

- Alternate Methods of Stabilizing Degrading Stream Channels in Western Iowa. Engineering Research Inst., Iowa State Univ., Ames, Rept. 81047, 1980.
3. C.R. Wilder. Soil-Cement for Water Resource Structures. Trans., American Society of Agricultural Engineers, St. Joseph, MI, Vol. 20, 1977, pp. 109-112.
  4. W.G. Holtz and K.D. Hansen. The Use of Compacted Soil Cement in Water Control Structures. 12th Congress on Large Dams, Mexico, Q44-R13, 1976, pp. 251-278.
  5. Soil-Cement for Water Control: Laboratory Tests. Portland Cement Assoc., Skokie, IL, 15166.02W, 1976.
  6. P.J. Nussbaum and B.E. Colley. Dam Construction and Facing with Soil-Cement. Portland Cement Assoc., Skokie, IL, RD010.01W, 1971.
  7. Suggested Specifications for Soil-Cement Slope Protection for Embankments. Portland Cement Assoc., Skokie, IL, 15052.03W, 1976.
  8. L.L. Litton. Soil-Cement for Use in Channel Grade Stabilization Structures, Iowa State Univ., Ames, M.S. Thesis, in preparation.
  9. G.F. Sowers. The Safety Factor in Excavations and Foundations. HRB, Highway Research Record 269, 1969, pp. 23-34.

#### Abridgment

## Reaction Products of Lime-Treated Southeastern Soils

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Six soils series (Cecil, Chewacla, Eutaw, Sumter, Tatum, and Wilcox) of the southeastern United States were investigated by using x-ray diffraction analysis, thermogravimetric analysis, and scanning electron microscope. The study compared the natural soil with lime-treated soil (by using 6 percent high-calcium-hydrated lime) after a 48-h accelerated curing period at 49°C (120°F). The compaction specimens were prepared in a Harvard miniature compaction mold by using impact compaction and were sealed in plastic wrap during the curing phase to prevent moisture loss. Lime-soil reaction products of calcium oxide-alumina oxide-hydrate ( $C_3AH_3$ ),  $C_3AH_6$ , calcium-silicate-hydrate (CSH) (gel) and CSH II were identified, although a different mixture of products was associated with each soil. Unknown products were also noted on the thermogravimetric analysis data at 440°, 450°, and 460°C. Both absorbed-solution and through-solution mechanisms appear to be involved in the formation of cementitious material. When compared with lime reactivity (i.e., unconfined compressive strength gain following the accelerated curing), the results for the montmorillonite dominated soils (Eutaw and Wilcox) suggest that excessive specific surface is a detriment to the development of significant cured unconfined compressive strength gains.

Six fine-grained soil series characteristic of those found in the southeastern United States were investigated by using x-ray diffraction, thermogravimetric analysis (TGA), and the scanning electron microscope (SEM) to determine the nature of lime-soil reaction products. Selected soil morphology, engineering physical property, and lime-reactivity data are presented in Table 1 (1-3). The soils exhibit a wide range of lime reactivity with the lowest strength gains noted for the montmorillonitic soils (Eutaw and Wilcox).

#### PREPARATION OF SOIL-LIME SPECIMENS

Specimens were prepared for SEM, x-ray diffraction, and TGA after an accelerated curing sequence (4). The soils were air dried, then dry mixed by hand with 0, 2, 4, and 6 percent high-calcium-hydrated lime by dry weight of the soil. Distilled water was added to each soil to achieve moisture contents approximately that of optimum for the lime-treated soil. Samples were compacted in a Harvard miniature mold with a 0.53-lb impact compaction hammer in 3 layers by using 25 blows/layer. Immediately after removal from the mold, the compacted specimens were sealed with plastic wrap to prevent moisture losses and then cured at 49°C (120°F) for 48 h.

#### SOIL TESTING PROCEDURES

##### X-Ray Diffraction

X-ray patterns for soils before and after treatment with 6 percent high-calcium-chemical lime were obtained by using a Norelco x-ray diffraction unit with a copper tube. For a detailed discussion of x-ray diffraction theory, see Jackson (5).

