaccurate.

The moisture content for maximum dry density of lime fly ash mixtures correlates reasonably well with the percent carbon content (figure 4), and to a lesser extent with specific surface of the fly ashes. This relationship between moisture content and carbon content agrees with previous findings^{3, 6}. Some of the exceptions to this are fly ashes samples 13 and 20 which are extremely coarse. These observations seem to indicate that the molding moisture requirements for lime fly ash is a function of the carbon content and the fineness of the fly ash.

It was observed in molding the lime fly ash mixtures that in some cases the mix behaved as a poorly graded sand, and in other cases "bulked" (e.g.: fly ash sample 7). This "bulking" was not unexpected, since many of the fly ash particles are spherical in shape. The moisture density curves generally were very flat over a range of 5 to 8 percent of moisture content, with a gradual slope on the dry side and a steep slope on the wet side of optimum moisture content.

In those data in which the moisture content for maximum strength and maximum dry density did not coincide, a mathematical procedure was used to estimate the moisture for final molding. This moisture content was a compromise at which there was the least total percent deviation from maximum strength and maximum density. The procedure is shown in Appendix A.

Final investigation

The fly ashes were mixed with both calcitic and dolomitic limes in ratios of 1:4 and 1:9 by weight at moisture contents previously determined, and specimens were prepared for unconfined compression testing. The results of the unconfined compression tests were analyzed to determine the relative strengths with different fly ashes and the influence of fly ash physical properties and chemical composition and type or quantity of lime on strength.

Unconfined compression test results. The lime fly ash mixtures were prepared, and nine specimens were molded for each mixture. The speci-



Fig. 3. Comparison of optimum moisture contents for maximum strength and maximum dry density for calcitic lime fly ash mixtures.



Fig. 4. Carbon content of fly ashes versus optimum moisture content for maximum dry density of calcitic lime fly ash mixtures.

mens were tested as follows: three after 7 day curing, three after 28 day curing, and three after 4 month curing. All specimens for this investigation were cured at $70 \pm 5^{\circ}$ F. and immersed in distilled water for 1 day before testing for strength. The strengths after immersion for calcitic lime fly ash mixtures (figure 5) and for dolomitic lime fly ash mixtures (figure 6) are arranged in order of decreasing 28 day cured strengths.







The strengths are for lime fly ash mixtures with no soil; soil could greatly influence the strengths.

The 7 day strength results for calcitic lime fly ash mixtures with fly ash samples 1 through 5 were much lower than obtained during the preliminary investigation. It is believed that this was a result of low temperatures during the first week of curing the final investigation specimens, the temperature averaging around 65-67°F. It is believed that tempera-



Fig. 6. Unconfined compressive strength of specimens of dolomitic lime fly ash mixtures moist cured 7 days, 28 days or 4 months and immersed in water 1 day.

ture variations did not seriously influence strengths of calcitic lime fly ash mixtures with fly ash samples 6 through 22 or any of the dolomitic lime fly ash mixtures. The results of strength tests for fly ash samples 1 through 5 mixed with calcitic lime are not included in this study because of their doubtful validity.

A comparison of unconfined compressive strengths indicates that there is no correlation between 7, 28 and 120 day strengths (figures 5, 6). This agrees with previous findings⁹. Some fly ashes react quickly with lime and achieve maximum strength early; other fly ashes react slowly and attain near maximum strengths after long curing times. The speed of the reaction is greatly influenced by the solubility of any chemical substances present at the start of the reaction¹⁰.

The 7 day strengths were not used in correlation studies because of the temperature variations that occurred during the final investigation and because in such a short period the pozzolanic activity has not developed sufficiently. In the presentation of results more emphasis will be given to those obtained after 28 day curing as this is a standard curing period used with these materials.

The Iowa produced fly ashes are compared with other fly ashes (table VII). Since all the Iowa fly ashes were collected by mechanical methods, they also have been compared with other mechanically collected fly ashes.

In both comparisons, the Iowa produced fly ashes are inferior pozzolans. One explanation for this is that the Iowa fly ashes are a random selection, and most of the other fly ashes have been accepted for commercial use in concrete.

Effects of fly ash physical properties and chemical composition on pozzolanic behavior. Comparisons were made between each fly ash's physical properties and chemical composition and the 28 and 120 day strengths obtained in lime fly ash mixtures. At the time this investigation was undertaken, it was generally recognized that finely divided, low carbon fly ashes were most beneficial in soil lime fly ash stabilization. However, there was no general agreement as to the limits which should be placed on fine-

TABLE VII. COMPARISON OF STRENGTHS OBTAINED FROM IOWA PRODUCED FLY ASHES WITH STRENGTHS OBTAINED FROM OTHER FLY ASHES.

	Average 28 day unconfined				
	compressive strengths, psi*				
	Iowa	mechanically	Other		
	fly	$\operatorname{collected}$	fly		
Lime: fly ash mixture	ashes†	fly ashes	ashes		
Calcitic lime-fly ash, 1:4	245	553	571‡		
Calcitic lime-fly ash, 1:9	222	444	507^{+}_{-}		
Dolomitic lime-fly ash, 1:4	295	662	635		
Dolomitic lime-fly ash, 1:9	245	547	505		

*After 1 day immersion.

All mechanically collected. Does not include fly ashes 1 through 5.

ness and carbon content or the extent to which these properties could be relied upon for selecting fly ashes. It was hoped that the information obtained in this investigation would provide a suitable basis for the selection of fly ashes for soil stabilization. Since the lime fly ash reaction is chemical, it seems a logical assumption that the chemical composition and physical properties of a fly ash should correlate with strengths obtained with the lime fly ash mixtures.

An attempt was made to divide the fly ashes into several groups having similar characteristics (e.g.: percent passing No. 325 sieve or silica content). It became obvious that the number of variables and range within each variable were too great for the number of observations. For this reason, all fly ashes were taken as a group for the correlation studies.

Values of 28 day cured strengths of lime fly ash mixtures were plotted against the percent by weight of fly ash passing the No. 325 sieve. The amount of material passing the No. 325 sieve showed a fairly definite relation to strength. A second trial was then made in which strength was plotted against values of percent passing the No. 325 sieve divided by percent retained on No. 325 sieve. The results of these trials are shown in figures 7 and 8. There are two factors contributing to the good correlation. The first is that fineness has been found to be a general indicator of those chemical components of fly ash which are considered to be reactive. The second is that the finer particles increase the surface area and hence increase the rate of reaction of fly ash with lime. The same condition was found for the 4 month cured specimens.

Values of fly ash specific surface were not plotted against strengths because the preliminary studies indicated that specific surface was a function of fineness and carbon content. Previous studies of fly ash in cement fly ash mortars indicated that no definite correlation existed between specific surface and unconfined compressive strength³.

In molding specimens, it was observed that several fly ashes having approximately the same specific gravity gave widely differing densities in compacted lime fly ash mixtures. Later, in testing and in attempting to correlate strengths with fly ash characteristics, it was found that some of the lime fly ash mixtures which gave exceptionally high strengths also gave exceptionally high densities. Conversely some of those fly ashes which gave low strengths also gave low densities.

The percent solids and void ratios of compacted lime fly ash mixtures were calculated from the dry densities of lime fly ash mixtures at the time of molding and from the appropriate specific gravity of each lime fly ash mixture. Closer analysis indicated that those fly ashes collected electrically generally gave mixes with a higher percent solids than fly ashes collected mechanically. Values of strengths were plotted against the product of percent solids in the lime fly ash mixtures and the percent by weight of fly ash passing the No. 325 sieve, and against the product of percent solids in the lime fly ash mixture and the percent by weight of fly ash passing the No. 325 sieve divided by the percent by weight of fly ash retained on the No. 325 sieve. These results, for 28 day curing, are shown in figures 9 and 10. The authors assumed that strength of lime fly ash mixes increases linearly with increase of percent solids. Two explanations are offered for the variations in percent solids: particle size distribution of the fly ash and electrostatic charges on the fly ash particles. If the lime fly ash mixture is well graded (influenced by gradation of both lime and fly ash particles) a much denser mix (higher percent solids) will be ob-



Fig. 7. Comparison of 28 day unconfined compressive strength of lime fly ash mixtures with fineness of fly ashes.

tained than with a poorly graded mix. If there is an electrostatic charge on the fly ash particles, there is a possibility that like charges on all particles could cause repulsion, thus preventing attainment of high percent solids.

The strengths were plotted against the total amounts of each of the inorganic components of the fly ashes given in table II. These include silica, iron oxide, alumina, calcium oxide, magnesium oxide and sulfur trioxide. The amounts of inorganic components were given both in percent by weight and in mole fraction. No definite relationship was found between strength of lime fly ash mixtures and the percent by weight of the different fly ash components. However, the mole fraction of silica correlated fairly well with strength. Plots of silica content (percent by weight



Fig. 8. Comparison of 28 day unconfined compressive strength of lime fly ash mixtures with percent of fly ash passing No. 325 sieve divided by percent retained on No. 325 sieve.

and mole fraction) are shown in figures 11 and 12. This gives an indication that mole fractions are more favorable for correlation studies.

Brink and Halstead³ have shown that the strengths of fly ash cement mortars (with standard sand) are adversely affected by fly ashes having high carbon contents. A similar trend is evident in figures 13 and 14, in which the relation between the 28 day pozzolanic strength and carbon content is shown. As with silica content, the plot of carbon content in mole fraction versus strength of lime fly ash mixtures gives a slightly better relationship than carbon content in percent by weight versus strength. Three factors are thought to be responsible for the reduced strength with increased carbon content. First it has been found from absorption tests that the organic material in fly ash does not react with Ca(OH)₂⁹. Second, the organic material occurs predominantly in the coarse fractions



Fig. 9. Comparison of 28 day unconfined compressive strength with percent solids of lime fly ash mixtures times percent of fly ash passing No. 325 sieve.

of a fly ash and its presence indicates coarse inorganic material of low reactivity. Third, the carbon particles are structurally weak.

The triangular chart concept was applied in which the various chemical components were grouped into three categories according to assumed influence on strength: favorable, neutral and unfavorable. The triangular chart procedure was similar to that used for textural classification of soils in which the three sides of the equilateral triangle are graduated from 0 to 100 percent representing sand, silt, or clay sizes. Triangular charts were prepared (see Appendix C for procedure) for percent by weight and for mole fraction of the chemical components of fly ashes used for the following combinations.

 SiO_2

R₂O₃

Trial No. 1

favorable

neutral



Fig. 10. Comparison of 28 day unconfined compressive strength with percent solids of lime fly ash mixtures times percent of fly ash passing No. 325 sieve divided by percent of fly ash retained on No. 325 sieve.

Trial No. 2	$egin{array}{c} { m SiO}_2\ { m R}_2{ m O}_3+{ m CaO}+{ m MgO}\ { m SO}_3+{ m C} \end{array}$	favorable neutral unfavorable
Trial No. 3	$egin{array}{l} { m SiO}_2+{ m Al}_2{ m O}_3\ { m Fe}_2{ m O}_3+{ m CaO}+{ m MgO}\ { m SO}_3+{ m C} \end{array}$	favorable neutral unfavorable
Trial No. 4	$egin{array}{l} { m SiO} + { m R_2O_3} \ { m CaO} + { m MgO} \ { m SO_3} + { m C} \end{array}$	favorable neutral unfavorable

The results obtained with mole fractions showed slightly better trends than those obtained for percent by weight in all four trials. Trial No. 4 appeared to give better results than did trials No. 1-No. 3. Those strengths on the triangular chart which varied considerably from adjacent strengths were probably influenced by fineness of fly ash or percent solids of lime fly ash mixtures. The application of the triangular chart



Fig. 11. Comparison of 28 day unconfined compressive strength with silica content (percent by weight) of fly ashes.

concept to the correlation of chemical components of fly ashes with strength of lime fly ash mixtures appears to be promising.

Twenty eight day strengths were also plotted against $SiO_2 + R_2O_3$ contents (percent by weight and mole fraction) of the fly ashes. The results using the mole fraction are shown in figure 15. As before, the mole fraction percentages gave better trends, showing a definite relationship between increase in strength and increase in $SiO_2 + R_2O_3$.

The next correlations attempted were between unconfined compressive strength of lime fly ash mixtures and combinations of the physical properties and chemical components of fly ashes which have shown the most promise in previous trials.

The following combinations were tried:

Percent of fly ash passing No. 325 sieve divided by carbon content





Product of percent solids in the lime fly ash mixture and percent

 $SiO_2 + R_2O_3 - C$ in the fly ash.

Product of void ratio of the lime fly ash mixture and carbon content of the fly ash. None of these gave correlations that were considered to be better than obtained in previous trials.

Variations of type of lime and lime fly ash ratio. The averages of the results indicate that the dolomitic lime gave 28 day strengths approximately 30 percent greater than calcitic lime. This average figure is very misleading, because the use of dolomitic lime in lime fly ash mixtures can give lower strengths than calcitic lime when mixed with some fly ashes.

The dolomitic lime fly ash mixtures generally gave higher densities than the calcitic lime fly ash mixtures. No correlation was found between the ratio of densities (dolomitic lime fly ash + calcitic lime fly ash) of com-



Fig. 13. Comparison of 28 day unconfined compressive strength with carbon content (percent by weight) of fly ashes.

pacted lime fly ash mixtures and the ratio of strengths. The strength ratios were then compared with contents of SiO_2 , CaO, MgO, and Ca/Mg ratio of fly ashes to determine if there was any correlation between chemical composition and effect of calcitic and dolomitic limes. Since no correlation between chemical composition of fly ash and effectiveness of the two limes was obtained, it is believed that one of the chemical components of fly ash not determined could have influenced the reaction. A good possibility is the alkali content of the fly ash¹⁰.

Tests were made to determine the alkalinity of the fly ashes using a





Leeds and Northrup pH meter and pH paper. No good correlations were obtained between pH and effectiveness of the limes. Variations in pH readings between the two test methods indicate the presence of electrostatic charges that could influence the lime fly ash reaction. Further study should be made of this problem.

CONCLUSIONS

Results obtained from the investigations indicate the following: 1. The specific surface of fly ash is a function of its fineness and carbon

content and hence is not a good criterion of strength producing quality or reactivity with lime.

 2. Fly ashes collected in power plants by electrical methods generally are finer and contain less carbon than fly ashes collected by mechanical means.
 3. The percent by weight of fly ash passing the No. 325 sieve gives a general indication of the carbon content and amount of reactive material.



Fig. 15. Comparison of 28 day unconfined compressive strength with $SiO_2 + R_2O_3$ content (mole fraction) of fly ashes.

4. The optimum moisture content for maximum strength of lime fly ash mixtures is generally below optimum moisture content for maximum dry density.

5. The optimum moisture content for maximum dry density of lime fly ash mixtures is a function of carbon content and fly ash fineness.

6. Optimum moisture contents for maximum strength and maximum dry density are approximately the same for lime:fly ash ratios of 1:4 and 1:9.

7. The average strengths obtained with seven Iowa produced fly ashes are approximately 50 percent of the corresponding strengths obtained with the other fly ashes tested.

 No correlation was found between the chemical composition of fly ash and the influence on strength by varying type of lime or lime:fly ash ratio.
 Generally, the use of dolomitic monohydrate lime produces strengths 30 percent greater than calcitic hydrated lime. However, with some fly ashes calcitic lime produces greater strength than dolomitic.

10. Generally, with standard Proctor compaction, dolomitic lime fly ash mixtures are denser than calcitic lime fly ash mixtures. No correlation was found between increased densities and strengths.

11. When curing lime fly ash mixtures at a temperature of 70°F., prolonged variations in temperature of \pm 5°F. produce significant variations in strength.

12. No correlation was obtained between 7, 28, and 120 day strengths when lime fly ash mixtures were cured at 70°F.

13. Compacted lime fly ash mixtures generally have a higher percent solids with electrically collected fly ashes than with mechanically collected fly ashes.

14. The chemical composition of fly ash expressed in mole fractions correlates with strength better than chemical composition expressed in percent by weight.

15. The application of the triangular chart concept to the correlation of chemical composition with strength appears to offer promising results.

16. The simplest, most reliable and most economical tests on which to base selection of a fly ash for soil lime fly ash stabilization are: sieve analysis using No. 325 sieve, carbon content determined by loss on ignition, and compaction tests to determine percent solids.

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APPENDIX A.

PROCEDURE FOR SELECTING MOISTURE CONTENTS FOR MOLDING STRENGTH SPECIMENS

The curves for unconfined compressive strength versus moisture content and dry density versus moisture content were plotted and optimum moisture contents for maximum strength and maximum dry density were determined. The moisture content for molding strength specimens is a compromise, and the moisture content at which the total percent deviation from maximum strength and maximum dry density is a minimum.

An example of this procedure is shown for fly ash sample 18 in a calcitic lime fly ash mix with a ratio of 1:4. The results of the moisture-strength and moisture-density study have been plotted on figure 16 and curves drawn. The optimum moisture contents obtained are:

Maximum unconfined compressive strength = 131 psi at 24 percent MC Maximum dry density = 91 pcf. at 27 percent MC

The total percent deviations were calculated based on figure 16 and are shown in table VIII. The curve of total percent deviation versus moisture content is shown in figure 17, which indicated a minimum point at a moisture content of 24.5 percent.





TABLE VIII. TOTAL PERCENT DEVIATION FROM MAXIMUM STRENGTH AND MAXIMUM DRY DENSITY FOR FLY ASH SAMPLE 18 FOR/OR CALCITIC LIME FLY ASH RATIO = 1:4.



Fig. 17. Molding moisture content versus total percent deviation from both maximum 7 day unconfined compressive strength and dry density for fly ash sample 18 at calcitic lime to fly ash ratio = 1:4.

APPENDIX B.

PROCEDURE USED FOR OBTAINING MOLE FRACTIONS

In determining the mole fractions of the various chemical components, a two-step procedure was used as follows:

Step 1. Divide the percent by weight of each component by the formula weight to obtain the proportional parts of molecules.

Step 2. Divide the proportional parts of each component by the total proportional parts to determine the percent of all molecules present or mole fraction.

An example of this procedure is shown below for fly ash sample 18.

COMPONENT	STEP 1	STEP 2
$\begin{array}{c} \mathrm{SiO}_2 \\ \mathrm{Fe}_2\mathrm{O}_3 \\ \mathrm{Al}_2\mathrm{O}_3 \\ \mathrm{CaO} \\ \mathrm{MgO} \\ \mathrm{SO}_3 \\ \mathrm{C} \end{array}$	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$\begin{array}{c} .71 \ /1.433 = 49.6 \\ .126/1.433 = 8.8 \\ .229/1.433 = 16.0 \\ .102/1.433 = 7.1 \\ .020/1.433 = 1.4 \\ .029/1.433 = 2.0 \\ .217/1.433 = 15.1 \end{array}$
e	1.433	100.0





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APPENDIX C.

USE OF TRIANGULAR CHART TO CORRELATE CHEMICAL COMPOSITION OF FLY ASHES WITH STRENGTH OF LIME FLY ASH MIXTURES

The chemical components of the fly ashes were divided into three groups: favorable, neutral, and unfavorable, according to an assumed effect on the lime fly ash reaction. When considering the components in percent by weight, the undetermined components were assumed to be unfavorable. A point was plotted for each fly ash on the triangular chart (separate chart for each mixture) and the 28 day unconfined compressive strength was written beside it. It was hoped that the strengths would be grouped so that iso-strength contours could be drawn.

An example of this procedure is shown in table IX and figure 18 for fly ash samples 9, 13 and 21 when mixed with dolomitic lime at a ratio of 1:4.

TABLE IX. CHEMICAL COMPOSITION OF SELECTED FLY ASHES ACCORDING TO COMPONENTS ASSUMED EFFECT ON LIME FLY ASH REACTION.

	Total percents* of components in each fly ash				
Component groups	Sample 9	Sample 13	Sample 21		
Favorable $(SiO_2 + R_2O_3)$	53.3	21.9	80.7		
Neutral $(CaO + MgO)$	7.4	5.1	11.5		
Unfavorable $(SO_3 + C)$	39.3	73.0	7.8		

*Percent by mole fraction as shown in table VI. .

PHYSICAL AND MINERALOGICAL FACTORS IN STABILIZATION OF IOWA SOILS WITH LIME AND FLY ASH

by

Manuel Mateos, Instructor, Civil EngineeringD. T. Davidson, Professor, Civil Engineering(Progress Report, August, 1961.)

ABSTRACT

The purpose of this investigation was to study the physical and mineralogical factors or variables that may influence the stabilization of Iowa soils with lime and fly ash. Four natural soils (a dune sand, a friable loess, a gumbotil and an alluvial clay), eight different fly ashes, and five limes were used.

