MINERALOGICAL AND ENGINEERING PROPERTIES OF COMPACTED SOIL-LIME MIXES

By

MOHAMED MAMDOUH ALY SABRY

Bachelor of Science in Civil Engineering
Cairo University
Cairo, Egypt
1969

Master of Science
Oklahoma State University
Stillwater, Oklahoma
1974

Submitted to the Faculty of the Graduate College
of the Oklahoma State University
in partial fulfillment of the requirements
for the Degree of
DOCTOR OF PHILOSOPHY
May, 1977

MINERALOGICAL AND ENGINEERING PROPERTIES OF COMPACTED SOIL-LIME MIXES

Thesis Approved:

James V. Parcher
Thesis Adviser

John C. Moore

Zuhair al-shaieb

Lester W. Reed

Norman N Durham
Dean of the Graduate College

ACKNOWLEDGMENTS

The author's debt to Dr. James V. Parcher goes far beyond this research. Without the encouragement, advice, friendship, and the equally important constructive criticism freely given by Dr. Parcher, the continued advanced study that has proved to be so beneficial might never have been undertaken and completed. The author is sincerely and deeply grateful.

Sincere appreciation is also expressed to Dr. John C. Moore, Dr. Lester W. Reed and Dr. Zuhair Al-Shaieb, committee members, for their invaluable guidance and inspiration throughout this study; and to Dr. T. A. Haliburton, for his counsel and excellent instruction associated during the author's graduate study.

The author also expresses his deep affection and gratitude to his parents for their unfailing support, wise criticism and never-failing confidence in his ability to accomplish any task; and to his wife, Magda, for her encouragement, understanding, and continuous help in performing the laboratory tests involved in the study.

The financial assistance of the Oklahoma Department of Transportation made completion of this work possible and is acknowledged with gratitude. The encouragement, friendly attitude, and stimulating presence of fellow graduate students also aided significantly in completion of the effort.

Finally, the writer expresses sincere thanks to Miss Kareta Casey for the excellence of her work in preparing the manuscript.

TABLE OF CONTENTS

Chapter Page

Chapter Page

LIST OF TABLES

LIST OF FIGURES

LIST OF PHOTOMICROGRAPHS

Photomicrograph Page

CHAPTER I

INTRODUCTION

In recent years lime has been extensively used to alter the engineering characteristics of clayey soils at reasonable cost with good quality control. The improvements of soil workability and plasticity are usually achieved with the addition of a small amount of lime. Although the amount of lime required for soil improvement is highly dependent on soil type and origin, most soils are improved by the addition of lime. Soil properties are modified by a small amount of lime but greater amounts are required to stabilize soils. There is some difference in the two terms, based primarily on considerations of strength. Lime-modified soils are usually assumed to have only enough lime to render them nonplastic, whereas stabilized soils exhibit marked improvement in strength and elastic modulus.

Hydrated lime is widely used to modify or stabilize clay soils. Nevertheless, the nature of its reaction with clay is not yet well understood. In particular, differences in the reaction products resulting from variations in clay mineralogy and from differences in mixing and curing procedures are largely unknown.

Research during the past two decades has established the fact that new minerals are formed under laboratory conditions by the lime-soil reaction, but there is some difference of opinion as to what these are. Calcium silicate hydrate, calcium aluminate hydrate, calcium silicate aluminate hydrate, and calcium carbonate have been variously reported. Only a few field investigations have been performed, but in these, the only new mineral (reaction product) reported was calcium carbonate. Very little is known about the reaction products in compacted soils and the relation of this reaction to soil strength.

Statement of the Problem

It seems reasonably clear that the new minerals formed by the reaction depend on controllable, environmental factors. Since the strength and long-term stability of the stabilized soil depends markedly on these new minerals, it becomes important to determine the mineralogical and environmental conditions under which the various new minerals are formed. With this knowledge, it should be possible to formulate recommendations for field controls (pertaining to mixing, compacting, and curing) that will permit maximum benefits to be derived from lime modifications.

X-ray diffraction and electron microscopy have been used to determine, with some degree of accuracy, differences in the new minerals formed when environmental factors are varied. Samples used in this study are natural soils, with no special treatment; and all tests, including X-ray diffraction, have been conducted on compacted samples.

The occurrence or development of new minerals during long periods of curing has been monitored, and the permanency or persistence of these minerals under leaching action and freeze-thaw cycles has been studied.

Scope of the Investigation

The principal objective of the study is to arrive at recommended field construction procedures and controls that will result in high long-term stability at reduced cost of construction.

The scope of this investigation includes: (1) evaluating the basic properties of natural and treated soils; and (2) investigating the effect of curing time, lime content, water content, and environmental alteration on the strength, pH, and mineralogy of compacted soils. Some selected samples have been subjected to durability and leaching tests.

Data and information provided by this study give an understanding of the relationship between the chemical reaction and soil behavior, and suggest ways in which current construction procedures may be advantageously modified.

CHAPTER II

LITERATURE SURVEY

Soil Stabilization

Natural soil is a complex and variable material. Yet, because of its universal availability and low cost, it offers great opportunities for skillful use as an engineering material. A soil exhibiting a marked and sustained resistance to deformation under repeated or continuing load application, whether in the wet or dry state, is said to be stable soil. If a soil deposit is not suitable, wholly or partially, for the requirements of the construction engineer, a basic decision must be made whether to:

1. Accept the site material as it is and design to standards sufficient to meet the restrictions imposed by its existing quality;
2. Bypass the unsuitable soil;
3. Remove the unsuitable soil, or
4. Treat the soil to improve its properties (soil stabilization).

When soil has been treated to improve its strength and its resistance to change, it is said to be stabilized soil. Thus, stabilization implies improvement in both strength and durability.

Stabilization is used in many soil engineering projects, the most commonly affected properties being volume change characteristics, strength, workability, permeability, and durability. Procedures used to achieve a stabilized soil may be categorized as:

1. Mechanical,
2. Hydraulic,
3. Electrical,
4. Thermal, and
5. Chemical.

Portland cement and lime are the cementing agents most widely used in the chemical stabilization of soils.

Lime Stabilization

The use of lime stabilization preceded the beginning of early recorded history as mentioned by McDowell (57). Perhaps its use in the construction of the pyramids of Shensi in the Tibetan-Mongolian Plateau is one of the oldest applications. These pyramids were constructed of a compacted mixture of clay and lime. They are so ancient that little was known about them when their existence was first recorded over 3000 years ago. In China and India lime stabilization has been used in various forms throughout the long history of these countries. The use of lime in sub-bases of the Roman roads dates back more than 2000 years, although its use as a road construction material in the United States dates back only to the 1920s. Highway and airport engineers found that lime greatly improved sub-grade soils containing a high percentage of clay by reducing the plasticity indexes and increasing the bearing values.

A brief listing of the effects of lime on clay soils shows why lime is currently one of the most popular soil stabilization agents:

1. It lowers the plasticity index.
2. It decreases the soil binder content substantially.
3. It increases the workability.
4. It aids in drying out wet soils.
5. It increases the shrinkage limit and reduces shrinkage cracking.
6. It eliminates almost all swelling problems.
7. It increases the CBR and soil strength; and
8. It reduces the permeability (3).

Lime

Lime, strictly defined, is calcium oxide (CaO) but as commonly used the term includes several forms of quick lime and hydrated lime ($Ca(OH)_2$) which are oxides and hydroxides of calcium or magnesium ($CaO + MgO$, $Ca(OH)_2 + Mg(OH)_2$, $Ca(OH)_2 + MgO$). The two sources of lime are calcitic and dolomitic rocks. As indicated above, there are three types of hydrated lime: $Ca(OH)_2$, $Ca(OH)_2 + MgO$, $Ca(OH)_2 + Mg(OH)_2$, and two types of quick lime: CaO and CaO + MgO. Reactive relations are as follows:

$$21 \text{ kg-cal} + CaCO_3 \longrightarrow CaO + CO_2$$

$$CaO + H_2O \longrightarrow Ca(OH)_2 + 15.3 \text{ kg-cal}$$

Alexander, Smith and Sherman (6) concluded that: (a) soil treated with quicklime exhibits higher strength than when treated with hydrated lime; (b) coarser limes are more effective than the fine ones; (c) lime may be applied either as a slurry or as a powder; (d) slurried quicklime produced higher strengths than the powdered quicklime; and (3) hydrated lime produces about the same strength slurried as powder does.

Engineering Characteristics of Soil-Lime Mixture

The addition of lime to soil will change most of, if not all, the soil properties. The greatest benefits of lime stabilization have been in the field of highway engineering, where a large proportion of the lime currently manufactured is used in stabilizing highway sub-grades and sub-bases. The reduction of plasticity, increase of strength, increase of workability, decrease of permeability, and increase of durability do, however, result in a variety of other applications. Troublesome moisture variations in highway sub-grades can be largely controlled through the use of lime. Sub-grades of airport runways and taxiways also are frequently stabilized with lime. At the Dallas-Fort Worth Regional Airport, 500 tons of lime were used daily for 95 days (4). The load-bearing capacity of the sub-grade and that of the pavement was vastly improved. In the case of swelling clay, lime is used to reduce the swelling pressure and decrease the volume change associated with moisture variation. Wright (85) reported that road beds for railroad tracks have been stabilized by injecting lime slurry. This method is fairly new, but it has been used with some success in Texas, Louisiana, Oklahoma, and recently in North Carolina. It is difficult to evaluate its degree of success at the present time. Erosion control is also a field in which lime treatment may play an important role in preventing the formation of gullies and tunnels in dispersive clays. Recently, Hayden (32) concluded that the addition of 1.5 percent to 2.0 percent lime by dry weight of soil will reduce dispersive soil erosion potential from 60% to less than 10%. There are still many questions that need to be answered with respect to the evaluation of the erosion potential of different soils. However, the effectiveness of lime has been proven.

The various effects of lime on soil properties are discussed separately in the following sections.

Atterberg Limits

Lime generally causes a marked reduction in the plasticity index, an increase in shrinkage limit, and improved workability. Many clay soils will be nonplastic after they have been treated with lime, and the capacity to shrink and swell is frequently eliminated.

The effect of lime on Atterberg Limits has been investigated by Hilt and Davidson (34), Mitchell and Hooper (59), Fossberg (24), Lund and Ramsey (50), and Ladd, Moh and Lambe (46). All of these investigators found that lime reduces the plasticity of clayey soils; however, the effect of lime on the Liquid Limit (LL) differs from one soil to another. Some authors report decreases in the LL with the addition of lime (72) (10) (50), while others report substantial increases in the LL (59) (46). It appears that the LL may either increase or decrease, depending on the type of soil. Herrin and Mitchell (33) indicate that in general, but not always, the LL is reduced in the more plastic clays and is increased in the less plastic soils.

It is concluded that the LL of lime-treated soils depends mainly on the type of clay mineral and lime, the amount of clay present in the soil, composition of the pore water, and the exchangeable cation originally present.

The Plastic Limit (PL) increases with an increase of lime. Hilt and Davidson (34) report that the PL will not increase beyond a certain maximum corresponding to a point of lime fixation beyond which additional lime has no effect. For montmorillonite Hilt and Davidson (34) state that the percent of lime at the point of fixation is:

$$L = \frac{C}{35} + 1.25$$

where

L = lime at fixation point, %; and

C = clay of $<2\mu$ size, %.

Stocker (72) found that the PL increases until the Plasticity Index (PI) is reduced to zero.

Regardless of whether the LL decreases or increases, the increase in PL is such that the PI of most clays is reduced by the addition of lime up to a certain lime content. The principal benefits of a reduction in plasticity come from the fact that nonplastic soils are more workable and have a greater volumetric stability, both of which are important factors in road construction. Lime-treated plastic clays take on the appearance and feel of a friable, silty soil which is easy to manipulate and compact.

Volume Change

Soils treated with lime usually do not exhibit shrink-swell behavior. As lime is added the Shrinkage Limit, Plastic Limit, and Liquid Limit tend toward a common value of the moisture content. Both swelling potential and swelling pressure normally are significantly reduced by treating clay with lime (36) (59). These reduced swell characteristics are generally attributed to a decreased affinity for water of the calcium-saturated clay and the formation of a cementitious matrix which resists volumetric expansion. Goldberg and Klein (27) state that shrinkage due to moisture loss from the stabilized soil may lead to the formation of some shrinkage cracks; however, the change of field moisture content in lime-treated soils is not large and shrinkage will not be extensive.

Grain Size Distribution

Changes in the effective grain size distribution (mechanical analysis) are observed almost immediately following the addition of lime to a clay soil. Lund and Ramsey (50) report that a clay having 88% of its particles finer than the U.S. No. 200 sieve exhibited only 57% of minus 200 sizes after 10% lime was added and the soil was cured for 14 days. After 240 days only 38% of minus 200 sizes were present. The grain size distribution affects the soil classification; for example, an A-7-6 soil was changed with 6% lime in 14 days to an A-4 soil, according to Brand and Schoenberg (10). However, Herrin and Mitchell (33) indicate that after a period of soaking in water, a small portion of the aggregated particles tends to break down.

Compaction Characteristics

According to Mitchell and Hooper (59), when soil-lime mixtures are compacted with a constant effort, the shape of the moisture-density curve, the maximum dry density, and the optimum moisture content depend on three factors:

1. Lime content.
2. Elapsed time between mixing and compaction.
3. Type of lime.

Generally, the lime-treated soil will have a lower maximum density and a higher optimum moisture content than is found for the untreated soil. The maximum density will continue to decrease as the lime content is increased, and the optimum moisture content will increase (59). These changes are accentuated by the increased flocculency of the mixture as it is allowed to cure.

Strength

The strength of a soil-lime mixture can be evaluated in several ways, such as by unconfined compression, CBR, Hveem stabilometer, and triaxial tests. The most convenient and popular procedure is the unconfined compression test, but it is not necessarily the most applicable or appropriate test for all purposes.

It should be emphasized that the strength of a soil-lime mixture is dependent on many variables (59):

1. Soil type.
2. Lime content.
3. Type of lime.
4. Curing time and method (temperature and water availability).
5. Water content and density.
6. Time elapsed between mixing and compaction.

The interactions among these variables exert a major influence on the soil strength. Generally, the strength will increase as the lime content, curing time, and density are increased. The influence of other variables is governed by the soil type (58) (6).

A clear distinction should be made between immediate stabilization and long-term stabilization of soil with lime. Immediate stabilization does not try to produce a high strength material but to obtain, within a short period of time, improvements of several geotechnical characteristics, mainly:

1. Decrease of the Plastic Index.
2. Better compaction characteristics.
3. Higher bearing capacities after compaction.

Most, but not all, clay soils respond immediately to lime. The short-time strength tests probably will not identify the optimum lime content for most types of construction, but it is essential that they be made to guard against the use of soils which are nonreactive with lime.

Van Gansel (82) reports that the amount of lime needed for maximum stability of specimens cured for two hours depends on the soil type but does not exceed 1.0% CaO. He also found that the Plastic Limit will increase by 25% of the Plasticity Index of the untreated soil. According to Van Gansel, the compaction curve of treated soil cured for two hours has a flatter shape than that of the untreated soil, with lower maximum density (ranging from 0.4 to 0.12 g/cm^3 lower) and higher optimum water content (1.2 to 3.3% higher). He also found that an addition of 3.0% CaO results in, after two hours curing, CBR values which are six to ten times larger than those for untreated soil with equal water content.

Neubauer and Thompson (62) concluded that the effects of the immediate lime-soil reaction are sufficient to greatly improve adverse in situ soil conditions for purposes such as expediting construction, providing increased sub-grade support, and constructing temporary roads. Brand and Schoenberg (10) reached the same conclusion in their study conducted in West Germany.

The cured soil-lime mixture is much stronger than the untreated and/or treated, uncured soil-lime mixture. Curing time and method greatly influence the rate of strength increase and the final strength of the soil mixture. According to Marks and Haliburton (52), an elevated curing temperature accelerates the reaction, resulting in higher early strength. Samples mixed with low lime content (2%) will reach maximum strength in less time than those mixed with higher lime content (6%).

Mitchell and Hooper (59) examined the effect of elapsed time between mixing and compaction on the final soil strength. They report that samples compacted within one hour after mixing had a higher strength than those compacted after 24 hours. All samples were cured for 215 days in a moist atmosphere prior to testing the strength.

The elastic properties of soil-lime mixtures were studied by Thompson (76) (77). He reported that lime treatment improved the shear strength of soils and that, although the cohesion of the mixtures was increased with the addition of lime, the angle of internal friction (ϕ) was very difficult to evaluate. In most cases, however, the cured soil-lime mixture had a larger ϕ than that of the untreated soil.

An improved tensile strength has been reported by Tulloch, Hudson and Kennedy (81), who give the following regression equation:

$$S_t = 6.8 + 50.6\ q_u$$

where

S_t = tensile strength (psi); and

q_u = unconfined compression strength in ksi.

