Comparison of Various Commercial Limes for Soil Stabilization

JERRY WEN-HANN WANG, DONALD T. DAVIDSON, ELMER A. ROSAUER, and MANUEL MATEOS, Respectively, Graduate Assistant, Professor of Civil Engineering, Assistant Professor, and Research Associate, Iowa Engineering Experiment Station, Iowa State University of Science and Technology, Ames

> The effect of lime manufacturing processes and lime properties of various commercial limes on the immersed unconfined compressive strength of soil-lime mixtures were investigated. The limes included eleven dolomitic monohydrate (Type N) and eight calcitic hydrated limes; several amounts of these limes were mixed with a Kansan-age till soil. Test specimens were compacted at standard Proctor density and strengths were obtained after 7 and 28 days of curing.

Strengths of test specimens containing calcitic hydrated limes were lower and less variable than strengths of test specimens containing dolomitic monohydrate limes. The variations that do occur using calcitic limes are believed related to calcination conditions, SiO₂, R_2O_3 and carbonate content.

Strenths of test specimens containing dolomitic monohydrate limes vary significantly. Factors influencing strength are type of kiln, calcination temperature and duration, amount of SiO₂, R_2O_3 , $Mg(OH)_2$ and carbonates, and crystallite size of the MgO. A calcining index is used to appraise lime quality with regard to the time and temperature factors of calcination. Data are given relating strength obtained to crystallite size of MgO.

• COMMERCIAL limes manufactured by different companies or from different limestones may vary considerably in their chemical and physical properties; i.e., even within the same type of lime. These differences are reflected in the performance of lime when it is used in soil stabilization. Thus, a knowledge of the physical and chemical properties of commercial limes may be used to predict their efficiency in the stabilization of soils.

The purpose of this research was to study the effects of composition and method of manufacturing limes on their quality as a soil-stabilizing agent. Several samples of calcitic hydrated (high calcium) and dolomitic monohydrate limes were obtained from lime companies throughout the United States. The characteristics of each individual lime were evaluated on the basis of the immersed unconfined compressive strength of soil-lime specimens, the assumption being that strength production is a positive function of the quality of the lime.

REVIEW OF THE LITERATURE

Chemistry of Lime Manufacture

Thermal Decomposition of Limestone. —Chemically speaking, lime refers only to calcium oxide (CaO); however, in common usage the term includes the calcination products of calcitic and dolomitic limestones. Calcitic (high calcium) limes are produced by calcination of calcareous materials (e.g., calcitic limestone, calcite, oyster shells, and chalk) containing from 95 to 99 percent calcium carbonate (CaCO₃). Dolomitic limes are produced from dolomitic limestone or dolomite which contains from 30 to 40

percent magnesium carbonate (MgCO₃), the rest being calcium carbonate.

At atmospheric pressure, calcitic limestone decomposes at a temperature of 1,650 F to form CaO and CO₂. The decomposition of dolomite, CaMg $(CO_3)_2$, is a two-stage process. At a temperature of 1,350 F dolomite decomposes to form MgO, CO₂ and CaCO₃. It is necessary to raise the temperature to 1,650 F to decompose the CaCO₃ (2, 9). This phenomenon is extremely important as is shown later.

Various investigators have studied the effects of stone size, temperature, and time of calcination of commercial limestones $(\underline{1}, \underline{5}, \underline{8}, \underline{14})$. The maximum temperature to which the limestone is subjected during the calcination process greatly affects the properties of the oxides formed. The retention time (1.e., the duration calcination) is also important. However, many investigators have found that the retention time has much less effect than the temperature on the properties of lime produced $(\underline{1}, \underline{6}, \underline{14})$. "Hard-burning" and "soft-burning" are descriptive terms frequently used to indicate the intensity of the heat obtained during calcination. Hard-burned limes consequently are more dense and less reactive than soft-burned limes because of sintering of the oxides (1, 5).

The mechanisms involved in sintering are complex but usually result in an increased bulk density, a decreased porosity, and growth of particle as well as crystallite size of the oxides. These changes reduce the reactivity of the sintered material. The presence of impurities increases the sinterability of the pure compound; and in general, the higher the temperature, the greater the amount of sintering (5, 7, 12, 13).

As has been mentioned, a temperature of at least 1,650 F must be reached to decompose a calcitic limestone fully. This temperature must also be obtained to calcine a dolomitic limestone, but this temperature is higher than that necessary for the formation of MgO, and at this elevated temperature the MgO previously formed undergoes severe sintering. Therefore, high temperature calcination has a definite negative effect on the reactivity of dolomitic limes, and specifically on the MgO component.

A part of this decrease in the reactivity of MgO has also been attributed to an increase in crystallite size with increasing calcination temperature and retention time * (16). The term "crystallite" is defined as the microscopic or submicroscopic units or blocks that compose a crystal (17). These mosaic fragments are believed to be the basic reacting units of materials. Their size correlates closely with chemical reactivity, inasmuch as it has been shown that hydration of MgO decreased with an increase in crystallite size *.

<u>Hydration of Lime</u>. —When lime reacts with water, the process is called "slaking" or hydration, and is accompanied by an increase in volume. The reaction is exothermic-highly so in the case of CaO, less so with MgO. The somewhat lower free energy of formation of Mg (OH)₂ may account for this slow hydration of MgO. Probably overburning of the MgO during the calcination process has a greater influence.

Commercial Lime Manufacture

In the commercial calcination of limestone two types of kilns are employed—the shaft kiln and the rotary kiln.