Test specimens 2 inches high by 2 inches in diameter were prepared from the different mixtures made with the proper moisture content. Stability evaluations were based on unconfined compressive strength of test specimens subjected to various curing and simulated weathering treatments.

The effects on stability studied included:

(1) moisture-density and moisture-strength relationships;

(2) proportions and amount of lime and fly ash;

(3) degree of densification;

(4) temperature at time of compaction and during curing;

(5) delay of compaction after wet mixing;

(6) use of small amounts of chemical additives; and

(7) modification of the quality of a fly ash by grinding or by discarding the coarse fraction.

Also a comparison was made of soil, lime, and fly ash, soil and lime, and soil and cement stabilization, which included freeze-thaw testing of selected mixtures.

The test results show that the pozzolanic activity of different fly ashes varies greatly, and that this activity varies with the type of lime used. The maximum strength occurs generally at a moisture content other than the optimum moisture content for maximum density. The compaction moisture content for maximum strength changes with the curing period; the longer the curing period the higher the moisture content required to give maximum strength. Modified Proctor compaction gives strengths about 100 percent greater than standard Proctor compaction. Compaction should be done as soon as possible after wet mixing clayey soils; it may be delayed for a day after wet mixing sandy soils. There is no optimum
amount of lime and fly ash for stabilizing all soils. The strength obtained is greatly influenced by the curing temperature: the higher the curing temperature the greater the strength obtained. After one day steam curing, compacted mixtures of soil, lime and fly ash may reach strengths comparable with the strength of concrete. The strength gain of friable soils stabilized with lime and fly ash may be accelerated by adding small amounts of some chemicals. Sodium carbonate appears to be the most promising chemical. The quality of a fly ash may be improved by grinding or by removing the coarse fraction.

It can be concluded that stabilization of soils with lime and fly ash is a sound method for building road base courses. Fly ash of a high quality should be used. A high quality fly ash with lime, dolomitic monohydrate lime generally preferred, may stabilize a soil to the extent that strengths after 28 days curing are comparable to those of soil cement (500 to 900 psi) on an economically competitive basis. Compacted mixtures of soil, lime, and fly ash withstand the destructive effects of alternate freezing and thawing.

INTRODUCTION

Soil, cheap and abundant, has been used for thousands of years as a construction material. In its natural state it generally has very poor engineering qualities, but they are improved by ramming. The introduction in the early Thirties of the concepts of lubrication effects of water and of an optimum moisture that produces a maximum density for a compactive effort gave the soil engineer new tools in the improvement of a soil for its use as an engineering material⁵⁹. This concept of the moisture density relationship was applied to soils treated with admixtures, and from this a separate science of soil stabilization has developed.

Several soil admixtures are used today to obtain a construction material with better engineering properties than those of the original soil. The most extensively used are cement and lime. Others, like lime with fly ash, appear to be satisfactory stabilizers; but they have not been much used because their characteristics and behavior when added to soils are not well known. Many other admixtures are being evaluated in the laboratory before subjecting them to field testing.

The importance of the construction program of the vast network of interstate highways has given additional emphasis to the investigations for new and better methods of soil stabilization. These investigations may bring some economy to the expenditures for the program. In a recent report on highway research the item for which the greatest amount of money was recommended was the improvement of knowledge of aggregates and soils; a total of ten million dollars annually was suggested³⁸.

During the last ten years the Engineering Experiment Station of Iowa State University, in cooperation with the Iowa State Highway Commission and the Iowa Highway Research Board, has been conducting an extensive evaluation of different methods of soil stabilization for road base and subbase courses. Special attention has been given to the use of cheap available chemicals and by-products or wastes. One of them is fly ash, which together with lime can be used in soil stabilization.

The work done until now to evaluate lime plus fly ash as an admixture to soils has been very restrictive. General conclusions as to the use of these materials have been drawn based on results obtained with a limited variety of the component materials—soil, lime and fly ash. And the testing has been limited. Insufficient knowledge of a method or process may lead to an erroneous evaluation of its qualities or properties. An attempt has been made in this investigation to introduce a reasonable number of variables in the main components: soil, lime and fly ash. Other factors had to be studied also: the investigation for this report was conducted to obtain information on the following aspects of soil, lime, and fly ash stabilization:

- 1. Lime and fly ash proportions and amount
- 2. Moisture to density to strength relationships
- 3. Effect of compactive effort
- 4. Effect of curing temperature
- 5. Influence of temperatures of component materials at time of compaction
- 6. Effect of delay of compaction after wet mixing
- 7. Effect of chemical additions on the lime fly ash reaction and their effects with soils
- 8. Study of the modification of fly ashes
- 9. Comparison with other methods of soil stabilization
- 10. Final evaluation including freezing and thawing tests.

REVIEW OF LITERATURE

As an artificial pozzolan, fly ash can be used in any of the numerous applications in which pozzolans are used, providing its quality competes with other available pozzolans^{7, 20, 21, 51, 56}. Mixtures of pozzolan, lime and water form a cement that was extensively used by the Romans. The name *pozzolan* comes from the city of Pozzuoli near Vesuvius and the bay of Naples, Italy, where the Romans quarried a volcanic ash. Roman structures built 2,000 years ago and still standing attest the quality and durability of pozzolanic cements.

Development of soil, lime, and fly ash stabilization

Much research has been done to find new uses for fly ash, but much more of the ash is produced than can be used. The principal uses have been as a filler in grouting materials, as an ingredient in the manufacture of building blocks and in Prepakt concrete, as a pozzolan in Portland cement concrete, and as an admixture with lime in soil stabilization^{9, 11, 47, 51, 66}. In 1934 a patent was granted on the use of fly ash with an aklaline earth base as a structural material⁵⁸. The cementitious properties of fly ash mixed with lime an water were studied in 1940; after that several compositions of soil, lime, and fly ash for use in base and subbase courses of pavements were studied^{13, 24}. Patents on the use of lime and fly ash with fine aggregate were issued in 1951, 1954, and 1957 on the use of lime and fly ash for stabilizing finely divided materials such as soils^{34, 35, 36}.

The first field trials of soil, lime and fly ash mixtures were made in the construction of a number of by-passes, interchanges and shoulders of the New Jersey Turnpike. They are reported to be satisfactory^{52, 53, 54}.

Work in the Iowa Engineering Experiment Station has indicated that about 25 percent lime and fly ash in ratios varying between one lime to nine fly ash and one lime to two fly ash can be used satisfactorily for stabilizing various textured soils^{14, 22, 28, 55, 64}. Higher ratios are required for clayey soils^{15, 40}. Dolomitic monohydrate lime produces higher strength than high-calcium hydrated lime in soil and lime mixtures with Iowa soils^{44, 48}. This was also true for soil, lime, and fly ash mixtures with Iowa soils^{14, 28, 63}. The highest compressive strength is at or just below the optimum moisture content for the standard Proctor compactive effort²⁸. High carbon fly ashes do not react with lime as well as the low carbon fly ashes; fineness is also a measure of the reactivity^{19, 63, 67}. The strength increases with the increase of fly ash content^{14, 49}. The addition of fly ash may not be necessary to lime stabilized soils containing large amounts of montmorillonite or kaolinite clays ³⁹ or of silt⁶⁴. The strength increases proportionately with the amount of compactive effort^{40, 68}. Increasing the time of mixing in a mechanical mixer, at constant speed, gives increased unconfined compressive strength²⁸. Test specimens were still gaining strength after a curing period of one year²⁸. The relative humidity during curing should be maintained as near 100 percent as possible²⁸.

The addition of calcium chloride to soil, lime, and fly ash mixtures increases its early strength^{28, 53, 54}. In field trials of soil, lime, and fly ash paving near Detroit, Michigan, the best field performance was obtained with a stony sand which had been treated with about 0.5 percent of calcium chloride six weeks prior to lime fly ash stabilization¹⁷. The higher early strength obtained in this road, and thus greater resistance to freezing, was attributed to an acceleration of the lime fly ash reaction by the calcium chloride.

The strength improvements when calcium chloride was added in small amounts to soil, lime, and fly ash mixtures suggested that other chemicals may produce similar strength increases. An investigation was made with 47 chemicals, and it was found that many of them improved considerably the early and long term strength of lime fly ash mixtures. Among the more promising are sodium carbonate, sodium and potassium hydroxides, lithium carbonate, potassium and sodium permanganates, potassium carbonates, sodium chloride, aluminum chloride, potassium and sodium bicarbonates, sodium sulfite and a sodium tetraphosphate^{18, 50}.

An evaluation of the most promising chemical additive, sodium carbonate, was then made^{22, 33, 49, 55}. As a result a patent was obtained on the use of sodium carbonate to accelerate the setting of lime, fly ash, and soil mixtures³².

A test road built with soil, lime, and fly ash was built near Colfax in 1958, and another near Fort Dodge in 1960. Both test roads have sections of base and/or subbase courses of soil treated with lime and fly ash⁴¹.

Mechanism of lime to fly ash reaction

When lime and fly ash are mixed with the soil, part of the lime combines with the soil particles, part with carbon dioxide present in the soil air and soil water, and part with fly ash in a pozzolanic reaction.

Lime reacts with the clay minerals in the soil in two ways. One, ionic in nature, is a complex reaction in which the excess of calcium cations supplied by the lime cause, by their crowding action on clay particles, a flocculation of the soil, and also an exchange of calcium for other cations in the clay structure. By this reaction soil plasticity is decreased, workability is greatly increased, and volume changes due to moisture are reduced. The other reaction, that takes place when the soil is in a compacted state, is pozzolanic in nature similar to the lime fly ash reaction. Fine silt size quartz minerals, in addition to clay minerals, are very likely involved in that reaction. Cementitious reaction products are formed which increase the bearing capacity of the soil.

Carbon dioxide combines with lime to form calcium carbonate or calcium magnesium carbonate, depending on the lime used. This combining is at a very slow rate in soil, lime, and fly ash mixtures. It has been found that the presence of carbon dioxide in the air does not affect the compressive strength of the silty and clayey soil, lime, and fly ash specimens¹⁴.

The main cementitious material created by the pozzolanic reaction is a hydrous calcium silicate; but since most pozzolans contain amounts of materials other than silica, other compounds involving iron, alumina, and the alkalies are likely formed $also^{10, 20, 21}$. Calcium silicates and aluminates have been identified in the reaction between lime and fly $ash^{8, 46}$. A compound has been isolated in the reaction between a lime and fly ash which is tentatively formulated as $[(Ca_{89} Na_{11}) O] [(Si_{75} Al_{25}) O_2] \cdot 9 H_2O^*$. Base exchange takes place between the pozzolan and lime, but this action is unlikely to be cementitious⁴⁵.

Pozzolans containing silica in amorphous forms react faster with lime than those containing silica in crystalline forms, and the rate of reaction varies inversely with crystal size²⁰. Strength increases with compacted density of soil, lime, and fly ash mixtures^{40, 68}. This may be due to an in-

^{*}Handy, R. L., Engineering Experiment Station, Ames, Iowa. Data on X-ray analysis of lime and fly ash mixtures. Private communication. February 24, 1961.

crease in the number of contact points among the soil particles providing greater bond by the cementitious microcrystals or gels.

The reactivity of pozzolans is correlated with the alkaline nature of lime pozzolan mixtures. The activation of silica by the hydroxyl ions plays an important part in the formation of calcium silicates. The maximum adsorption of calcium ions by quartz occurs at a pH of 11^{42} . A study of the adsorption of calcium by a clay showed that the amount of calcium adsorbed increases with increase of pH up to about pH 11^{12} . Therefore there seems to be an optimum pH for the formation of calcium silicates in the lime pozzolan reaction.

MATERIALS AND METHODS

Materials used

Soils

Four natural soils, a dune sand, a friable loess, an alluvial clay, and a heavily weathered glacial till, were selected as representative of important Iowa soil types (tables I and II).

Ottawa sand was used in the preliminary evaluation of the effects of chemical additives on the lime and fly ash reaction. It is a natural silica sand assumed to be unreactive with lime and water at the curing temperatures used. Its gradation met the requirements for graded standard sand (ASTM Designation: C109-58)⁴:

SIEVE SIZE			PERCENT PASSING		
No.	16	(1190-micron)	100		
No.	30	(590-micron)	98 ± 2		
No.	50	(297-micron)	28 ± 5		
No.	100	(149-micron)	2 ± 2		

Fly ashes

Eight fly ashes were selected to represent variations in the properties of this by-product material (table III).

TABLE I. DESCRIPTION OF NATURAL SOILS.

Soil	Dune sand (S-6-2)*	Friable loess (20-2)	Alluvial clay (627-1)	Kansan gumbotil (528-8)
Location	Benton County, Iowa	Harrison County, Iowa	Harrison County, Iowa	Keokuk County, Iowa
Geological description	Wisconsin-age eolian sand, fine grained, oxidized, leached	Wisconsin-age loess, friable, oxidized, calcareous	Recent fill, alluvial plastic, slightly calcareous	Kansan-age gumbotil, highly weathered, plastic, non-calcareous
Soil series	Carrington	Hamburg	None	Mahaska†
Horizon	С	С	Undefined	Fossil B
Sampling depth, ft	. 6-11	49-50	0-4	7.5-8.5

*Numbers in parentheses are those assigned by the Soil Research Laboratory of the Iowa Engineering Experiment Station. †Underlies C horizon loess of Mahaska series.

Fly ash No. 1, collected by multiple cyclone and electrical precipitators, was from coal from districts 3 and 8 in Ohio and from northern West Virginia. This fly ash was processed through pulverizing mills so that 70 percent passed a No. 200 mesh. The sample was sent from the St. Clair (Michigan) Power Plant of the Detroit Edison Company.

TABLE II. PROPERTIES OF NATURAL	SOILS.	
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Soil	Dune sand	Friable loess	Alluviai clay	Kansan gumbotil
Textural composition,* %: Gravel (> 2 mm.) Sand (2-0.074 mm.) Silt (0.074-0.005 mm.) Clay (< 0.005 mm.) Colloids (< 0.002 mm.)	0.0 95.5 1.5 3.0 2.6	0.0 0.7 82.3 17.0 14.0	0.0 2.4 25.6 72.0 61.0	$\begin{array}{c} 0.0 \\ 19.4 \\ 14.6 \\ 66.0 \\ 63.0 \end{array}$
Atterberg limits†: Liquid limit, % Plastic limit, % Plasticity index	Non-Plastic	32 25 7	$72\\26\\46$	76 26 50
Classification: Textural‡ Engineering (AASHO)§	Sand A-3(0)	Silty loam A-4(8)	Clay A-7-5 (20)	Clay A-7-5 (20)
Chemical: Cat. exch. cap.,** m.e./100 g pH†† Carbonates,‡‡ % Organic matter,§§ %	c. 1.0 6.6 0.4 0.1	14.5 8.4 10.4 0.1	$\begin{array}{r} 44.4 \\ 7.7 \\ 3.6 \\ 1.6 \end{array}$	39.2 7.4 2.0 0.1
Predominant clay mineral***	: Montmoril- lonite (trace)	Montmoril- lonite	Montmoril- lonite	Montmoril- lonite

*ASTM Method D422-54T³. †ASTM Method D423-54T and D424-54T³. †Triangular chart developed by U. S. Bureau of Public Roads 65, p. 47. §AASHO Method M145-49². **Ammonium acetate (pH = 7) method on soil fraction 0.42 mm. (No. 40 sieve). ††Glass electrode method using suspension of 15 g soil in 30 cc. distilled water. ‡†Versenate method for total calcium. §§Potassium bichromate method. ***X-ray diffraction analysis.

TABLE III. ANALYSIS OF FLY ASHES.

Fly ash no.	1	2	3	4
Source	St. Clair, Michigan	Cedar Rapids, Iowa	Louisville, Kentucky	Cedar Rapids, Iowa
Loss on ignition, %* Specific surface, Blaine (sq. cm./g.) Specific gravity Fineness (% passing no. 325 sieve)	3.92820.2.5891.8	$7.2 \\ 2663. \\ 2.39 \\ 49.8$	$2.6 \\ 3226. \\ 2.60 \\ 86.1$	$18.6 \\ 4550. \\ 2.37 \\ 54.9$
Silicon dioxide (SiO ₂), % Magnesium oxide (MgO), % Calcium oxide (CaO), % Aluminum oxide (Al ₂ O ₃), % Iron oxide (Fe ₂ O ₃), % Sulphur trioxide (SO ₃), %	$\begin{array}{c} 43.5 \\ 0.2 \\ 2.9 \\ 23.2 \\ 24.8 \\ 0.8 \end{array}$	36.7 1.0 3.5 21.3 24.3 2.0	$\begin{array}{r} 42.5\\ 0.8\\ 5.7\\ 23.4\\ 20.0\\ 2.3\end{array}$	$36.2 \\ 0.9 \\ 8.3 \\ 15.8 \\ 16.7 \\ 1.5$

*Approximately equal to carbon content.

Fly ash No. 2, collected by mechanical equipment, was from coal from northern Illinois. The coal was burned in a B & W boiler. This sample was sent from the Sixth Street power station in Cedar Rapids, Iowa, by the Iowa Electric Light and Power Company.

Fly ash No. 3 was collected by electrical precipitators from a dry bottom type of boiler using unwashed coal from western Kentucky. The sample was sent from the Paddy's Run Power Station at Louisville, Kentucky, by the Louisville Gas and Electric Company.

Fly ash No. 4, collected by mechanical precipitators, was from coal from northern Illinois burned in a Springfield boiler. This sample was sent from the Sixth Street power station in Cedar Rapids, Iowa, by the Iowa Electric Light and Power Company.

Fly ash No. 5 was collected by mechanical (centrifugal) precipitators. The coal from Illinois was pulverized in a ball mill prior to burning. The sample was sent from Riverside station power plant at Davenport, Iowa, by the Iowa-Illinois Gas and Electric Company.

Fly ash No. 6 was collected by mechanical precipitators (multicone dust collector). The coal from Iowa (Monroe, Polk, Marion and Mahaska Counties) was unwashed steam coal which was pulverized and tangential fired. The sample was sent from the Des Moines Power Plant by the Iowa Power and Light Company.

Fly ash No. 7 was collected by mechanical equipment (VGR multiclone). The coal from southern Illinois was washed, dried, and pulverized with Riley mills. The sample was sent from the Waterloo power plant by the Iowa Public Service Company.

Fly ash No. 8 was collected by mechanical precipitators (cyclone type). The coal from several Missouri and Kansas mines was pulverized and burned in suspension in combustion engineering boilers. The sample was sent from the Hawthorne Station power plant of the Kansas City Power and Light Company, Missouri.

TABLE III. Continued.

Fly ash no.	5	• 6	7	8
Source	Davenport, Iowa	Des Moines, Iowa	Waterloo, Iowa	Kansas City, Missouri
Loss on ignition, % Specific surface, Blaine (sq. cm./g.) Specific gravity Fineness (% passing no. 325 sieve)	$0.7 \\ 576. \\ 3.43 \\ 22.6$	$0.2 \\ 1460. \\ 2.82 \\ 31.8$	$13.9 \\ 4240. \\ 2.34 \\ 54.9$	$3.8 \\ 2048. \\ 2.68 \\ 64.8$
Silicon dioxide (SiO ₂), % Magnesium oxide (MgO), % Calcium oxide (CaO), % Aluminum oxide (Al ₂ O ₃), % Iron oxide (Fe ₂ O ₃), % Sulphur trioxide (SO ₃), %	$11.3 \\ 0.3 \\ 12.3 \\ 0.9 \\ 68.4 \\ 3.2$	$\begin{array}{c} 40.1 \\ 0.3 \\ 5.8 \\ 13.1 \\ 36.7 \\ 2.4 \end{array}$	38.5 0.2 3.2 18.1 16.2 1.1	$35.3 \\ 0.9 \\ 5.3 \\ 7.7 \\ 43.3 \\ 1.4$
	100			

Limes

Most of this investigation was made using two commercial grade limes furnished by the U. S. Gypsum Company. One is a hydrated calcitic lime, brand name *Kemikal*, and the other is a type N monohydrate dolomitic lime, brand name *Kemidol*. In the preliminary evaluation of chemical additives to Ottawa sand, lime, and fly ash mixtures a calcium hydroxide (calcitic hydrated) lime, reagent grade, from Fisher Scientific Company was used. Two dolomitic monohydrate limes, from Western Lime and Cement Company and from Rockwell Lime Company, were also used in a comparative study of some commercial dolomitic monohydrate limes (table IV).

Cement

The Portland cement used was commercial type I from the Penn-Dixie Cement Corporation of Des Moines, Iowa.