Using indirect tensile tests Thompson (76) found the tensile strength to be 0.13 q_u. He also suggested (77) that the elastic modulus of soil-lime mixtures, under a confining pressure of 15 psi, may be estimated by the equation:

$$E = 9.98 + .124\ q_u$$

where

E = compressive modulus of elasticity (ksi); and

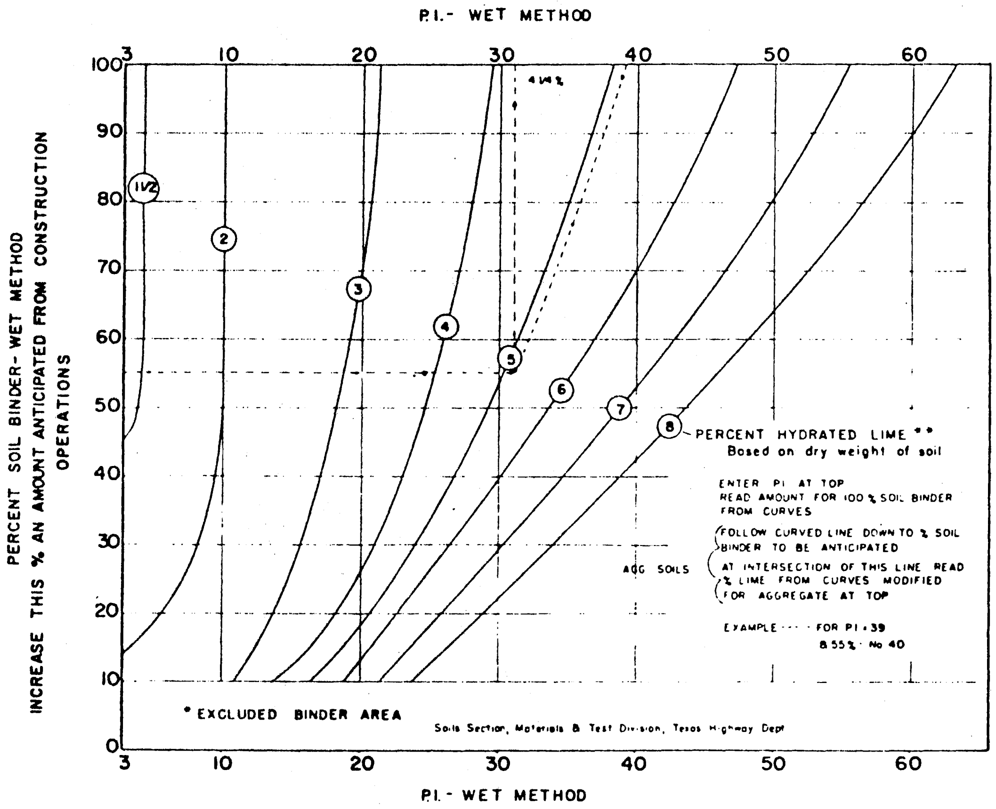
q_u = unconfined compressive strength (psi).

The long-term tests required to predict the strength of soil-lime mixtures under field conditions are often impractical from the standpoint of time and also might not suggest the use of enough lime due to the ideal conditions under which the tests are run. Accelerated curing under elevated temperatures may be useful in evaluating the long-term strength in a relatively short time. This procedure has been suggested by Anday (8) and Marks and Haliburton (52).

With respect to the rate of chemical reaction, 10 days curing at 20°C is equivalent to 5 days at 30°C, to 2½ days at 40°C, etc. However, a laboratory testing program must generally be used to verify the presumed equivalency in strength.

Selection of the amount of lime to be used with a given soil has been variously based on the following considerations:

1. The unconfined strength of treated samples cured at normal or elevated temperatures;
2. The lime content at which the pH value of a soil-lime mixture (20 gm + 100 ml deionized water) reaches 12.4 (20);
3. The lime content at which the PL is a maximum (34); and
4. Indirect prediction based on index properties of the soil (58) (see Figure 1).



PERCENT HYDRATED LIME **
Based on dry weight of soil

ENTER PI AT TOP
READ AMOUNT FOR 100 % SOIL BINDER FROM CURVES

FOLLOW CURVED LINE DOWN TO % SOIL BINDER TO BE ANTICIPATED

AT INTERSECTION OF THIS LINE READ % LIME FROM CURVES MODIFIED FOR AGGREGATE AT TOP

EXAMPLE ···· FOR PI = 39
& 55% - No 40

Soils Section, Materials & Test Division, Texas Highway Dept

* Exclude use of chart for materials with less than 10% - No 40 and cohesionless materials (P.I less than 3)

** Percent of relatively pure lime usually 90% or more of Ca and/or Mg hydroxides and 85% or more of which pass the No 200 sieve Percentages shown are for stabilizing subgrades and base courses where lasting effects are desired Satisfactory temporary results are sometimes obtained by the use of as little as 1/2 of above percentages Reference to cementing strength is implied when such terms as "Lasting Effects" and "Temporary Results" are used

Figure 1. Recommended Amounts of Lime for Stabilization of Subgrades and Bases (After McDowell (58))

Most of these methods are acceptable for use in connection with highway or airport construction, although additional testing is usually required to validate their use with a particular soil. The first method should generally be used to determine the mix design for foundation layers.

Durability

Durability tests for stabilized soil have been conducted by simulating weathering through such cyclic tests as (a) freezing and thawing, (b) wetting and drying, or (c) heating and cooling.

Generally, the durability of soil-lime mixtures in the field has been satisfactory. Rosen and Marks (67) reported that the residual strength after six freeze-thaw cycles decreases to 30% of the original strength. This appears to be a very severe loss of strength, but strength loss from a freeze-thaw cycle is much greater in the laboratory than in the field. Mateos and Davidson (54) reported that soil treated with lime and fly ash experienced a 20% loss of strength after ten cycles of freeze and thaw. Townsend and Klym (80) also indicated that the strength loss after seven cycles of freeze and thaw was only 20%. Walker and Karabulut (83) report over a 50% strength loss after five cycles of freeze and thaw for clayey soils.

Thompson and Dempsey (78) indicated that in most cases it is thought that the damage caused by freezing and thawing can be healed if the material is permitted to recure under the original conditions.

Durability must be considered in establishing the mix design and selecting design strength parameters.

Permeability

The effect of lime on the soil permeability is still not clear. Fossberg (9) reported that the permeability decreased with increasing lime content and curing time. He based this conclusion mainly on the results of consolidometer tests. Townsend and Klym (80) reported a substantial increase in the coefficient of permeability of heavy clay soils (from 2×10^{-8} to 4×10^{-6} cm/sec). The writers claimed that their results differ from those of Fossberg because of the additional flocculation that occurs during a mellowing period perior to compaction. Fossberg, who compacted his samples immediately after mixing, reported a reduction in the coefficient of permeability from 10^{-6} to 10^{-8} in/min after 28 days curing, at a void ratio of 1.5. Townsend and Klym, on the other hand, reported an increase in k from 2×10^{-8} cm/sec to 4×10^{-6} cm/sec for samples mellowed 24 hours before compaction and cured for 19 weeks, with most of the increase occurring during the first 4 weeks.

Soil-Lime Reaction

Lime stabilization of highway sub-grade soil has been in use for about 30 years. During the past 15 years the use of hydrated lime to reduce the plasticity and increase the strength of clay soils has grown tremendously. To understand the effects of lime on the physical and mechanical properties of fine-grained soil, the chemical reactions of soil and lime must be examined. Several researchers have investigated this reaction using different types of clay and lime. However, very few have investigated the reactions occurring in compacted soil, particularly under field conditions.

In general, it has been found that the addition of lime to a fine-grained soil initiates several reactions. Short-term reactions consist of cation exchange and flocculation-agglomeration, and the extent to which these take place depends on the availability of water and the time permitted for curing. The decrease of plasticity, increase in workability, and shift of the compaction curve result primarily from these reactions.

Several studies have been conducted to evaluate the significance of cation exchange and flocculation in the stabilization process. Hilt and Davidson (34) introduced the term "lime fixation" which was defined as "the process by which lime is held by the soil and is not available for pozzolanic reactions, or it is the percentage of lime additive at which strength just begins to increase." They also found that cation exchange does not play a major role, but that the crowding of additional cations onto the surfaces of the clay particles is an important mechanism. The addition of lime will reduce the water surface tension in minute pores and cause the clay particles to become attracted to each other, causing flocculation or aggregation and giving the clay soil a silt-like character.

Herrin and Mitchell (33) reported that when lime and moist clay soil are mixed together and allowed to mellow in a loose condition for a few days, the calcium cations of the lime will replace the weaker metallic ions, such as sodium and hydrogen, on the surfaces of the clay particles. They also reported that replacement of the univalent ions by the divalent calcium ions will attract the soil particles together, making clayey soils more friable and less plastic.

Slone (71), in his study of early reactions of lime with pure kaolinite, showed that some reaction takes place as early as one day. However, his study was made on a soil-lime suspension (5 gm kaolinite + 65 ml of saturated $Ca(OH)_2$ solution at 25°C). He found a pronounced etching and bevelling to be evident along the edges of many particles. He also found that a cementing agent was formed after 14 days of curing which he identified as calcium aluminum silicate hydrate. Ormsby and Bolz (64) conducted a similar study on kaolinite, using compacted samples with 0.8%, and 6% lime based on the dry weight of soil. The reaction product was identified as calcium silicate hydrate. A differential thermal study of the clay-lime reaction by Glenn (25) showed that calcium silicate hydrate having a low grade of crystallinity is formed after four or five days of curing at 40°C. His mixtures were prepared as slurries with 45% lime and 162% water by dry weight of soil. Kobashi (40), in his study of lime's reaction with an allophane-kaolinite clay mineral, reported that amorphous hydrated gehlenite was formed within one day. Kobashi believes that in the reaction of metakaolin and calcium hydroxide solution calcium hydroxide is adsorbed by the metakaolin in the first place, then is diffused into the metakaolin lattice, and finally is coordinated to form a crystalline product. The amount of lime required for complete reaction is related to the area of the clay particles, which influences the adsorptivity of the clay mineral.

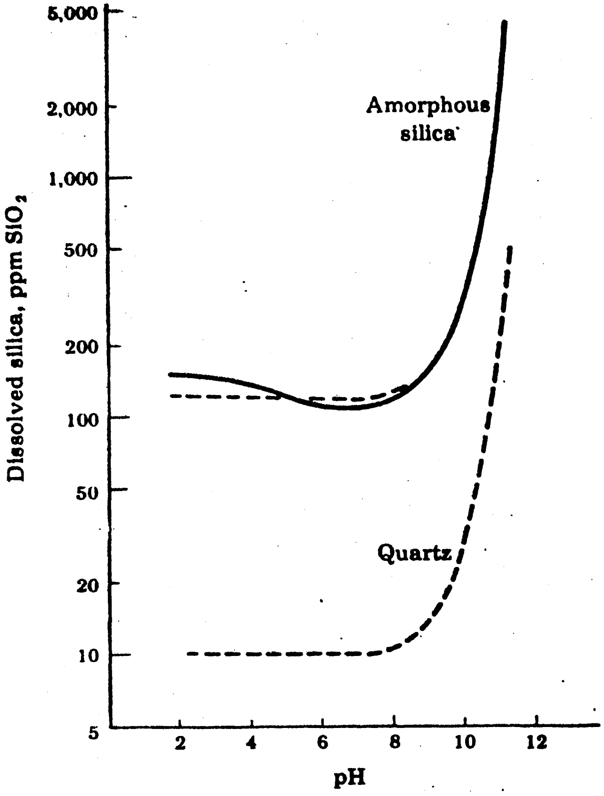
Diamond and Kinter (17) reviewed several hypotheses and concluded that cation exchange, flocculation, and carbonation cannot be regarded as essential factors in the instant modification of soil. They believed that the primary phenomenon is a very rapid physical adsorption of both calcium and hydroxyl ions on the clay surfaces, even when these are already calcium saturated. They suggested that an immediate reaction occurs between the alumina-bearing edges of clay particles and the lime adsorbed on the faces of adjacent particles. This results in the formation of bonds of tetracalcium alumina hydrate and, possibly, calcium silicate hydrate which links the clay particles together.

Stocker (73) believes that the cementing products are hydrated calcium silicates and aluminates, produced by lime attacking the edges of 2:1 clay minerals and that the reaction products are attached along the crystal edges from which they are generated. These products were described as a foil-like habit of high surface area. In Stocker's opinion, reactions of this type are a primary cause of flocculation. This study differs from those on compacted soils in that it was carried out on diffused cells of soil, whereas particles of compacted soils are surrounded by the stabilized system.

It may be concluded that even if cation exchange occurs it does not play a major role in changing the soil properties. Flocculation and agglomeration are a result of the rapid formation of calcium aluminate hydrate and some form of a silica gel cementing the clay particles together.

Soil-Lime Pozzolanic Reaction

When enough lime is mixed with soil and water, a slow, long-term reaction occurs in which the calcium ions react with dissolved silica and alumina. The source of silica and alumina may be the clay mineral, feldspar, mica, or amorphous silicate or alumina silicate minerals. The solubility of silica and alumina increases as the pH of the clay-lime mixtures rises toward its limiting value of 12.4 (Figure 2, Reference



Solubility of silica at 25°C. The solid line shows the variation in solubility of amorphous silica with pH as determined experimentally. The upper dashed line is a calculated curve for amorphous silica, based on an assumed constant solubility of 120 ppm SiO_2 *at pH's below 8 and on a value of −9.9 for log* K_1 *for* H_4SiO_4. *The lower dashed line is the calculated solubility of quartz, based on the approximately known solubility of 10 ppm* SiO_2 *in neutral and acid solutions.*

Figure 2. Variation of SiO_2 solubility with pH (After Krauskopf (41))

(41)). Eades and Grim (21) indicate that clay particle edges are attacked first in some cases involving illite and montmorillonite "the whole clay mineral structure deteriorates without the formation of a substantial new crystalline phase." However, Stocker (73) showed that only 3% of the clay is involved in the initial cementing. He also found that strong modification of all the clay does not completely eliminate the activity of the clay surfaces. Even very strongly cemented clay has much of its original crystalline structure.

Most of the work suggested a "through solution" mechanism in which clay edges are dissolved from the clay's structure to form a cementing material. Ormsby and Bolz (64) indicated the possibility that surface chemical reactions can occur and new phases can form directly on the surface of the clay lattice.

Both "through solution" and "hydration-crystallization" appear to be possible mechanisms in soil stabilization. It is unlikely that clay particles will be completely destroyed, but severe alteration of the clay structure is possible.

Since the available ions in a soil-lime system are silica, alumina, and calcium, it is not surprising that reaction produces compounds falling largely in two classes: hydrated calcium silicate (CSH) and hydrated calcium aluminates (CAH) (in the shorthand notation commonly used for these compounds, C = CaO, A = Al_2O_3, H = H_2O, and S = SiO_2). Only under hydrothermal conditions are the calcium silicate hydrates well crystallized and easy to detect. The principal known phases are: (a) tobermorite gel, also called CSH (gel), (b) CSH (I), and (c) CSH (II). Taylor (74) reports that these products vary in crystallinity as well as calcium content; the highest calcium content is found in CSH (gel), and the best crystallinity is found in CSH (II). There are several CAH compounds. The one commonly formed at normal temperatures is a member of the tetra-calcium hydrate group. This product may proceed to $C_4AH_{12}\cdot CO_2$, which is a highly stable phase. If the reaction takes place at slightly elevated temperatures (30°C), a different phase is introduced, C_3AH_6, which is a stable crystal differing from C_4AH_{13} in its structure and degree of crystallinity.

Eades and Grim (21) report that a poorly crystallized CSH of unspecified type is found when pure kaolinite is treated with lime at 60°C. They failed to detect any new crystalline product from a lime-montmorillonite reaction, although it was obvious that a reaction had occurred. A lime-illite reaction was also observed but no positive conclusion was reached as to the nature of the product. Hilt and Davidson (35) reported that montmorillonite will react with lime at room temperature forming C_4AH_{13} and a weak phase of CSH(gel). Diamond, White and Dolch (18) stated that montmorillonite reacted with lime at 60°C forming CSH(I) and that the reaction with kaolinite produced the cubic crystalline C_3AH_6; no crystalline calcium aluminate compound was formed from montmorillonite. At a lower temperature both clays form tobermorite gel and CAH CO_3. Ormsby and Bolz (64) performed electron microscopic studies to investigate the kaolinite-lime reaction. They reported that the products of reaction were calcium silicate hydrates, either CSH(I), CSH(II), or both; no quaternary phases were detected. Kobashi (40) reacted lime with allophane-kaolinite clay minerals and concluded that hydrated gehlenite (C_2ASH_{11}) was formed in most of the samples. There were also indications that C_4AH_{13}, C_3AH_6, and C_3AH_{8-12} were formed but not as prominently as C_2ASH_{11}. The samples were treated at 30°C with the water content varying from 80 to 120% and with relatively high lime contents of 5 to 20%. All samples were sealed during curing (two months). There was also some indication that $CaCO_3$ had been formed.

Glenn (25) examined the lime-bentonite reaction by differential thermal and thermogravimetric analysis (DTA and TGA). Samples were cured at temperatures of 23°, 40°, and 80° to 170°C. CSH(I) and CSH(II) were formed at temperatures of 40°, 80°, 105°, and 145°C, but at 23°C only CSH(gel) was found after three years. Tobermorite was found at temperatures of 105° and 170° after 12 hours curing.

Kronert and Wetzel (42) (43) (44) (45) examined the reaction of kaolinite, montmorillonite and fire clay with lime at an elevated temperature (60°C) for curing times of up to 56 days. The samples were prepared with 43% lime and a water content of 400%. The kaolinite reaction was analyzed after 5 days of curing, by which time some crystallized C_4AH_{13} had been formed; there was an indication that CSH (tobermorite) was evolving after 14 days of curing. C_4AH_{13} was altered to form C_3AH_6, which is more stable at room temperature. The reaction with fire clay was very similar to that with kaolinite, but was more pronounced and the reaction was much more rapid. No free lime was detected after 14 days in the fire-clay tests, whereas in the case of kaolinite there was some lime still available after 56 days (Figure 3). In the case of montmorillonite, C_4AH_{13} was present after 5 days but diminished after 28 days with an increase in the production of hydrogranite (Figure 4).