A shaft kiln is essentially a vertical metal cylinder lined with refractory brick and with a hopper for stone storage at the top. Many shaft kilns are fired from dutch ovens which open into the shaft near its base. The kiln may be designed to use coal, producer gas, natural gas, or oil as fuel. Generally limestone ranging in size from 5 to 12 in. is charged into the top of the kiln. At periodic intervals of about 8 hr the calcined product is drawn off at the bottom and fresh stone moves down into the hot zone of the kiln.

The rotary kiln is also a metallic cylinder lined with refractory brick, but is nearly horizontal. Rotary kilns are many times the length of shaft kilns, and are set up at an inclination of $\frac{1}{2}$ to $\frac{3}{6}$ in. per ft. The same fuels employed in shaft kilns can also

^{*}Unpublished research data, E.A. Rosauer and R.L. Handy, Engineering Experiment Station, Iowa State Univ., Ames.

be used in rotary kilns. In rotary kiln practice the limestone is crushed to a maximum size of 2 in. and then screened into several size groups, each of which is fed separately into the kiln at a prescribed rate dependent on size. The proper range in each size group for uniform calcination is such that the diameter of the largest piece is about twice that of the smallest. A wide size range does not contribute to uniform calcination. Due to the rotary motion and inclination of the kiln, the stone continuously moves down through a counter flow of heat toward the lower hot end where it is discharged.

After calculation using either type of kiln the calcined product is cooled and may then be further processed.

The calcined product comes from rotary kilns in pebble form; from shaft kilns, in lumps. Before slaking, both types are ground to a fairly uniform size, usually finer than $\frac{1}{2}$ in., and sieved. Dry hydrated lime is then produced when quicklime is reacted with just enough water to satisfy its chemical affinity for moisture under conditions of hydration. The hydrated product is essentially dry. High calcium lime reacts readily with water to produce calcitic hydrated lime, in which all the CaO is converted to Ca(OH)₂. However, dolomitic lime, specifically the less reactive MgO component, does not hydrate readily in ordinary processing equipment. Dolomitic limes, hydrated in equipment operated at atmospheric pressures and low retention times, are known as dolomitic normal hydrated limes (Type N), or dolomitic lime can be hydrated in special equipment using longer retention periods or elevated temperatures and pressure. These pressure-hydrated limes are commercially known as dolomitic dihydrate, or special hydrated limes (Type S), and contain both Ca(OH)₂ and Mg(OH)₂.

Because slaking is accompanied by an increase in volume, lumps of lime change to powder during the process. Any unhydrated lime can be removed from the finished product by screening through a No. 30 sieve or by using an air separation system. After slaking, the product is usually stored in bins for 48 hr or so to allow for evaporation of excess moisture.

Because of the larger stone size and the longer retention time used in the shaft kiln, the quality of the calcined product is less uniform than that of the rotary kiln. In other words, lime has a greater tendency toward overburning in the shaft kiln. Rotary kilns have the advantage of better control of temperature and feed rate, which in turn results in a better quality of the calcined product.

Lime in Soil Stabilization

Lime has been successfully used to stabilize plastic soils. Studies have been conducted at Iowa State University in an effort to determine what type of lime produces highest strength and other beneficial physical changes to soils.

Results of research thus far indicate that in some cases dolomitic quicklime has been found to give higher strengths and in other cases dolomitic monohydrate lime gives higher strengths. In all cases, the dolomitic dihydrated lime gives lower strengths than the dolomitic monohydrate lime and dolomitic quicklime (10, 11).

It has been found that small amounts of calcitic quicklime may be better than calcitic hydrated lime. No general conclusions as to superiority can be made using greater amounts.

At ambient temperatures dolomitic limes (quicklime and monohydrate) were found to give greater strengths than calcitic limes except with some kaolinitic soils. In the latter case calcitic hydrated and dolomitic monohydrate limes gave approximately the same strengths (10, 11, 15).

The role played by the magnesium oxide in dolomitic limes is not exactly known. The hypothesis is that it acts as a catalyst in the soil-lime reaction (11). Research has shown that magnesium in limes must be present in the oxide form to have maximum effectiveness. The optimum molar ratio of calcium to magnesium in dolomitic lime is between 1:1 and 2:1 (11). Most commercial dolomitic monohydrate limes have a calcium to magnesium ratio within these limits.

Materials

A plastic calcareous glacial till of the Kansan stage from the south central part of Iowa was chosen as being representative of many soils of the north central United States, because it is composed of approximately equal portions of sand, silt and clay. Its properties are given in Table 1.

Nineteen samples of limes commonly used in construction were obtained from manufacturers throughout the United States. Eleven were dolomitic monohydrate limes, $Ca(OH)_2$ and MgO; and eight, calcitic hydrated limes, $Ca(OH)_2$. Upon reception they were kept in air-tight containers. The description of the manufacturing processes and physical and chemical properties are given in Tables 2, 3 and 4. These data were supplied by the manufacturers.

Methods

Preparation and Curing of Specimens. - The batches were mixed in a Hobart mixer

SOIL PROPERTIES	
Property	Value
Textural composition ^a (\$ by wt.):	
Gravel (above 2 mm) Sand (2.0-0.074 mm) Silt (0.074-0.005 mm) Clay (below 0.005 mm) Colloidal (below 0.001 mm)	0.0 30.6 31.9 37.5 28.0
Atterberg limits ^b :	
Liquid limit Plastic limit Plasticity index	40 15 25
Chemical properties:	
pH ^C Cation exchange capacity ^d , (me/100 g) Carbonates ^e (\$) Organic matter ⁱ (\$)	8.7 17.5 6.4 0.1
Predom. clay mineral ^g	Montmorillonite
Textural class. ^h	Clay
Engineering class. (AASHO) ⁱ	A-7-6 (13)

TABLE 1

^aASTM designation: D422-54T.

bASTM designation: Dh23-5hT and Dh2h-5hT.