Chemicals

The following chemicals evaluated as additives to lime fly ash mixtures were reagent grade, except magnesium oxide which was USP grade:

CHEMICAL
Sodium carbonate
Sodium hydroxide
Sodium metasilicate
Sodium chloride
Aluminum chloride
Calcium chloride
Lithium carbonate
Magnesium oxide
Manganese chloride
Phosphoric acid
Potassium permanganate
Sodium phosphate

FORMULA

 $\begin{array}{c} Na_{2}CO_{3} \\ NaOH \\ Na_{2}SiO_{3} \bullet 9H_{2}O \\ NaCl \\ AlCl_{3} \bullet 6H_{2}O \\ CaCl_{2} \\ Li_{2}CO_{3} \\ MgO \\ MnCl_{2} \bullet 4H_{2}O \\ 85\% H_{3}PO_{4} \\ KMnO_{4} \\ Na_{3}PO_{4} \bullet 12H_{2}O \end{array}$

Water

Distilled water was used throughout to eliminate the variable that might result from impurities added with ordinary tap water.

Procedures

Mixture proportions

The proportions of soil plus lime or lime fly ash or cement were made based on the dry weight of the soil and lime, the soil, lime, and fly ash, or the soil and cement mixtures. The chemical additive, when used, was computed on a dry basis excluding the water of crystalization, and is expressed as a percentage of the dry weight of the total Ottawa sand or soil, lime, and fly ash mixture. Chemicals were added either in powder form or as a component of the mix water.

Mixing and molding

Mixing of batches for preparing test specimens was done in a kitchen mixer at low speed in the following sequence of operation: The dry in-

	TABLE IV. ANALYSIS OF LIMES.		·			
	Kind of lime	Calcitic hydrated	Dolomitic monohydrate	Calcitic hydrated	Dolomitic monohydrate	Dolomitic monohydrate
ч	Type	Commercial	Commercial type N	Reagent grade	Commercial type N	Commercial type N
	Source	New Braunfels, Texas	Genoa, Ohio			
84	Company	U. S. Gypsum	U. S. Gypsum	Fisher	Western	Rockwell
	Brand name	Kemikal	Kemidol			
	Silicon dioxide, % Iron and aluminum oxide, % Calcium oxide, % Magnesium oxide, % Sulfur trioxide, % Loss on ignition, % Passing no. 325 sieve, %	$\begin{array}{c} 0.3 \\ 0.6 \\ 73.8 \\ 0.6 \\ 0.3 \\ 24.1 \\ 95.5 \end{array}$	$0.4 \\ 0.2 \\ 49.6 \\ 31.8 \\ 1.1 \\ 17.0 \\ 91.0$		0.6 1.1 48.3 33.2 16.8 99.2	0.4 0.6 45.4 36.3 21.0 91.0

gredients were machine mixed for 30 seconds, the mix water was added and machine mixed for one minute, the mixture was hand mixed for about

30 seconds to clean the sides and bottom of the mixing bowl, and the mixture was machine mixed for one more minute.

Molding of test specimens was started immediately after a batch was mixed, except where otherwise indicated. A double plunger drop-hammer apparatus was used to mold 2 inch diameter by 2 ± 0.05 inch high specimens. With this apparatus the equivalent of standard Proctor compactive energy was obtained when giving 5 blows on each side of the specimen using a 5 pound hammer dropping 12 inches with the molding apparatus fastened to a wooden table^{3, 28}. The equivalent of modified Proctor compactive energy was obtained with 10 blows on each side with a 10 pound hammer dropping 12 inches with the molding apparatus fastened to a concrete pedestal^{3, 40, 68}. The standard Proctor compaction was used in these studies except where otherwise specified. After being molded, the specimen was extruded, weighed to the nearest 0.1 gram and measured to the nearest 0.001 inch. During molding, a wet cloth was kept over the bowl to prevent drying of the mixture.

Curing

Specimens of each batch were moist cured at $70 \pm 4^{\circ}$ F., except where otherwise indicated, at a relative humidity of over 90 percent for the desired periods of time. To retain moisture better and to reduce absorption of carbon dioxide from the air, the specimens were wrapped in wax paper and were sealed with cellophane tape before being placed in the humid room.

Specimens cured at higher temperatures were wrapped in Saran wrap and kept in watertight containers with free water inside to assure a high relative humidity during the curing period. Steam cured specimens were wrapped in Saran wrap and put in an autoclave at 15 atmospheres of pressure and 248°F. (120°C.); the temperature was increased slowly up to the 248°F. in order to prevent cracking of the specimens. Specimens cured at low temperatures were kept in a refrigerator after being wrapped in Saran wrap. The loss of moisture in no specimen was greater than 5 percent of the total moisture content.

> TABLE V. ANALYSIS OF PORTLAND CEMENT. Des Moines Source: Company: Penn-Dixie Silicon dioxide, % 21.6Aluminum oxide, % 5.13.0 Iron oxide, % 64.1 Calcium oxide, % Magnesium oxide, % 2.9Sulfur trioxide, % 2.30.6 Loss on ignition, %

Strength testing

After each curing period, specimens were unwrapped and immersed in distilled water for one day. Then they were tested for unconfined compressive strength using a load travel rate of 0.1 inch per minute. Tests were run in triplicate, and the average strengths are reported in psi. This is in accordance with ASTM specification designation C109-58, which requires a minimum of three specimens for each set of curing conditions⁴. Any readings which deviate excessively can be detected in three observations. Specimens that differed by more than 10 percent from the average value of test specimens made from the same mix and tested at the same age were not considered in determining compressive strength. If two specimens were rejected, new specimens were prepared. Durability tests

The Iowa freeze-thaw test was used to evaluate the durability of selected mixtures²⁶. Four 2 inch by 2 inch specimens from each mixture were cured 28 days in the moisture room. Two specimens, designated the control specimens, were then left immersed for 10 days; and the other two specimens designated the freeze and thaw specimens, were exposed alternately to temperatures of $20 \pm 2^{\circ}$ F. (16 hours) and $77 \pm 4^{\circ}$ F. (8 hours) for ten cycles, each cycle lasting 24 hours. A vacuum flask specimen container was used to cause freezing to occur from the top down and to supply unfrozen water, kept at $35 \pm 2^{\circ}$ F. by a light bulb, to the bottom of the specimen throughout the test¹⁶. After these treatments, the unconfined compressive strengths of the freeze-thaw specimens ($p_{\rm f}$) and of the control specimens ($p_{\rm c}$) were determined. These values were used to evaluate the durability of the stabilized soils. The index of resistance to the effect of freezing ($\mathbf{R}_{\rm f}$) was calculated from the formula:

$$R_{f} = \frac{100 P_{f}}{P_{c}} (\%).$$

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 soil, lime, and fly ash mixtures showed some differences between the optimum moisture for maximum dry density and that for maximum 7 day strength of a silty soil²⁸.

Since the information on the effects of molding moisture on the strength of lime fly ash stabilized soils is scarce and often contradictory, an investigation was conducted to find if there is any correlation between the moisture for maximum dry density and the moisture for maximum strength. The strength tests had to be made including short and long term curing periods; consequently specimens molded at different moisture contents were kept curing for 7, 28 and 90 days. Two compactive efforts were used, one approximating the standard Proctor and the other approximating the modified Proctor³. 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 with dune sand and gumbotil. In these tests the proportions were 76.5 percent soil, 6 percent lime and 17.5 percent fly ash (figures 1 to 8).

Dune sand

The moisture for maximum dry density and the moisture for maximum 7 or 28 day strengths in any of the six sets of mixtures show no correlation (table VI). The moistures for maximum strength are far to the dry side of optimum moisture for maximum density. Both moistures of the specimens cured 90 days are closer, but there is still 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 still on the dry side of the optimum moisture for maximum density. The strength curves for 7 and 28 days 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 den-



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



sity (table VII). 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 (table VIII). That is not so for modified Proctor compaction, in which 7 and 28 day curing strength curves, although rather flat, show a maximum strength





Fig. 3. Moisture-density and moisture-strength relationships of a 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash no. 3 for standard and modified Proctor compactive efforts. Fig. 4. Moisture-density and moisture-strength relationships of a 76.5:6:17.5 mixture of gumbotil, calcitic hydrated lime, and fly ash no. I for standard and modified Proctor compactive efforts.

TABLE VI. MOISTURE CONTENTS FOR MAXIMUM DRY DENSTY AND MAXIMUM STRENGTHS OF DUNE SAND, CALCITIC HYDRATED LIME, AND FLY ASH MIXTURES FOR STANDARD AND MODIFIED PROCTOR COMPACTIVE EFFORTS.

	Moisture contents For maximum For maximum strength,			
	density, %	7 day	28 day	90 day
Fly ash no. 1 Standard Modified	$\begin{array}{c} 11.5\\ 8.0 \end{array}$	4.0 4.0	5.5 4.0	$9.0 \\ 7.0$
Fly ash no. 2 Standard Modified	13.8 10.0	No strength No strength	$\begin{array}{c} 11.0\\ 8.5\end{array}$	11.0 10.0
Fly ash no. 3 Standard Modified	12.0 10.0	9.0 7.5	$9.0 \\ 8.5$	$\begin{array}{c} 10.0\\ 9.5\end{array}$

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 the alluvial clay used do not show a peak for maximum dry density and the density increases as the moisture con-

TABLE VII. MOISTURE CONTENTS FOR MAXIMUM DRY DENSTY AND MAXIMUM STRENGTHS OF GUMBOTIL, CALCITIC HYDRATED LIME, AND FLY ASH MIXTURES FOR STANDARD AND MODIFIED PROCTOR COMPACTIVE EFFORTS.



Fig. 5. Moisture-density and moisture-strength relationships of a 76.5:6:17.5 mixture of gumbotil, calcitic hydrated lime, and fly ash no. 2 for standard and modified Proctor compactive efforts. Fig. 6. Moisture-density and moisture-strength relationships of a 76.5:6:17.5 mixture of gumbotil, calcitic hydrated lime, and fly ash no. 3 for standard and modified Proctor compactive efforts. tent decreases (figure 8). The strength curves show, however, a definite optimum moisture that changes conspicuously with curing time for standard compaction and slightly for modified.

Discussion

The results obtained here are significant in that they present new facts on the relations between maximum density and maximum strength in soil stabilization. The common practice has been to compact the stabilized soil at 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 cemen-





Fig. 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.

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

titious compounds by hydration, as that of the lime fly ash reaction, the role of the water is of paramount importance.

Analyzing the results it is observed that, in general:

a) The optimum moisture for maximum strength increased with the increase in curing time;

b) 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, gumbotil and alluvial clay, it was on the wet side. With the friable loess the two optimums are rather coincident.

The results indicate that a supply of water is needed for the hydration processes to continue. With dune sand an amount of water two percentages below the optimum moisture 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 hydration, therefore that should be the recommended moisture to stabilize the friable loess. The moisture should be on the dry side of the optimum rather than on the wet side.

The clayey soils showed great avidity for water. This is because complex reactions, apart from the lime fly ash reactions, take place between the lime and soil particles. 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 the H₂O molecules. Based on long term strengths, it seems advisable to use amounts of water much greater than the optimum for maximum density with clayey soils containing high percentages of montmorillonitic clay. The moisturedensity curves for both clayey soils are rather flat. The maximum density in some is not sharp, being undefined.

Lime and fly ash proportions and content

One of the questions in soil, lime, and fly ash stabilization is as to the amount of lime and fly ash needed with the soil. The optimum amount and proportions of the lime and fly ash admixture are governed by the desired strength in the stabilized soils and by economy.

An unconfined compressive strength after 28 days curing of at least 300 psi after 28 hour immersion may be indicative of adequate stability for a base course mixture to withstand the imposed loads and the detrimental effects of freezing and thawing^{6, 37}.

Lime fly ash stabilization has to compete economically with other admixtures that might give the soil the same strengths more cheaply. The price of lime ranges between 15 and 25 dollars a ton, including transportation to the job site. Fly ash sells for about one dollar a ton at the power plants. Even after transportation expenses, the price of fly ash is much below that of lime. Economic reasons favor the use of greater amounts of fly ash than of lime.

Much work has been done to find the best proportions and amounts of lime and fly ash, but this work has never been so comprehensive as to include enough kinds of fly ashes. In the work reported herein, eight fly ashes were evaluated with the dune sand and three fly ashes with the other three soils. The fly ashes are produced in Iowa or within a radius which make them economical.

The reason for using eight fly ashes with the sand is that sandy and granular soils respond better to lime fly ash stabilization than silty or clayey soils. These eight fly ashes represent a wide range in characteristics, sources, and pozzolanic activity. The results obtained with them may indicate the best proportions and amount to be used.

The number of fly ashes to use with the loess and clayey soils was narrowed to three. These three represent such a variety in properties and composition that the effectiveness of fly ash addition to silty and clayey soils stabilized with lime and their optimum lime and fly ash proportions and amount may be determined.

Two types of commercial limes, a calcitic hydrated and a dolomitic monohydrate, were used with all the fly ashes and soils. Two more dolomitic monohydrate limes were used with fly ash no. 3 and dune sand to check on the effectiveness of available commercial dolomitic monohydrate limes.

The amounts of lime used were 3, 6 and 9 percent with all soils; with gumbotil 12 percent lime was also tried. For each of the amounts of lime four mixes were prepared, one without fly ash and three with 10, 17.5 or 25 percent fly ash. All the percentages were based on the dry weight of the total soil, lime, and fly ash mixture. The above combinations of lime and fly ash gave sufficient data to plot strength contours, which was done for the 28 day strength results. The strength developed after 7 days curing was rather low. Contour graphs made for 7 day strength did not show very much and are not presented here.

In preliminary work, not included here, moisture-density and moisturestrength relationships were determined to select the molding moisture content for every combination of soil, lime, and fly ash. At least four sets of tests were run for every combination of soil and fly ash. Maximum strengths for calcitic hydrated lime and the same amount of dolomitic monohydrate lime were obtained for practically the same optimum amount of water. The molding moisture content needed for maximum 28 day strengths was chosen.

Specimens were molded and then cured for 7 and 28 days. The specimens were also immersed in distilled water for 24 hours before testing for unconfined compressive strength (figures 9 to 26, inclusive).

Dune sand

Strength contours. The plotted strength contours (figures 9 to 14, inclusive) indicate there is no optimum amount and ratio of lime and fly ash that might be used with any kind of lime and fly ash to stabilize dune sand. There is a great similarity among the contours obtained with the same fly ash, but with different limes. In general the proportions and amount of

Materials



Fig. 9. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. I for 7 and 28 day curing periods, and strength contour lines for 28 day results.

lime and fly ash needed to stabilize dune sand vary according to the kind of fly ash used.

The inclination of the strength contours, which approach a vertical position, except that for fly ash No. 10, indicates that with dune sand lower amounts of lime than of fly ash should be favored. The recommended amounts are between 3 and 6 percent lime and between 15 and 30 percent

Materials



Fig. 10. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. 2 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

fly ash. The best amount within these limits differs with the kind of fly ash.

Density. The density varied with the kind and amounts of lime and fly ash. There is no consistency in the densities attained with calcitic hydrated or dolomitic monohydrate lime. The densities apparently depend on the kind of fly ash and the admixture proportions.



Fig. 11. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. 4 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

Lime. It has been observed by other investigators that in lime fly ash stabilization, dolomitic monohydrate lime produces greater strength than calcitic hydrated lime^{15, 19, 28, 49, 55, 63}. The analysis of the effectiveness of the limes which follows is based on the variety of lime and fly ash combinations used in this investigation.

In mixtures of dune sand, lime, and fly ash No. 1, No. 2, No. 4, or No. 7,



Fig. 12. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. 5 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

dolomitic monohydrate lime was more effective than calcitic hydrated lime for both 7 and 28 day curing periods. With fly ash No. 5 test results were erratic, and conclusions cannot be made as to which lime was more effective. With fly ash No. 6, calcitic hydrated lime was more effective than dolomitic monohydrate lime. With fly ash No. 8, 7 day strengths of mixtures with calcitic hydrated lime were greater than with dolomitic lime.

Materials dune sand lime

fly ash no.6



Fig. 13. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. 6 for 7 and 28 day curing periods, and strength contour for 28 day results.

but dolomitic monohydrate lime gave better 28 day strengths. Therefore no general conclusion is possible as to which kind of lime, whether calcitic hydrated or dolomitic monohydrate, is best in lime fly ash stabilization of dune sand; the kind of lime to use depends on the properties of the fly ash. Nevertheless it can be concluded on the basis of 28 day strengths only that dolomitic monohydrate limes generally give better strengths than calcitic

Materials dune sand lime fly ash no. 7



Fig. 14. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. 7 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

hydrated lime. The only exception to this was found in mixtures containing fly ash No. 6.

Tests with fly ash No. 3 deserve special discussion (figures 16, 17). Three dolomitic monohydrate limes were used with this fly ash. Comparing the effectiveness of calcitic hydrated lime with the dolomitic monohydrate limes, the U. S. Gypsum calcitic lime was better for 7 day strength

Materials dune sand lime

fly ash no. 8



Fig. 15. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. 8 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

than the dolomitic lime from the same company, but slightly less effective than the dolomitic limes from Rockwell and Western. All three dolomitic limes gave 28 day strengths much higher than the calcitic lime. Of the three dolomitic monohydrate limes tested the one from Rockwell was most effective. No explanation was found for the differences in strength produced by the dolomitic limes. An investigation is presently being con-

Materials dune sand lime fly ash no. 3



Fig. 16. Immersed unconfined compressive strength values obtained for several combinations of dune sand, lime, and fly ash no. 3 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

ducted to compare the effectiveness of various commercial dolomitic and calcitic limes⁶⁹. The effectiveness of dolomitic limes seems to depend on the temperature and period of burning, the amount of impurities, the gradation, and probably other factors.

Fly ash. The strength of mixtures made with fly ash No. 3 attained very high strengths. Mixtures made with dolomitic monohydrate limes either from U. S Gypsum or Rockwell showed a strength of 1000 psi after 28

Materials

dune sand dolomitic monohyrate lime fly ash no.3



Fig. 17. Immersed unconfined compressive strength values obtained for several combinations of dune sand, dolomitic monohydrate limes, and fly ash no. 3 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

days of curing. This strength approaches that of a lean concrete. Mixtures made with the other dolomitic monohydrate lime from Western and fly ash No. 3 showed a strength after 28 days curing of about 600 psi, which is also very good. Strengths of about 500 psi for the same curing period were obtained with calcitic hydrated lime. Seven day strengths of 200 or 300 psi, depending on the type of lime used, were obtained with this fly ash.

Fly ash No. 1 also gave good strengths. Six hundred psi was obtained after 28 days curing in mixes with dolomitic monohydrate lime. The 7 day strength for the same mixes was close to 300 psi, but the results obtained with this fly ash and calcitic hydrated lime after 28 days curing were very poor, barely reaching 100 psi.

Other fly ashes that gave strength over 300 psi after 28 days curing were fly ash No. 6 in mixes with calcitic hydrated lime, and fly ash No. 7 with dolomitic monohydrate lime. Many fly ashes did not reach the desired 300 psi after 28 days curing in mixes with either of the limes used.

The above results point out that the strengths obtained depend very greatly on the fly ash used. This indicates the great disparity of pozzolanic properties of fly ashes. Some of them with lime may give strengths comparable with those obtained with cement, while others develop scarcely any strength.

Fly ash No. 3 was used with three different dolomitic monohydrate limes; the densities varied also for mixtures with these three dolomitic limes, but the strengths were not in relationship to the density but to the admixture content and amount. Fly ashes of low specific gravity (Nos. 2, 4, and 7) imparted very low dry densities to the sand, lime, and fly ash mixtures.

Friable loess

Strength contours. The strengths obtained in the friable loess mixtures with lime only were decreased by the addition of fly ash No. 1. Additions of fly ash No. 2 did not increase the strength of the friable loess and lime mixtures to any great extent. Additions to fly ash No. 3 increased the strength some, but not greatly. The strength contours with friable loess are therefore sparse and difficult to draw (figures 18, 19, 20).

The only type of fly ash that may be recommended for use with lime to stabilize friable loess is a high quality fly ash like No. 3. The verticality of the contours with fly ash No. 3 favors the use of small amounts of lime and large amounts of fly ash. The recommended amounts are 3 percent dolomitic monohydrate lime, 25 percent fly ash No. 3, and 72 percent friable loess. If the price of the fly ash is prohibitive, this soil can be stabilized with lime alone.

Density. Calcitic hydrated lime gave lower density than equal amounts of dolomitic monohydrate lime. Fly ash No. 2, of low specific gravity, lowered the density in proportion to the amount of fly ash in the mixture. No correlation was found between density and strength.

Lime. Dolomitic monohydrate lime with or without fly ash always gave better strengths than calcitic hydrated lime. Nine percent dolomitic monohydrate lime added to friable loess showed an immersed strength of 400 psi, which is considered adequate for a road base or a subbase course.