Stocker (73) found that all lime-clay reaction products had the composition $C_{11}S_{4.5}A_1H_x$. Diffused lime reacted with montmorillonite clay at all ages, giving a product with the $SiO_2:Al_2O_3$ molar ratio of

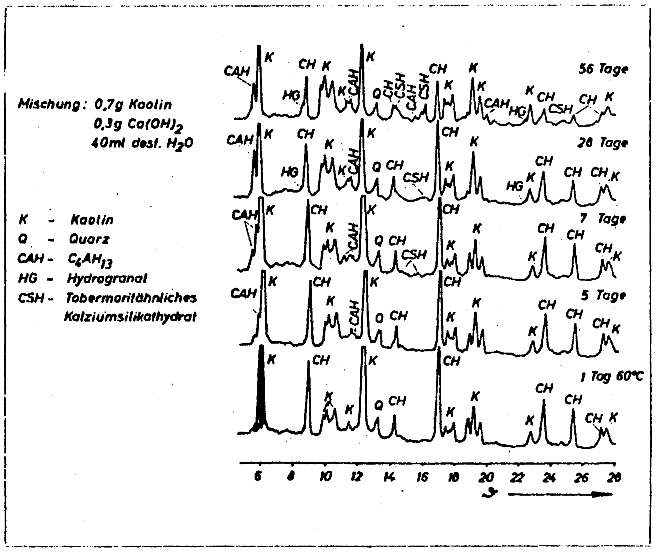


Figure 3. X-ray Diffraction Diagrams for Kaolin After Reaction With $Ca(OH)_2$ at 60°C (After Kronert and Wetzel (42))

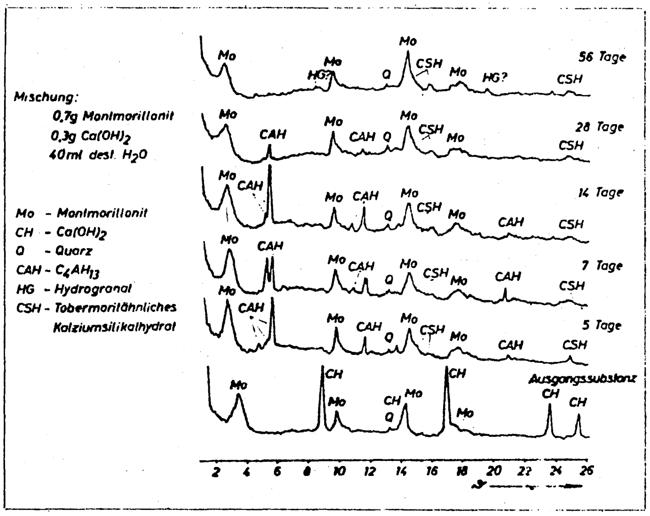


Figure 4. X-ray Diffraction Diagram of Montmorillonite After Reaction With $Ca(OH)_2$ at 60°C (After Kronert and Wetzel (42))

4.5:1.0. Reaction was exclusively with the particle edges. The low release of potassium suggested that there is a preferential attack on the edges of montmorillonite crystals.

It is clear that the experimental conditions in the above investigations do not favor the formation of calcium carbonate. However, Eades, Nicholas and Grim (22) found during field experimentation that a great portion of the cementing product was $CaCO_3$. It appears far more appropriate to study the reaction and fabric present in a compacted soil than to study those in a suspension. Matsuo and Sung (55) found that there was a definite relation between strength increase and carbonation. They also found that the relation between degree of reaction and unconfined strength is a linear one.

In any study in which laboratory results are to be extrapolated to field conditions, it is important to evaluate the influence of $CaCO_3$. If the presence of $CaCO_3$ is not favorable to the soil properties, a feasible way of preventing its formation in the field should be developed. An attempt by Sabry (69) was made to prevent the formation of $CaCO_3$ in the laboratory. The soil used was Georgia kaolinite. The samples were cured unwrapped with 6% lime content at 140°F for 3 days. The samples were prepared at optimum moisture content; some of them were soaked before curing to prevent the infiltration of CO_2 from the air. The results (see Figure 5) show the differences in the new minerals that were formed. Soil strength was not investigated.

Factors Affecting Lime Reactivity

It has been proven that most clay soils will react with lime and that the soil properties will be affected by lime. However, the degree

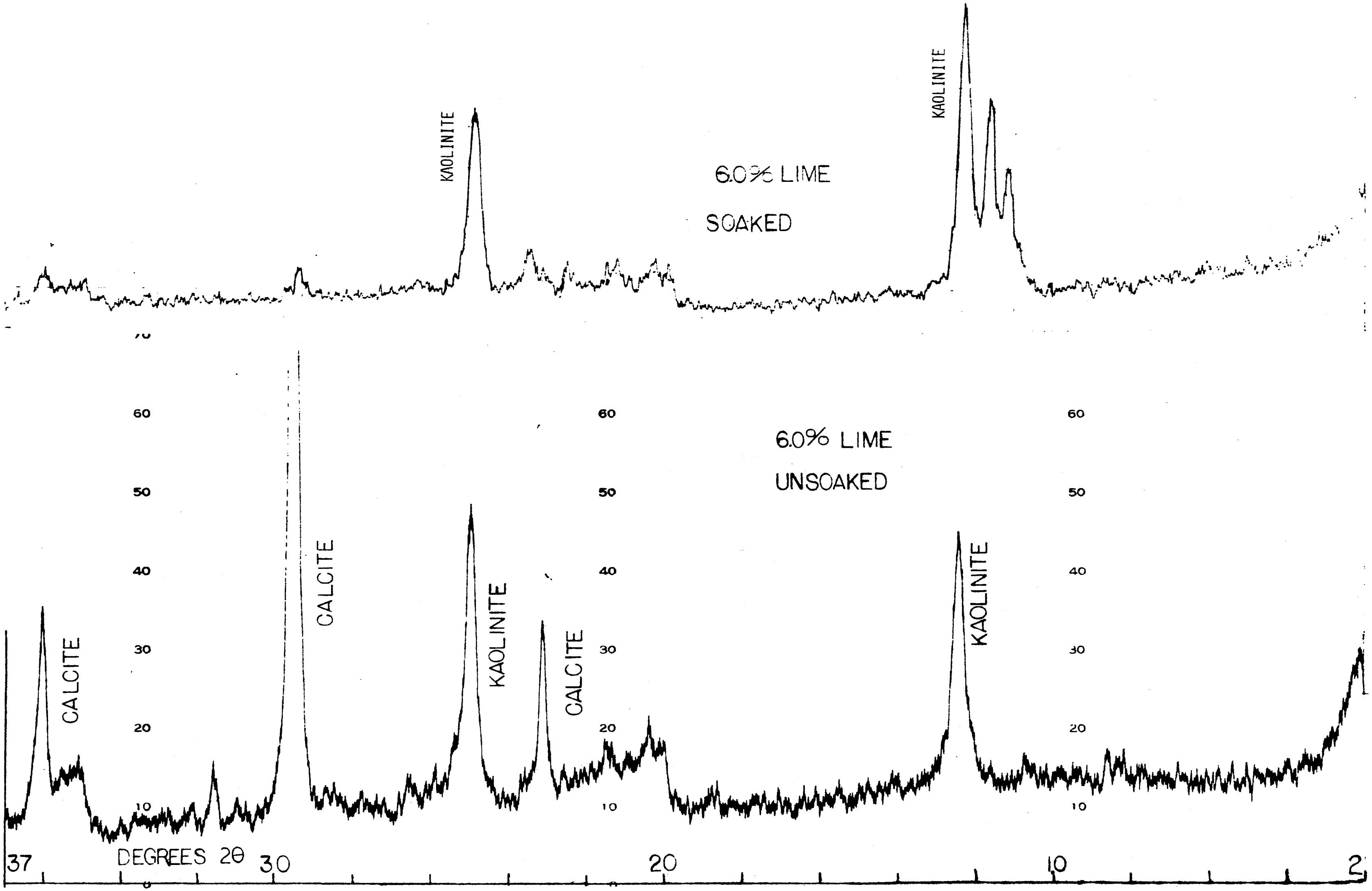


Figure 5. X-Ray Diffraction Patterns for Georgia Kaolinite Indicating the Effects of Soaking on Soil-Lime Reaction (After Sabry 69)

of reactivity varies from one soil to another. Thompson (75) and Arman and Munfakh (9) reported that some factors that will retard the soil-lime reaction are: (a) organic content, (b) good natural drainage, (c) low soil pH (<7.0), and (d) the presence of A-horizon soils. Harty and Thompson (32), Moore and Jones (61), Shen and Li (70), and O'Flaherty and Gray (63) reported that several other factors that influence the reactivity of soil are the silica/alumina ration, cation exchange capacity, type of exchangeable cation, type of clay mineral, clay content, and carbon content. There is no defined trend for the relative influence of these factors at the present time, but they may be useful in predicting the behavior of a particular soil during and following the application of lime.

CHAPTER III

MATERIALS SAMPLE PREPARATION AND TESTING PROCEDURES

This chapter provides background information concerning the selected clayey soils and sample preparation procedures employed throughout the research. Physical characteristics and geological origins will be given in considerable detail, since most of these factors influence the soil reactivity. Every effort was made to employ uniform procedures in the preparation of both raw and treated soil mixtures to assure that, insofar as possible, behavioral differences would be attributable to differences in the reaction processes. Testing procedures were also standardized in all phases of the research.

Materials

Soils: Origin and Nature

The soils used in this research were natural materials obtained from diverse locations in the state of Oklahoma. Some of these soils are also found in the neighboring states of Kansas and Texas. Two samples were selected on the basis of their relative purity with respect to the dominant mineral occurring in the clay fraction of the soil. The other two samples were selected by the Research Division of the Oklahoma Department of Transportation. They are classified as troublesome soils for highway construction purposes.

The Union City clay is a material of medium plasticity, obtained from the Permian deposits of central Oklahoma. These marine deposits are the dominant geological formation found in a wide north-south band passing through central Oklahoma. The formations have a distinctive red color from their high iron oxide content and are commonly termed the Permian Red Beds. The red beds are composed of Permian red clay overlying soft, variable, red clay shales and sandstones and are usually interbedded. The clay and clay shale have moderately high volume change characteristics associated with any variation in moisture content. The sample was obtained from a clay pit operated by the Oklahoma Brick Company, two miles north of Union City, Oklahoma, in Canadian County. The sample was dug from the bottom of the open pit, about 15 feet below the natural ground surface. There was a noticeable occurrence of gypsum sheets in the area, most of them formed in shrinkage cracks in sheets of up to two inches in thickness. The collected samples include no parts of the gypsum sheets. The sample was re-examined in the laboratory to insure that there was no gypsum imbedded in the large lumps. The selection of this site was based on the high purity of the deposit and the high clay content. The clay fraction of this soil is entirely illite.

The Camargo bentonite was obtained from Dewey County in northwestern Oklahoma. A study by Patrick (65) indicated that much of northwestern Oklahoma is covered with a veneer of alluvial sands and gravels of the Tertiary age. These sediments are generally uncemented, cross-bedded, yellow to light brown soils, locally as much as 400 feet thick, and overlying the red shales and sandstones of the Permian Period.

The sample of Camargo bentonite used in this study is a calcium montmorillonite of exceptional purity, ranging from white to pinkish-gray in color. Bentonite is usually thought to be a result of the chemical alteration (weathering) of volcanic ash. In the Camargo area it is found in a stratum 5 to 6 feet thick, underlying 20 to 30 feet of the Tertiary soils described above. The sample came from a pit 6½ miles north and 1 mile east of Camargo, Oklahoma that was operated commercially for several years but is no longer in operation. Large chunks of the material were taken from the bottom of the pit. It is highly plastic and soapy to the touch in the wet state and is hard, brittle and light in weight in the dry state.

The predominant clay minerals in Oklahoma soils are illite and montmorillonite. Each of the samples so far described contains one of these minerals in a nearly pure form and contains a high percentage of clay size particles.

Two other soils were provided by the Oklahoma Department of Transportation. Pavements have been found to perform poorly when either of these soils occurs in a highway sub-grade. The Burleson soil was sampled in Bryan County in southwestern Oklahoma. The sample was taken from a depth of 24 to 48 inches in a pit located 350 feet south of the northwest corner of Section 10, T5S, R9E. The general description of the soil profile is shown in Table I.

The soil selected from the AC horizon exhibits swelling characteristics typical of this soil series. Burleson soils are on ancient stream terraces with slope gradients usually less than 2%. The soil, formed from alkaline clayey sediments, is moderately well drained, having slow runoff in nearly level areas, medium runoff in gently sloping areas, and a very low permeability. Water enters the soil rapidly when it is dry and cracked, and very slowly when it is moist. A cyclical geometric pattern is observed in the soil strata. The pit dug for sampling was only 20 feet long; yet, in this interval the bottom of the AC horizon extended to a depth of 55 inches twice, and to a depth of only 18 inches in between. Lytton and Meyer (47) report a similar geometric pattern in other areas of heaving soils, and believed it to be a result of the volumetric instability of such soils.

TABLE I

BURLESON SOIL PROFILE AT SAMPLING LOCATION

Horizon	Depth (in.)	Description
A1	0-10	Clay, black, firm, disturbed layers
A12	10-24	Clay, black, very firm
AC	24-48	Clay, very dark gray, very firm; many slick surfaces, increasing with depth, especially numerous between 36 and 48 in.
C	48-55	Clay, olive gray, very firm, highly slickensided

The other soil provided by the Oklahoma Department of Transportation is from Osage County, northern Oklahoma. The Oklahoma Department of Transportation has encountered many problems along State Highways

11, 60, and 99 that traverse this particular soil, which is a member of the Summit series. The Summit soil was sampled from the Barnard Ranch at a location described as the center of the E 1/2 of the E 1/2 of the NE 1/4 of Section 27, T28N, R8E of the Indian Meridian. The sample was taken from the side of a small gully about 50 feet west of a county road. The description of the soil profile exposed in the gully is shown in Table II.

TABLE II

SUMMIT SOIL PROFILE AT SAMPLING LOCATION

Horizon	Depth (in.)	Description
1. A1	0-9	Black, silty clay loam, firm when moist (hand pressure)
2. B1	9-17	Black, silty clay loam, firm when moist
3. B21	17-26	Black, silty clay, very firm, moist
4. B22	26-34	Dark grayish brown, silty clay, very firm, moist
5. B23	34-57	Dark grayish brown, silty clay, very firm, moist
6. B3	57-72	Silty clay, very firm, moist, coarsely mottled with olive and brown
7. Cr	72+	Shale, olive, weathered soft rock

Horizons 4 and 5 were selected for sampling and exhibited a considerable number of slickensides, but these were most numerous in Horizon 5.

This soil is a residual soil that typically occurs on limey clays, limestones, or on colluvium from those materials. The slope generally varies from 1.0 to 5.0%. The underlying rock formations are Fayetteville shale, Hindsville limestone, Moorefield limestone, St. Joe limestone, and the Boone formation (cherts). The Summit soil extends into Kansas and the Burleson soil extends into Texas. The study of these soils is therefore of some interest to states other than Oklahoma (28).

All samples were obtained in a disturbed condition and the sampled water content for each soil was determined. The samples were then oven-dried for 24 hours at 100°C, after reducing the size of clods to 2.0 in. or less in diameter. Dried material was processed in the Model 4E Straub laboratory mill which was adjusted to produce soil particles passing the U.S. No. 40 sieve. As material came through the grinder it was checked frequently by sieving small samples of the soil to insure that all of the material would pass the U.S. No. 40 sieve. Processed soils, totaling approximately 400 lbs for each material, were stored in large, heavy-duty plastic bags.

Physical and Engineering Properties

Index properties of all soils used in this study, as well as the results of grain size analyses, are presented in Table III; the maximum dry density, optimum moisture content (OMC), and soil permeability and strength at OMC are also reported. As can be seen, the four selected soils have quite different physical characteristics. Although the textures of the chosen soils differ noticeably, the same processing procedure was used for all of them. It must be emphasized that none of these soils was selected on the basis of the properties shown in Table

TABLE III

PHYSICAL AND ENGINEERING PROPERTIES

Soil	Specific Gravity	Natural Water Content	Liquid Limit	Plastic Limit	Shrinkage Limit	Plasticity Index	Activity Number	Grain Size Analyses (%)			Compaction		Permea-bility Kav cm/sec at OMC	U/C Strength tsf at OMC
								Sand +.06mm	Silt -.06mm	Clay -.002mm	γd pcf	w		
Burleson Soil	2.70	36.02	66.0	23.8	11.22	41.2	.66	6	32.0	62.0	88.4	24.75	3.8×10^{-9}	4.64
Summit Soil	2.74	25.80	55.5	21.3	12.33	34.2	.62	8	36.5	55.5	101.9	20.30	4.5×10^{-9}	3.72
Union City Clay	2.78	22.40	43.3	25.5	16.96	16.8	.22	0	24.0	76.0	103.2	22.00	1.6×10^{-8}	3.09
Camargo Clay	2.82	71.20	132.7	69.9	15.89	62.8	1.50	8	50.0	42.0	--	--	9.0×10^{-9}	1.74

TABLE IV

CHEMICAL ANALYSIS

Soil	pH	Organic Content	Leaching Test ppm				Relative Sulfur Content ppm	CEC meq/100g	Total Cation meq/100g	Total Chemical Analyses (s)						
			K^+	Na^{++}	Ca^{++}	Mg^{++}				SiO_2%	Al_2O_3%	FeO%	CaO%	MgO%	Na_2O%	K_2O%
Burleson Soil	8.17	1.45	295	783	8438	345	17	39.4	49.2	75.07	15.45(.23)	2.80(.04)	3.47(.20)	1.05(.03)	.55(.01)	1.61(.04)
Summit Soil	7.75	0.62	163	65	4960	300	30	21.1	28.0	71.40	16.49(.14)	3.11(.06)	4.77(.22)	1.15(.05)	.77(.02)	2.31(.16)
Union City Clay	8.31	0.09	423	660	2890	645	395	21.1	23.8	63.09	20.34(.11)	4.12(.03)	3.12(.15)	2.71(.08)	1.20(.01)	5.42(.10)
Camargo Clay	7.50	0.00	80	90	10313	1225	14	96.7	62.4	68.93	19.16(.76)	2.02(.03)	5.20(.17)	4.26(.02)	.17(.01)	.26(.01)

III: however, the range of variation in these properties enhances the value of this study. The Oklahoma State University Miniature Impact Compaction Device was used to prepare all compacted samples used in this study. This device is a miniature mechanical impact compactor which uses a freely-falling hammer lifted by a rack-and-pinion gear arrangement. Application of the standard Proctor compactive effort (in terms of energy per unit volume) to a Harvard miniature size compaction sample is accomplished by allowing the hammer to drop 4.0 in. on each of the four layers using 19 blows per layer. For more information about the Oklahoma State University miniature compaction device, see Reference (66).