^CGlass electrode method using suspension of 15 g of soil in 30 cc distilled water.

d Ammonium acetate (pH = 7) method on soil fraction below 2 mm (No. 10 sieve).

eversenate method for total calcium.

Potassium bichromate method.

EDetermined by X-ray analysis of the fraction passing No. 200 sieve.

^hFrom triangular chart developed by U.S. Bureau of Public Roads; 0.074 mm used as lower limit of sand fraction.

AASHO designation: M145-49.

model C-100. The lime was first mixed with the dry soil and then the materials were wet mixed for 3 min.

Cylindrical 2- by 2-in. test specimens were prepared at a density near the standard AASHO using the Iowa State compaction apparatus (10, 11, 15, 18).

The specimens were wrapped in waxed paper and sealed with cellophane tape to prevent carbonation of lime from carbon dioxide of the air. They were then placed in a moisture room having a relative humidity between 90 and 100 percent and a temperature of 70 ± 3 F.

	MANUFAC	TURING INF	ORMATIO	N OF LIMES	
		Cal	cining Cond	lition	
Lime	Kiln	Stone	Burning	Burning	Hydration
Designation ^b	Type	Size	Time	Temp.	Temp.
	- 7 8	(in.)	(hr)	(°F)	(°F)
D1	Shaft	-	-	1,800-2,400	_
D2	Rotary	2¼ - 1	3.75	2,200-2,400	250
D3	Shaft	-	8	1,600-1,700	176
D4	Rotary	-	-	2,400-2,500	-
D5	Shaft	-	2.5	3,000	-
D6	Shaft	8 x 4	8-12	2,000-2,100	225
D7	Shaft	-	_	2, 300-2, 600	-
D9	Rotary	1½ by ¾-	1	2, 150-2, 200	260
		∛4 by %			
D10	Shaft	5-10	8	-	-
D11	-	4-6	21/3	2, 250	200
D12	-	-	-	2,300-2,500	212
C1	Shaft	-	-	1,800-2,400	200-225
Č2	Rotary	2-%	2-4	2,400	200
C3	Shaft		-	2,200	•
C4	Rotary	-	-	2,750	-
C5	Rotary	-	-	2, 450	-
C6	-	6	8	2,200	122
C7	Rotary	-	5	2,200	-
C8	Rotary	-	3.5	2, 350-2, 400	215-225

TA	זמ	P.	- 9
- 10			

^aData supplied by lime manufacturers.

^bD = dolomitic monohydrate lime; C = clacitic hydrated.

TABLE 3 PROPERTIES OF DOLOMITIC MONOHYDRATE LIMES⁸

**		C	hemical_Co	nstituents (\$ b	y wt)	Loss on	Co Ma	Sieve	Settling	
Designation	CaO	Ca(OH)	MgO	Mg(OH) _a b	SiO	R ₂ O ₃ C	Ignition	Ratiod	(\$ Passing No 325)	Rate See (min) ^e
 D1	49 60	65 52 ^f	32 00	15	0 34	0 17	16 97	1 83	91.00	43 5
D2	47 13	61 86	34.49	5	2 20	1 00	16.08	1 62	65,80	37.5
20	48 30	63 801	33 20	TR	0.60	1.10	16,80	1 73	99.24	32 0
54	47 99	64 00	89 11	5	1 10	0 75	16.10	1 43	92 928	15 5
75	47 80	82 86	32 00	5	0.81	0 56	19 03	1 76	96 80	13 5
55	46 97	A1 09	99 08	5	0 24	0 30	16 50	1.64	98 848	27.0
10	40 01	49 00	99 00	, s	0 15	0 99	17 50	1 68	98 918	49.0
Di Di	41 90	03 40 ⁻	33 80		0.40	0 49	20.89	1 47	-	98.0
D9	40 30	39 92	30 40	20	0.40	0 00	10 50	1 84	_	35.0
D10	46 50	61 43	33 60	10	0.15	0.33	10 50	1 04		
D11	48.19	63, 66 ¹	33.82	TR	0 37	1 00	16.82	1.69	78,00	27 0
D12	47 00	62 09 ^f	33 00	-	1.00	0.70	17.00	1 69	96 208	37 0

^aData supplied by lime manufacturers.

^bEstimated from I-ray diffraction analysis.

R203 - 1203 + Po203.

^dCalculated as ratio of calcium to magnessium by weight from amount of materials present as their oxides.

ASTM designation: Cllo-49.

Calculated by molecular weight ratios from amount of CaO present.

SANTM designation: Cllo-49.

PROPERTIES OF CALCITIC HYDRATED LIME*										
Lime Designation		Chemi	cal Constit	Loss on	Sieve	Sottling				
	CaO	Ca(OH) ₂	MgO	SiO ₂	R₂O₃ ^b	CaCO ₃	Ignition	(\$ passing No. 325)	Rate S ₅₀ (min) ^C	
C1	72.00-	95.11- ^d	0,40-	1.00	0.10-	-	23.50-	95.00-	31.0	
	75.00	99.08 d	1.40	1.60	0.40		24.50	96.00		
C2	73.00	95.58	0.70	1.10	0.90	1.14	24.25	96, 50	144.0	
C3	74.18	93.74	0.09	0.45	0.37	3.62	-	99.25	103.5	
C4	73, 10	96, 56 ^d	1.20	0.90	0.45	-	24.10	-	132.0	
C5	73.00-	96.43d	0.34	1.30	0.66	-	23.00	95.00	41 0	
	75.00						25.00			
C6	73.15	96, 63 ^d	0.48	0.60	0.22	-	24 53	-	102 0	
C7	73.50	94.30	0.69	1.05	0.18	-	24 00	98 70	165 0	
C8	73.94 ⁶	97.68	0.64	0,69	0.59	0.10	24.05	97.00	108.0	

TABLE 4 PROPERTIES OF CALCITIC HYDRATED LIME[®]

^aData supplied by lime manufacturers.