Materials



fly ash no.l



Fig. 18. Immersed unconfined compressive strength values obtained for several combinations of friable loess, lime, and fly ash no. I for 7 and 28 day curing periods, and strength contour lines for 28 day results.

Fly ash. Fly ashes Nos. 1 and 2 either did not greatly improve the strength of friable loess and lime mixtures or were detrimental to the point where they actually lowered the strength in some cases. This may be due to the fact that friable loess may have greater pozzolanic activity with lime than fly ashes Nos. 1 or 2. Fly ash No. 3 gave strength improvements to friable loess and lime mixtures, particularly for mixtures with low lime contents. This is the only fly ash tested that may be recommended to use

Materials

friable loess lime fly ash no.2



Fig. 19. Immersed unconfined compressive strength values obtained for several combinations of friable loess, lime, and fly ash no. 2 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

with lime, preferably dolomitic monohydrate, in the stabilization of friable loess.

Gumbotil

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Strength contours. Strength contours tend to be horizontal for low lime contents and become vertical for high amounts (figures 21, 22, 23). This indicates that lime up to a certain amount increases strength, and then fly



Fig. 20. Immersed unconfined compressive strength values obtained for several combinations of friable loess, lime, and fly ash no. 3 for 7 and 28 day curing periods, and strength contour lines for 28 day results.



Fig. 21. Immersed unconfined compressive strength values obtained for several combinations of gumbotil, lime, and fly ash no. I for 7 and 28 day curing periods, and strength contour lines for 28 day results.

Materials gumbotil lime fly ash no.2



Fig. 22. Immersed unconfined compressive strength values obtained for several combinations of gumbotil, lime, and fly ash no. 2 for 7 and 28 day curing periods, and strength contour lines for 28 day results.



Fig. 23. Immersed unconfined compressive strength values obtained for several combinations of gumbotil, lime, and fly ash no. 3 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

ash becomes important in the development of strength. No definite ratio of lime to fly ash gives the highest strengths. Recommendations on the amounts of lime and fly ash to be used should be based on the need of a minimum amount of lime, which is about 5 percent. Low amounts of lime required high amounts of fly ash, and high amounts of lime required low amounts of fly ash. Several combinations of lime and fly ash may be chosen



Fig. 24. Immersed unconfined compressive strength values obtained for several combinations of alluvial clay, lime, and fly ash no. I for 7 and 28 day curing periods, and strength contour lines for 28 day results.

depending on the desired strength. The amount of lime required will be between 5 and 9 percent, and that of fly ash between 10 and 25 percent.

Density. Density values did not correlate with strength, neither did they correlate with the kind of lime used. The fly ash of low specific gravity, No. 2, gave lower densities than the other two fly ashes used.

Lime. The calcitic hydrated lime in low amounts gave greater strengths than low amounts of dolmitic monohydrate lime. Dolomitic monohydrate lime was better than calcitic in high amounts. This was observed for mixtures with and without fly ash. High amounts of lime may stabilize gumbotil soil satisfactorily. For instance, 12 percent dolomitic monohydrate lime gave a 7 day strength of 190 psi and a 28 day strength of 298 psi.

Fly ash. All three fly ashes tested were effective in improving the strength that may be obtained with gumbotil and lime alone. Strengths of from 400 to over 500 psi were obtained after 28 days curing. Consequently the use of fly ash with lime may be recommended to stabilize gumbotil to meet the standards of a base course.

Alluvial clay

Strength contours. There is no definite optimum ratio of lime to fly ash in the tests made with alluvial clay soil (figures 24, 25, 26). The dolomitic monohydrate lime content of mixtures was very critical for the development of strength. For high amounts of dolomitic lime the fly ash content was more critical. With calcitic hydrated lime, the fly ash content was almost the only component contributing to strength as seen by the verticality of the contours for mixtures with calcitic lime.

The recommended amounts and kinds of lime and fly ash to stabilize alluvial clay are from 5 to 7 percent dolomitic monohydrate lime with from 10 to 25 percent of any fly ash used, or else 3 percent calcitic hydrated lime with 25 percent fly ash No. 3. Fly ashes Nos. 1 and 2 are not recommended with calcitic hydrated lime because the same strengths may be obtained with dolomitic monohydrate lime only, in amounts from 6 to 9 percent.

Density. No relationship was found between density and strength. The statements made as to the relationship between specific gravity of fly ash and density of mixtures also apply here.

Lime. The calcitic hydrated lime gave better strengths than dolomitic monohydrate for the lowest amount of lime, 3 percent. The effectiveness is reversed for higher amounts. Without fly ash, 9 percent of plain dolomitic monohydrate lime may properly stabilize alluvial clay. Strengths of 173 psi after 7 day curing, and 345 psi after 28 days were obtained.

Fly ash. The overall effectiveness of fly ash No. 3 exceeded that of the other two fly ashes. Fly ash No. 1 was better than fly ash No. 2 with dolomitic monohydrate lime, but the effectiveness was reversed with calcitic hydrated lime; fly ash No. 2 was better than fly ash No. 1.

Strengths from 400 to 500 psi may be obtained with dolomitic lime and fly ash. This is an adequate strength level. Only fly ash No. 3 could be used with calcitic lime to stabilize alluvial clay. This is due to the low amount of calcitic hydrated lime required, although the strengths obtained, of the order of 350 psi, are rather low.



alluvial clay lime fly ash no.2 28 day strength and contours 7 strength day 308 164 183 166 237 273 175 109[,] (psi) 200 psi 300 6 ⁶243 **2**88 304 **1**53 •188[¯] 182 193 129 Calcitic hydrated lime, % 3 288 229 **1**49 132 199 204 125 188 ο C no. 2,% 30 25 0 10 17.5 0 ٥I Fly ash no. 2, % Fly ash 28 day strength and contours 7 day strength 173 (psi) 234 213 214 345 390 381 42 300 274 358 192 173 215 193 Dolomitic monohydrate 200 psi lime, % 100 168 3 3 . 153 48 232 152 48 130 137 ο 0 20 30 0 10 0 10 17.5 25 no. 2, % Fly ash no. 2, % ash Fly

Fig. 25. Immersed unconfined compressive strength values obtained for several combinations of alluvial clay, lime, and fly ash no. 2 for 7 and 28 day curing periods, and strength contour lines for 28 day results.
Discussion

Based on this study, no conclusions can be drawn as to the best ratio of lime to fly ash or as to the amount of lime and fly ash that could be used to stabilize any kind of soil.

Based on the results obtained with dune sand the amount of lime recom-





Fig. 26. Immersed unconfined compressive strength values obtained for several combinations of alluvial clay, lime, and fly ash no. 3 for 7 and 28 day curing periods, and strength contour lines for 28 day results.

mended for sandy or granular soils is from 3 to 6 percent and that of fly ash from 10 to 25 percent.

Unless fly ash is of a very high pozzolanic value, it should not be used with friable loess. If such a fly ash is available, 3 percent lime and 25 percent fly ash are recommended. The use of dolomitic monohydrate lime is favored.

The amounts of lime and fly ash best for both alluvial clay and gumbotil soils vary. For gumbotil, between 5 and 9 percent lime and between 10 and 25 percent fly ash are recommended. For alluvial clay, between 5 and 7 percent dolomitic monohydrate lime and between 10 and 25 percent fly ash are recommended. Smaller amounts of lime may be used if it is a calcitic hydrated lime.

In general, dolomitic monohydrate limes gives better strength with fly ash than calcitic hydrated lime for the curing temperatures used (70°F.). It should be pointed out that with one fly ash, No. 6, calcitic hydrated lime was more effective that dolomitic monohydrate lime. For small amounts of lime, the calcitic hydrated is more effective than the dolomitic monohydrate in the stabilization of clayey soils with lime and fly ash; at higher lime contents, dolomitic monohydrate gives better strengths than calcitic hydrated.

Fly ash, unless of a high quality, is detrimental in the stabilization of friable loess; in all other soils it was beneficial, giving better strengths than mixtures of soil and lime without fly ash.

Twenty-two fly ashes were studied as to their pozzolanic behavior, among them those used in these tests⁶⁷. No new information was found here as to the relation between pozzolanic activity of a fly ash and its physical or chemical characteristics.

The maximum dry density, for the same compactive effort, of soil, lime, and fly ash mixtures does not correlate with strength. Density varies with amounts and kind of lime and fly ash. Dolomitic monohydrate lime gives consistently greater densities in friable loess, lime, and fly ash mixtures than calcitic hydrated lime. Fly ashes of low specific gravity produce lower densities than fly ashes of higher specific gravity.

Effect of compactive effort on strength of soil, lime, and fly ash mixtures

The present trend in compaction of earth embankments, subgrades, and stabilized soils is toward compactive efforts greater than the standard Proctor. The Corps of Engineers specifies 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^{65, 40}, only one fly ash was used, and the specimens were cured only up to 28 days.

TABLE IX. MAXIMUM STRENGTHS OBTAINED AT DIFFERENT CURING PERIODS FOR STANDARD AND MODIFIED PROCTOR COMPACTION OF 76.5:6:17.5 DUNE SAND, CALCITIC HYDRATED LIME, AND FLY ASH MIXTURES.

Fly ash used, no.	Compaction	Maximum compr 7 day	n immersed u essive streng 28 day	inconfined th, psi 90 day
1 1	Standard Modified	$\begin{array}{c} 55\\ 105\end{array}$	90 170	$\begin{array}{c} 240 \\ 570 \end{array}$
2 2	Standard Modified	0 0	$\begin{array}{c} 150\\ 390 \end{array}$	$\begin{array}{c} 560 \\ 1025 \end{array}$
3 3	Standard Modified	$\begin{array}{c} 165 \\ 280 \end{array}$	390 [°] 750	$\begin{array}{c} 930 \\ 1780 \end{array}$





Fig. 27. Effect of compactive effort on strength of a 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash.

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 strength versus time are plotted (figures 1 to 8 and 27 to 30, tables IX to XII).

In all the eight comparative studies made, the modified compaction gave strengths considerably greater than the standard compaction. This increase is appreciated in all curing periods, and ranges from a minimum of 50 percent increase to a maximum of 160 percent without any correlation whatsoever and depending on the kind of soil and fly ash and probably on the kind of lime also.



TABLE X. MAXIMUM STRENGTH OBTAINED AT DIFFERENT MIXING PERIODS FOR STANDARD AND MODIFIED PROCTOR COMPACTION OF A 76.5:6:17.5 FRIABLE LOESS, CALCITIC HYDRATED LIME, AND FLY ASH NO. 3 MIXTURE.

Fig. 28. Effect of compactive effort on strength of a 76.5:6:17.5 mixture of gumbotil, calcitic hydrated lime, and fly ash.

The rate of strength increase for 7, 28 and 90 days curing is almost a straight line relationship, except for those mixes made with the gumbotil. Greater rate of increase 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 days curing, all the mixtures show that the strength increase also takes place at longer curing periods.

The convenience of compacting the soil, lime, and fly ash mixtures 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.

TABLE	XI.	MAXIN	AUM	STRENGTH	OBTAINED	AT	DIFFERENT	MIXING	PERIODS
FOF	STA	NDARD	AND	MODIFIED	PROCTOR	сомі	PACTION OF	76.5:6:	17.5
(TIMR	VEIL C	ALCIT	IC HYDRA'	ED LIME	AND	FLY ASH	MIXTURES	3.

Fly ash used, no.	Compaction	Maximun compr 7 day	n immersed u essive streng 28 day	inconfined sth, psi 90 day
1 1	Standard Modified	$\begin{array}{c} 170 \\ 490 \end{array}$	$\begin{array}{c} 260 \\ 700 \end{array}$	$\begin{array}{r} 440 \\ 1000 \end{array}$
$2 \\ 2$	Standard Modified	$270 \\ 570$	$\begin{array}{c} 430\\ 835\end{array}$	$\begin{array}{c} 675\\1170\end{array}$
3 3	Standard Modified	$\begin{array}{c} 255 \\ 620 \end{array}$	$\begin{array}{c} 445\\ 890 \end{array}$	$\begin{array}{c} 685\\ 1260\end{array}$





When lime and fly ash are used to stabilize friable soils, account for the steady increase in strength with time has to be made (figures 27 to 30). 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 time of compaction on soil, lime, and fly ash mixtures has not been studied. The ambient mean temperature between two consecutive days in Iowa may in extreme cases be 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 extreme cases of ambient temperature during the working season on the strength of soil, lime, and fly ash mixtures.

TABLE XII.MAXIMUM STRENGTHS OBTAINED AT DIFFERENT CURING PERIODSFOR STANDARD AND MODIFIED PROCTOR COMPACTION OF A 76.5:6:17.5MIXTURE OF ALLUVIAL CLAY, CALCITIC HYDRATED LIME, AND FLY ASH NO. 3.

Compaction	Maximum compre 7 day	Maximum immersed unconfined compressive strength, psi 7 day 28 day 90 day			
Standard Modified	240 445	$310 \\ 585$	$\begin{array}{c} 460 \\ 810 \end{array}$		





The soils used were dune sand and gumbotil in mixes with 76.5 percent soil, 6 percent calcitic hydrated lime and 17.5 percent fly ash No. 3. A very reactive fly ash was used because it should accentuate the findings. A series of batches were 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, and fly ash mixtures were molded at several water contents, and then stored in the moist room at $70 \pm 3^{\circ}$ F. The maximum immersed unconfined compressive strength and density values were obtained from these specimens (table XIII).

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. No noticeable effects are seen in the tests with sand.

According to the results, the basic reaction between lime and fly ash is not influenced by the temperature, in the range 54 to 104° F., of the materials at the time of mixing. This statement is based on the results obtained with sand, which may be considered as an aggregate inert to lime and fly ash. 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 prior to compaction.

Further tests were made in which the materials were mixed at the same temperatures and then stored at the same temperatures of mixing for four hours before compaction. The specimens were cured in the moist room. Dune sand was the only soil used. The maximum results obtained, from batches made at different water moisture contents, are recorded (table XIV).

The results obtained prove further 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. The lime reacts in clayey soils in several ways with the clay particles, and some of these reactions may be activated by temperature. These reactions subtract part of the lime or make it inactive 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 on strength of soil, lime, and fly ash mixtures

When interruptions in road construction occur right after mixing of lime and fly ash with 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 to soil, lime, and fly ash mixtures between wet mixing and compaction.

Selected mixes using dune sand or gumbotil, calcitic hydrated lime, and fly ashes Nos. 1, 2, or 3 were made. The mixtures were prepared with

pacted; another set was stored for 24 hours in the same moist room before compaction of specimens. The maximum values for strength and density for 4 hours in the moist room at 70°F., and then specimens were comtures was immediately compacted into specimens; another set different amounts of water to obtain maximum values for strength and density. After mixing the soil, lime, fly ash, and water, one set of mixare given in tables XV and XVI. compaction of specimens. was stored

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

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	Temperature,	Maximun compr	n immersed u essive streng	inconfined th, psi	Maximum dry	Optimum M. C. for
Soil	°F.	7 day	28 day	90 day	density, pcf	maximum density, %
Dune sand Dune sand Dune sand	54 70 104	$154 \\ 165 \\ 158$	422 390 382	$1004 \\ 930 \\ 1010$	$123.8 \\ 124.2 \\ 124.2$	12 12 12
Gumbotil Gumbotil Gumbotil	$54\\70\\104$	302 255 238	$455 \\ 445 \\ 350$	620 685 492	94.1 93.0 92.5	25 25 25

TABLE XIV. INFLUENCE OF MIXING TEMPERATURE OF MATERIALS ON THE STRENGTH OF A 76.5:6:17.5 MIXTURE OF DUNE SAND, CALCITIC HYDRATED LIME, AND FLY ASH NO. 3, IN WHICH COMPACTION WAS DELAYED FOUR HOURS AFTER MIXING.

Temperature,	Maximun compr	n immersed u essive streng	nconfined th, psi	Maximum dry	Optimum M. C. for
°F.	7 day Î	28 day	90 day	density, pcf	maximum density, %
54	140	369	960	124.0	12
$\frac{70}{104}$	$141 \\ 148$	$\frac{348}{342}$	935 973	122.0	12

Dune sand

Strength and density of the mixture with dune sand decrease slightly as the time between wet mixing and compaction increases. Regarding strength, the greatest decrease is found in mixtures made with fly ash No. 3, in which for 7 days curing it dropped from 165 psi for no delay in molding to 118 psi for a 24 hour delay; the drop for 28 days curing is from 390 to 243 psi; for 90 days curing there is no difference between the strength of specimens molded after mixing and of those molded after a 24 hour delay. With fly ash No. 2 specimens there is also a great difference after 90 days curing between the strength of mixtures with no delay in compaction and those with 24 hours delay, the strengths for these two are 560 and 417 psi respectively. The decrease with fly ash No. 1 is not very significant although it is steady with time of delay.

In general the decrease in strength is very slight in mixtures in which compaction was performed 4 hours after wet mixing. The decrease is more accentuated for the mixtures stored 24 hours 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 hour delay.

TABLE XV. 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.

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ash no.	Delay between mixing and molding	Maximum dry density, pcf	Maximum compre 7 day	i immersed u essive streng 28 day	inconfined gth, psi 90 day
1 1 1	Molded after mixing Molded 4 hrs. after mixing Molded 24 hrs. after mixing	$121.2 \\ 120.3 \\ 118.6$	55 45 41	90 81 60	$240 \\ 219 \\ 210$
$2 \\ 2 \\ 2$	Molded after mixing Molded 4 hrs. after mixing Molded 24 hrs. after mixing	$112.3 \\ 112.5 \\ 110.8$	0 0 0	$150 \\ 159 \\ 141$	$560 \\ 532 \\ 417$
3 3 3	Molded after mixing Molded 4 hrs. after mixing Molded 24 hrs. after mixing	$124.1 \\ 122.6 \\ 122.6 \\$	$165 \\ 141 \\ 118$	$390 \\ 348 \\ 243$	930 935 945

TABLE XVI. 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.

ash	Delay between	elay between Maximum dry		Maximum immersed unconfined			
no.	mixing and molding	ng and molding density, pcf		compressive strength, psi			
			7 day	28 day	90 day		
1	Molded after mixing	undefined	170	260	$440 \\ 431 \\ 327$		
1	Molded 4 hrs. after mixing	undefined	151	260			
1	Molded 24 hrs. after mixing	undefined	136	279			
3 3 3	Molded after mixing Molded 4 hrs. after mixing Molded 24 hrs. after mixing	undefined undefined undefined	$255 \\ 260 \\ 173$	$445 \\ 405 \\ 244$	685 596 351		

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 hour 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 delay of compaction increased. As the maximum dry density was undefined in mixtures with gumbotil, the moisture-dry density relationships are plotted for the range in moisture content in which the maximum strengths were obtained (figures 31, 32). 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 hour delay and about 5 pcf for a 24 hour delay.

Discussion

The results stress the importance of proceeding with compaction as soon as possible after wet mixing of soil, lime, and fly ash mixtures. This is highly recommended with montmorillonitic clayey soils in which strengths



Fig. 31. Moisture-density relationships of a 76.5:6:17.5 mixture of gumbotil, calcitic hydrated lime, and fly ash no. 3, in which compaction was carried at different intervals of time after wet mixing.



may drop by about 40 percent and dry density by about 6 percent if compaction is delayed one day after wet mixing. With sandy soils, the drop in strength and dry density is not very significant, 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 three different 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.

A very small reduction in strength and density in sandy soils indicates that the first two processes are not developed to a great extent. Because the carbonization of lime takes place at a rapid rate in a moist condition and because pozzolanic reactions are unlikely 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 soil particles are very important in clayey 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 follow wet mixing of clayey soil, lime, and fly ash mixtures.

Effect of temperature on strength of soil, lime, and fly ash mixtures

High temperature is known to accelerate the reaction between lime and fly ash. The knowledge of the rate of strength increase with temperature of curing is important as a determinant of the working season for lime and fly ash stabilization. It also may throw some light on the prediction of long-term strengths at ambient temperatures by curing for a short period of time at high temperatures.

Dune sand was used in these studies with calcitic hydrated lime and fly ashes Nos. 1, 2 or 3, or with dolomitic monohydrate lime and fly ash No. 3 (tables XVII, XVIII, figures 33 to 36).

Calcitic lime

The results point out the beneficial effects of high curing temperatures on the strength of soil, lime, and fly ash mixtures. The rate of strength increase varies with temperature. With calcitic lime the lowest increase in psi per degree F. is found between 50° F. and 70° F. as seen by the small value of the tangent of the lines joining the strength values at 50° F. and 70° F. The strength then increases at a higher rate between 70° F. and 104° F. At 104° F. there is a break in the rate of strength for specimens cured for 28 days. Between 104° F. and 140° F., specimens cured for 3 and 7 days experience the highest rate of increase in psi per degree F.; those cured for 28 days are still gaining strength, but the rate is a little lower than that at the previous range of temperatures. Between 140° F. and





Fig. 33. Effect of temperature on strength of a 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash no. 1. Fig. 34. Effect of temperature on strength of a 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash no. 2.