Chemical and Mineralogical Analyses

The results of chemical analyses of the four soils are shown in Table IV. The total chemical analyses were conducted on six samples of each soil. The table shows the mean value of the result and, in parentheses, the standard deviation, s. The leaching, cation exchange capacity, and organic content were determined in the manner recommended by Jackson (39); the pH value of the natural soil was determined by the Eades and Grim method (20). It may be seen in Table IV that all soils contain a rather high calcium content. The X-ray charts (Figures 6 - 9) indicate the type of clay mineral in each soil. The equipment employed throughout this study were: (1) Sergeant-Welch Model NX pH meter, (2) Atomic Absorption Spectrophotometer (Parkin-Elmer 40), and (3) Norelco X-ray unit by Phillips, Inc., type 120 45 B/3. All tests were conducted on fully representative samples of the soils.

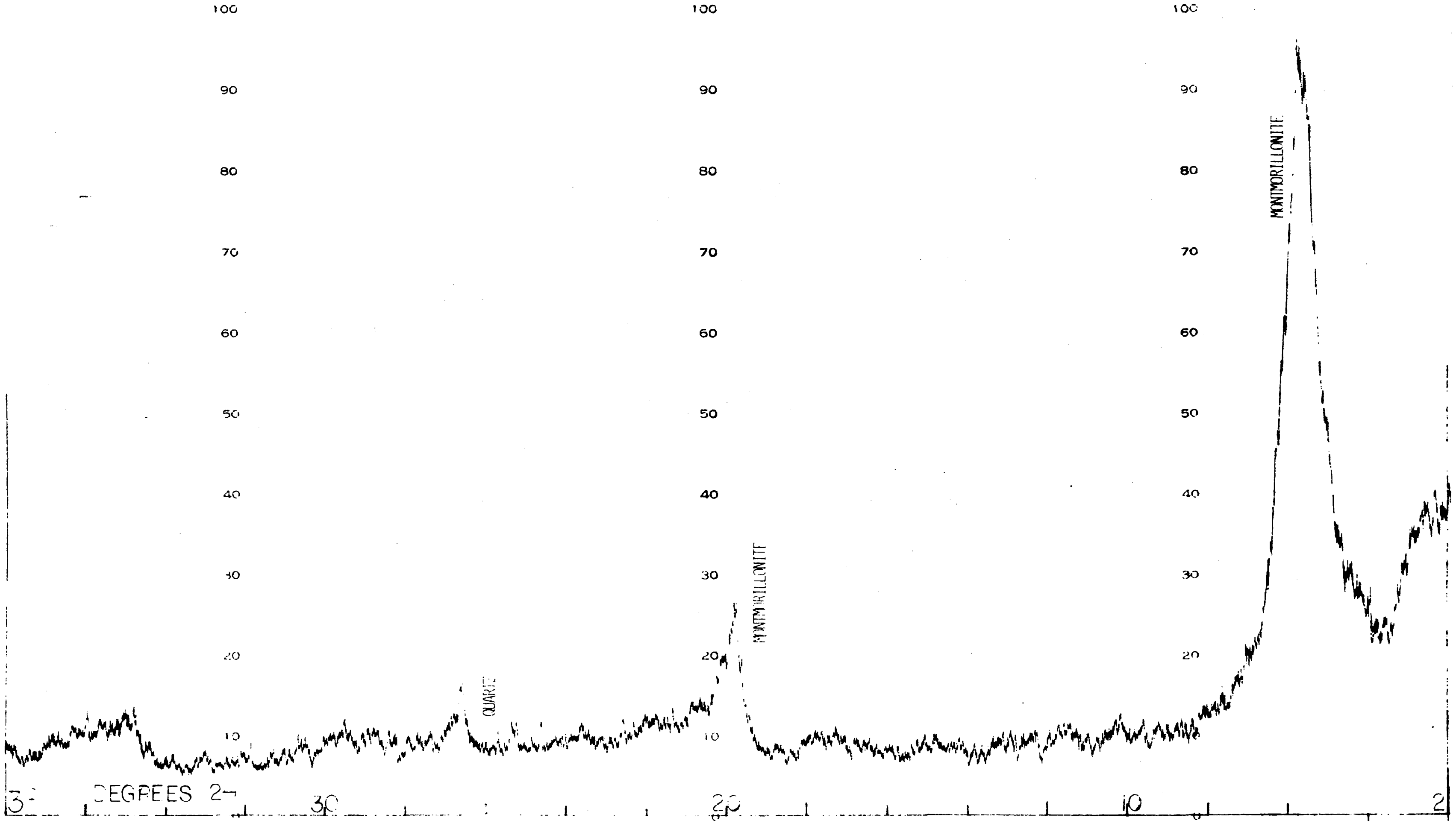


Figure 6. X-Ray Diffraction Pattern for Camargo Clay

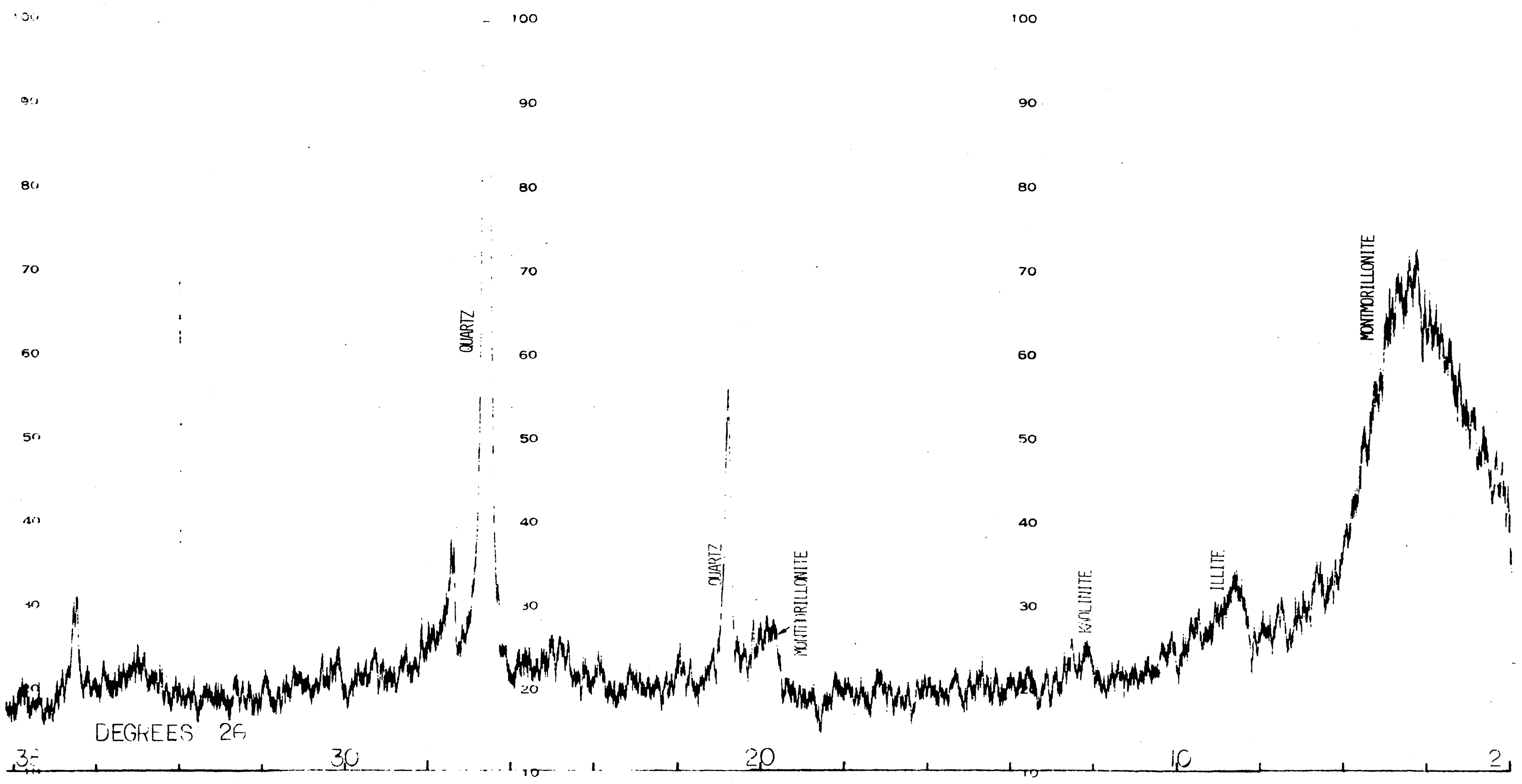


Figure 7. X-Ray Diffraction Pattern for Burleson Soil

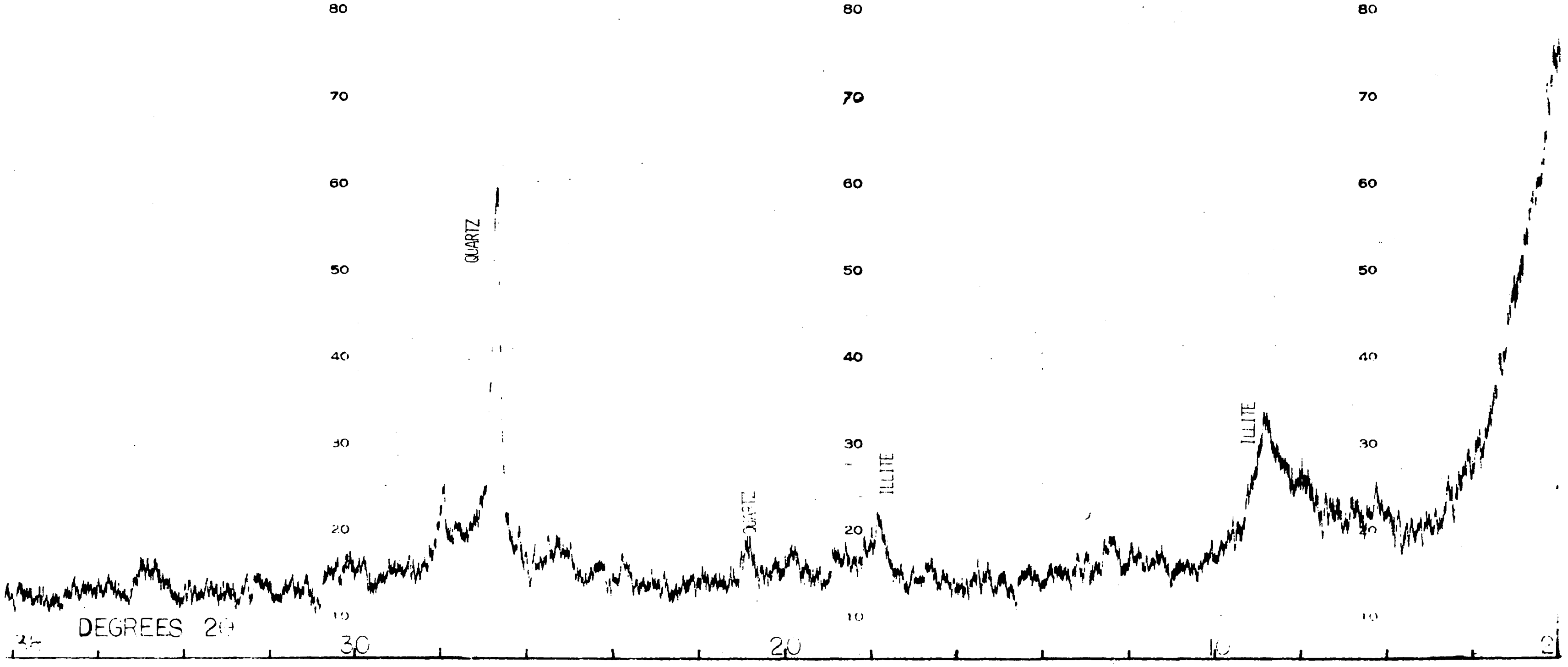


Figure 8. X-Ray Diffraction Pattern for Union City Clay

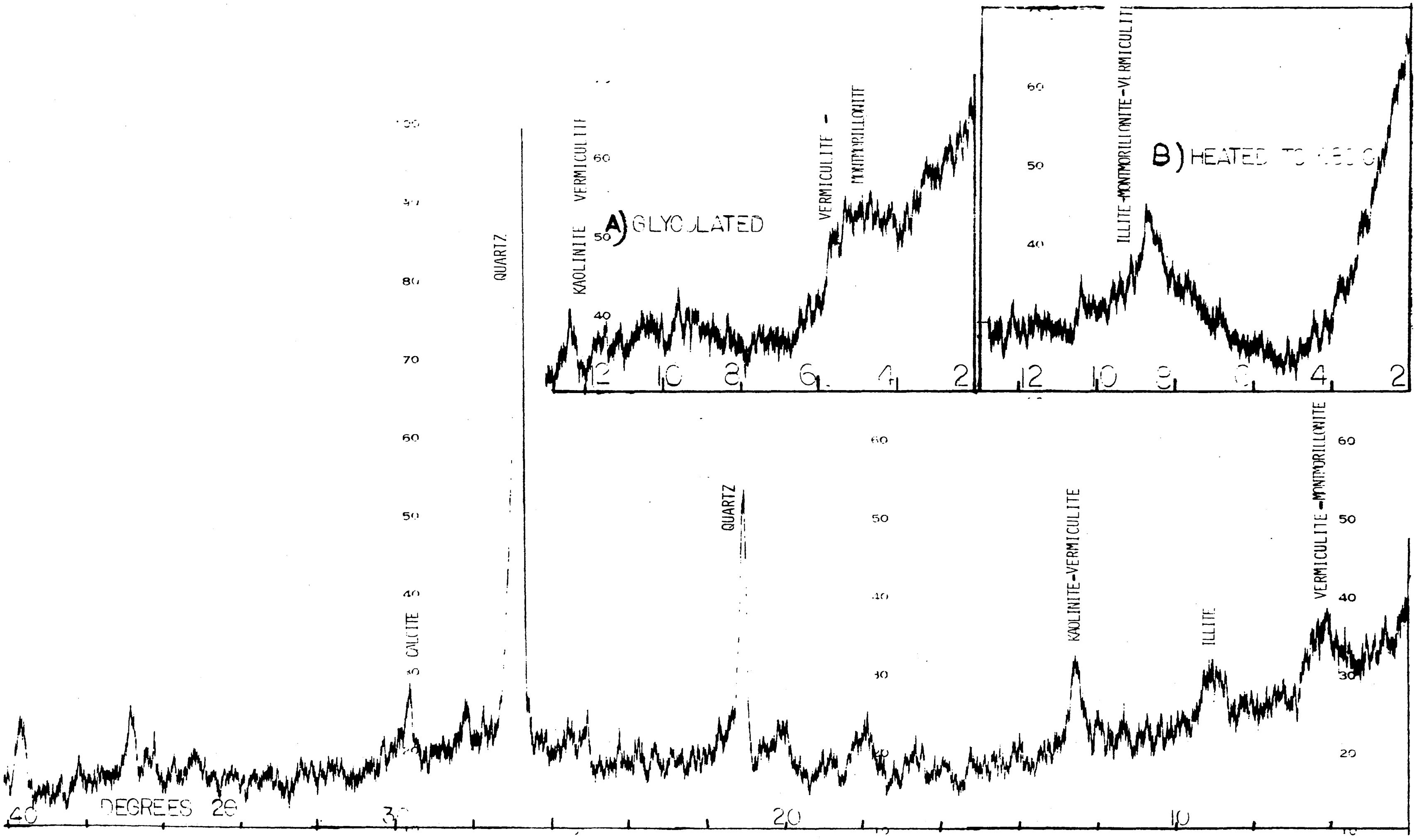


Figure 9. X-Ray Diffraction Pattern for Summit Soil

Lime

The lime used in this study was finely ground calcium hydroxide from the Fisher Scientific Company. Slurried lime was mixed with the soil to reduce carbonation and to simulate commonly used construction procedures. The bulk lime container was kept tightly closed and small amounts of lime were placed in sealed, dark glass bottles for immediate use. The chemical analysis of the calcium hydroxide (as given by the manufacturer) is shown in Table V.

TABLE V

CHEMICAL ANALYSIS FOR LIME

$Ca(OH_2)$	F.W. 74.09
Chloride (Cl)	0.005%
Iron (Fe)	0.040%
Sulfur compounds (as So_4)	0.020%
Other heavy metals (as Pb)	0.001%
Magnesium alkali salts	0.800%
Insoluble in HCL	0.030%

Water

The water used throughout the study was distilled, demineralized water. A Barnstead Model DO 803 steam-powered distillation unit produced distilled water for storage in a tin-lined tank. The water is drawn through a Bantam demineralizer unit not more than ten hours prior to use. The pH of the water was between 6.5 and 7.0.