^bR₂03 = A1203 + Fe203.

CASTM designation: C110-49.

^CCalculated by molecular weight ratios from amount of CaO present.

^eCalculated by molecular weight ratios from amount of Ca(OH)₂ present.

Determination of Optimum Molding Moisture. -Moisture-density determinations were made on mixtures containing 2, 6, 8, and 12 percent of each lime used; five different moisture contents were used and three specimens molded for every point. The specimens were cured for 7 days, immersed, and tested for strength. Thus, moisture-density and moisture-strength curves were obtained for most combinations of soil and lime to be investigated. From these curves the optimum moisture for maximum dry density and the optimum molding moisture for maximum 7-day strength were derived. If the optimum moisture contents for maximum dry density and maximum moisture content did not coincide, a compromise moisture content was used (<u>18</u>). The molding moisture for mixtures containing 4 and 10 percent lime was determined as a straight line interpolation between their respective neighbors.

Once the optimum molding moisture was determined, specimens were prepared at this moisture content and cured for 7 and 28 days.

<u>Strength Testing</u>. —After the specified curing period, the specimens were immersed in distilled water for 24 ± 1 hr and then tested to failure under unconfined compression at a loading rate of 0.1 in. per min. Three specimens were used in each determination and the average strengths are reported. If one sample deviated from the average by more than 10 percent, the other two samples supplied the average.

<u>X-ray Measurement of Crystallite Size.</u>—The average crystallite sizes of the limes were found by a method originally suggested by Scherrer and applied by Rosauer and Handy (<u>16</u>). This method is based on the relation between the breadth of an X-ray diffraction maximum and the size of the diffracting crystallites, more specifically, the mean dimension normal to the diffracting planes of the crystallites.

Individual samples of dolomitic lime were mounted in disc-shaped sample holders so that they could be rotated during analysis to avoid effects of preferred orientation and to increase statistical accuracy. A small press and a pressure of 400 psi were used in mounting. A G.E. XRD-5 X-ray diffraction unit with a copper tube was operated at 50 kvp and 16 ma. The radiation was nickel filtered. Proper intensity and resolution of the diffraction peaks were obtained by using an optical system consisting of a 1° beam slit, medium resolution Soller slit, and 0.2° detector slit. A time constant of 8 sec, a slow scanning rate of $0.2^{\circ} 2\theta$ per min, and a chart speed of 1 in. per min were used to reduce pen jiggle and give good reproducibility.

The peak breadth, B_0 , is the width of the peak (in degrees 20) at half maximum intensity and is an inverse measure of crystallite size. The Scherrer equation was used in the calculation of the mean crystallite dimensions from the B_0 values. The reflection of fine quartz powder (20-40 μ) was recorded in a similar fashion to furnish the instrument broadening. The breadth of the MgO (200) line, and (100) and (001) lines for Ca(OH)₂ were measured in five repeat tracings. Because the unit cell of MgO is cubic, the dimension calculated from the (200) reflection represents the total thickness of the diffracting planes. The unit cell of $Ca(OH)_2$ is hexagonal, therefore, the broadening of the (001) line gives the thickness of the average $Ca(OH)_2$ crystallite prism, and the broadening of the (100) line gives the average distance between two opposite faces of the prism. Crystallite size calculations for components of several dolomitic limes are given in Table 5.

RESULTS

Moisture-Density Studies

<u>Maximum Density.</u>—The decreases in maximum dry density with an addition of 2 percent dolomitic lime were 6.2 to 7.7 percent, depending on the brand of lime whereas addition of 2 percent various commercial calcitic lime gave decreases between 7.4 and 9.0 percent. Further decreases were about 1 percent for every additional 2 percent of lime, either calcitic or dolomitic, up to 12 percent (Fig. 1).

The differences in maximum dry densities due to the same type of commercial limes, calcitic or dolomitic, were 2 to 3 pcf at any specific percentage of lime content. These differences are believed to be caused by variations in the gradation of the various brands of commercial lime. No correlation was found between strength and the variation in density by using different limes.

The lower density obtained from mixtures made with calcitic limes may be partly explained by the fact that calcitic limes are usually finer than dolomitic limes, as shown by the settling rate data in Tables 3 and 4. The finer particles require a higher moisture content for optimum compaction. The increased amount of water takes the place of part of the soil particles in the same unit volume, and a lower maximum dry density for soil-calcitic lime mixtures results. In addition, calcitic limes have a somewhat lower specific gravity than dolomitic limes. At the same lime content, calcitic lime would be larger and replace more soil particles in a unit volume of soil-lime mixture, which, perhaps, also contributes to the reduction of the density.



Figure 1. Variation of maximum dry density of soil stabilized with calcitic hydrated and dolomitic monohydrate commercial limes.



Figure 2. Variation of OMC for maximum dry density of soil stabilized with calcitic hydrated and dolomitic monohydrate commercial limes.

Optimum Moisture Content. —The increase in optimum moisture content (OMC) for maximum dry density as the amount of lime is increased is shown in Figure 2. Initial increases are large but tend to level off. From 0 to 2 percent lime the OMC increases from 2 to 4 percent. Between 2 and 12 percent lime content the increase in OMC is about 1 percent for every 4 percent increase in lime.