TABLE XVII. EFFECTS OF CURING TEMPERATURE ON STRENGTH OF A 76.5:6:17.5 MIXTURE OF DUNE SAND, CALCITIC HYDRATED LIME, AND FLY ASH.

Fly ash	temperature	Immersed	unconfined	compressive
no.	° F.	3 day	strength, ps 7 day	28 day
1 1 1 1 1	$50 \\ 70 \\ 104 \\ 140 \\ 248$	$0\\0\\41\\813\\1783$	$\begin{array}{c} & 0 \\ & 42 \\ & 295 \\ 1216 \\ & 2342 \end{array}$	$\begin{array}{r} 0 \\ 78 \\ 1018 \\ 1488 \\ 2572 \end{array}$
2 2 2 2 2 2	$50 \\ 70 \\ 104 \\ 140 \\ 248$	0 0 43 449 1477	$0\\0\\208\\712\\1595$	0 141 718 971 1627
3 3 3 3 3	$50 \\ 70 \\ 104 \\ 140 \\ 248$	$\begin{array}{c} & 0 \\ & 37 \\ 268 \\ 1530 \\ 3407 \end{array}$	$\begin{array}{r} 0 \\ 159 \\ 635 \\ 1789 \\ 3862 \end{array}$	155371149621994263

TABLE XVIII. EFFECTS OF CURING TEMPERATURE ON STRENGTH OF A 76.5:6:17.5 MIXTURE OF DUNE SAND, DOLOMITIC MONOHYDRATE LIME, AND FLY ASH NO. 3.

Curing	Immersed	unconfined c	ompressive
°F.	3 day	7 day	28 day
50	0	0	193
70	52	145	783
104	717	1097	1755
140	1464	1622	2079
248	1997	2605	2947

248°F. the strength is still increasing; but the rate of increase, although still very important, is smaller than for some of the other temperature ranges. The shape of the curves indicate that the strength should still be increasing for curing temperatures over 248°F. Steam curing mixtures made with fly ash No. 3 at temperatures higher than 248°F. may make them reach strength of 4000 psi or over after a few hours curing.

Dolomitic lime

The pattern of strength increase for mixtures made with dolomitic monohydrate lime is very different from the one given by the mixtures made with calcitic hydrated lime (compare figures 35 and 36). The rate of strength increase at low temperatures is greater than with calcitic lime, but at high temperatures it is not as great. At about 135° F. the strengths are the same for both limes; dolomitic lime gave better strengths below that curing temperature; above that temperature calcitic lime was the best one.

Discussion

The pozzolanic activity between lime and fly ash is greatly influenced by temperature. After curing periods of 3 and 7 days, all specimens cured at 50°F. failed during immersion, but those cured at 248°F. developed strengths comparable with those of concrete. At ambient temperatures, dolomitic monohydrate lime gave higher strengths than calcitic hydrated lime, but at high temperatures calcitic lime was better than dolomitic.





Fig. 35. Effect of temperature on strength of a 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash no. 3.

Fig. 36. Effect of temperature on strength of a 76.5:6:17.5 mixture of dune sand, dolomitic monohydrate lime, and fly ash no. 3.

The importance of high temperatures in the development of strength emphasizes the necessity for early summer construction when using lime fly ash stabilization. The pavement courses will have time to cure for several weeks at temperature high enough to aid in developing strength enough to withstand the adverse effects of winter freezing temperatures.

The strengths obtained for every temperature and curing period are in relation to the reactivity of the fly ash. Fly ash No. 3 is a good quality fly ash, and all the strengths obtained with it are above those obtained with fly ashes Nos. 1 or 2. Fly ash No. 1 is considered of medium quality and generally performed better than fly ash No. 2, considered of poor quality. The inherent strength production, or quality, of a fly ash shows up on the unconfined compressive strength of its mixes with lime for any temperature of curing. The standardized methods of selecting a fly ash include a variety of tests cumbersome and expensive to make, and some do not select a fly ash properly. The selection of a fly ash must be made on the basis of its reactivity with lime, except when, as in cement concrete, a gradation of the fine material is very important.

Although these tests are not statistically enough, it appears that the quality of a fly ash is reflected in the strength values of its mixtures with lime at any temperature, and it is possible that a fly ash might be selected on the basis of a simple strength test, three days after molding the specimens.

For instance, the fly ash mixed with calcitic hydrated lime and dune sand in the proportions used here should be a good quality fly ash if after three days it gives strengths of 3000 psi cured at 248°F., 1400 psi cured at 140°F., and 220 psi cured at 104°F. More studies of this kind should be made to establish criteria for use in the selection of a satisfactory fly ash by the simple method of determining its reactivity with lime for short curing periods at high temperatures.

Some investigators have used a short curing period at high temperature to predict the strength that may be expected after long curing periods at ambient temperatures. To check for possible relationships of this kind, the strength after 90 days curing at 70°F. has been compared with the strength-curing time relationship (figure 37). The results indicate that strengths equal to those obtained after 90 days curing at 70°F. may be obtained:

a) after 6 days curing at 104°F. with fly ash No. 1 and calcitic lime

b) after 19 days curing at 104°F. with fly ash No. 2 and calcitic lime

c) after 12 days curing at 104°F. with fly ash No. 3 and calcitic lime

d) after 7 days curing at 104°F. with fly ash No. 3 and dolomitic lime

The range is from 6 to 19 days with both limes and even with calcitic lime only. This points out the difficulty of predicting long-term strengths at ambient temperatures by finding short-term strengths at high temperatures. Curing the specimens at temperatures higher than 104°F. will give









a less realistic correlation because of the probable formation of compounds different from those formed at ambient temperatures.

It should also be mentioned that the strength may be accelerated after the specimens have been cured for a time at a certain temperature by submitting them to higher temperatures. The lower the initial temperature of curing the higher the strength is boosted (table XIX). These findings indicate that the strength of soil, lime, and fly ash mixtures may be increased at any time by submitting them to higher curing temperatures.

Steam curing soil stabilized mixtures

After the temperature curing studies were made, further investigation was carried on the effect of steam curing on the strength of stabilized soil specimens.

In a report presented to the Highway Research Board an additional 10 million dollars was recommended for research on aggregates and soil stabilization during the next four or five years³⁸. The same report suggested some research in the use of nuclear energy in highway construction. Based on the need for new sources of aggregates and the future use of nuclear energy, the study on steam curing of soil, lime, and fly ash specimens was expanded to include soil cement and soil lime. This was done because of the concrete-like strengths obtained with soil, lime, and fly ash mixtures, and to gather information on the effects of steam curing on other kinds of soil stabilization.

This was not approached as a systematic study since it is beyond the purpose of the lime fly ash stabilization investigation.

Extensive research has been done on sand lime bricks^{23, 27, 29, 43, 57}. These brick are made by submitting the sand lime paste to temperatures of 150-200°C. (302-392°F.) for about 8 hours in autoclaves with pressures from 5 to 10 atmospheres. The addition of clay has been tried, and about 10 percent clay has been found to increase the strength of sand lime bricks^{27, 57, 60}.

Immersed unconfined Curing compressive strength, psi Mixture 7 days at 120°C. 234276.5% dune sand, 6.0% calcitic 28 days at 10° C. + 7 days at 120° C. 2104 hydrated lime, and 17.5% fly 35 days at 10°C. 40 ash no. 1 28 days at 40°C. + 7 days at 120°C. 2104 35 days at 40°C. 107928 days at 60° C. + 7 days at 120°C. 1895 35 days at 60°C. 1336 159576.5% dune sand, 6.0% calcitic 7 days at 120°C. 28 days at 10°C. + 7 days at 120°C. hydrated lime, and 17.5% fly 191535 days at 10°C. ash no. 2 0 28 days at 40°C. + 7 days at 120°C.

35 days at 40°C.

35 days at 60°C.

TABLE XIX. EFFECTS OF HIGH-TEMPERATURE CURING ON SPECIMENS PREVIOUSLY CURED AT LOWER TEMPERATURES.

228

28 days at 60°C. + 7 days at 120°C.

1520

905

1204

The treatment of cement concrete by steam is a well known process, and the curing of lime and fly ash mixtures at high temperatures has already been mentioned. A comparative study of the autoclaving of soil specimens stabilized with lime, cement, or lime and fly ash at 248°F., 15 atm., was undertaken (table XX).

Discussion

Test specimens of soil stabilized with lime, lime and fly ash or cement attained very high strengths by exposing them for a few hours to elevated temperature and steam. The specimens after one day in the autoclave at 248°F. gave strengths above those attained by specimens moist cured for 90 days at 70°F.

Greater strengths after steam curing were obtained with soil, lime, and fly ash mixtures. A mixture of 76.5 percent dune sand, 6 percent calcitic hydrated lime and 17.5 percent fly ash No. 3 developed a concrete-like strength of 2548 psi after 24 hours in the autoclave; after 7 days the strength was 3662 psi. The same mixture had a strength of only 930 psi after 90 days curing at 70°F. A great increase in strength was also obtained with the same fly ash and different lime percentages in mixtures with friable loess. Mixtures of dune sand, lime, and fly ash No. 1 or No. 2 also were very good in strength. It appears that soil, lime, and fly ash mixtures give strengths after one day in the autoclave that may not be reached, even for years, by curing at ordinary temperatures.

Mixtures of soil and lime also increase very greatly in strength when cured in the autoclave. For example a mixture of 94 percent friable loess and 6 percent calcitic hydrated lime gave a 24 hour strength of 1792 psi steam cured, and only 403 psi after 90 day moist curing at 70°F. The same strength increases were found with other soil and lime mixtures. Sand and lime mixtures, that have practically no strength at ordinary temperatures, reached 1030 psi after 3 days in the autoclave. It was observed that calcitic hydrate lime gave better strength improvement to soils than dolomitic monohydrate lime when the specimens were cured in the autoclave. But at 70°F., dolomitic monohydrate lime gave better strengths than calcitic hydrated lime.

Cement treated soil also benefited from the accelerated curing with steam, but not as much as the lime or lime and fly ash treated soil. A mixture of 92 percent dune sand and 8 percent cement showed a strength of 654 psi after 24 hours in the autoclave. The same mixture showed a strength of 541 psi after 90 days moist curing at 70°F. The same relation in strength for both curing methods and time of curing is observed with other mixtures of soil cement. When the specimens were cured at ordinary temperature, cement gave better strengths than the same amounts of lime or comparative amounts of lime and fly ash. However, lime or lime and fly ash proved to give better results than cement when the specimens were cured in the autoclave.

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1. High temperature curing in the autoclave with a supply of moisture in the form of steam increases the strength of soils stabilized with lime, or cement, or with lime and fly ash.

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TABLE	$\mathbf{X}\mathbf{X}$.	Comparison	\mathbf{OF}	STRENGTHS	OF	SOIL	STABILIZED	MIXTURES	CURED	AT	248°F.	AND	70°	°F
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230

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Materials and proportions	Immersed strengt	unconfined o h after stear at 248°F.	compressive n-curing	Immersed strengt	ompressive t-curing	
	1 day	3 day	7 day	7 day	28 day	90 day
Dune sand + 8% calc. lime + 8% cement. + 6% calc. lime + 17.5% F. A. no. 1 + 6% calc. lime + 17.5% F. A. no. 2	$311 \\ 654 \\ 1668 \\ 1087$	$1030 \\ 968 \\ 1783 \\ 1477$	${ m ND}^{*}$ 1162 2342 1505	5 398 55	20 474 90	$30 \\ 541 \\ 240 \\ 520$
+ 6% calc. lime + 17.5% F. A. no. 3 + 6% calc. lime + 17.5% F. A. no. 3	2548 ND	3407 2014	3662 ND	$165\\145$	$\begin{array}{c}150\\390\\783\end{array}$	930 1030
Friable loess + 3% calc. lime + 3% dolo. lime + 3% cement + 6% calc. lime + 6% coment + 9% calc. lime + 9% dolo. lime + 9% dolo. lime + 9% coment + 3% calc. lime + 17.5% F. A. no. 3 + 6% calc. lime + 17.5% F. A. no. 3	$\begin{array}{c} 630\\ 254\\ 366\\ 1792\\ 1396\\ 955\\ 1441\\ 1344\\ 1140\\ 1432\\ 1780\\ 2063\\ \end{array}$	$\begin{array}{c} 654\\ 271\\ 420\\ 1977\\ 1630\\ 1084\\ 1820\\ 1524\\ 1425\\ 1624\\ 1969\\ 2182\\ \end{array}$	ND ND 2118 1561 1244 ND ND ND ND ND ND	72 117 ND 59 151 330 78 174 423 140 142 126	$110 \\ 249 \\ ND \\ 105 \\ 354 \\ 495 \\ 158 \\ 400 \\ 566 \\ 226 \\ 225 \\ 203 \\$	287 287 ND 403 584 715 499 621 1001 ND 655 ND
Alluvial clay + 9% calc. lime + 9% dolo. lime + 3% calc. lime + 6% cement	921 613 717	969 597 715	1054 ND 711	$109 \\ 173 \\ 328 \dagger$	166 345 469†	218 336 501†
Gumbotil + 9% calc. lime *Not determined.	1188	1318	1350	125	. 215	386
†Dolomitic monohydrate lime used.						

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2. The strength increases with length of curing in the autoclave. This may indicate that strengths can be increased to even higher values by curing at higher temperatures than were used here $(248^{\circ}F.)$.

3. Compacted mixtures of soil, calcitic hydrated lime, and a high quality fly ash develop concrete-like strengths after a few hours of steam curing.

4. When cured in the autoclave, the mixtures with lime and fly ash gave best strengths followed by those with calcitic hydrated lime, dolomitic monohydrate lime, and cement in that order; sand and lime mixes are regarded as special, requiring higher temperatures than those used here. Calcitic hydrated lime also rates better than dolomitic in steam cured soil, lime, and fly ash mixtures.

5. The strengths obtained after 24 hours in the autoclave are not attained by moist curing for 90 days at 70° F.

The results of these experiments may be important in the development of the technique of soil stabilization. The results obtained reinforce the recommendations made by the Highway Research Board to promote research for studying the applications of nuclear power in road construction. Cheap energy from a nuclear reactor may be used in soil stabilization and probably in other phases of road construction. The development of a nuclear reactor which can heat economically a 4 to 6 inch layer of compacted stabilized soil to temperatures above 250°F. could revolutionize the practice of soil stabilization. By regulating the amount and time of application of heat, and by using the proper admixtures with a soil, layers of various strength could be obtained to comply with the engineering requirements of a road subbase, base, or surface course. If the application of heat to road courses is economically feasible, further work may determine such details as the amount of stabilizer to use with different soil types, the time and temperature of application as related to the heat conductivity of the soil and to the strength desired, and the feasibility of the use of steam.

Preliminary survey of chemical additives to mixtures of lime and fly ash

The preliminary survey was made using twelve chemicals in varying amounts to determine the minimum amount of each required for substantial improvement of the lime fly ash reaction and to serve as the basis for selecting a smaller number of chemicals for more detailed studies. Ottawa sand was used as the soil component because its gradation and monomineralic composition, silica, may make it behave as an inert material at the curing temperatures used, thus minimizing the effect of the soil component on the lime fly ash reaction. A calcitic hydrated lime was chosen because, although of reagent grade, it was representative of a great amount of commercial limes produced in the U. S. A medium quality fly ash from the midwest (St. Clair Power Plant) was used as the pozzolan component. The Ottawa sand, lime, fly ash mix proportions were 75 percent, 5 percent, 20 percent, respectively, near optimum for these materials. Specimens were molded at optimum moisture for strength (figure 38).

Any or certain amounts of all the chemicals used increased the strength of the Ottawa sand, lime, and fly ash mixture. Following is an analysis of each chemical evaluated.





Sodium carbonate

Even the smallest amount of sodium carbonate tried, 0.05 percent, increased the strength substantially. Seven and 28 day strengths were in-



Fig. 38. Continued. (e, f, g.)

creased over thirty times with amounts of chemical greater than 0.5 percent. Some differences in strength are shown between the use of sodium carbonate in powder form or in liquid solution, but the great increase in strength warrants the use of the chemical in either form. The optimum amount is about 1.0 percent when used in powder form. The commercial



Fig. 38. Continued. (h, i, j, k.)

price of this product, 35 to 65 dollars a ton, makes it a promising additive for lime fly ash stabilization.

Sodium hydroxide

This chemical is also very effective. A noticeable improvement of strength started with amounts of sodium hydroxide as low as 0.03 percent. A recommended amount is about 1.0 percent. This chemical, priced at about 100 dollars a ton, may also be an economical activator of the pozzolanic reaction.

Sodium chloride and calcium chloride

The effects of these two additives are somewhat parallel. They gave little improvement to 7 day strength, but gave a substantial increase to 28 day and 4 month strengths even with small concentrations of chemical. The price difference, 20 dollars a ton for sodium chloride and 60 dollars for calcium chloride, and the small amounts of sodium chloride required for a maximum increase in strength, makes sodium chloride the choice when improvements of long-term strengths is the main interest. Threetenths of a percent of sodium chloride increased the 28 day strength by about ten times, and the optimum amount was about 1.0 percent.

Sodium metasilicate

This chemical increased the strength greatly, even in small amounts. The strength increase was more or less proportional to amount used; the optimum was above 3.0 percent. The strength of 1,000 psi was found after 7 days curing with the largest amount of sodium metasilicate tested, 3.0 percent. The commercial price of this chemical is about 120 dollars a ton on a dry basis, which makes it a promising chemical additive when used in small amounts.





Lithium carbonate, potassium permanganate, manganese chloride, aluminum chloride and sodium phosphate

These chemicals increase strengths, but the rate of increase, amounts required, and economical considerations make them less desirable.

Phosphoric acid

Although very small amounts of phosphoric acid improved soil strength, concentrations larger than 0.03 percent caused a decrease in strength. Its use is therefore not recommended.

Magnesium oxide

One of the components of dolomitic monohydrate (Type N) lime is magnesium oxide; consequently the effects on strength caused by addition of this chemical should give an indication on the effects of using dolomitic monohydrate lime instead of calcitic hydrated in lime fly ash stabilization.

Small amounts, up to 0.5 percent, resulted in a slight decrease of strength, but increased amounts up to the largest amount tried, 5.0 percent, increased the strength (figure 38, k). The results indicate that dolomitic monohydrate limes are more effective with the fly ash used here, but they are not as effective as calcitic hydrated lime plus treatment with some of the other chemical additives. The results also warranted an investigation on the effects of chemical additives to dolomitic lime and fly ash mixtures.

Extended evaluation of chemical additives

To complement the tests made with Ottawa sand, the study was extended to include four natural soils: a dune sand, a friable loess, an alluvial clay and a gumbotil (tables I and II).

The evaluation of magnesium oxide indicated that dolomitic monohydrate lime might be more effective than calcitic hydrated lime, and that the use of dolomitic lime might make unnecessary the addition of chemicals; therefore the use of both limes, calcitic hydrated and dolomitic monohydrate, was evaluated. Comercial type limes were used.

Three fly ashes were selected to include such desired variations in their properties as coarseness, carbon content, and specific surface.

From the preliminary studies, four chemicals warranted further evaluation based on strength improvement and economics: sodium carbonate, sodium hydroxide, sodium metasilicate and sodium chloride.

The proportions of soil, lime, and fly ash used were 76.5 percent, 6 percent and 17.5 percent. The amount of chemical used was 1.0 percent in mixtures prepared with all soils, limes, and fly ashes, except that 0.5 percent was also used with dune sand and fly ash No. 1. The evaluation was not intended to be an economic comparison of lime fly ash and chemical stabilization of soils with other methods of soil stabilization, but rather to be a check on the possible beneficial effects of the selected chemicals on soil, lime, and fly ash mixtures. Therefore, the mixture proportions are within the range commonly recommended for lime fly ash stabilization, and the amount of chemical added is probably near the optimum amount, except that for sodium metasilicate.

The molding moisture content for mixtures was deducted from the moisture-density and moisture-strength curves of soil, lime, and fly ash mixtures without chemical additives. With friable loess, maximum density and maximum strength occurred at the same moisture content, and this was considered the optimum. The moisture requirements for maximum density and maximum strength of mixtures with sand were not the same, and as the moisture content for maximum density gave very low strengths, the moisture content for maximum strength was used as the optimum. The molding moisture to get maximum strengths of mixtures with alluvial clay and gumbotil was about two percent above the optimum for maximum density.