##### TGA

A Dupont 951 thermogravimetric analyzer and a Dupont 990 thermal analyzer and record console were used for TGA. About 10 mg of the entire soil sample were used. The sample was heated from 25° to 800°C at a constant rate of 20°C/min. Weight loss was a result of the release of surface water and structural hydroxyls. Minerals loose these hydroxyls within specific ranges of temperatures and at constant percentages of weight. Therefore, some minerals (gibbsite and kaolinite) may be identified quantitatively (5).

##### SEM

An AMR-100 SEM was used on the soils at various magnifications.

#### DISCUSSION OF RESULTS

##### Cecil

A sharp reduction in kaolinite and illite x-ray diffraction peaks indicated some degradation of those minerals. This was supported by TGA results where kaolinite peaks at 500°C were reduced in size. The new peak (reaction products of lime treatment) was identified by Glenn (6) at 140°C to be calcium-alumina oxide-hydrate ( $C_4AH_{13}$ ), calcium-silicate-hydrate (CSH) (gel), and CSH I. Data by Ruff and Ho (7) preclude the existence of the latter. The peak at 230°C indicates that  $C_4AH_{13}$  makes up at least part of the 140°C weight loss. The new mineral found at 320°C was identified as  $C_3AH_6$  (6). The 670°C peak could

Table 1. Soil properties.

Soil Series	Family	Natural pH	Liquid Limit (%)	Plastic Limit (%)	Percentage Passing No. 200	Unconfined Compressive Strength (lb-f/in <sup>2</sup> )		Lime Reactivity (lb-f/in <sup>2</sup> )
						0 Percent Lime	6 Percent Lime	
Cecil	Clayey, kaolinitic	4.9	52	33	85	20	184	164
Chewacla	Fine-loamy, mixed, thermic	8.2	24	17	43	48	201	153
Eutaw	Very fine, montmorillonitic, thermic	5.4	70	37	99	57	60	3
Sumter	Fine-silty, carbonic, thermic	8.4	51	24	88	59	90	31
Tatum	Clayey, mixed, thermic	4.3	33	24	83	36	77	41
Wilcox	Fine, montmorillonitic	4.3	72	42	90	12	14	2

Note: 1 lb-f/in<sup>2</sup> = 6.894 kPa.

correspond to the 700°C mineral described by Glenn as CSH II.

SEM photographs of the natural soil indicated a matrix of angular plates that grade from silt to clay particles of about 0.1  $\mu\text{m}$  in diameter. The plates were in a card house or face-to-edge arrangement. The addition of 2 percent lime rearranged the particles into stacks. When Cecil was treated with 6 percent lime, the larger particles remained stacked, but the smaller clay plates were attached to these with their edges. Cementing products were not visible in the photographs. The edge-to-face structure of the soil supports the adsorbed state mechanism of stabilization described by Diamond and Kinter (8).

#### Chewacla

The x-ray diffraction pattern exhibited a large reduction of the 4.2 Å quartz peak and almost total destruction of the kaolinite peak. The reduction of the kaolinite was not so evident in TGA data, which indicated a breakdown in the crystal structure as well as a breakdown of the mineral itself. New minerals included CSH (gel) at 160°C and CSH II at 670°C. Another product was found at 440°C but was not identified in the literature.

The SEM photograph of natural Chewacla showed that clay platelets covered larger particles in a face-to-face arrangement. The addition of 2 percent lime produced an amorphous material that coated and bridged all particles. A cementing agent, which was apparently amorphous in nature since x-rays did not detect it, was clearly visible. This supports the presence of a through solution mechanism of stabilization (8).

#### Eutaw

The x-ray peaks in this soil were reduced, and the creation of a reaction product was shown at 18 Å. New peaks in the TGA data indicated the presence of C<sub>3</sub>AH<sub>6</sub> and CSH II at 310° and 670°C, respectively.

By using SEM photographs, Eutaw in its natural state had a tissue-like structure (montmorillonite) throughout its matrix, and the clay particles dominated in the soil in a more or less random arrangement. The addition of 2 percent lime reduced the amount of montmorillonite but did not destroy it all. Clay-sized particles still had a random arrangement. Montmorillonite disappeared after the addition of 6 percent lime and clay platelets had a more oriented face-to-face pattern. There is no visual evidence of a cementing agent.