Within the same type of lime different brands gave OMC differences of as much as 2 percent at any specific lime content. This difference is important in stabilization of soils with lime. It indicates that a separate moisture-density relationship should be made when using limes of different brands, although of the same type.

In general, calcitic limes showed higher OMC for maximum dry density than dolomitic limes, which is related to the differences in maximum densities.

<u>OMC of Maximum Dry Density and Moisture Content for Maximum Strength.</u>—With all the commercial limes used, the moisture content for maximum strength was at or very close to the OMC for maximum dry density at the standard AASHO compaction. In general, the deviation was of the order of less than 0.5 percent. Only a few exceptions occurred; nevertheless, the deviation was never greater than 1 percent.



Figure 3. Immersed unconfined compressive strengths of various mixtures of soil and dolomitic monohydrate commercial limes at standard Proctor (AASHO) density.

Strength Studies

Dolomitic Monohydrate Limes. — The 7- and 28-day curing strength increases up to an addition of 4 percent dolomitic lime and then it gradually levels off (Fig. 3). The amount of strength increase between 7 and 28 days varies with the amount and brand of lime. In general, the strength at 28 days 15 between 50 and 100 percent higher than at 7 days.

At any specific curing period the strengths vary considerably with the brand of lime. At a certain lime content, a lime may give twice the strength of another. This indicates that some dolomitic limes are superior to others. Consequently, the properties of the



Figure 4. Effect of SiO₂ + R₂O₃ content on immersed unconfined compressive strengths of soil stabilized with 6 percent commercial dolomitic monohydrate limes, cured 28 days.

strength of the soil and lime mixture are discussed. In this discussion the 28-day strengths are used exclusively inasmuch as longer curing strengths give more reliable results (4).

Effect of $SiO_2 + R_2O_3$ Content. -Figure 4 shows an increase in strength with an increase in the amount of SiO_2 and R_2O_3 (Fe₂O₃ + Al₂O₃) in dolomitic limes. The trend fit by means of the least squares method, is

$$y = 365 + 22x$$
 (1)

in which

 $x = percent SiO_2 + R_2O_3$; and

y = 28-day immersed unconfined compressive strength, psi, with 6 percent dolomitic lime content.

In obtaining Eq. 1 corrections have been applied to the strength of limes D2 and D3 because of their respectively fine and coarse MgO crystallite sizes. As discussed later, the finer the MgO crystallite size in lime, the higher its stabilized strength. Therefore, in accordance with Figure 5, a deduction of approximately 30 psi was made from the strength obtained with lime D2 and an increase of 70 psi was made to strength obtained with lime D3. The other limes do not differ appreciably from an average MgO crystallite size of about 2,500 Å. In accordance with Figures 5 and 6 an increase of approximately 130 psi was also made to the strength obtained with lime D5 because of its relatively coarse MgO crystallite size and overcalcined condition. Lime D12 is not included here because it was found to be recarbonated.

Silicon dioxide and sesquioxides are the main components of natural cements, but they are generally considered as impurities in lime. When they react with water, silicate and aluminate gels are formed. These gels possess cementing properties and may contribute to part of the strength of lime-stabilized soil. It was found in this investigation that the amount of strength increase depends on the amount of these impurities present. Further study is needed to determine the individual effects of SiO₂, Al_3O_3 , and Fe_3O_3 on the strengths of lime-stabilized soil.

Effect of Crystallite Size. —The sizes of MgO and Ca(OH)₂ crystallites in the dolomitic limes are given in Table 5. The size of Ca(OH)₂ crystallites in dolomitic limes ranges from 280 to 718 Å. The MgO crystallite size is much larger and varies much more from one lime to another, ranging from 2, 100 to 10,000 Å. However, most of the MgO crystallites fall in the size range of 2, 100 to 3,080 Å.

An attempt was made to correct the stabilized strengths of some limes to find the real relationship between the MgO crystallite size in dolomitic limes and the unconfined compressive strength of soil-lime mixtures. The strength data in Figure 4 was corrected assuming an average $SiO_2 + R_2O_3$ content of 1 percent for each lime. Corrections

....

	MgO, (200)			Ca(OH) ₃ , (001)			Ca(OH) ₂ , (100)		
Lime Designation	B ₀ (degree 29) ²	σb	D (Å)	B _o (degree 2θ) ^a	σb	D (Å)	B _o (degree 20)a	σ ^b	D (Å)
D 1	0.2602	0.0041	2.340	0.3492	0.0105	526			
D2	0.2630	0.0018	2, 100	0.4760	0.0053	280	0 3451	0 0084	- 647
D3	0.2398	0.0108	10,000	0.4484	0.0094	311	0 3272	0.0004	890
D4	0.2538	0.0047	3,090	0.3758	0 0118	445	0.3212	0.0001	740
D5	0.2478	0.0024	4,410	0.3172	0 0002	890	0.0010	0.0103	100
D6	0.2548	0.0036	2,920	0.3138	0 0084	701	0.2022	0.0080	909
D7	0.2538	0.0020	3,080	0.3634	0 0115	490	-	-	•
D9	0.2600	0.0050	2,340	0.3668	0.0042	470	0 3054	0 0197	-
D10	0.2608	0.0016	2,340	0.3356	0 0034	582	0.0004	0.0137	(16
D11	0.2580	0.0020	2,530	0.3110	0.0032	718	-	-	-

TABLE 5

Average of 5 runs.

^bStandard deviations.