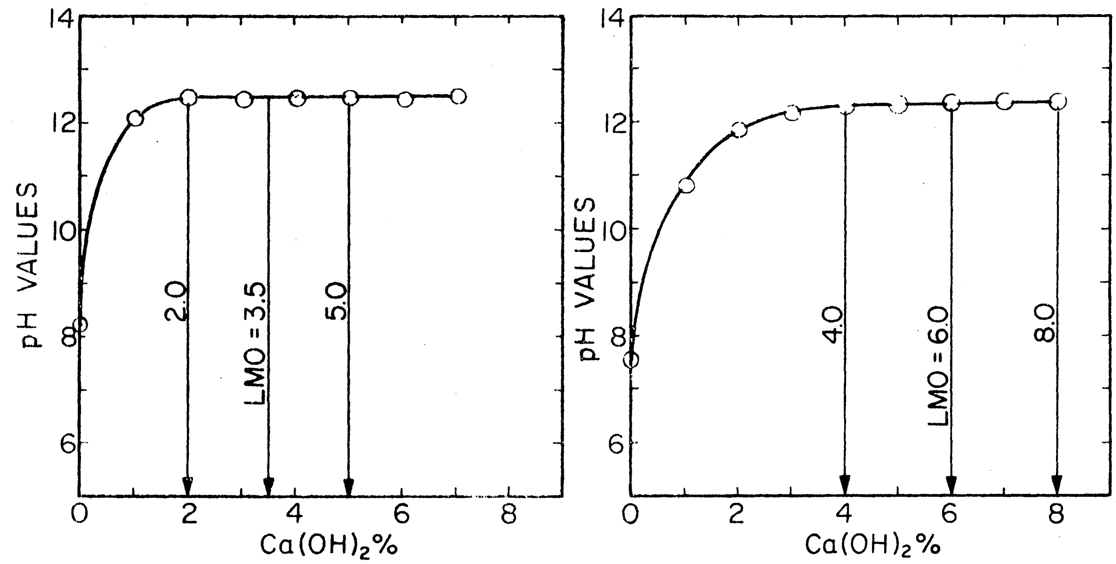
Sample Preparation

Standard procedures for sample preparation and testing were adopted for use in this study. The procedures were found to provide excellent control of moisture content and to insure a high degree of homogeneity in test specimens. For guidance in designing the soil-lime mixes that were to be tested, the relationship between lime content and pH for each soil was determined and the lime modification optimum was found using the method suggested in Reference (20). These results are given in Figure 10.

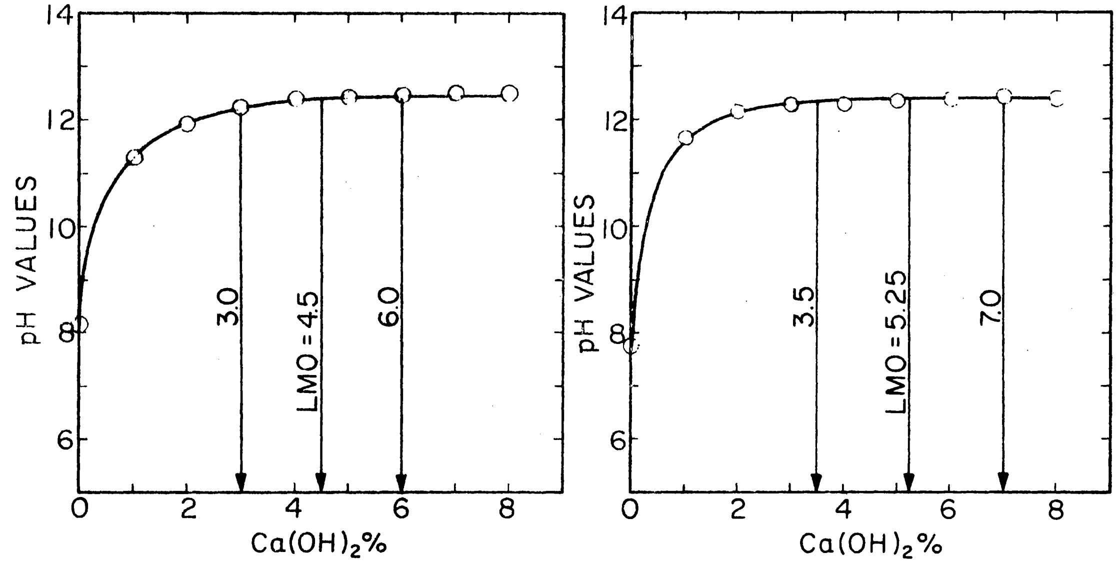
Compacted Specimens

The compaction curve (moisture-density relationship) was obtained for the untreated soil and for the treated soil for each of the three different percentages of lime selected for study (see Figure 11). The procedure used in preparing all compacted specimens of lime-treated soil is as follows:

1. Oven-dry all materials at 100°C for 24 hours;
2. Weigh out 100 g of soil (± 0.1g) and place in a plastic bag;
3. Weigh out the required amount of lime to the nearest 0.01 g and place it in a beaker;
4. Add to the lime the amount of water required to produce the design moisture content in the soil-lime mixtures, and stir gently to produce a smooth slurry;
5. Add the slurry to the dry soil in the plastic bag;