Dune sand

The data on tests made with this soil and combinations of calcitic hy-

Mixture Proportions 76.5 % dune sand 6.0 % lime

17.5 % fly ash No.1

A 7 day curing ○ 28 day curing + 90 day curing



Fig. 39. Effect of 0.5 percent chemical additive on strength of a 76.5:6:17.5 mixture of dune sand, lime, and fly ash no. 1.

drated or dolomitic monohydrate lime and fly ashes Nos. 1, 2 and 3 are plotted as bar graphs in figures 39 to 42.

Sodium carbonate, sodium metasilicate and sodium hydroxide in amounts of 1.0 percent increased 7, 28 and 90 day strengths of all dune sand, lime, and fly ash mixtures considerably. Sodium chloride increased 28 and 90 day strengths of dune sand, calcitic lime, and fly ash mixtures to a great extent and also increased substantially the 90 day strength of dune sand, dolomitic lime, and fly ash mixtures except those made with fly ash No. 2, in which the strength increase was quite small.

The strengths obtained using 0.5 percent chemical in mixtures with fly ash No. 1 are smaller than those obtained with 1.0 percent chemical additive, but the strength increases follow the same trend for both amounts.

Friable loess

All four chemicals increased the strength of loess, calcitic lime, and fly ash mixtures except for 90 day strength of specimens made with sodium metasilicate and fly ash No. 2 (figures 43, 44, 45). Loess, dolomitic lime, and fly ash mixtures were not appreciably benefited by the addition of the chemicals.

Mixture Proportions						A 7	day cu	iring		
76.5.% dune sand 6.0% lime						0 2	8 dayc	uring		
17.5 % fly ash Na.I						+ 9	O day d	uring		
	c	ALCIT	IC HY	DRAT	ED LI	ME				
		1	mmerse	d Com	prassiva	Stren	oth.p.s.	L. '		
Chemical Additive	0 200	400	600	800	1000	1200	1400	1600	1800	2000
None	A0 7	.,	1					-1-	1	
Sodium chlorida (%	Δ									
	,			<u> </u>						
Sodium metasilicate,17	°	<u> </u>		T						
Sodium carbonate,1%		Δ		<u> </u>	1					
Sodium hydroxide,1%		Δ					>	ŀ		
							!		`	
	DO10	MITIC	MON	OHYDE	RATE	LIME				
		. 1	mmerse	d Con	npressiv	a Stre	ngth, p.	s. i.		
Chemical Additive	0 <u>200</u>	400		800	1000	-1200	1400	1600	1800	200
None		<u> </u>			,		•	÷. '		
Sodium chloride, 1%	<u>Δ</u>				⊒					
Sodium metasilicate, 1%	•	Δ		<u> </u>	7					
Sodium carbonate,1%		Δ		0						
Sodium hydroxide, l%	<u> </u>	Δ				0		•		

Fig. 40. Effect of 1.0 percent chemical additive on strength of a 76.5:6:17.5 mixture of dune sand, lime, and fly ash no. 1.

The use of sodium chloride, sodium carbonate or sodium hydroxide in mixtures of friable loess, calcitic hydrated lime and fly ash No. 1 or No. 3 could be recommended. The strengths produced by the addition of these chemicals in mixtures containing calcitic hydrated lime surpassed that of the similarly proportioned mixtures containing dolomitic monohydrate lime, with or without chemicals.

Alluvial clay and gumbotil

The effect of chemical additives on these clayey soils stabilized with lime and fly ash was nil and sometimes detrimental; consequently the results are not graphed. Specimens treated with sodium carbonate, sodium hydroxide or sodium metasilicate and cured for 90 days were so weakened during the 24 hour immersion period that strength testing was impossible, or strengths were much lower than the strengths of specimens made without treatment or with sodium chloride as the additive. Sodium carbonate, sodium hydroxide and sodium metasilicate are therefore not recommended for use as additives to montmorillonitic clay soils stabilized with lime and fly ash. Sodium chloride was neither harmful nor beneficial; so there appears no reason to use it as an additive.

6.0 % lime 17.5 % fly osh No.2	+	90 day curing
76.5 % dune sand	o	28 day curing
Mixture Proportions	A	7 day curing

	c	ALCIT	IC HY	DRATI	ED LI	ME		•		
		Li Ii	mmersed	Com	pressive	s Strer	ngth', p.s.i	•		
CALCITIC HYDRATED LIME Immersed Compressive Strength, p.s.i. Chemical Additive Sodium chloride, 1% Sodium metasilicate,1% Sodium hydroxide, 1% DOLOMITIC MONOHYDRATE LIME Immersed Compressive Strength, p.s.i. DOLOMITIC MONOHYDRATE LIME Immersed Compressive Strength, p.s.i. Chemical Additive Sodium chloride, 1% Sodium chloride, 1% Sodium metasilicate, 1% Sodium hydroxide, 1% Sodium	1800	2000								
None	•		_							
Sodium chloride, 1%										
Sodium metasilicate,1%	A	0								
Sodium carbonate,1%	Δ	0								
Sodium hydroxide,1%	Δ		0		}					
	DOLO	MITIC	MONO	DHYDF	RATE	LIME	nath n'	- 1		
Chemical Additive (200	400	600	8 <u>00</u>	1000	1200	1400	1600	1800	200
None	•		<u> </u>	•					L.	
Sodium chloride,1%										
Sodium metasilicate, 1%	<u>A</u>	0		3						
Sodium carbonate, 1%	Δ	0								
Sodium hydroxide.l%	Α	0	·	+						

Fig. 41. Effect of 1.0 percent chemical additive on strength of a 76.5:6:17.5 mixture of dune sand, lime, and fly ash no. 2.

Sodium carbònate

This chemical was very effective in the improvement of 7 and 28 day strengths of sandy soil, lime, and fly ash mixtures, regardless of the kind of hydrated lime used. Ninety day strengths were also benefited, but to a lesser extent. Sodium carbonate also improved the early strength of friable loess, lime, and fly ash mixtures containing calcitic hydrated lime, but it did not improve the early strength of mixtures containing dolomitic monohydrate lime.

Owing to its relatively low cost, sodium carbonate in amounts of 0.5 to 1.0 percent is a most promising additive for sandy soils stabilized with lime and fly ash.

Neither sodium carbonate, sodium hydroxide, nor sodium metasilicate are recommended as additives to montmorillonitic clay soil, lime, and fly ash mixtures because they reduce the long-term immersed strength, and they do not increase early strength.

Sodium hydroxide

This chemical greatly improved the strength of sand and friable loess stabilized with hydrated lime and fly ash. The overall effectiveness was

Mixture Proportions 76.5 % dune sand 6.0 % lime 17.5 % flyash No.3

▲ 7 day curing
 ○ 28 day curing
 + 90 day curing



Fig. 42. Effect of 1.0 percent chemical additive on strength of a 76.5:6:17.5 mixture of dune sand, lime, and fly ash no. 3.

greater with calcitic hydrated lime than with dolomitic monohydrate lime. As an example of the strength increases possible, dune sand stabilized with calcitic hydrated lime and fly ash No. 1 showed the following strength improvements by the addition of 1.0 percent of sodium hydroxide:

CURING PERIOD	UNTREATED MIXTURE	TREATED WITH 1.0% NaOh	INCREASE
7 days	42 psi	443 psi	10.5 times
28 days	74 psi	1,291 psi	17.4 times
90 days	241 psi	1,493 psi	6.2 times

Its use is therefore recommended with sand and friable loess.

Sodium chloride

This chemical used as an additive increased the 90 day strength of dune sand, lime, and fly ash mixtures, in some to a considerable extent. Seven day strength was slightly reduced, and 28 day strength was sometimes greatly improved and sometimes was reduced. All 90 day strengths were increased by the addition of sodium chloride. The same trends were observed in mixtures with friable loess as a soil. Thus sodium chloride may be a promising additive to friable soils stabilized with lime and fly ash

Mixture	Proportions						▲ 7	day cu	ring		
76.5 %	friable loess	`					o 26	3 day cu	uring		
6.0 %	lime			-			+ 94) day cu	arina		
17.5 %	fly ash No.L							,			
			·								
		1	CALCIT	IC HY	DRAT	ED LI	ME				
			١m	mersed	Comp	ressive	Strengt	h, p.s.i.			
Chemi	ical Additive .	200	400	600	800	1000	1200	1400	<u> </u>	1800	2000
None											
Sodium	n chloride, 1%	A	· · · · ·	<u> </u>							
Sodium	metasilicate,1%	A	0	_							
Sodium	carbonate,1%		Δ	0							
Sodium	n hydroxide,⊺%		Δ		0		}				
		l· 1	(· ·
		0010		MONO	uvna	TE 1					
		DUL	JAILIC	MONO	n 10K#						
Chami	ical Additive		In.	nmersed	Comp	ressive	Streng	th, p.s.i.			
Cheith		<u> </u>	400	600	800	1000	1200	1400	1600	0081	2000
None		Δ	•	ł							
Sodium	chloride, 1%	∆		0	}						
Sodium	n metasilicate,1%		Δ	<u> </u>							
Sodium	n carbonate, 1%		Δ	<u> </u>							
Sodium	n hydroxide, 1%		Δ		0						
		L L	· · · ·						h		

Fig. 43. Effect of 1.0 percent chemical additive on strength of a 76.5:6:17.5 mixture of friable loess, lime, and fly ash no. 1.



when long-term strengths are desired. The strength of montmorillonitic clay soil, lime, and fly ash mixtures was not affected by adding sodium chloride.

Sodium metasilicate

Mixture Proportions

Sodium metasilicate in the amount of 1.0 percent increased the strength of the dune sand, lime, and fly ash mixtures. It can also improve friable loess, lime, and fly ash mixtures containing some fly ashes. For the percentage used, this chemical rates lower than sodium carbonate or sodium hydroxide. Greater amounts may improve the strength of friable soils; they were not tried here for economic reasons.

Calcitic hydrated and dolomitic monohydrate limes

The dolomitic monohydrate lime used produced better strengths than the calcitic hydrated lime when the mixtures were not treated with chemicals. However, the calcitic lime mixture responded better to chemical treatments.

Effects of additives at low curing temperatures

The strengths obtained with lime and fly ash mixtures depend greatly on

7 day ouring

	• • • • • • • • • • • • • • • • • • • •								ring		
76.5 %	friable loess						o 28	3 day c	uring		
6.0%	lime flucestation						+ 90) day c	uring		
17.5 %	TIY OSH NO.2										
		C	CALCIT	пс ну	DRATI	ED LIN	ΑE				
			. Іл	nmersed	Comp	ressive	Streng	th, p.s.i.			
Chem	ical Additive (200	400	_600	800	1000	1200	1400	IEÓO	1800	2000
None				·	•	·		•		•	
0		×									
Sodium	n chioride, 1%										
Sođium	n metasilicate,1%	∆		ł							
Sodium	n carbonate,1%	Δ		<u> </u>							
Sodium	n hydroxide,1%		Δ.	¢.	⋺						
		I			<u>_</u>	1	<u>_</u>	i			I
		DOLC	MITIC	MONO	HYDR.	ATE L	IME				
Ch			In	nmersed	Comp	oressive	Streng	ith, p.s.i			
Cnem	ical Additive (<u>200</u>	400	600	800	1000	1200	1400	1600	1800	2000
None		A	0		ł						
Sodium	n chloride. 1%	Δ	0		3						
0 - dt		Δ	0]								
300100	n merosiiicate, 176			ı							
Sodium	n carbonate, 1%	A	0	J							
Sodiun	n hydroxide,i%		Δ	<u> </u>							
-							<u></u>				

Fig. 44. Effect of 1.0 percent chemical additive on strength of a 76.5:6:17.5 mixture of friable loess, lime, and fly ash no. 2.

curing temperatures. When soils are stabilized with lime and fly ash in the late part of the summer in temperate climates, they may not develop sufficient strength to withstand the imposed stresses of the colder seasons. This may lead to failure of the pavement.

The effect of chemical additives at low temperatures was investigated. Dune sand and fly ash No. 1 were used with both calcitic hydrated and dolomitic monohydrate limes. The specimens were molded at ambient temperatures. The curing temperature was $43 \pm 1^{\circ}$ F. Results for 7 and 28 day strengths were recorded (figure 46).

Calcitic lime

The mixture of dune sand, calcitic hydrated lime and fly ash No. 1 without additive, cured for 7 days, failed during the period of immersion in water. The same happened with the mixture with 1.0 percent sodium chloride as additive. Additions of 1.0 percent metasilicate, sodium carbonate or sodium hydroxide, however, gave strengths of about 100 psi.

After 28 days curing, the mixture without additive showed some immersed strength, 41 psi. This strength was increased five or sixfold by



Fig. 45. Effect of 1.0 percent chemical additive on strength of a 76.5:6:17.5 mixture of friable loess, lime, and fly ash no. 3.

Mixture Proportions 0 7 day curing 76.5% dune sand + 28 day curing 6.0% lime 17:5% fly ash No.i



Fig. 46. Effect of 1.0 percent chemical additive on strength of a 76.5:6:17.5 mixture of dune sand, lime, and fly ash no. I cured at a temperature of 43°F.

additions of 1.0 percent sodium metasilicate, sodium carbonate or sodium hydroxide. Sodium chloride produced a slight strength improvement.

Dolomitic lime

The untreated dune sand, dolomitic lime, and fly ash mixture did not show any immersed strength after 7 days curing. Additions of 1.0 percent sodium metasilicate gave a strength of 107 psi; 1.0 percent sodium carbonate gave 57 psi; and 1.0 percent sodium hydroxide gave 76 psi. Sodium chloride was not beneficial.

After 28 days, the untreated mixture had a strength of 111 psi. Additions of 1.0 percent sodium metasilicate or sodium carbonate more than doubled the strength. One percent sodium hydroxide increased the strength almost three times, to 298 psi. Specimens with sodium chloride did not show any immersed strength.

Discussion

The beneficial effects of some additives to the lime fly ash pozzolanic reaction are very important when low temperatures are expected during the curing period. Addition of promising chemicals may lengthen the working season for stabilization of soils with lime and fly ash.

The strengths obtained with dune sand, lime, and fly ash No. 1 mixtures cured at $43 \pm 1^{\circ}$ F. may be from 200 to 300 psi by the addition of a small amount of sodium metasilicate, sodium carbonate or sodium hydroxide. Those strengths may be sufficient in a base course to withstand the adverse effects of traffic and lower winter temperatures. Untreated sand, lime, and fly ash No. 1 mixtures after 28 days curing showed strengths of 100 psi or less, which are insufficient for a base course. The same beneficial effects may be expected with other fly ashes. Sand, lime, and fly ash mixtures made with either calcitic hydrated or dolomitic monohydrate lime increased in strength by the addition of sodium metasilicate, sodium carbonate or sodium hydroxide, but the data obtained herein were not sufficient to indicate which lime is more beneficial.

The chemical additives as salts also assist by lowering the freezing point of the free water in stabilized soil mixtures. By depressing the temperature at which the free soil water freezes, more time is allowed to gain strength; and the stabilized sail is exposed for shorter periods to the damaging effects caused by ice formation.

Mechanism of chemical additives in lime, and fly ash mixtures

A complete evaluation of the mechanism of the effects of chemical additives in lime and fly ash mixtures must involve extensive chemical analysis. Based on the strength data and on the assumption that strength is indicative of the extent of the pozzolanic reaction, the mechanism may be explained.

The effects of chemical additives on lime and fly ash may be due to one or more of the three following:

1) Speeding up of the pozzolanic reaction;

- 2) Production of secondary cementitious products; and
- 3) Combination with the primary, or pozzolanic, cementitious products.

The first should probably be of a catalytic nature. It may show up particularly in the curve for 7 day strength versus additive content, with a sharp increase in strength for small amounts of chemical added.

In the second, the chemicals combine or react with lime to form cementitious products like $CaCO_3$, $Ca(PO_4)_2$, $Al(OH)_3$, etc.

In the third are included those chemicals that may combine or react with the pozzolanic cement produced, with the pozzolanic materials in fly ash or with the soil. This combination or reaction may be a complex one producing better cementitious materials or speeding up the reaction or be a reaction that activates some of the materials, increasing their pozzolanic value.

For a separate evaluation of the different chemicals, they may be grouped on the basis of their reactions—basic, neutral or acidic. Bases and basic salts, also known as alkalies and alkaline salts, produce hydroxyl ions in water solution to varying extents. Acid salts produce hydrogen ions in water solutions to varying extents. Neutral salts in water solution do not upset the natural balance of hydrogen and hydroxyl ions. Another group is formed with phosphoric acid, and magnesium oxide is in a miscellaneous group.

This evaluation is made based on the results obtained with mixtures with Ottawa sand as a soil in this and in a previous investigation^{18, 50}. The characteristics of this sand make it, supposedly, an inert material in the lime and fly ash reaction or the lime, fly ash, and chemical reaction.

Bases and basic salts

Alkaline additives increase the amount of available hydroxyl ions in the moistened Ottawa sand, lime, and fly ash system. As a result, the pozzolanic reaction may be accelerated by the increased solubility of the siliceous material caused by the alkalinity³⁰.

The base, sodium hydroxide, acts as a catalyst supposedly in the following way:

a) It first reacts with the siliceous material to produce intermediate sodium silicates;

b) The over-all reaction goes to completion when the intermediate sodium silicates subsequently react with lime (calcium hydroxide) to form sodium hydroxide and cementitious insoluble calcium silicates;

c) The sodium hydroxide is then free for further reaction with unreacted siliceous material.

In the alkaline salts, sodium carbonate very likely reacts with lime in the moist Ottawa sand, lime, and fly ash mixture to form calcium carbonate and sodium hydroxide

 $Na_2CO_3 + Ca(OH)_2 \rightarrow CaCO_3 + 2NaOH.$

The precipitated calcium carbonate contributes cementation to the system, and as hypothesized the sodium hydroxide acts as a catalyst.

The other alkaline salts used, sodium phosphate, sodium metasilicate and lithium carbonate, may act similarly to sodium carbonate. Sodium phosphate reacts with lime to form calcium phosphate, which may be cementitious, and sodium hydroxide, which acts as a catalyst. Sodium metasilicate forms highly cementitious calcium silicates with lime and also releases sodium hydroxide. Lithium carbonate reacts with lime and precipitates calcium carbonate releasing lithium hydroxide, an alkali that produces the same catalitic effects as sodium hydroxide in the lime fly ash reaction. *Acid salts*

Acid salts undergo an hydrolysis reaction with the precipitation of weak bases (hydroxides). With calcium hydroxide (lime) and aluminum chloride this reaction proceeds as follows:

 $2\text{AlCl}_3 + 3\text{Ca}(\text{OH})_2 \rightarrow 2\text{Al}(\text{OH})_3 + 3\text{CaCl}_2.$

The weak base formed, $Al(OH)_3$, has some cementing properties that may be beneficial. The calcium chloride formed may also benefit through complex effects of the third category.

With calcium chloride, the principal long-term strength benefits obtained are thought due to a different type of chemical mechanism and are included in the third category of effects. Calcium chloride being highly hygroscopic and deliquescent ensures a relatively high concentration of calcium ions over a long period of time by providing moisture for a solution. Since lime has a low solubility and a lower ionization constant than calcium chloride, the concentration of calcium ions from lime is lower than that from calcium chloride.

The other acid salt used, manganese chloride, probably produces effects analogous to those of calcium chloride.

Neutral salts

Sodium chloride, although a neutral salt, may act as does calcium chloride; but it gives less benefit to long-term strength, perhaps because sodium chloride is less hygroscopic and deliquescent than calcium chloride.

The mechanism of the action of potassium permanganate in lime and fly ash mixtures is also included in the third category. Potassium permanganate, a strong oxidizing agent, may oxidize the carbon in the fly ash with subsequent production of potassium carbonate and the precipitation of manganese dioxide. The potassium carbonate formed may then give rise to further reactions, of the first and second category, similar to those of sodium carbonate, previously discussed, which are beneficial to strength. Potassium permanganate may also clean the surface of fly ash by oxidation of possible organic matter. This may make the fly ash more reactive with lime.

Acid

Very small amounts of phosphoric acid somewhat improved the strength. This improvement may be brought about by the formation of complex calcium phosphates or by the activation of fly $ash^{1, 25}$. Increased amounts of acid caused a decrease in strength, due to the neutralization caused by the acid which reduced the alkalinity and subsequently the silica release.

Miscellaneous chemical

Magnesium oxide is supposed to react with lime and fly ash producing effects of the third category. It may enter into the pozzolanic reaction and form complex silicates of calcium and magnesium. The effectiveness of magnesium oxide, a component of dolomitic monohydrate lime, in calcium hydroxide and fly ash mixtures corresponds with the findings of previous research which indicated that dolomitic monohydrate lime gives better strengths than calcitic hydrated lime in soil, lime, and fly ash mixtures cured at ambient temperatures.
Chemical additives in soil, lime, and fly ash mixtures

Four chemicals were evaluated with soils; sodium carbonate, sodium hydroxide, sodium metasilicate and sodium chloride. The greater benefits were obtained with the sandy soil; and the benefits decreased with the increase in the amount of clay in the soil.