Figure 5. Effect of crystallite size of MgO on immersed unconfined compressive strengths of soil stabilized with 6 percent commercial dolomitic monohydrate limes, cured 28 days.

thus made were approximately the deductions in strength of 30, 10, and 20 psi for the limes D2, D3, and D4, respectively, and an increase in strength of 20 psi for both limes D7 and D10. In accordance with Figure 6, an increase of approximately 100 psi was made to the strength of lime D5 because of its overcalcined condition. Limes D1, D6, D9, and D11 did not need correction. Using the least squares method a straight line was plotted through the corrected points (Fig. 5). The finer the MgO crystallite size, the higher the stabilized strength. This relationship for 6 percent of dolomitic monohydrate lime can be expressed as

$$\log y = 3.17 - 0.17 \log x$$

(2)

in which

x = MgO crystallite size in A; and

y = 28-day immersed unconfined compressive strength, in psi.

No correlation, however, was found between the size of $Ca(OH)_2$ crystallites and the strengths. This may be explained by the fact that the range of the $Ca(OH)_2$ crystallite sizes was too small to show significant influence, and that the presence of a large

amount of MgO in the dolomitic lime might overshadow the influence on strength due to the variation of $Ca(OH)_2$ crystallite size. Thus, it is suggested that this is an indirect indication that the reactivity of MgO has a definite influence on the strength obtained with soil stabilized with dolomitic lime.

It appears that additional grinding during the manufacturing process of dolomitic limes would be beneficial in soil stabilization with lime. Grinding would reduce the size of the lime particles and increase the surface area (i.e., expose more crystallite surfaces for reaction) but would add appreciably to cost.

Effect of Kiln Type. —Figure 7 shows that the strengths obtained with rotary kiln limes not only are higher but also have less variation than strengths obtained with shaft kiln limes. The nature of the operation of the type of kiln explains this result. Table 6 gives some of the pertinent data supplied by the lime manufacturers.

TABLE 6

(in	.)	(hr)	(°F)	
Range	Avg.	Range	Avg.	Range	Avg.
$\frac{4-10}{3}$	6 ³ / ₄	$2^{1/2}-12$ 1-3 ³ /.	7.1	1,600-3,000 2,150-2,500	2,250 2,380
	(10) Range 4-10 $3/8-2^{1}/4$	$\begin{array}{c c} (11.) \\ \hline Range & Avg. \\ \hline 4-10 & 6\frac{3}{4} \\ \frac{3}{6}-2\frac{1}{4} & 1\frac{1}{4} \\ \end{array}$	(11.) (hr Range Avg. Range $4-10$ $6\frac{3}{4}$ $2\frac{1}{2}-12$ $\frac{3}{8}-2\frac{1}{4}$ $1\frac{1}{4}$ $1-3\frac{3}{4}$	(in.) (nr) Range Avg. Range Avg. 4-10 $6^{3}/_{4}$ $2^{1}/_{2}$ -12 7.1 $^{3}/_{8}$ - $2^{1}/_{4}$ $1^{1}/_{4}$ 1 - $3^{3}/_{4}$ 2.4	(in.) (inr) (1r) Range Avg. Range Avg. Range $4-10$ $6^{3}/_{4}$ $2^{1}/_{2}-12$ 7.1 1,600-3,000 $^{3}/_{8}-2^{1}/_{4}$ $1^{1}/_{4}$ $1-3^{3}/_{4}$ 2.4 2,150-2,500

KILN PROPERTIES

Table 6 suggests that uniformity and quality of limes produced are a function of stone size, and calcination time and temperature, and that these are interdependent. It is seen that shaft kilns allow for greater variation in calcining conditions than rotary kilns, and it is expected that the properties of limes produced from different shaft kilns will also show significant differences. The higher quality of rotary kiln limes may be due to smaller stone size, shorter calcination time, and better control of temperature and feed rate.

Effect of Firing Conditions. -Because time and temperature of firing affect the





Figure 6. Effect of calcining condition on immersed unconfined compressive strengths of soil stabilized with 6 percent commercial dolomitic monohydrate limes, cured 28 days.

Figure 7. Effect of kiln type on immersed unconfined compressive strengths of soil stabilized with 6 and 8 percent various commercial dolomitic monohydrate limes. properties of the limes, a calcining index was used in this investigation as a measure of the relative amount of heat used in the calcining process. This calcining index is defined as the square root of the calcining time in hours multiplied by the difference of the actual calcining temperature and the theoretical decomposition temperature of 1,650 F. The square root of the calcining time was used because the effect of temperature is greater than that of time (1, 6, 14).

Corrections were made to strengths of soil-dolomitic lime mixtures to reduce the effects of $SiO_2 + R_2O_3$ content and MgO crystallite size, in accord with earlier equations. Lime D2 has the highest $SiO_2 + Fe_2O_3$ content and the finest MgO crystallite size of all the limes, and according to Figures 4 and 5, a correction of approximately 70 psi should be deducted from the strength of this soil-lime mixture. Limes D3 and D5 have $SiO_2 + Fe_2O_3$ contents close to the average of all the limes but have the coarsest MgO crystallite sizes. According to Figures 4 and 5, approximately 60 and 30 psi of strength, respectively, should be added for these limes. Limes D6, D9, and D11 need no correction.

The corrected strengths show a significant relationship to the calcining index (Fig. 6) and indicate an optimum condition of calcination; i.e., at a calcining index of 0 to 1,000 with the units $\sqrt{\text{hour}}$ (°F). In other words, a well-decomposed, non-sintered lime has better soil stabilizing qualities than hard-burned lime. Sintering lowers the quality of the lime by reducing its reactivity (5, 7, 12, 13).