(a) UNION CITY ILLITE (b) CAMARGO BENTONITE



(c) BURLESON SOIL (d) SUMMIT SOIL

Figure 10. Variation of pH Values With Lime Content

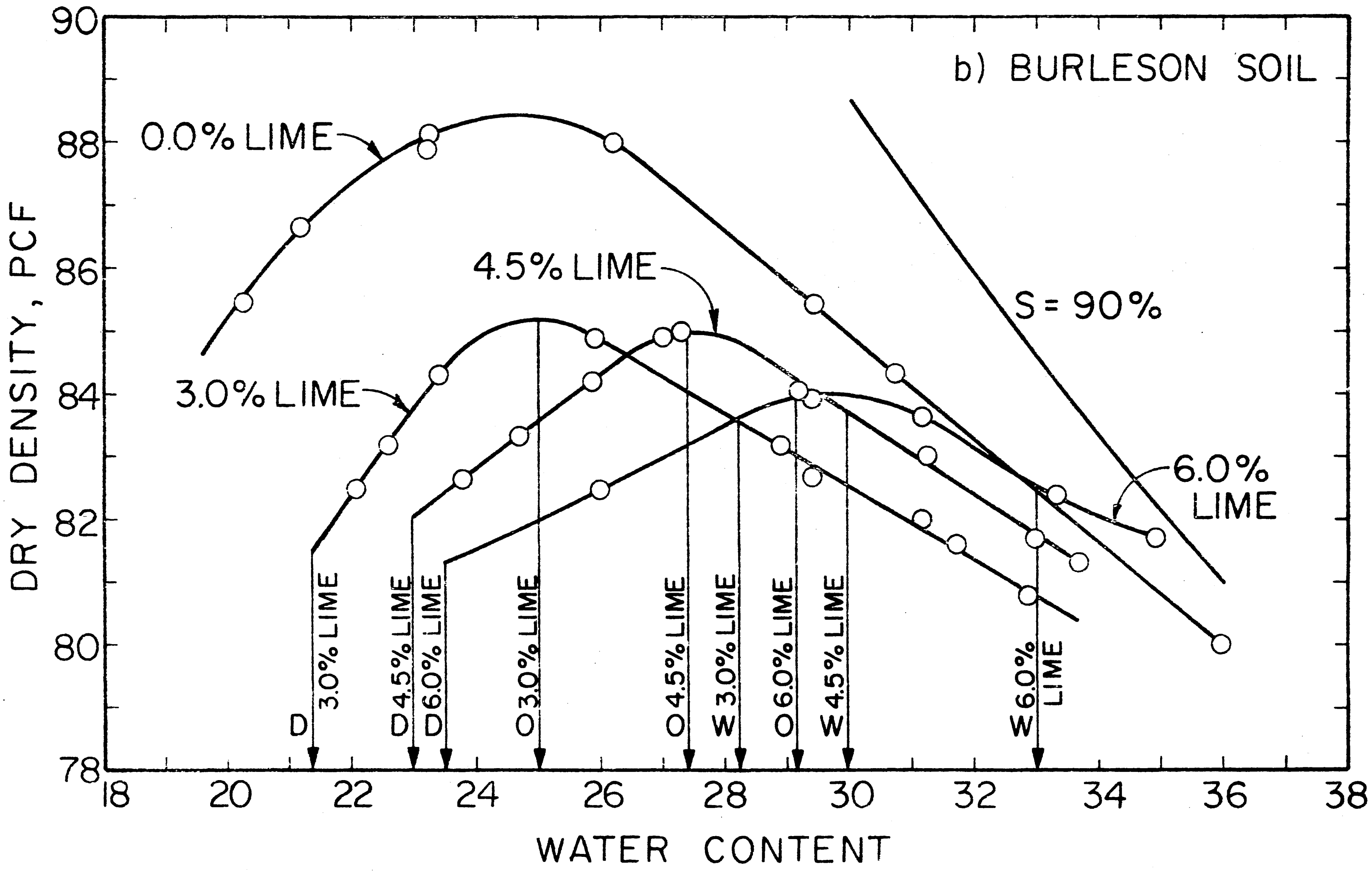


Figure 11. Continued

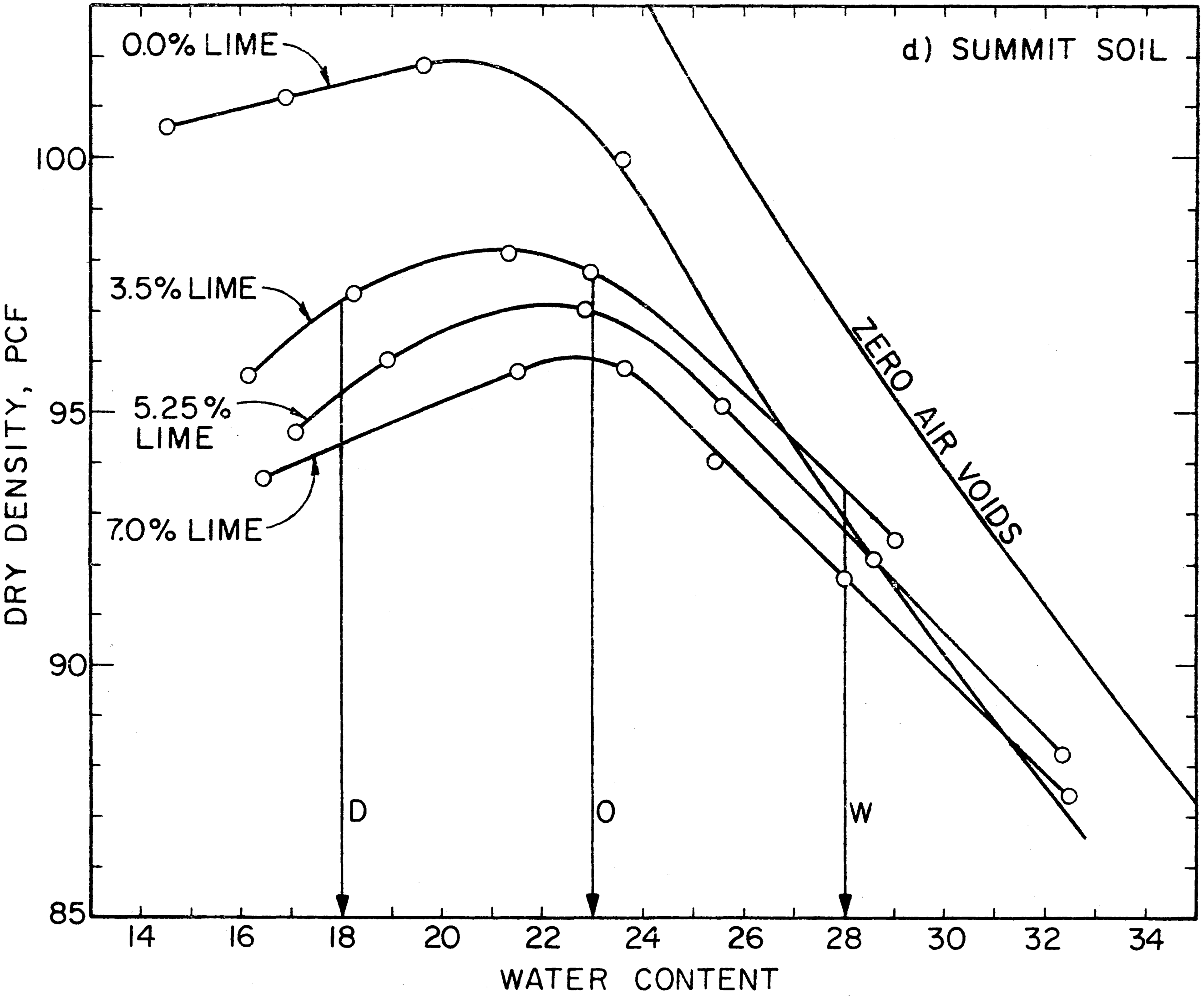


Figure 11. Continued

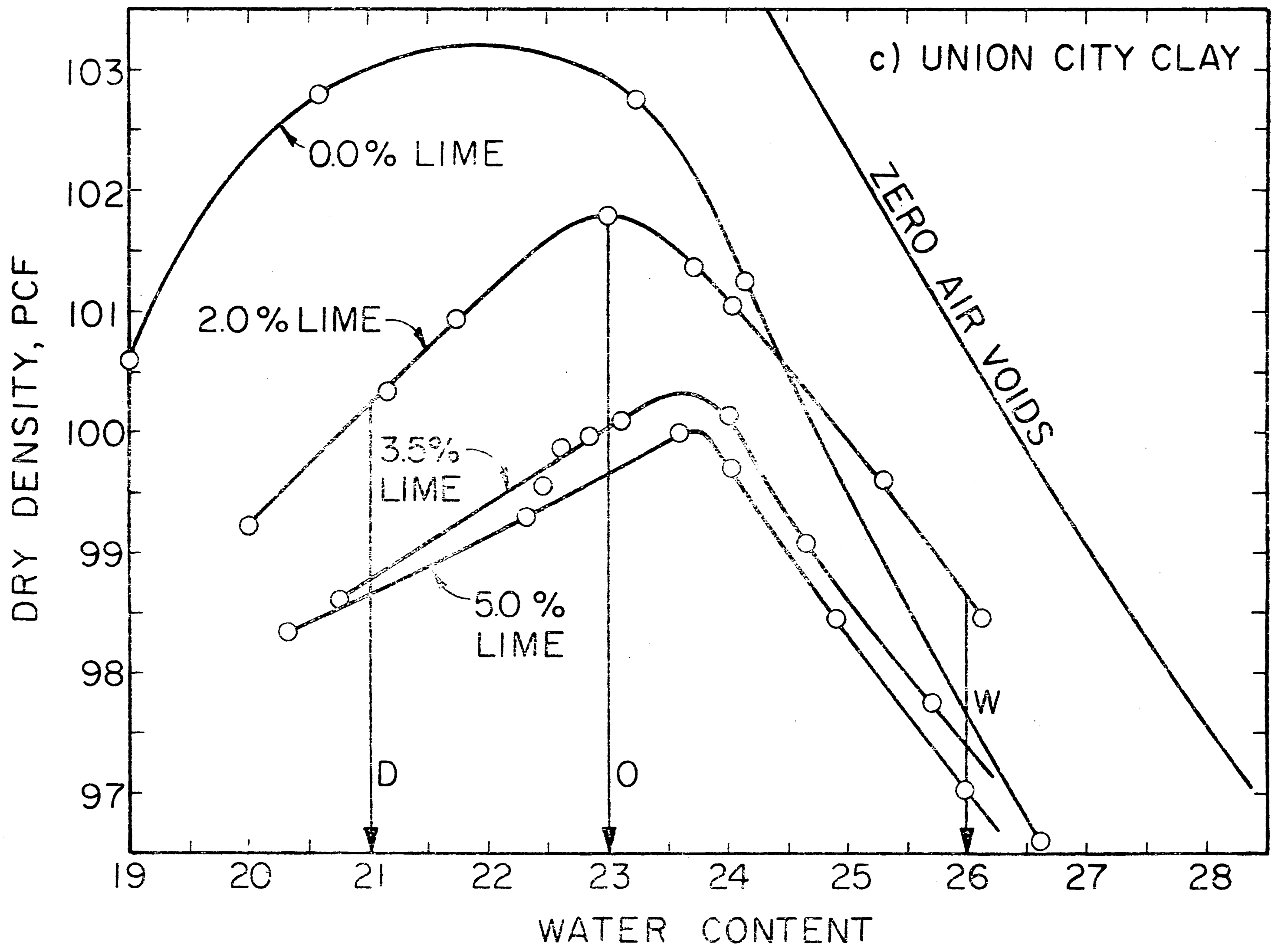


Figure 11. Continued

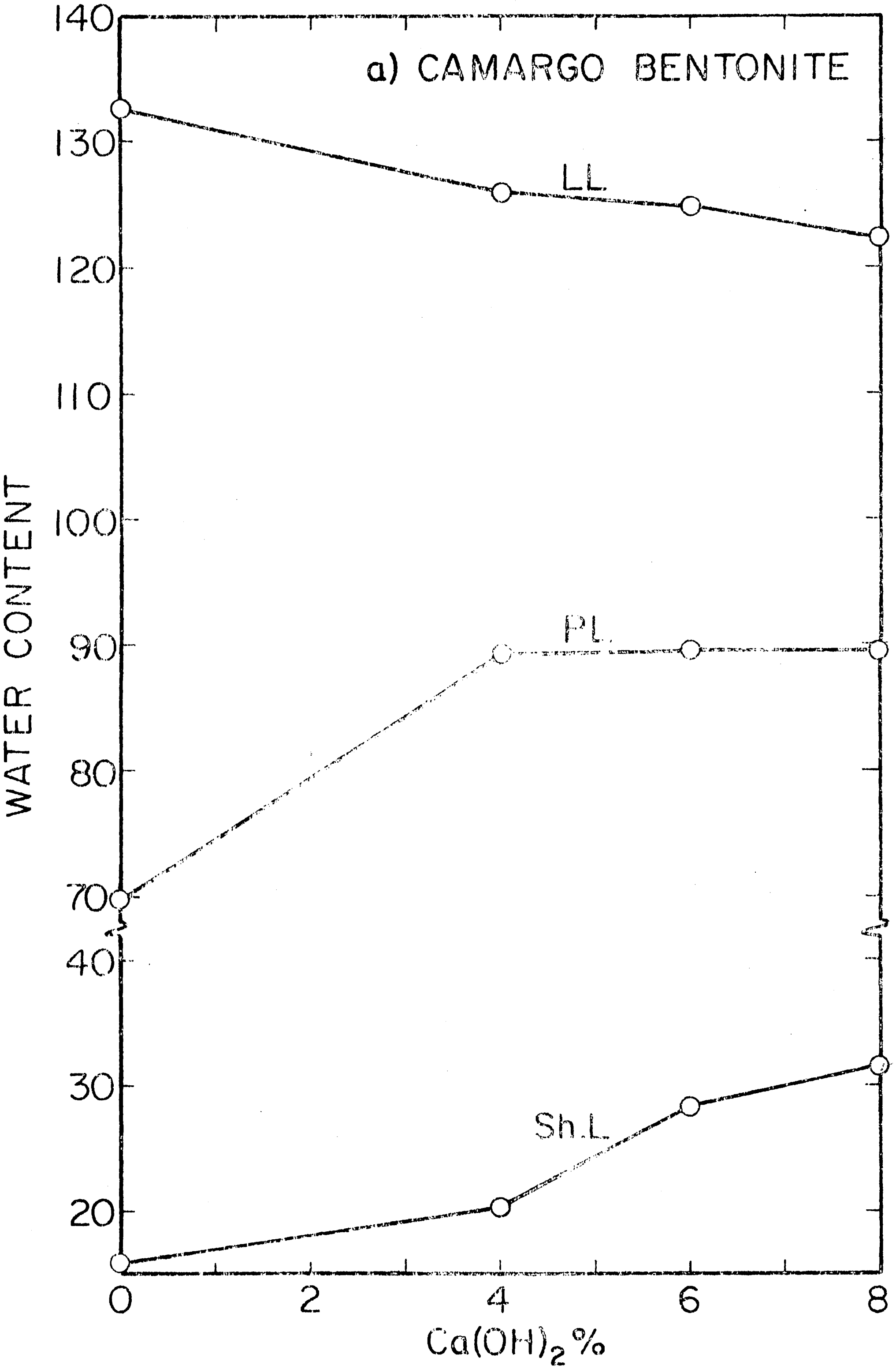


Figure 12. Effect of Lime Treatment on Atterberg Limits

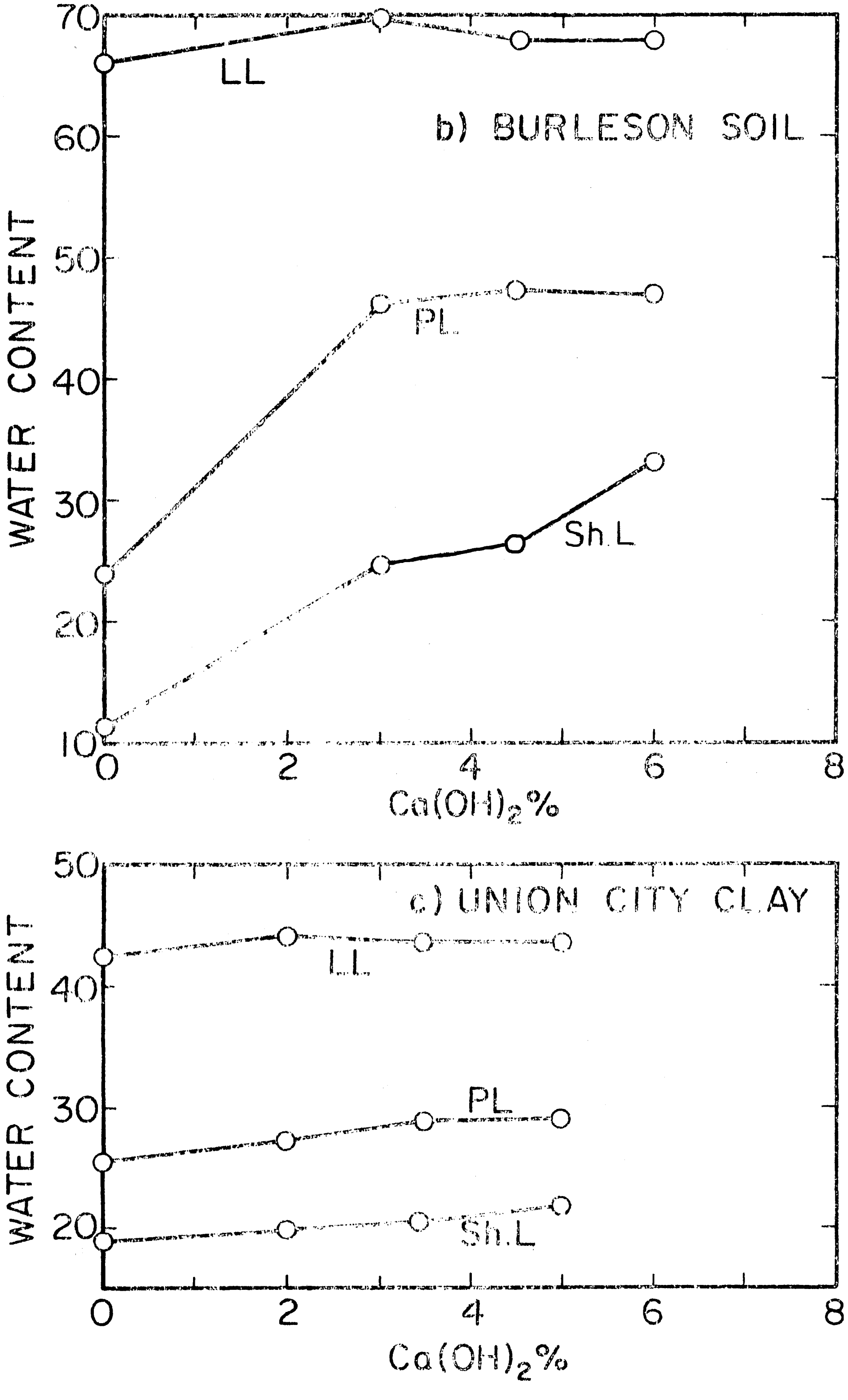


Figure 12. Continued

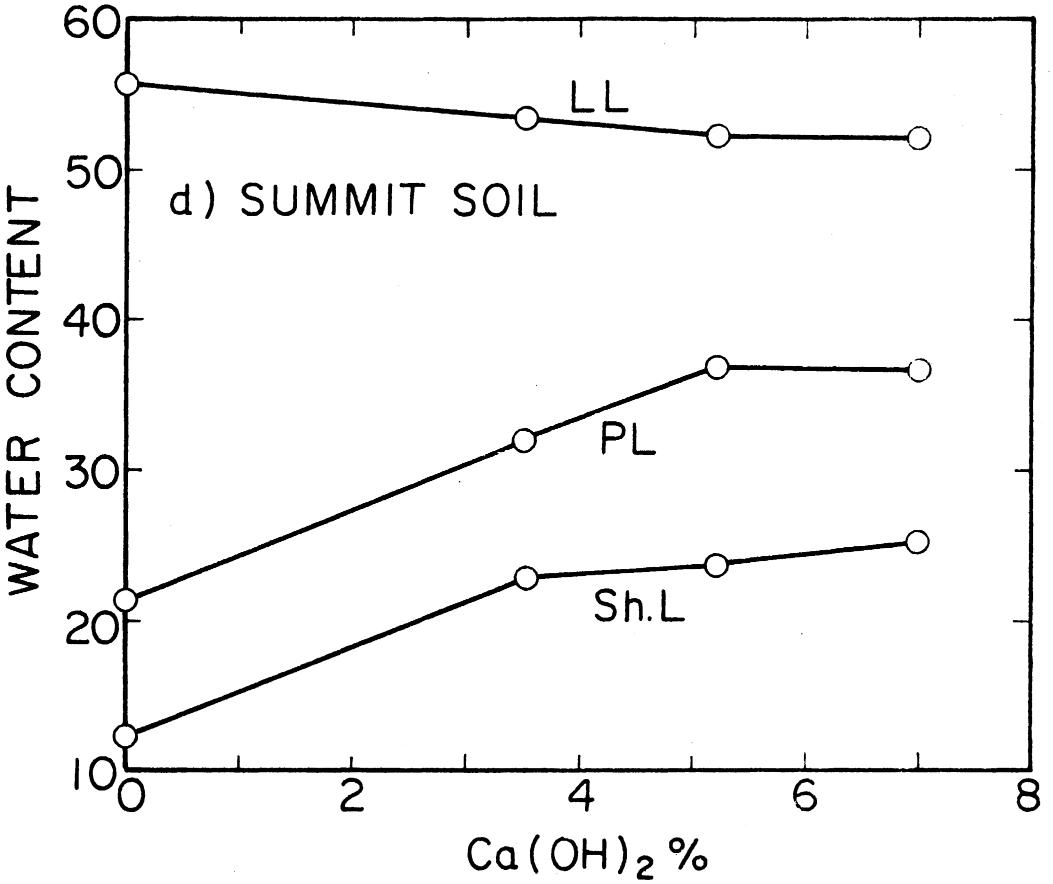


Figure 12. Continued

6. Mix the soil and slurry by continuously kneading the bag until the mixture is homogeneous;

7. Tie the bag closely in such a way as to exclude air;

8. Allow the mixture to mellow for one hour prior to compaction;

9. Compact the sample in the Harvard Miniature Apparatus, using an impact procedure designed and calibrated to produce a standard proctor density;

10. Determine the wet and dry weight of the sample, and compute the moisture content;

11. Repeat the procedure for each data point of the compaction curve.

Atterberg Limits Samples

Soil samples for determining the Liquid, Plastic, and Shrinkage Limits of the soil-lime mixtures were prepared following steps 1 through 8 above. These results are given in Figure 12.

Replicates

To provide some statistical validity of test results, the procedures described for determining the compaction characteristics and Atterberg Limits of soil-lime mixes were repeated twice to provide three replications of the data.

Untreated Soil

The compaction characteristics and Atterberg Limits of the natural, untreated soils were determined using ASTM Standard Procedures, generally without replication.

Strength Test Specimens

The unconfined compression test was selected as the most appropriate method for evaluating the results obtained by varying the lime content, moisture content, and curing conditions. In preparing the test specimens, steps 1 through 9 given previously were followed except that 220 g of soil were used rather than 110 g. From that point the procedure was continued as indicated by the following steps:

12. Immediately upon extraction from the mold, wrap the specimen in Saran Wrap and label it.

13. Apply a double coat of microcrystalline wax (Mobil Oil Corporation M-498-AS) to the wrapped specimen.

14. Place the waxed specimen in a glass jar that is one-third filled with saturated sand and screw the lid on tightly.

15. Submerge the jar in a water bath maintained at a temperature of about 40°C, where it will remain during the desired curing period. The sand provides a humid environment inside the jar and its weight reduces the buoyancy. Jars should be checked occasionally to make sure that no water is leaking into them from the water bath.

pH Test Samples

To gain additional information concerning the nature of the soil-lime reaction process in the specimens prepared for strength tests, samples for pH tests were prepared concurrently with those for strength tests. The pH test samples were prepared, compacted, and cured in exactly the same manner as the strength test specimens, except that abbreviated samples using only 20 g of dry soil were made. The compaction procedure was adjusted to provide the same compaction energy per unit volume as was used for the full-size specimens. At the time each strength test specimen was tested in unconfined compression, the pH of the corresponding pH test sample was determined. Since the samples had been compacted, it was necessary to pulverize them in order to prepare the soil suspension for the pH test. The suspension, using 100 ml of water, was mixed for one minute in a blender and then placed on a shaker for one hour prior to determining the pH.

Mineralogical Test Specimens

The specimens to be used in X-ray diffractometer and electron microscopy studies were compacted discs containing 5 g of dry soil and were prepared concurrently with the strength and pH test samples previously described. The same mode and unit energy of compaction were employed in an attempt to obtain about the same unit weights as those of the corresponding strength test specimens; identical curing procedures were followed. The use of soil discs, rather than a suspension dried on a glass slide, in the X-ray diffractometer has not been reported in any of the references studied. However, it offers several advantages over the slide procedure, at least for this application. In particular, the specimen analyzed by X-ray diffractometry could also be examined directly in a scanning electron microscope.

Permeability Test Specimens

Compacted specimens for the permeability tests were cured in the compaction molds, which also served as the soil chambers for the permeability tests. A limited number of samples was prepared using compaction procedures previously described. The test series consisted only of mixtures having a lime content corresponding to the Lime Modification Optimum content (LMO), and all samples were compacted at the Optimum Moisture Content. All specimens were cured for 30 days. In one group of specimens additional water was applied to the mixture after compaction and before curing to duplicate the curing conditions used in some of the strength test groups. In this case half of the added water was sprayed on the surfaces of the soil lifts in the compaction mold; the other half was sprayed onto the end surfaces before the specimens were wrapped and sealed for curing. This procedure did not result in any greater sample densities than would otherwise have been present.

Durability Test Specimens

Compacted specimens for the durability tests were prepared in the manner described for strength test specimens. However, the same lime content, corresponding to the LMO, was used for all samples and all were cured for 30 days prior to testing.

Testing Program

Some of the simpler tests, such as determining the Atterberg Limits, are so well understood that it is not necessary to give details beyond those given under the preceding section, Sample Preparation. In this section details will be given for those tests not widely understood or for which more or less arbitrary procedures were adopted in this research. In addition, certain tests embraced extensive and complex variations of the principal influential factors--lime content, moisture content, and curing time--and these variations must be clearly described.

Strength Tests

A total of 720 compacted specimens were tested--180 for each of the four soils used in the study. Out of each group of 180, 60 specimens were prepared for each of three different lime contents, corresponding to the LMO and to the LMO ±2%.

Each group of 60 specimens at a given lime content was further subgrouped as follows:

(a) 24 "O" specimens compacted at optimum moisture content;

(b) 24 "dry-side" or "D" specimens compacted at a moisture content of approximately 0.8 x OMC; and

(c) 12 "wet-side" or "W" specimens compacted at a moisture content of approximately 1.2 x OMC.

Groups (a) and (b) were then divided further into groups of 12. In one of these groups the specimens were sealed and cured at the moisture contents used during compaction, while the moisture contents of the other groups were increased, prior to sealing and curing, by spraying onto their surfaces an amount of water equal to the difference in the amount used in compacting the group (c) specimens and that used for compacting the O or D group to which the specimens belonged. The shorthand notations adopted for these groups in various figures and tables are D→W and O→W, respectively, for D and O specimens sprayed with water to bring them to moisture contents equal to those of the W specimens.

The foregoing procedure resulted in 5 groups of 12 specimens, in each of which some differences in character or mineralogy might be anticipated. Each group of 12 is then finally divided into 4 groups of 3, corresponding to the 4 different curing periods of 2, 10, 30, and 90 days selected for this study.

Mineralogical Tests

X-ray diffractometer data were obtained for the intact compacted disc samples using a Norelco Model 120 54B/3 X-ray unit. Selected discs were also microscopically examined and photographed using a scanning electron microscope Model JEOLCO JSM-2.

Permeability Tests

The coefficient of permeability of compacted specimens was obtained from a constant head permeability test conducted in a Soil Test Model K620 permeameter. To produce the relatively high heads needed to cause measurable flow through soils of low permeability, a constant air pressure of 50 psi was maintained above the water in the water supply reservoir. Through a manifold connected to the reservoir it was possible to conduct several tests simultaneously. Data pertaining to the leaching of calcium from treated soils were also obtained from these tests by analyzing the collected effluents.

Durability Tests

The durability of lime-treated soils alternately frozen and thawed was evaluated on the basis of the resulting reduction in unconfined compressive strength. Specimens were subjected to three freeze-thaw cycles in which they were frozen for 16 hours at 0°F, and thawed for 8 hours at 78°F. At the end of three cycles, the unconfined compression test was conducted as previously described.

In summary, for each soil type there were 60 sets of 3 specimens each (replicates), each set differing in some respect from every other set in order to provide the desired combinations of the four variables: lime content, compaction moisture content, alteration of ambient moisture during curing, and curing period.

The 180 test specimens for a given soil were generally prepared during a three-day period, at the rate of 60 per day, and in no case did the procedure take more than four days. The 60 specimens that were prepared in one day, more or less, contained no replicates; that is, one specimen from each of the 60 sets described above was prepared in each day's run. Moreover, to avoid the bias that might result from following a fixed pattern of preparation, the order in which the 60 specimens were made was selected randomly using random number tables.

After it had cured for its designated period, each specimen was tested in unconfined compression using a Karol-Warner Inc. Model 550 compression test machine. Load was applied at a constant rate of deformation, 0.014 in. per minute, with load and deformation being recorded at intervals until failure occurred. The final moisture content was then determined.

pH Tests

Eades and Grim's method (20) was followed generally in determining the pH of the treated compacted and cured soil samples, with minor procedural alterations (described under Sample Preparation) required to prepare a proper suspension from a hard and compact disc of soil. This procedure is described fully in Appendix A.

Concluding Remarks

The described testing program was designed specifically for the study of compacted lime-treated soils, using mix proportions that are economically feasible in highway construction, procedures that simulate those that are (or may be) used in field work, and ambient moisture and temperature conditions that may be present or practicable in the field. The slightly elevated curing temperature of 40°C used in this study is perhaps only occasionally present during field curing; however, it permitted the study to progress more rapidly and caused no soil-lime reactions that would not have occurred at a lower temperature.

CHAPTER IV

ENGINEERING PROPERTIES OF SOIL-LIME MIXTURES

The unconfined compression test is a rapid and a simple means of evaluating the shear strength of clayey soils. The main precaution that must be taken is to minimize the loss of moisture during the course of the test. In order to do so the test should be conducted as rapidly as possible without sacrificing the accuracy of the results. The rate of loading selected was 0.014 in./min, which is equivalent to 5% strain within 10 minutes. The weight of each specimen was recorded before the test and compared with its weight before curing. Throughout the testing program the weight was found to differ not more than 1/2 g from the original weight. Immediately after failure in unconfined compression, the entire test specimen was used to evaluate the final water content. This moisture content was found to be within ±1.0% of the designed water content.