With the available data it is difficult to evaluate the influence of the soil factor in soil, lime, fly ash, and chemical mixtures. The chemical additives used were beneficial in mixtures with friable soils and detrimental in mixtures with montmorillonitic clay soils. The decrease in strength in the clayey soils is likely brought about by the excess of sodium ions and high alkalinity in the pore fluid of the soil, lime, and fly ash mixtures. Both factors introduce in the clay structures disruptive forces that are not overcome by the cementitious bond of the pozzolanic reaction.

Modification of fly ashes

The processing of fly ash to broaden its use or to improve its qualities has not been extensively tried. In the manufacture of lightweight aggregate, a fly ash is sintered by a process developed a few years $ago^{5, 61}$. By the sintering process spherical particles $\frac{1}{8}$ to $\frac{1}{2}$ inch in size are made. This is carried out at a temperature high enough to cause the particles to adhere, but not to fuse. The spherical uncompacted pellets produced contain about 40 percent voids with a density of about 42 pcf.

Some work is now being done on the modification of fly ash by grinding. The results of this work show that it is possible to improve some of the properties of a fly ash, such as specific surface and specific gravity, and strength of mortars with lime.

The reaction between lime and fly ash is apparently a surface reaction, as the reactivity of a fly ash with lime is closely related to fineness and specific surface. It was supposed that by grinding or by scalping the coarse fraction, a fly ash might be improved for its use in soil stabilization. Consequently two low quality fly ashes, Nos. 2 and 4, were selected to be processed and used with dune sand and calcitic hydrated or dolomitic monohydrate lime.

The proportions used were 76.5 percent dune sand, 6 percent lime and 17.5 percent fly ash. The mixtures were run at several water contents and the maximum results are recorded (tables XXI, XXII).

Fly ash No. 2. This fly ash, with a 7.2 percent carbon content, was selected because it did not show any strength after 7 days curing for any combination of sand, lime and fly ash. The results show that neither grinding it to pass a No. 270 sieve nor using only the fraction passing the No. 270 sieve gave any improvements in 7 day strengths. For 28 and 90 day curing periods, the mixtures with the processed fly ash showed an increase in strength over the unprocessed, but this increase does not warrant the cost of processing this fly ash. Fly ash No. 4. This fly ash was chosen because it has a very high content of carbon, 18.6 percent. A different lime, dolomitic monohydrate, was used with this fly ash, and the No. 200 was used as a selector sieve instead of the No. 270.

Grinding the coarse part to pass the No. 200 sieve results in a slight increase in strength. Discarding the material retained in the No. 200 sieve, the strength is increased 64 percent after 28 days curing and 40 percent after 90 days. The processing of this high carbon fly ash may then be economical.

Discussion

The density of sand, lime, and fly ash mixtures increased greatly when the fly ash was modified by grinding or by scalping the coarse fraction. This increase in density is caused by an improvement in the gradation of the fly ash or by breaking down the hollow spheres, which are in any fly ash. An increase in strength is brought about by the increase in density and by a closer contact and/or more contact points between the lime and fly ash. A finer fly ash has more surface area available for the pozzolanic reaction to take place, which also brings an increase in strength. The increase in strength was partly a contribution of the higher densities and of the great surface area of the modified fly ashes.

A fly ash of high carbon content may be beneficially processed by sieving. The coarse material will contain most of the carbon, which is not reactive with lime and can be reused as a fuel. The fine material will be more reactive with lime, and can be used in soil stabilization or as a pozzolan in concrete.

The above tests suggest that the quality of fly ashes may be improved

TABLE XXI. COMPARATIVE RESULTS OBTAINED BY THE MODIFICATION OF THE FLY ASH OF A 76.5:6:17.5 mixture of dune sand, calcitic hydrated lime, and fly ash no. 2.

Process of fly ash	Maximum dry density, pcf	Maximum immersed unconfined compressive strength, psi				
		7 day	28 day	90 day		
As it is (unprocessed)	112	0	158	554		
Ground to pass the no. 270 sieve	116	0	203	631		
Discarded coarser than no. 270 sieve	118	0	175	633		

TABLE XXII. COMPARATIVE RESULTS OBTAINED BY THE MODIFICATION OF THE FLY ASH OF A 76.5:6:17.5 mixture of dune sand, dolomitic monohydrate lime, and fly ash no. 4.

Process of fly ash	Maximum dry density, pcf	Maz uncor	timum imme fined compr strength, ps	rsed essive i
v	• • • •	7 day	28 day	90 day
As it is (unprocessed) Ground to pass the no. 200 sieve Discarded coarser than no. 200 sieve	$105. \\ 110.5 \\ 126.5$	91 116 103	$309 \\ 408 \\ 506$	650 700 892

by grinding and/or sieving, which will broaden their use as a construction material.

Lime stabilization

It has been found in this investigation that the lime stabilization of some soils may sometimes not be appreciably benefited by the addition of fly ash. To obtain data to evaluate the use of lime or lime and fly ash, an extensive study of lime stabilization was made. Maximum strengths up to 90 days were recorded, and up to 25 percent of lime was used (tables XXIII to XXVI, and figures 47, 48, 49).

Presentation and discussion of results

Dune sand. Though sandy soils do not benefit by the addition of small amounts of lime, it was suspected that large percentages of lime might impart some strength. Therefore, quantities of lime up to 25 percent were studied in mixtures with dune sand (table XXIII).

The large quantities of lime strengthened the dune sand; for instance a mixture of 25 percent dolomitic monohydrate lime and 75 percent sand, had 7 and 28 day strengths of 112 and 215 psi. respectively. But the additions of so much lime is not economical. It was also observed that dolo-

Lime		Molding dry density, pcf	lmm compre	ersed uncon essive streng	fined rth. psi
Kind	%		7 day	28 day	90 day
Calcitic hydrated	$3 \\ 9 \\ 12 \\ 15 \\ 25$	110 113 117 119 120.5 112	0 8 19 30 64	11 11 25 30 51 73	11 12 31 42 ND* ND
Dolomitic monohydrate	$3 \\ 6 \\ 9 \\ 12 \\ 15 \\ 25$	110 113 116.5 119 120.5 120.0	$egin{array}{c} 0 \\ 0 \\ 21 \\ 32 \\ 53 \\ 112 \end{array}$	$11\\15\\29\\51\\120\\215$	14 31 57 93 ND ND

TABLE XXIII. STRENGTHS OF DUNE SAND STABILIZED WITH LIME.

*Not determined.

TABLE XXIV. STRENGTHS OF FRIABLE LOESS STABILIZED WITH LIME.

Lime		Molding dry density, pcf	Imm compre	ersed uncon essive streng	fined th, psi
Kind	%		7 day	28 day	90 day
Calcitic hydrated	$\begin{array}{c}3\\6\\9\\12\end{array}$	99.9 99.0 99.0 97.7	72 59 78 64	$110 \\ 105 \\ 158 \\ 144$	$287 \\ 403 \\ 499 \\ 435$
Dolomitic monohydrate	$3 \\ 6 \\ 9 \\ 12$	$100.9 \\ 100.8 \\ 100.6 \\ 100.5$	$117 \\ 151 \\ 174 \\ 182$	$249 \\ 354 \\ 400 \\ 369$	234 584 621 588

mitic monohydrate lime produced much higher strengths than calcitic hydrated lime. The strengths obtained with lime may be greatly increased by the addition of a fly ash.

The added strength obtained by the addition of lime to sandy soils probably comes mainly from carbonation of the lime. But part of the strength may have been caused by the formation of calcium silicates, although this is not likely to have occurred at the curing temperatures used in this research.

Friable loess. This soil shows a great pozzolanic activity with lime. It has been pointed out in another section, that based on 7 and 28 day curing periods the addition of some fly ashes diminishes the strength obtained with this loess and lime only, but the pozzolanic action between loess and lime continues and is important beyond 28 days (figure 47). Very small amounts of lime are needed to develop the full strength that may be obtained by addition of lime. Six percent of dolomitic monohydrate lime appears to be the best amount; use of greater percentages do not appear warranted.



Fig. 47. Strengths obtained by additions of different amounts and kinds of lime to friable loess.

Friable loess should not be stabilized with lime and fly ash unless a very good quality fly ash is used. Six percent dolomitic monohydrate lime gave strengths of 150, 354 and 584 psi for 7, 28 and 90 days curing respectively. These strengths were actually lowered by the addition of a medium or low quality fly ash.

Alluvial clay. The strengths obtained with alluvial clay stabilized with lime were relatively low (figure 48). The desirable value of 300 psi after 28 days curing may be obtained with 9 percent dolomitic lime, but for this amount the strength is not improved beyond 28 days. A recommended amount of lime is 12 percent, but the strengths obtained with this amount



Fig. 48. Strengths obtained by additions of different amounts and kinds of lime to alluvial clay.

TABLE XXV. STRENGTHS OF ALLUVIAL CLAY STABILIZED WITH LIME.

÷.	-	D	Immersed unconfined				
Lime		Density, pcf	compr	essive streng	rth, psi		
Kind	%	,	7 day [°]	28 day	90 day		
Calcitic	3	92.4	125	132	124		
hydrated	6	91.1	129	182	194		
	9	90.6	128	166	218		
	12	89.8	112	158	241		
Dolomitic	3	93.5	48	48	35		
monohydrate	6	92.2	173	274	250		
v	9	91.5	173	345	336		
	12	90.8	194	334	415		

may also be obtained with an economically competitive lime and fly ash admixture.

Gumbotil. In the experiments with gumbotil, lime was added in amounts up to 25 percent (figure 49). In every curing period a percentage of lime was found above which there was no appreciable increase in strength. This "breaking" percentage tends to be higher for the longer curing periods. (This was also observed in the results with alluvial clay.)

At least 9 percent of either dolomitic or calcitic lime is recommended. With dolomitic lime, 200 and 300 psi. may be obtained after 7 and 28 days curing respectively. These figures are rather low and may be increased by the addition of fly ash, or by substituting some lime for fly ash. Lime and fly ash may compete economically and strengthwise with the minimum amount of lime required.

Discussion

These researches change the concept that in lime stabilization a small



Fig. 49. Strengths obtained by additions of different amounts and kinds of lime to gumbotil.

TABLE XXVI. STRENGTHS OF GUMBOTIL STABILIZED WITH LIME.

Lime	0/0	Molding dry density, pcf	Imm compre 7 day	ersed uncon essive streng 28 dav	fined th, psi 90 day
mu	70		, aug	_0 u,	oo uuj
Calcitic hydrated	$3 \\ 6 \\ 9 \\ 12 \\ 15 \\ 25$	93.5 89.5 87.1 87.1 87.0 86.4	$100 \\ 116 \\ 125 \\ 132 \\ 141 \\ 173$	$145 \\ 155 \\ 215 \\ 228 \\ 240 \\ 307$	97 317 386 478 ND* ND
Dolomitic monohydrate	$3 \\ 6 \\ 9 \\ 12 \\ 15 \\ 25$	93.8 92.5 92.3 92.3 89.8 86.2	$0\\89\\191\\190\\197\\211$	$\begin{array}{c} 0 \\ 104 \\ 274 \\ 298 \\ 296 \\ 326 \end{array}$	0 188 429 495 ND ND

*Not determined.

amount of lime added to soil is sufficient to obtain the maximum benefits of lime. It is possible that this concept was the result of a testing program limited in time. Observation of 7 and 28 day strengths may lead to that erroneous concept (figures 47, 48, 49). But when curing periods were continued up to 90 days, the strength gain with time was found to be influenced by the amount of lime. It might be concluded, from the 7 and 28 day strengths of friable loess (figure 47), that 3 percent lime is the best amount to stabilize this soil; higher amounts do not particularly add to strength. But a study of 90 day strengths shows that 6 percent should be the recommended amount of lime. Therefore the amount of lime needed to stabilize a soil should be determined on the basis of long as well as short curing periods. If it is desirable to obtain a high long-term strength, the highest economically possible amount of lime should be used.

It was also found (figures 47, 48, 49) that calcitic hydrated lime was more effective than dolomitic monohydrate lime in low amounts of around 3 percent of lime. Dolomitic monohydrate lime was more effective than calcitic hydrated in amounts of lime of 6 percent or higher. Consequently when small amounts of lime are used, the calcitic hydrated type should be favored. For high amounts, dolomitic monohydrate lime should be used.

Moisture-density of clayey soils treated with lime

The moisture-density curves for gumbotil and alluvial clay treated with lime and fly ash had a peculiar shape (figures 4, 5, 6 and 8). There was not a distinctive maximum density; it being undefined in many instances. Fly ash was found not to be the cause of this.

The shape of the curves of moisture-density relationships of a friable loess and lime mixture follow the concept of a maximum density at an optimum moisture content (figure 50). This soil, friable loess, has a relatively low amount of clay, 17 percent. But for mixtures of gumbotil and lime or alluvial clay and lime there is no defined maximum density for an optimum moisture content, and the drier the mixtures the greater the dry density obtained (figures 51, 52). Both gumbotil and alluvial clay have a very high content, about 70 percent, of montmorillonitic clay. It was suspected that high amounts of clay, at least of the montmorillonitic type, were the cause of the poorly defined shape of moisture-density curves.

To find if the soil without lime had the same shape of moisture-density curves, some comparative tests were made. For instance in figure 54 moisture-density curves for alluvial clay are plotted with and without lime, compacted with the same compactive effort (figure 53). A wide range of moisture contents was used in these tests. The curve for straight soil shows a continuous increase in density as the water content increases, up to a maximum density; higher amounts of water decrease the density. The curve for the soil lime mixture shows a small increase in density with increase in water content for very low amounts of moisture; from then on, the density decreases with the increase in water content. The addition of lime to soils of high content of montmorillonitic clay distorted the shape of moisture-density curves.

The moisture-density curves for montmorillonitic clay soils stabilized with lime are probably affected by the flocculating effects of lime. The lime alters the characteristics of clayey soils converting them into a material with the workability of friable soils. At low moisture contents the flocculating effects of lime impart to clayey soils a highly open structure. This facilitates the expulsion of air which becomes more important to the increase of density than does the lubricating effects of water. The free expulsion of air from a mass containing about one-third void space can easily influence greatly the final compacted dry density at low moisture contents.

The maximum strength does not occur at a point of maximum density (figures 51, 52, 53). A second point of maximum strength is initiated for high moisture contents. This is more clearly seen in the 28 day strength curve. Therefore the present concept used in soil stabilization of compaction at the optimum moisture content for maximum dry density should be reviewed. The strength gain or hardening of mixtures stabilized with lime and fly ash comes from the formation of cementitious products rather than



Fig. 50. Moisture-density and moisture-strength relationships of a mixture of 91 percent friable loess and 9 percent calcitic hydrated lime, compacted at standard Proctor compactive effort.



Moisture content,



from density. A high moisture content maintains a larger supply of water for the hydration process to proceed at a faster rate and/or for longer periods. It is therefore recommended that in the stabilization of soils with lime or lime and fly ash, the molding or compacting moisture content be chosen on the basis of the maximum strength rather than the maximum density of the mixture.

TABLE XXVII. IMMERSED UNCONFINED COMPRESSIVE STRENGTH OF MIXTURES OF SOIL,

*	STAL	SILIZED WITH	FORTLAND	CEMENT.		
Soil	Lime treatment % and kind	Cement, %	Dry density, pcf	Imm compr 7 day	ersed uncon essive streng 28 day	fined gth, psi 90 day
Dune sand Dune sand Dune sand	None None None	5 8 10	$110.8 \\ 112.7 \\ 117.1$	$127 \\ 398 \\ 591$	$184 \\ 474 \\ 770$	$228 \\ 541 \\ 802$
Friable loess Friable loess	None None	$6\\9$	$\begin{array}{c} 101.3\\ 103.5\end{array}$	$\begin{array}{c} 330\\ 423\end{array}$	$\begin{array}{c} 495\\ 566\end{array}$	$\begin{array}{c} 715 \\ 1001 \end{array}$
Alluvial clay Alluvial clay Alluvial clay	3, dol. 3, dol. 3, dol.	3 6 9	$93.5 \\ 94.0 \\ 94.9$	$266 \\ 328 \\ 391$	$341 \\ 469 \\ 574$	369 501 ND*
Gumbotil Gumbotil Gumbotil	4, calc. 4, calc. 4, calc.	3 5 8	$94.2 \\ 93.4 \\ 94.4$	$317 \\ 440 \\ 515$	376 493 586	$463 \\ 687 \\ 870$
Gumbotil Gumbotil	4, dol. 4, dol.	5 8	$95.0 \\ 94.7$	$\begin{array}{c} 432 \\ 534 \end{array}$	$\begin{array}{c} 507 \\ 692 \end{array}$	590 830

*Not determined.





Fig. 52. Moisture-density and moisture-strength relationships of a mixture of 91 percent alluvial clay and 9 percent calcitic hydrated lime, compacted at standard Proctor compactive effort.



Portland cement stabilization

An evaluation of lime fly ash stabilization is not complete without a comparison of its effectiveness with that of cement stabilization. Strength results for several percentages of cement are presented and discussed here.

Plastic soils to be stabilized with cement should be pre-treated with lime to flocculate the soil particles and thus facilitate the mixing process. Alluvial clay and gumbotil are soils of high plasticity needing the lime pretreatment. Consequently alluvial clay was treated with 3 percent lime and gumbotil with 4 percent in addition to cement. Both lime and cement were added together.

The same water content found optimum for soil lime specimens was used here (table XXVII, and figures 54 to 57).

Discussion

Portland cement in the proper amounts stabilized any of the four soils tested. Good strengths were obtained with at least 8 percent cement in dune sand, 6 percent in loess, 3 percent lime plus 6 percent cement in alluvial clay and 4 percent lime plus 5 percent cement in gumbotil. These mixes gave 7 day strengths over 300 psi.

Most of the final strength was developed in the first seven days. The rate of increase after seven days was not very pronounced, except with the loess. In the length of time needed to develop strength lies an important difference between cement and lime fly ash stabilization (compare figures 54 and 27; figures 55 and 29; figures 56 and 28, and figures 57 and 30). The early strengths for lime fly ash were low, but the strength steadily increased with time at a fairly good rate. For long curing periods the



Fig. 54. Strength of dune sand cement mixtures.

strengths with lime and fly ash and with cement tended to equalize; being in many instances greater for lime fly ash than for cement treated soils.

Both calcitic hydrated and dolomitic monohydrate limes were used with gumbotil to compare their effectiveness in changing the plasticity in the lime treatment. The results were erratic and do not show consistently



Fig. 55. Strength of friable loess cement mixtures.



Fig. 56. Strength of gumbotil cement mixtures.

better improvements, based on strength, with one or the other lime (table XXVII). Further tests should be conducted to compare the effectiveness of both limes in treatments for soil cement stabilization. In the meantime the cheapest one available is recommended.

Durability evaluation

The effectiveness of lime fly ash stabilization was compared with that of other methods of soil stabilization. A few mixes were selected with the proper amount of lime and fly ash for each soil, to compare them with mixes in which lime and/or cement was the stabilizer. The comparison included freeze-thaw testing of selected mixes.

Dolomitic monohydrate lime and fly ash No. 3 were the most suitable lime and fly ash for stabilizing any of the four Iowa soils evaluated here. The addition of chemicals is highly recommended with sandy soils; therefore chemical additives were used in three mixes with dune sand. Sodium carbonate and sodium chloride were chosen as additives based on strength improvements, cost of the chemicals, and practicability of their use in field construction. The composition and proportions of the selected mixtures vary somewhat with each soil.

The proportions used in the soil, lime, and fly ash mixtures were calculated to compete with the required amount of cement and/or lime needed to stabilize the same soil. Use was made of the Iowa State equal cost line method for soil, lime, and pozzolan mix design⁷¹.

It was assumed that:

a) Eight percent cement is required to stabilize dune sand.

b) Ten percent cement or 9 percent dolomitic monohydrate lime is required to stabilize friable loess.

c) Three percent lime and 9 percent cement are required to stabilize alluvial clay.



Fig. 57. Strength of alluvial clay cement mixtures.

d) Four percent lime and 8 percent cement are required to stabilize gumbotil.

e) The cost of lime or cement is the same, about \$22 a ton.

f) The cost of fly ash is one-sixth that of lime or cement.

g) The cost of handling two materials (lime and fly ash; lime and cement), instead of one if stabilized with cement or with lime only, is equal to the cost of one percent of cement.

h) The cost of sodium carbonate and handling this extra material is 2.5 times that of an equal amount of cement, and the cost of one percent of sodium chloride and extra handling is the same as one percent of cement.