As mentioned earlier, individual crystallites increase in size with increased calcining temperature and retention time (16). However, in the present investigation no relationship was found between the calcining index and the crystallite size of MgO in the lime. This may be due to the calcining index, as it was calculated, being only a rough measure of the relative amount of heat used during calcination. Perhaps a different method of calculating the calcining index might reveal a correlation. Furthermore, possible inaccuracies in the data supplied by the manufacturers and sampling errors contributed to this lack of correlation. It is believed that a correlation between calcining index and MgO crystallite size would be found if more accurate data on the conditions of calcination could be obtained.

Effect of Carbonate Content of Lime. -X-ray diffraction analysis showed that lime D12 contained a large amount of CaCO₃ and probably some MgCO₃. This may explain why this lime gave very low stabilized strength. As carbonates do not react with the soil to produce strength (4), the presence of a large amount of carbonate reduces the available lime by an equivalent amount.

Effect of $Mg(OH)_2$.—It was found that many of the dolomitic monohydrate limes used in this investigation were not true monohydrates. It was estimated from X-ray diffraction analysis that in some limes between 40 and 50 percent of the MgO had hydrated (Table 3). Because dolomitic dihydrate lime generally gives lower stabilized strength than dolomitic monohydrate lime (10, 11), precautions should be taken in the hydration and storage of lime to prevent the MgO from hydrating.

In the present study, however, the effect of $Mg(OH)_2$ on the strength properties of soil-lime mixtures was overshadowed by the variables previously discussed. In addition, the amount of $Mg(OH)_2$ in lime could not be accurately determined. Hence, no relationship was found between strength obtained and the amount of $Mg(OH)_2$ present in the limes. In finding the relationship shown in Figure 7, no correction was made on limes D9 and D1, which contained the most $Mg(OH)_2$ among all the limes. It was assumed that the very small MgO crystallite size was able to counteract the strength decrease due to the large $Mg(OH)_2$ content. This assumption was also used in deriving the relationship given in Figure 6. In finding the relationship shown in Figure 5, a correction was not applied to the lime D9 because it was assumed that the optimum calcining index of lime D9 was able to counteract the decrease of strength that would normally result from the presence of a large amount of $Mg(OH)_2$ in the lime.

Strengths with Calcitic Hydrated Limes. —Strength curves for different lime contents obtained with the eight commercial calcitic hydrated limes show that the amount of calciti lime that gave the highest 7- and 28-day strengths was between 2 and 4 percent (Fig. 8). For amounts of lime from 4 to 12 percent the strength with any brand of lime is within 10 percent of the average value. This is within the range of operational deviation



Figure 8. Immersed unconfined compressive strengths of various mixtures of soil and calcitic hydrated commercial limes at standard Proctor (AASHO) density.

(3). Consequently, it may be assumed that all the calcitic limes gave practically the same strength when used in amounts greater than 2 percent.

A further comparison of the qualities of the limes was made by studying the strengths obtained using 2 percent. On this basis lime C3, which contained a relatively large amount of CaCO₃, and lime C4, which was calcined at an extremely high temperature, gave rather low strengths. These factors also influenced the strength of mixtures made with the dolomitic limes.

As already reported (<u>10</u>, <u>11</u>), the calcitic hydrated limes were also found to give lower stabilized strengths than the dolomitic monohydrated limes. The increase between 7 and 28 days with the calcitic limes was about 80 percent based on 7-day strengths.

SUMMARY AND CONCLUSIONS

Results of research on lime stabilization of soils conducted at Iowa State University indicate that certain factors in the manufacture of lime influence the strength obtained in soil-lime test specimens. Other properties of limes also influence the strength obtained. Detailed studies using several commercial dolomitic monohydrate and calcitic hydrated limes suggest the following conclusions:

1. Strengths of soil-lime test specimens using various commercial dolomitic monohydrate limes show significant variations.

a. Some factors influencing the strength obtained with dolomitic limes are type of kiln used in calcination; firing conditions of the kiln; amount of silicon dioxide and sesquioxides in the lime; crystallite size of MgO, and carbonate and $Mg(OH)_2$ content of the lime.

b. Limes from rotary kilns produce higher strengths than limes from shaft kilns. The data also indicate that rotary kiln limes are more uniform in strength production than shaft kiln limes. c. An optimum calcining condition for the production of lime for soil stabilization is indicated by a calcining index of 0 to 1,000 with the units $\sqrt{\text{hours}}$ (⁰F). The calcining index is defined as the square root of the calcining time, in hours, multiplied by the difference of the actual calcining temperature and the theoretical decomposition temperature of 1,650 F. This optimum calcining condition indicates that a welldecomposed lime is better for use in soil stabilization than hard-burned lime.

d. Strengths increase with an increase in silicon dioxide and sesquioxide content of the lime.

e. The crystallite size of magnesium oxide influences the strength characteristics of soil-lime mixtures; generally, the finer the MgO crystallite size, the higher the strength. Calcium hydroxide crystallites are much smaller than MgO crystallites and do not show a significant effect on the strength obtained.

f. Limes containing appreciable amounts of carbonate or $Mg(OH)_2$ produce lower strengths. Proper processing and storing is advisable.

2. Calcitic hydrated limes produce lower strengths than dolomitic monohydrate limes, and do not show large variations between one another. The variables control-ling the strength properties of calcitic limes are believed to be the SiO_2 and Al_2O_3 content, the calcining conditions, and the carbonate content.

3. Soils treated with dolomitic monohydrate limes show slightly higher standard AASHO density and lower optimum moisture content than when treated with calcitic hydrated limes.

4. Inasmuch as the optimum moisture content among various commercial limes investigated differed by as much as 2 percent from each other at any specific lime content and within the same type of lime, it seems advisable to determine the optimum moisture content of available limes before construction is begun.