All samples were tested as soon as possible after the design curing time had elapsed. Samples cured for 2 days were tested within 51 hours after compaction. Samples cured for 10, 30, and 90 days were tested within 9 hours after the curing period had elapsed.

In the following section, strength data and discussion of these data will be presented for the four tested soils. It is clear from Chapter III that the four soils differ greatly in dry density as well as design water content. Only qualitative comparisons of soil strength will be presented among the tested soils.

Camargo Soil

Analyses of strength data obtained after 2 days of curing for the treated clay reveals a very interesting behavior (Figure 13). Maximum strength gains were obtained from samples compacted wet of optimum. This is true regardless of lime percentage and curing time; however, the difference between the W and O samples is proportional to the lime percent. This result indicates that samples compacted at a higher water content produce higher strength in spite of their lower dry densities. It is also significant that samples designated O→W and D→W cured for more than 10 days had strengths higher than those of O and D samples but always lower than those of W samples. The strength increase was much higher in D→W samples than in O→W samples. It seems that the availability of water and lime are the principal factors governing strength gain. It is evident that W samples always had strengths higher than those of D and O samples, regardless of the curing time.

The strength improvement of the O→W and D→W samples varies with curing time and lime content. The final strength after 90 days of curing for the Camargo soil indicates that W samples maintain their superiority in strength over all other samples. The D→W and the O→W samples exhibit a very high rate of strength gain between 6% to 8% lime content where the strength of D and O samples had begun to level off, which indicates that the latter samples had very nearly attained a maximum strength that would preclude any benefits from a further increase in lime content. However, it is clear from Figure 13 that soil strength

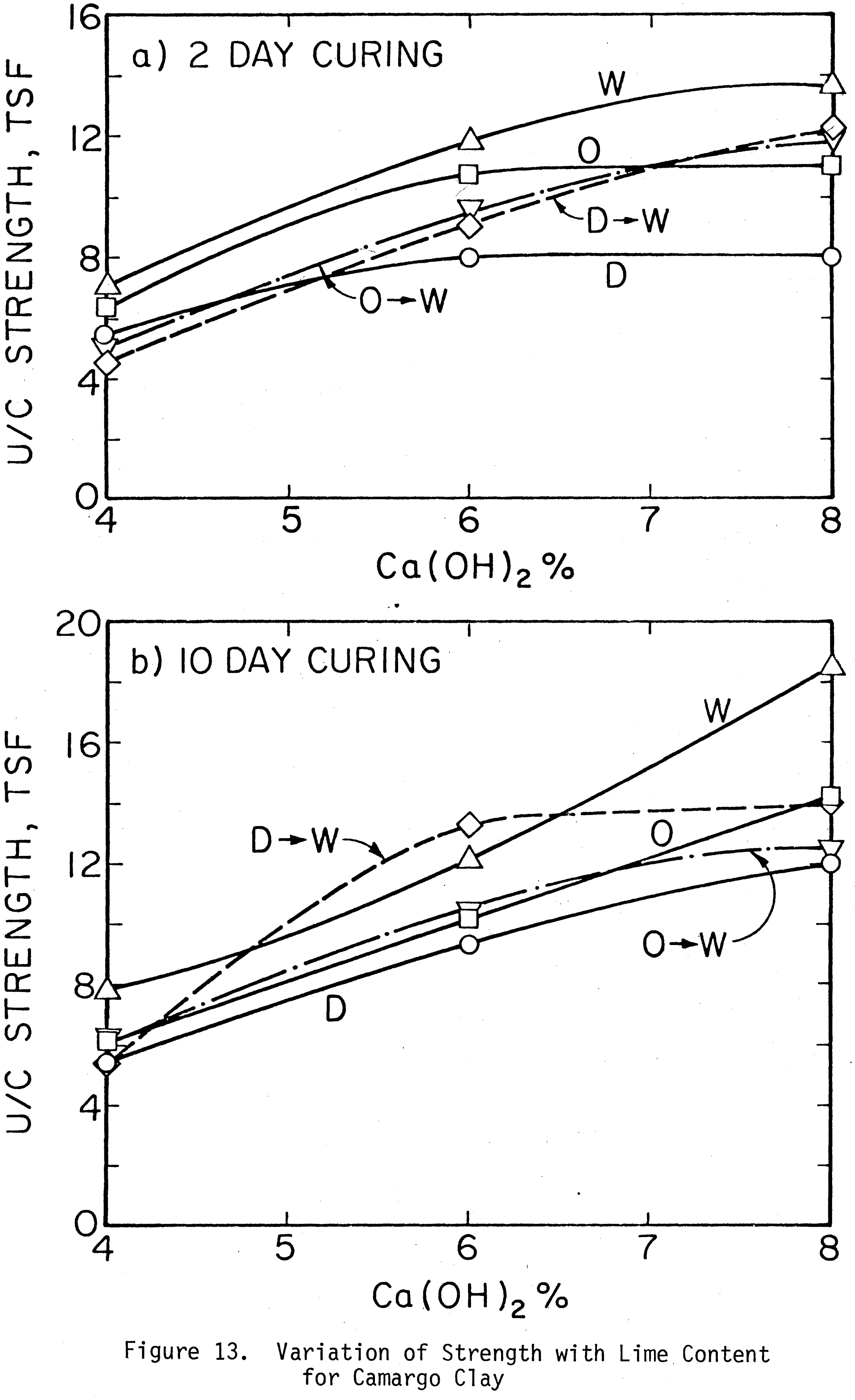


Figure 13. Variation of Strength with Lime Content for Camargo Clay

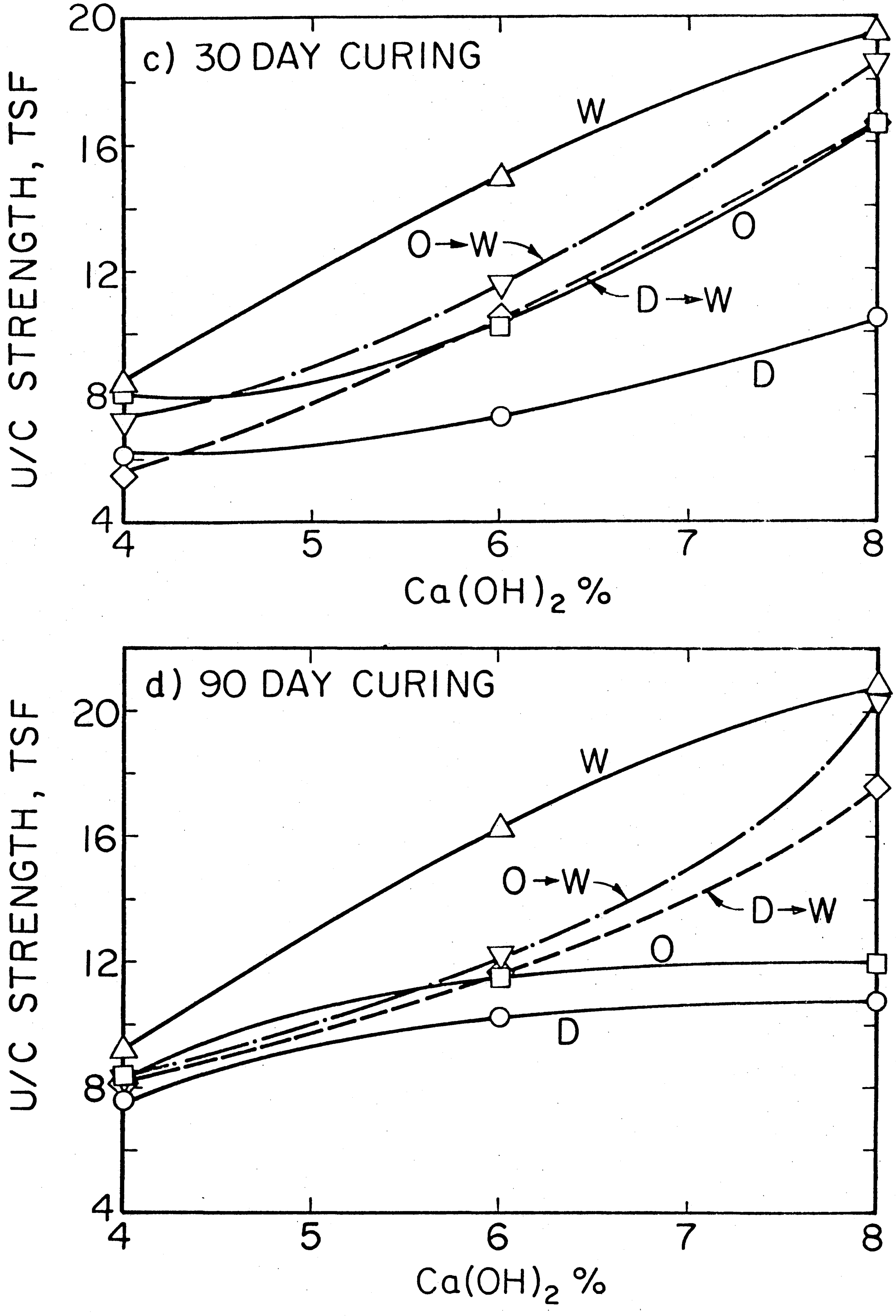


Figure 13. Continued

is directly proportional to the lime content, without any exceptions, for the range of lime contents investigated.

The effect of curing time on samples treated with different lime contents is presented in Figure 14. The first thing to notice in these curves is the relatively high strength of the W samples. In the case of 4% lime content it is clear that the post-compaction addition of water to the D and O specimens to produce the D→W and O→W groups was of no benefit.

After 90 days of curing, D→W specimens were slightly stronger than the D samples. On the other hand, the slopes of the lines connecting the strength at 30 and 90 days of curing for O and D samples are much flatter than those of the O→W and D→W groups. The steeper slopes of these latter lines leave some hope that the O→W and D→W specimens may have strengths superior to those of the O and D samples in the long run, perhaps exceeding D and O specimens that contain 30% more lime. Different behavior was observed in samples containing 6% lime (the LMO): (a) the O→W and the D→W samples exhibit strengths greater than those of the O and D samples for a short curing time, and (b) a drop in stength after 30 days of curing is observed for the D and D→W samples, which indicates that some distinctive phenomena take place in samples compacted dry of optimum. The strength loss could be a result of a change of the cement phase (to be discussed later in the section). At a higher lime content the strength superiority of D→W and O→W samples over D and O samples is quite pronounced. Surprisingly, the strength of O→W samples is almost as great as that of W samples, after 30 days of curing.

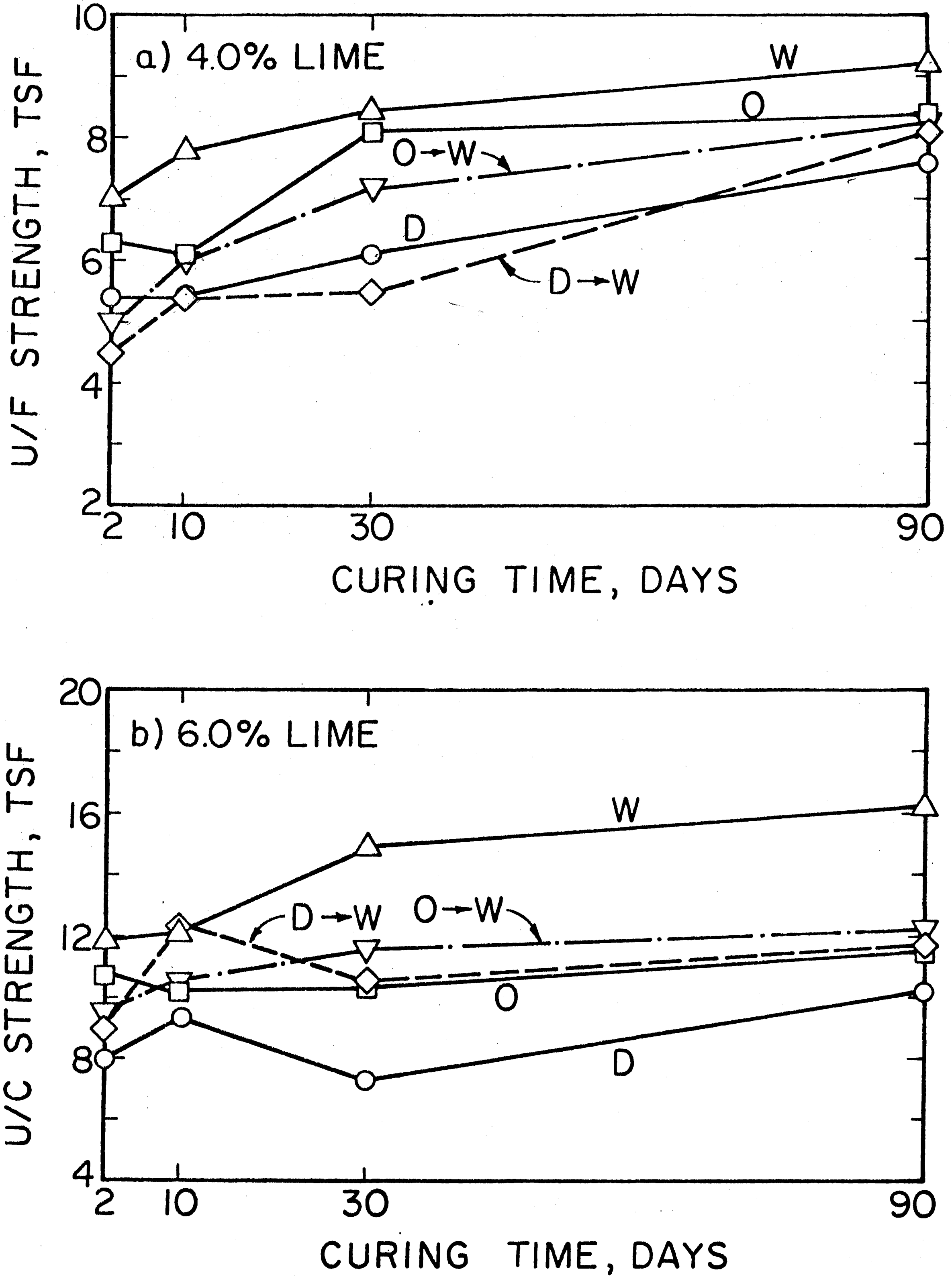


Figure 14. Variation of Strength with Curing Time for Camargo Clay

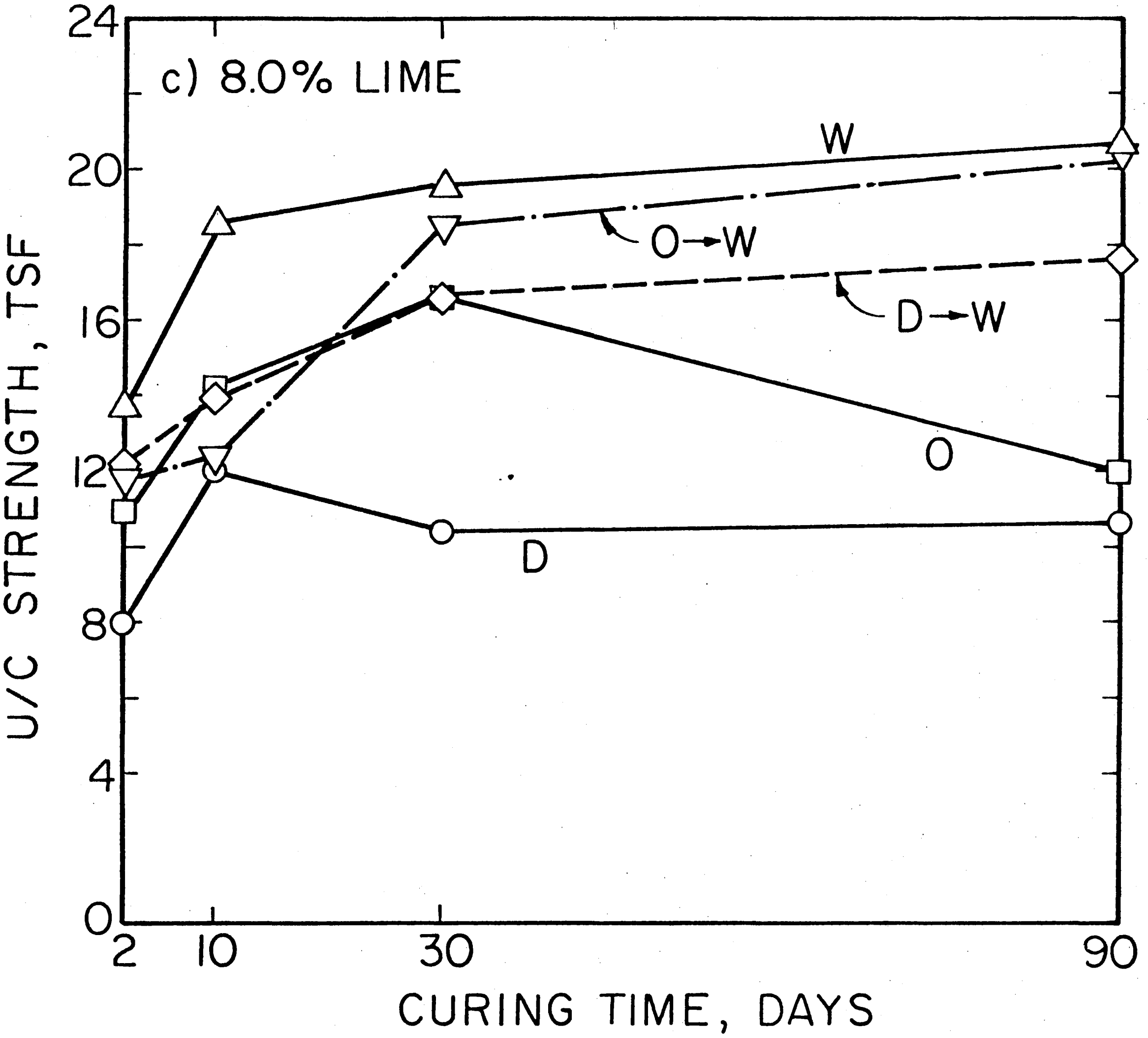


Figure 14. Continued

The Camargo clay samples show several interesting characteristics. For example, the strength superiority of W over O specimens and of O over D specimens is absolutely consistent for all lime contents and all curing periods investigated. Except for the lowest lime content used (4%), it is also observed that soil strength is improved by the addition of water after compaction. These results appear contradictory to what is generally believed, in that samples having the lower dry densities and higher moisture contents were the strongest. It is well known that adding water to a compacted soil will generally weaken it, but in this case the water appears to foster two helpful phenomena: (a) efficient calcium diffusion, and (b) insuring the existence of a high pH environment around most of the clay particles. The diffusion reduces the possibility of local lime concentrations and provides most of the sample with uniform amounts of Ca^{++} and OH^- ions. The D samples, compacted at low water content, particularly benefit from that kind of migration and gain strength when water is added after compaction. At the LMO (6% lime) the soil pH reaches 12.4. It is understandable that the pH of a compacted soil depends highly on the dilution factor. In the case of saturated soil, where all voids are filled with lime slurry, the dilution factor is much less than that used in evaluating the soil pH value. For maximum reactivity the pH value in the microscopic voids should be at least 12.4. The solubility of silica and alumina at this pH is very high, providing many Si and Al ions in the soil voids and furnishing maximum opportunity for pozzolanic reactions to occur.