#### Sumter

Sumter reacted to the addition of lime by the reduc-

tion of x-ray kaolinite peaks. The TGA data indicated the creation of the 460°C mineral after a 2 percent lime treatment.

A distinguishing feature in the SEM photograph was the presence of skeletal remains of microscopic animals that are found in Selma Chalk, Sumter's parent material. These remains measured approximately 4  $\mu\text{m}$  in diameter. Clay particles were in a face-to-face array. After 2 percent lime was added, the soil matrix became loose and random, and the shallow sea skeletons were still intact and abundant. A lime content of 6 percent oriented the clay plates into a face-to-face arrangement, and the skeletons disappeared from the sample.

#### Tatum

The kaolinite x-ray peak deteriorated with the addition of lime and a peak at 8.0 Å disappeared. Other peaks were reduced, but not as drastically. The 280°C TGA peak (gibbsite) was reduced while CSH (gel) and CSH II appeared at 150° and 670°C after lime treatment. The unknown 450°C was also present.

The SEM photographs indicated that natural Tatum consisted of irregularly shaped silt-sized and clay-sized plates. The small clay platelets exhibited a card house pattern. Two percent lime oriented the plates into stacks.

After the addition of 6 percent lime, the plates returned to the card house structure. Again, no cementing agent was observed. The card house structure and the moderate strength gains after the addition of lime could be caused by the adsorbed state process.

#### Wilcox

Both the two-to-one expandable minerals and the illite x-ray peaks almost entirely disappeared as a result of lime treatment. The quartz peak was reduced drastically. Gibbsite's TGA peak was reduced and a slight CSH II peak was the only observable new mineral.

The soil particles in natural Wilcox appear to be coated with montmorillonite in the SEM photographs. Most of the montmorillonite disappears after lime was added (2 percent), and the plates were in a face-to-face structure. The soil was rearranged into a card house structure after 6 percent lime was added. A trace of montmorillonite was still noted.

As with Eutaw, Wilcox has a high specific surface. Much of the lime was apparently used to break down the montmorillonite. The lime treatment created an edge-to-face arrangement, although the soil gained little strength after treatment and accelerated curing with 6 percent lime. The edge-to-face structure may be caused by the reduction of repulsive forces because of increased cation concentration. If actual stabilizing material had been

Table 2. Summary of results from TGA patterns.

Soil	Mineral	
	Reduced Peak <sup>a</sup>	New Peak <sup>b</sup>
Cecil	Gibbsite, kaolinite	C <sub>4</sub> AH <sub>13</sub> , CSH (gel), C <sub>3</sub> AH <sub>6</sub> , CSH II
Chewacla	Gibbsite, kaolinite	CSH (gel), 440°C (unknown), CSH II
Eutaw		C <sub>3</sub> AH <sub>6</sub> , CSH II
Sumter		460°C (unknown)
Tatum	Gibbsite	CSH (gel), 450°C (unknown), CSH II
Wilcox	Gibbsite	CSH II

<sup>a</sup>These peaks were found to decrease in intensity after the addition of 2 percent lime.

The reduction continued with each increase in lime percentage.

<sup>b</sup>These peaks appeared in the TGA curves after the addition of 2 percent lime. They increase in intensity after each addition of lime.

produced through the adsorbed-state mechanism, strength gain would have been greater. Cementing materials could have been produced by the through solution process; however, the surface area was too large for the cementitious material to affect its unconfined compressive strength.

#### CONCLUSIONS

A summary of the lime-soil reaction product identifications is presented in Table 2. As noted, new TGA peaks were produced by reaction products at 440°C in Chewacla, 450°C in Tatum, and 460°C in Sumter that have not been identified.

The Eutaw and Wilcox soil series have clay fractions that are dominated by montmorillonite. The specific surface of the clay fraction is, therefore, quite high. Much of the lime was apparently used to break down or degrade the montmorillonite. The lime treatment created an edge-to-face arrangement, which may be created by the reduction of repulsive forces associated with double-layer compression in response to increased cation concentration. The net result in terms of unconfined compressive strength increase of lime reactivity, as defined by Thompson (4), was practically nil. If actual stabilization material had been produced through the adsorbed-solution mechanism, larger gains in strength would have been expected. Furthermore, the reaction products produced by the through solution process would also be ineffective because the surface area is too large for the cementitious material to create significant increases in unconfined compressive strengths, although C<sub>3</sub>AH<sub>6</sub> and CSH II were detected following accelerated curing. Thus, it appears that too much fine clay will be detrimental to the development of lime reactivity even though soil-lime reaction products may develop.