5. At present, the most reliable tests for evaluating a dolomitic lime for use in soil stabilization are determinations of crystallite size of MgO and the amount of SiO₂ and R_2O_3 in the lime.

6. There are some obvious limitations in the present study; e.g., use of strength as a criterion of lime quality and use of only one soil. Nevertheless, the correlations between lime properties and strengths obtained are believed to be real, as studies have shown that the relative position of any lime on a quality vs strength scale remains the same when several soils are used (15). However, at the present time no conclusion can be drawn as to how lime quality would affect another soil property, such as plasticity. Further studies on the quality of lime as a soil stabilizing agent are in progress at the Engineering Experiment Station of Iowa State University.

ACKNOWLEDGMENTS

This investigation is part of the research being carried out under Projects HR-82 and HR-48 of the Iowa Highway Research Board (Projects 449-S and 340-S of the Iowa Engineering Experiment Station) with funds supplied by the Iowa State Highway Commission.

The authors extend their appreciation to R.L. Handy, Associate Professor of Civil Engineering, for his advice and assistance.

The following lime manufacturers supplied samples and data: Ash Grove Lime and Portland Cement Co., Kansas City, Mo.; Basic Incorporated, Cleveland, Ohio; Batesville White Lime Co., Batesville, Ark.; G & W. H. Corson Inc., Plymouth Meeting, Pa.; Gibsonburg Lime Productions Co., Gibsonburg, Ohio; Kaiser Aluminum and Chemical Sales Inc., Oakland, Calif.; Linwood Stone Products Co. Inc., Davenport, Iowa; Marblehead Lime Co., Chicago, Ill.; Moorse Lime Co., Springfield, Ohio; National Gypsum Co., Buffalo, N. Y.; National Lime and Stone Co., Carey, Ohio; Ohio Lime Co., Woodville, Ohio; Rockwell Lime Co., Manitowoc, Wisc.; St. Clair Lime Co., Oklahoma City, Okla.; U.S. Gypsum Co., Chicago, Ill.; Warner Co., Philadelphia, Pa.; and Western Lime and Cement Co., Milwaukee, Wis.

REFERENCES

- 1. Azbe, V.J., "Fundamental Mechanics of Calcination and Hydration of Lime and Methods of Control." In "Symposium on Lime." Pp. 88-102, ASTM (1939).
- Conley, J.E., "Calcination Conditions for Limestone, Dolomite and Magnesite." Trans. Amer. Inst. Mining Met. Engrs., 148:330-346 (1942).
- 3. David, H.T., Davidson, D.T., and O'Flaherty, C.A., "Pinpointing Suspect Triplicate Unconfined Compressive Strength Values in a Series of Soil-Additive Strength Determinations." Materials Research and Standards, 1: No. 12 (Dec. 1961).
- Davidson, D.T., and Handy, R.L., "Lime and Lime-Pozzolan Stabilization." In K. B. Woods (ed.), "Highway Engineering Handbook." Pp. 21-98 to 21-108, McGraw-Hill (1960).
- Fischer, H.C., "Calcination of Calcite: I. Effect of Heating Rate and Temperature on Bulk Density of Calcium Oxide." Jour. Amer. Ceram. Soc., 38:245-251 (1955).
- 6. Fischer, H.C., "Calcination of Calcite: II. Size and Growth Rate of Calcium Oxide Crystallites." Jour. Amer. Ceram. Soc., 38:284-288 (1955).
- Glasson, D.R., "Reactivity of Lime and Related Oxides: VII. Crystal Size Variation in Calcium Oxide Produced from Limestone." Jour. Applied Chem., 11:201-206 (1961).
- 8. Haslam, R.T., and Hermann, E.C., "Effect of Time and Temperature of Burning on the Properties of the Lime." Industrial Engineering Chem., 18:960-963 (1926).
- Haul, R.A.W., and Markus, J., "On the Thermal Decomposition of Dolomite: IX. Thermogravimetric Investigation of the Dolomite Decomposition." Jour. Applied Chem., 2:298-306 (1952).
- 10. Laguros, J.G., Davidson, D.T., Handy, R.L., and Chu, T.Y., "Evaluation of Lime for Stabilization of Loess." Proc., ASTM, 56:1301-1319 (1956).
- 11. Lu, L.W., Davidson, D.T., Handy, R.L., and Laguros, J.G., "The Calcium-Magnesium Ratio in Soil-Lime Stabilization." HRB Proc., 36:794-805 (1957).
- 12. Nelson, J.W., and Cutler, I.B., "Effect of Oxide Addition on Sintering Magnesia." Jour. Amer. Ceram. Soc., 41:406-409 (1958).
- 13. Quirk, J.F., "Factors Affecting Sinterability of Oxide Powders: BeO and MgO." Jour. Amer. Ceram. Soc., 42:178-181 (1959).
- 14. Ray, K.W., and Mathers, F.C., "Effect of Temperature and Time of Burning on Properties of High Calcium Lime." Industrial Engineering Chem., 20:415-419 (1928).
- 15. Remus, M.D., and Davidson, D.T., "Relation of Strength to Composition and Density of Lime-Treated Clayey Soils." HRB Bull. 304, 65-75 (1961).
- Rosauer, E.A., and Handy, R.L., "Crystallite-Size Determination of MgO by X-Ray Diffraction Line Broadening." Iowa Academy of Science, Proc., Vol. 68 (1961).
- 17. Sproull, W.T., "X-Ray in Practice." McGraw-Hill (1946).
- 18. Vincent, R.D., Mateos, M., and Davidson, D.T., "Variation in Pozzolanic Behavior of Fly Ashes." To be published in ASTM Proc., (ca. 1961).