Dune sand

The sand, lime, and fly ash equal cost line graph for the selected mixtures is given in figure 58. All the mixtures within the triangle ABC have the same cost or are cheaper than the required 8 percent of cement needed to stabilize dune sand.

Based on 28 day strength requirements, lime and fly ash may be economically used to stabilize sandy soils (table XXVIII). Either lime and fly ash mixtures or lime and fly ash mixtures with chemical additives withstood the severity of freezing and thawing tests and had enough residual



Fig. 58. Equal-cost-line charts for soil stabilized with selected admixtures of lime and fly ash or of lime, fly ash and chemical compared with mixtures of soil, lime and cement or of soil and cement.

strength to be considered adequately stable. A good quality fly ash (No. 3) was used in these tests; these results may not be reproduced with all kinds of fly ash.

All five selected dune sand, lime, and fly ash mixtures gave 28 day strengths equal or greater than dune sand and cement for the same curing period. It has been estimated that after freezing and thawing, the stabilized soil specimens should have a minimum strength of 250 psi¹⁶. This value was surpassed by all mixtures (col. p_f , table XXVIII). It is desirable that soil stabilized specimens show an index of resistance to the effects of freezing (R_f) of at least 80 percent to satisfactorily withstand Iowa climatic conditions¹⁶. Only mixes Nos. 4 and 6 gave indexes of resistance lower than 80 percent; however, they had R_f values of 78 percent, which should be adequate, since the values of p_f and p_c are over 400 psi.

Some mixtures continued gaining strength during freezing and thawing cycles and/or during immersion. None of the mixtures showed any visible damage from freeze-thaw, neither did they show any expansion.

The as-molded dry density of the several mixtures changed by as much as 12 pcf, but there was no relationship between density and strength values.

Friable loess

Only one loess, lime, and fly ash mixture could compete economically and on a strength basis with loess and cement or loess and lime mixtures. That loess, lime, and fly ash mixture was 72 percent loess, 3 percent dolomitic monohydrate lime and 25 percent fly ash No. 3 (table XXIX). It was compared with mixtures of the same soil stabilized with 9 percent dolomitic monohydrate lime or with 10 percent cement. The amount of 9 percent dolomitic lime was based on a previous evaluation using different amounts of lime (table XXIV). Ten percent cement was chosen based on the ASTM requirements to stabilize this kind of soil³.

Strengths of 400 psi were obtained with all selected mixtures after a curing period of 28 days. The mixtures exposed to 10 cycles of freezing and thawing showed a strength either close to 400 psi or well over this value, which is very adequate for a base course. The indexes of resistance were over the minimum of 80 percent desired.

Friable loess can be stabilized with cement, lime, or lime and fly ash for use as a road base course material. The 10 percent cement mixture gives strengths that are much higher than those obtained with mixtures with lime or with lime and fly ash. It appears that a lower amount of cement might also adequately stabilize friable loess. For instance, mixture No. 15 (table XXIX) of 6 percent cement and 94 percent loess gave a strength of 495 psi after 28 days. This strength is comparable with that obtained with the selected mixtures of loess and lime and of loess, lime, and fly ash. Therefore, it is possible that 6 percent cement would be an adequate

Mix no.	Proportions	As-molded dry density, pcf	Unconfi 28 day*	ined compres P _f ‡	ssive stren P _c ‡	gth, psi R ₁ §. %
$egin{array}{c} 1 \\ 2 \\ 3 \\ 4 \end{array}$	92% sand, 8% P. cement 73% sand, 3% dol. lime, 24% fly ash no. 3 76% sand, 4% dol. lime, 17.5% fly ash no. 3 82% sand, 3% dol. lime, 15% fly ash no. 3	$112.6 \\ 124.3 \\ 124.4$	$474 \\792 \\646$	$\begin{array}{c} 507\\821\\634 \end{array}$	$517 \\ 966 \\ 674$	98 85 94
4A 5	+ 0.5% sodium carbonate 82% sand, 3% dol. lime, 15% fly ash no. 3 82% sand, $\frac{3}{2}$ % calc. lime, 15% fly ash no. 3	$\begin{array}{c} 117.2\\ 123.8\end{array}$	$\begin{array}{c} 554 \\ 390 \end{array}$	452 ND**	583 ND	78 ND
6	+ 0.5% sodium carbonate 82% sand, 3% calc. lime, 15% fly ash no. 3	116.1	644	596	570	104
5A-6A	+ 0.5% sodium chloride 82% sand, 3% calc. lime, 15% fly ash no. 3	$\begin{array}{c} 124.1\\ 123.1 \end{array}$	$\begin{array}{c} 453 \\ 120 \end{array}$	414 ND	454 ND	78 ND
*After 28 c †After 28 c	lays curing and 24 hours immersion in distilled water. lays curing, 24 hours immersion in distilled water and ten fi	eeze-thaw cycles.				

\$After 28 days curing and 11 days immersion in distilled water and $R_{f} = \frac{100 P_{f}}{P_{e}}$. **Not determined.

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TABLE XXIX.	DURABILITY	EVALUATION	OF	SELECTED	ADMIXTURES	то	STABILIZE	FRIARLE	LOESS
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Mix no.	Proportions	As-molded dry density, pcf	Unconfi 28 day*	ined compres P _f †	ssive stren P.‡	gth, psi R ₁ §, %
11 12 13	90% loess, 10% cement 91% loess, 9% dol. mon. lime 72% loess, 3% dol. mon. lime.	$\begin{array}{c} 103.5\\ 100.8\end{array}$	$\begin{array}{c} 645\\ 396\end{array}$	$\frac{567}{387}$	682 428	83 90
$\begin{array}{c} 14 \\ 15 \end{array}$	25% fly ash no. 3 91% loess, 9% cement 94% loess, 6% cement	$99.1 \\ 103.5 \\ 101.3$	$462 \\ 566 \\ 495$	441 ND** ND	$521 \\ \mathrm{ND} \\ \mathrm{ND}$	85 ND ND

*After 28 days curing and 24 hours immersion in distilled water. †After 28 days curing, 24 hours immersion in distilled water and ten freeze-thaw cycles. ‡After 28 days curing and 11 days immersion in distilled water. $R_{\rm f} = \frac{100 \ P_{\rm f}}{P_{\rm c}}$. *Not determined.

TABLE XXVIII. DURABILITY EVALUATION OF SELECTED ADMIXTURES TO STABILIZE DUNE SAND.

is much below that of cement, or a high quality fly ash is cheaply available.

Gumbotil

Two fly ashes, No. 2 and No. 3, were used with dolomitic monohydrate lime to stabilize gumbotil and to make an evaluation of the durability of these mixtures. The proportions used, based on previous results, were 69 percent gumbotil, 6 percent lime and 25 percent fly ash (table XXX). The strengths previously obtained with lime and gumbotil were rather low (table XXIV) and do not recommend the use of straight lime stabilization for base course construction with gumbotil; therefore the use of lime was not evaluated. The amount of cement to stabilize gumbotil, based on ASTM requirements is about 12 percent³; this was the amount used in the durability studies.

Without using lime it would be impossible to field mix gumbotil with cement, because gumbotil is an extremely plastic clay soil. Hence, four percent of the required amount of cement was replaced by lime to decrease the plasticity of the soil.

Both mixtures in which lime and fly ash was the stabilizing agent gave strengths comparable with that of the mixture of gumbotil stabilized with lime and cement. The strengths after 28 days curing were above 600 psi for both immersion periods and for all three mixes selected for the freezethaw studies. The strengths after freezing and thawing cycles were about 540 psi for the three mixes. These strengths are very good for this highclay content soil and warrant the use of these mixtures as a base course material. The indexes of resistance are adequate for mixes Nos. 21 and 22 (table XXX). Mix 23 had a rather low index of resistance of 68 percent. This index value is due to a substantial gain of strength during the 11 day immersion period. Provided that the strength after the Iowa freeze-thaw test is still 529 psi, gumbotil may be used in a base course when stabilized with the materials and proportions of mix 23; that is 69 percent gumbotil, 6 percent dolomitic monohydrate lime and 25 percent fly ash No. 3.

As evident by the strength obtained with mix 24, good strengths may be secured with lesser amounts of lime and cement. However, strengths equivalent to those of mix 24 may be also obtained with lesser amounts of lime and fly ash than those used in mixes 21 and 22. It may therefore be concluded that gumbotil can be stabilized with lime and fly ash, competing economically with cement.

The strengths obtained with the specimens prepared with gumbotil for the durability evaluation studies had greater strengths than specimens made with the same admixtures in previous studies. This lack of reproducibility of strength was found only with gumbotil. It is possible that specimens prepared for the durability studies were benefited during curing by temperatures slightly higher than in the other studies, causing the strength differences noted.

the smallest amount needed for stabilizing

TABLE XXX. DURABILITY EVALUATION OF SELECTED ADMIXTURES TO STABILIZE GUMBOTIL.

Mix no.	Proportions	As-molded dry	Unconfi 28 dou*	ned compre	ssive streng	th, psi
		density, per	20 uay	- c †	₽f∔	R19, %
$\frac{21}{22}$	88% gumbotil, 4% dol. mon. lime, 8% cement 69% gumbotil, 6% dol. mon. lime, 25% fly	95.1	705	634	550	87
23	ash no. 2 69% gumbotil, 6% dol. mon. lime, 25% fly	90.0	606	642	534	83
24	ash no. 3 91% gumbotil, 4% dol. mon. lime, 5% cement	$\begin{array}{c} 94.1 \\ 93.3 \end{array}$	$\begin{array}{c} 682 \\ 534 \end{array}$	780 ND*	529 ND	68 ND
*After 28 da †After 28 da ‡After 28 da ‡After 28 da $R_{f} = \frac{100 \text{ H}}{P_{c}}$ **Not determ	ys curing and 24 hours immersion in distilled water. ys curing, 24 hours immersion in distilled water and ten fro ys curing and 11 days immersion in distilled water. 	eeze-thaw cycles.	-			- 12

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TABLE XXXI. DURABILITY EVALUATION OF SELECTED ADMIXTURES TO STABILIZE ALLUVIAL CLAY.

Mix no.	Proportions	As-molded dry density, pcf	Unconfi 28 day*	ned compres	ssive stren Pat	gth, psi
31	88% alluvial clay, 3% calc. hyd.		•	- 1	- 04	1013, 70
32	lime and 9% cement 69% alluvial clay, 6% dol. mon.	94.9	574	498	527	94
33	lime and 25% fly ash no. 3 91% alluvial clay, 3% dol. mon.	93.6	513	475	563	84
	lime and 6% cement	94.0	470	ND**	ND	ND

*After 28 days curing and 24 hours immersion in distilled water. †After 28 days curing, 24 hours immersion in distilled water and ten freeze-thaw cycles. ‡After 28 days curing and 11 days immersion in distilled water. $R_{\rm f} = \frac{100 \ P_{\rm f}}{P_{\rm c}}$. **Not determined.

alluvial clay, according to ASTM tests³. The lime and fly ash combinations that might give strengths comparable with those obtained with cement were those made with dolomitic monohydrate lime plus fly ash No. 3 (table XXXI). Mixtures of alluvial clay and lime did not show high strength (table XXVI), so they were not evaluated here.

Instead of using the full cement requirement of 12 percent, 3 percent lime and 9 percent cement were used. The lime was used primarily to give the soil friable characteristics which would allow better mixing with the cement. Lime also may counteract any adverse effects from the somewhat high organic matter content of the alluvial clay.

Both mixtures when tested by freezing and thawing gave strengths of around 500 psi for any of the three testing treatments tried. The indexes of resistance were also above the minimum desired. It appears that alluvial clay stabilized with the proper lime and fly ash admixture may have strengths and durability comparable with those of alluvial clay stabilized with cement, and may be economically competitive as well (figure 58).

Mix 33, of 91 percent alluvial clay and 9 percent lime and cement, was not evaluated in freezing and thawing, but gave seemingly adequate strength. It is also possible that mixtures containing smaller amounts of fly ash than mix 32 might give strengths equal to those of mix 33.

CONCLUSIONS

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

2. Maximum strength of soil and lime mixtures also may be at a compaction moisture content different from the optimum moisture content for maximum density.

3. The required compaction moisture content to produce maximum strength changes with the curing period of soil, lime, and fly ash mixtures: the longer the curing period the greater the compaction moisture content needed for maximum strength.

4. Increasing the compactive effort from standard Proctor to modified Proctor increases the strength of soil, lime, and fly ash mixtures. The strength increase obtained is variable, but usually is from 50 to 160 percent.

5. There is no optimum amount or ratio of lime and fly ash for stabilizing all soils. The amount and proportions of lime and fly ash to use depend greatly on the kinds of fly ash and soil, and somewhat on the kind of lime. For granular soils the amount of lime should be between 3 and 6 percent; the amount of fly ash between 10 and 25 percent. For clayey soils the amount of lime should be between 5 and 9 percent; the amount of fly ash between 10 and 25 percent.

6. Dolomitic monohydrate lime generally gives better strengths in soil, lime, and fly ash mixtures than calcitic hydrated lime in normal amounts and when cured at ambient temperatures.

7. At low lime contents, of around 3 percent, calcitic hydrated is more effective than dolomitic monohydrate for stabilizing clayey soils with or without fly ash; at higher lime contents, dolomitic monohydrate gives better strengths than calcitic hydrated.

8. The fly ashes used were beneficial to soil and lime mixtures for all soils except friable loess. With the friable loess, only a high quality fly ash was beneficial to loess and lime mixtures.

9. Heating of the materials to high temperature at the time of mixing lowers the compacted density and cured strength of clayey soil, lime, and fly ash mixtures.

10. 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. At the most, with clayey soils, wet mixing and compaction should be done the same day; but when sandy soils are used, compaction could be delayed until the day after wet mixing without appreciable loss of strength.

11. Increase of temperature accelerates the lime fly ash pozzolanic reaction, and the strength of soil, lime, and fly ash mixtures may be greatly increased by moist curing at higher than ambient temperatures. Soil and lime and soil and cement mixtures are also benefited by high temperature moist curing.

12. Steam cured specimens of soil stabilized with lime, with lime and fly ash, or with cement after a few hours attain strengths comparable with those of concrete.

13. At ambient temperatures, dune sand or dune sand and fly ash stabilized with dolomitic monohydrate lime reaches generally higher strengths than when stabilized with calcitic hydrated lime, but at high temperatures (above 140°F.) calcitic is better than dolomitic lime.

14. The quality of a fly ash for soil stabilization is reflected in the unconfined compressive strength developed in mixes with lime after curing at any temperature. A mixture made with a high quality fly ash will always show greater strength than a mixture made with a low quality one, regardless of the curing temperature at which both mixtures were cured.

15. No correlation was shown between long-term strength at ambient curing temperatures and short-term strength at elevated curing temperatures for soil, lime, and fly ash mixtures. The strength correlation depends on the kind of fly ash, the kind of lime, and probably also on the type of soil.

16. The quality of a fly ash can be improved by removing the coarse fraction and/or by grinding.

17. The strength attained with soil, lime, and fly ash mixtures may be increased by the addition of small amounts of some chemicals. Sodium carbonate, sodium metasilicate, and sodium hydroxide seem to be the most promising ones, as indicated by strength improvements and economic considerations. This benefit is greatest in mixtures with sandy soils followed by mixtures with soils of low plasticity. Clayey soils stabilized with lime and fly ash do not benefit from the addition of sodium hydroxide, sodium carbonate, or sodium metasilicate.

Although the increase of strength gained from the use of chemical additives occurs over the ordinary range of temperatures, the additives are especially needed at temperatures close to freezing when they may permit extending the working season of the soil, lime, and fly ash stabilization.

Sodium carbonate is the chemical most highly recommended for use in sandy or silty soils stabilized with lime and fly ash. The addition of 0.5 percent sodium carbonate permits a reduction in the amounts of lime and fly ash needed to attain the same strength that may be obtained by using greater amounts of lime and fly ash.

18. The amount of lime needed to stabilize a soil should be determined on the basis of short as well as long curing periods. Small amounts of lime give early strengths equal to or higher than larger amounts of lime, but after long curing periods the larger amounts will produce the greatest strengths.

19. The moisture-density curves of montmorillonitic clay soils stabilized with lime are affected by the flocculating effects of lime. Sometimes the curves do not show a maximum density.

20. Cement is a very effective stabilizer for most soils. The strength gain of mixtures of soil and cement is rapid, and a large percentage of ultimate strength is developed in a relatively short time. But compacted soil, lime, and fly ash mixtures gain strength slowly, and full strength may not be developed for several years. The comparison of soil and cement and soil, lime, and fly ash test specimens should be made on the basis of 28 day curing. After this period, soil cement should have developed about 90 percent of the ultimate strength, and soil, lime, and fly ash only about 50 percent, depending on the soil, lime, and fly ash used.

21. Selected compositions of dune sand, lime, and fly ash, or of dune sand, lime, fly ash, and chemicals can compete in strength, freeze-thaw resistance, and cost with mixtures of the same soil stabilized with cement.

22. Friable loess is most effectively stabilized with cement. If lime is cheap and a good quality fly ash is available, lime or lime and fly ash may compete with cement for stabilizing friable loess.

23. Additions of fly ash are beneficial to gumbotil and lime mixtures. Se-

lected gumbotil, lime, and fly ash mixtures show good resistance to freezing and thawing, and may compete with gumbotil cement stabilization.

24. Additions of fly ash are beneficial to alluvial clay and lime mixtures. Lime fly ash stabilization of alluvial clay may compete economically and strengthwise with cement stabilization.

RECOMMENDATIONS FOR FURTHER STUDY

The following suggestions for further research are an outgrowth of this investigation:

1. Moisture-density and moisture-strength relationships for mixtures of soil, lime, and fly ash should be compared with the effect of molding moisture content on strength determined at curing periods up to one year. With clayey soils these studies should include specimens made with the highest moisture contents possible.

2. The same moisture-density and moisture-strength studies should be made for mixtures of soil and lime.

3. Moisture-density and moisture-strength relationships should be compared for mixtures of soil and cement, with the effect of molding moisture content on strength determined at different curing periods up to 90 days or longer.

4. A basic investigation be made to determine the products formed in the lime fly ash reaction.

5. A basic investigation be made to determine the effects of lime in clayey soils in both compacted and uncompacted states and at different moisture contents.

6. A method be developed for finding the pozzolanic activity of a fly ash by curing lime and fly ash specimens for short curing periods at elevated temperatures.

7. A machine might be designed to heat layers of road courses built with stabilized soil. A portable nuclear reactor could be the source of cheap energy.

8. The effect of fineness of lime on strength of soil, lime, and fly ash mixtures should be studied.

9. A further evaluation ought to be made of the effect of chemical additives on the strength of soil, lime, and fly ash mixtures.

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APPENDIX

INTERPOLATION OF OPTIMUM MOISTURE AND MAXIMUM DENSITY VALUES IN SOIL, LIME, AND FLY ASH MIXTURES

A method of estimating the optimum moisture content of lime, fly ash, and soil mixtures has been proposed previously^{23, 71}. In this method it is assumed that the optimum moisture content and maximum density for mixtures of soil and lime, with lime contents between 0 percent and 50 percent, change linearly. It has been found that this does not happen; there is a decrease in maximum density and an increase in optimum moisture content for very small amounts of lime, of the order of 1 to 3 percent, in a soil and lime mixture as compared with the increase in the untreated soil. This is very conspicuous with clayey soils. Additions of larger amounts of lime cause only small additional changes in the optimum moisture or maximum density of soil and lime mixtures. Similar variations of these properties, although to a much smaller degree, may occur for soil and fly ash mixtures containing amounts of fly ash between 0 percent and 50 percent.

It is felt that the estimation of optimum moisture content and maximum dry density of soil, lime, and fly ash mixtures by the previously proposed method should be discontinued. A much greater degree of accuracy may be obtained by interpolating from the values obtained for mixtures of soil, lime, and fly ash proportioned within the range of the recommended quantities for a proper stabilization. The optimum moisture and maximum density changes in this range of mixtures have more nearly a linear relationship.

The same triangular chart can be used, but instead of interpolating the optimum moisture and maximum density between values obtained for mixtures in the vertices, they can be interpolated from values obtained for mixtures that are located inside the triangle.

For example, with a clayey soil the following control mixtures might be used:

1) Mixture composed of 4 percent lime, 15 percent fly ash and 81 percent soil;

2) Mixture composed of 7 percent lime, 10 percent fly ash and 83 percent soil;

3) Mixture composed of 7 percent lime, 25 percent fly ash and 68 percent soil.

The optimum moisture contents for these three mixtures would be plotted on the triangular chart, and the optimum moisture contents for other mixtures interpolated between them, assuming a straight line variation.

The same procedure could be followed for the maximum dry densities.