Thompson (9) has stated that montmorillonite soils generally react well (in terms of lime reactivity) on the basis of a study that included 39 soils, but only three of the selected soils had montmorillonite percentages in excess of 20 percent of the total soil composition by weight. The Eutaw soil composition had 42 percent expanding 2:1 minerals (predominantly montmorillonite) and the Wilcox had 43 percent.

Cecil and Chewacla demonstrated significant gains in strength after lime treatment and accelerated curing. The SEM photographs suggest that adsorbed-solution mechanisms created the cementitious material for the Cecil, but Chewacla may have benefited from through-solution derived compounds. Although

Tatum did not have as large an increase in strength, the SEM photographs suggest a through-solution cementitious material.

Subsequent research by Rosser and Moore (10) and Lockett (11) has determined that the moisture content used for the compaction of Sumter and Eutaw soil specimens as reported in this paper created a moisture deficiency for the lime-treated specimens. The effect of lime modification increased the plastic limit of Sumter by 20 points and of Eutaw by 10 points. Since the optimum moisture content of the lime-treated material would increase by approximately the same amount, the shift would mean that dry-side compaction was used for the lime-treated specimens for the Eutaw and Sumter soils. The use of a higher optimum moisture content (35 percent) for the Eutaw still produced no lime reactivity in terms of a gain in strength of unconfined compression. However, the Sumter lime reactivity was increased to 62 lb·f/in<sup>2</sup> when a compaction moisture content of 41 percent was used. It appears that the high percentage of montmorillonite in the Eutaw still is accountable for the absence of strength gains. The response of the Sumter soils affirms the suspected moisture deficiency effect for that particular soil series.

#### REFERENCES

1. B.F. Hajek, F.L. Gilbert, and C.A. Steers. Soil Associations of Alabama. Agricultural Experiment Station, Auburn Univ., Auburn, AL, Agronomy and Soils Department Series No. 24, Nov. 1975.
2. R.K. Moore, and G.C. Brown. Development of Soil Stabilization Guidelines for Alabama Soils. State of Alabama Highway Department, Montgomery, HPR Rept. 84, Oct. 1977.
3. C.M. Ford. Reaction Products of Lime-Treated Alabama Soils. Auburn Univ., Auburn, AL, MS thesis, Dec. 1978.
4. M.R. Thompson. Mixture Design for Lime-Treated Soils: A Report of the Investigation of Lime Stabilization of Soils for Highway Purposes. Department of Civil Engineering, Univ. of Illinois, Urbana-Champaign, Jan. 1969.
5. M.L. Jackson. Soil Chemical Analysis Advanced Course. Univ. of Wisconsin, Madison, 1956.
6. G.R. Glenn. Differential Thermal and Thermogravimetric Analysis of Reacted Bentonite-Lime-Water Mixtures. HRB, Highway Research Record 315, 1970, pp. 122-132.
7. C.G. Ruff and C. Ho. Time-Temperature Strength-Reaction Product Relationships in Lime-Bentonite-Water Mixtures. HRB, Highway Research Record 139, 1966, pp. 42-60.
8. S. Diamond and E.B. Kinter. Mechanisms of Soil-Lime Stabilization: An Interpretative Review. HRB, Highway Research Record 92, 1965, pp. 83-102.
9. M.R. Thompson. Lime Reactivity of Illinois Soils. Journal of Soil Mechanics and Foundation Division, ASCE, Vol. 92, No. SMS, Sept. 1966.
10. G.K. Rosser and R.K. Moore. Lime-Treatment of Alabama Black-Belt Soils. State of Alabama Highway Department, Montgomery, HPR Rept. 93, April 1980.
11. L.W. Lockett. Lime-Soil Mixture Design for Alabama Black-Belt Soil. Auburn Univ., Auburn, AL, MS thesis, Dec. 1980.