Without adequate water the diffusion of lime necessary to dissolve silica and alumina in a microsopically uniform manner throughout the sample is not likely to occur. Samples of group D treated with 6%

lime and cured for 30 days exhibited a decrease in stength. This strength loss may be attributable to the occurrence of an intermediate phase of the cementing products. The cementing material formed during the early days will be rich in calcium since there is an abundance of it. When the free calcium has been depleted, the strength will level off or continue to improve only slightly. An improvement may be interpreted as an increase in the degree of crystallinity of the cementing product; this is believed to be the reason for the strength gain of the group D samples at 4% lime content (Figure 14). If calcium ions are more available, more of the cementing material will be developed in a short time, and the excess calcium will either alter this product to another cementing material or will be diffused to release more free silica and alumina. In group D samples with 6% lime a strength of 9.2 tsf was reached after 10 days, whereas at 8% lime these samples attained strengths of 12 tsf in the same length of time.

Samples of group D compacted with 6% lime reached strengths of 10.2 tsf after 90 days, with a gain of 2.9 tsf from those cured for 30 days. The gain is principally caused by the alteration of cementing materials or by the generation of additional cementing material. The same thing occurred in samples compacted with 8% lime, although no initial loss of strength was observed, and the gain of strength between 30 and 90 days is slower. No strength loss occurred at all in the O samples, because the soil particles are originally closely packed and thus require less cementing material, and the higher water content increased the efficiency of the lime reaction. The W samples had a still higher degree of saturation which enhanced the dissolution of silica and alumina to react with the lime. The higher degree of saturation facilitates lime diffusion and provides the samples with greater homogeneity.

The foregoing hypotheses are consistent with the observed behavior and differences in behavior of the test specimens. Even though some strength loss may occur in the early stages of curing dry-side mixtures, those losses are recovered during later stages of curing. The Camargo soil is composed of montmorillonitic clay mineral grains ranging downward in size from about 0.06 mm. The silt-size particles are believed to be poorly weathered volcanic ash containing a small percentage of quartz. The principal point is that this soil is composed of materials with a very high surface area, which makes it very reactive in the lime stabilization process.

Burleson Soil

The dominant clay mineral of this soil is calcium montmorillonite similar to that of the Camargo clay. The main difference is that the Burleson soil contains more quartz and an organic content of 1.45%. It is generally known that organic material will retard the soil-lime reaction. The Burleson soil is an A-horizon soil, usually thought to be least responsive to lime stabilization; however, it is not clear that the effect of lime on the Burleson soil was much inhibited by these circumstances. Reference to Figure 15 indicates that the behavior of the Burleson soil is similar to that of the Camargo soil. The W group exhibits a higher strength than is found in the O and D groups. A slight improvement is apparent in the O→W and D→W groups almost from the beginning of cure, with the strength gain becoming more pronounced at higher lime contents and with increased curing time. The

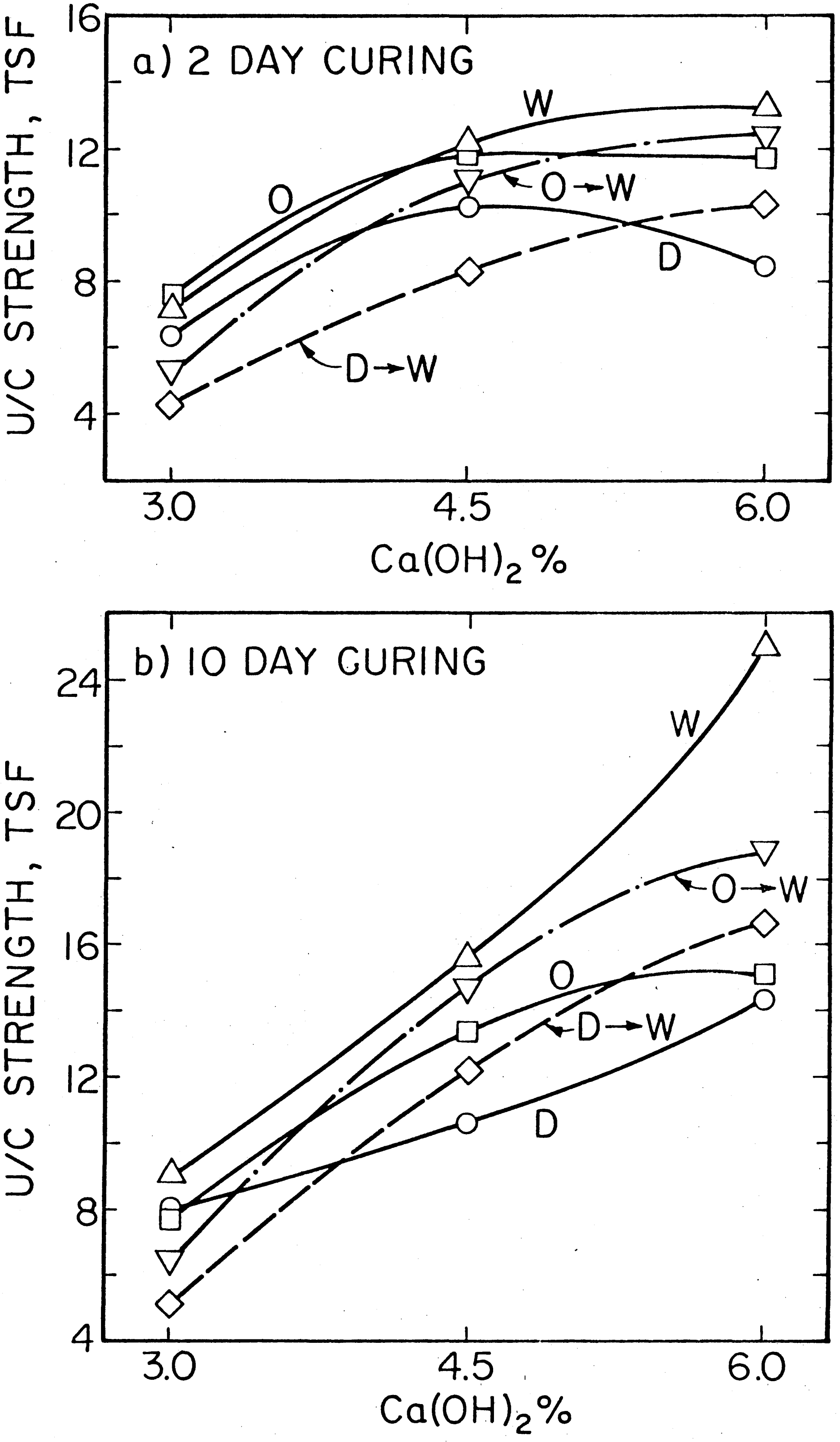


Figure 15. Variation of Strength with Lime Content for Burleson Soil

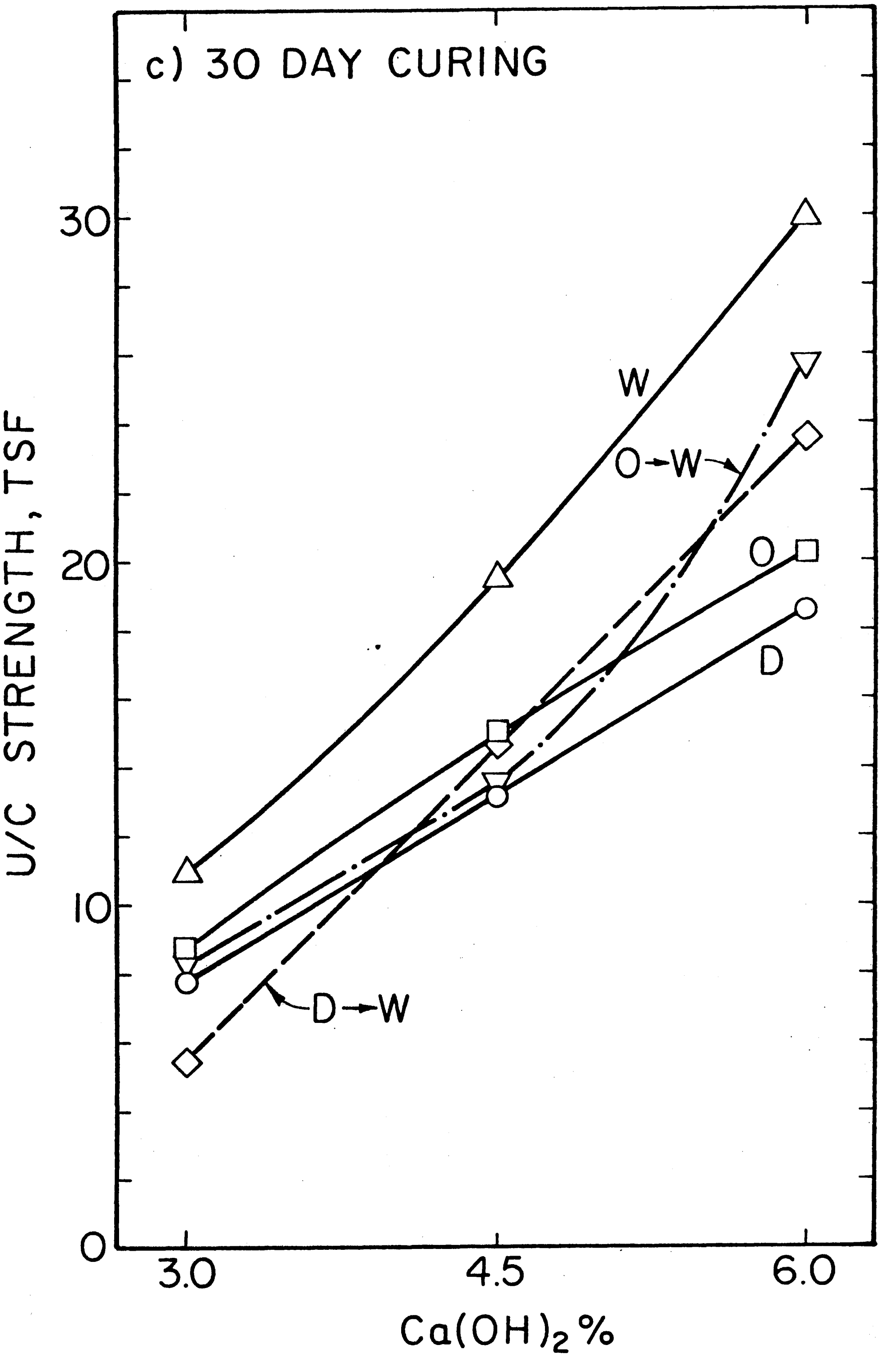


Figure 15. Continued

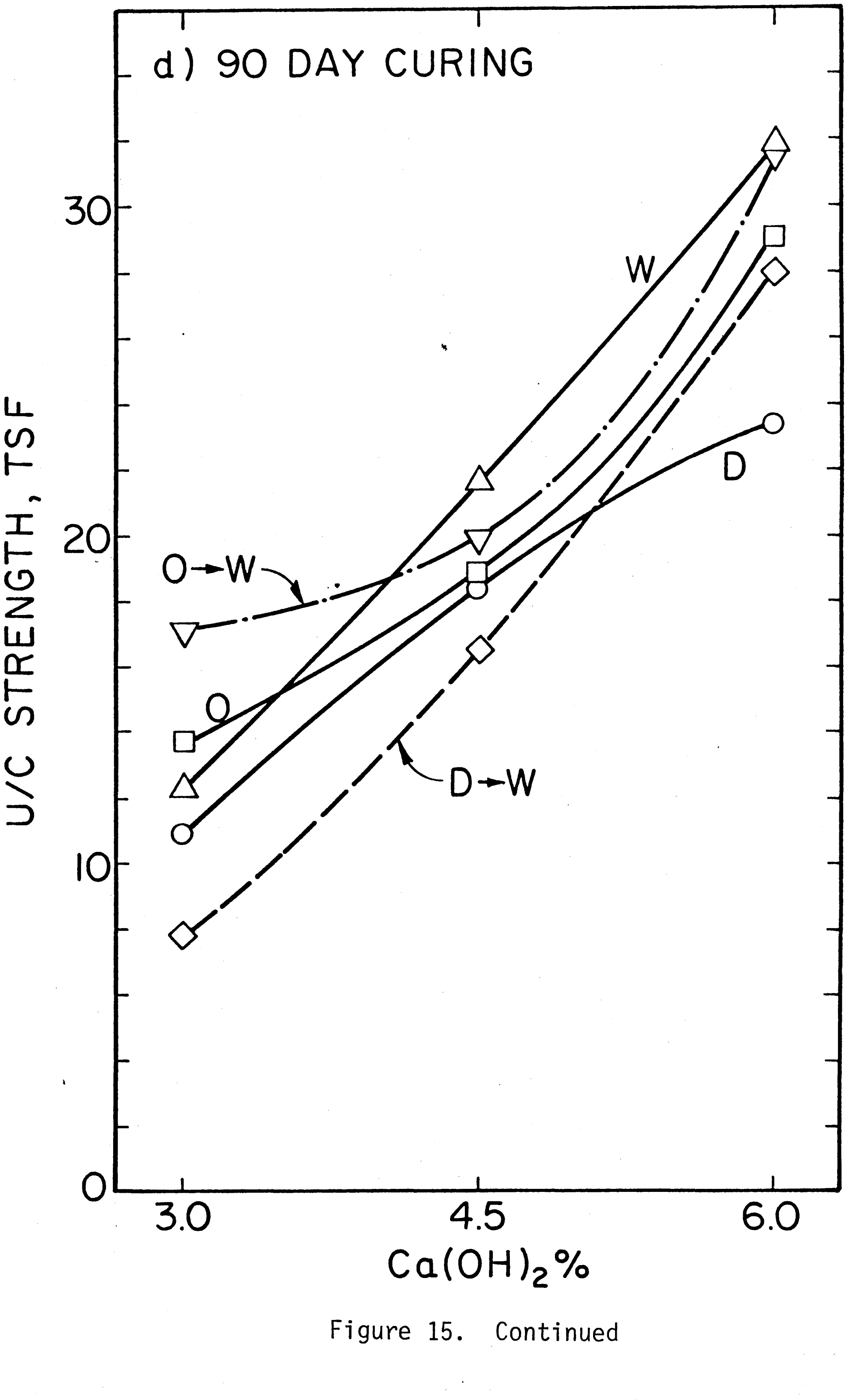


Figure 15. Continued

D→W samples were stronger than those of the D group after 30 days of curing, while the O→W samples were stronger than the O samples only when the lime content was higher than the LMO. It is worth mentioning that the O→W samples nearly attain the strength of the W samples at 6% lime content when cured for 30 days. After 90 days of curing the O→W samples had also increased substantially in strength until the difference between the O→W and W specimens was 2 tsf or less. A further interesting point with this soil is that there is no indication that soil strength will level off at any lime content within the modification range (LMO ± 1.5%). It appears that all groups of samples gain strength as the lime content increases. The strength gain with increasing lime content (up to 6%) is very nearly a linear relationship for specimens cured 90 days.

The time effect on the treated Burleson soil was highly dependent on the lime percent used. The rate of strength gain after 30 days for samples treated with 3% lime was very slow with the exception of the O→W samples. The situation changes completely at the 4.5% lime level, where the O→W, D, and O samples continued to show a very high rate of strength gain even after 30 days of curing. The W and D→W groups had a moderately low rate of gain. The D→W samples did not exhibit any promising results until a lime content of 6% was reached; then the D→W samples were higher in strength than the O samples, and the O→W samples reached almost the strength of the W samples.

Although this soil is an A-horizon soil with 1.45% organic content, it reacted quite well with lime. This result contradicts what has usually been reported. For example, soil reactivity has been evaluated, based on its strength gain over 28 days at room temperature

(73°F). Soils are classed as nonreactive if the strength gain is less than 50 psi. Some investigations of A-horizon soils have shown that as little as 0.8% organic content may effecively limit strength gains to less than 14 psi. It is evident that this is not the case in the Burleson soil.

Just as for the Camargo clay, Burleson samples compacted wet of optimum achieved the highest strength. The strength loss in the O→W samples tested after curing for 30 days (Figure 16) is a temporary anomaly (perhaps a sample preparation error) that was reversed after 90 days of curing. It was generally found that the Burleson soil, contrary to expectations, responds well to lime stabilization (see also Figures 11 and 12). There is no doubt that the cementing material formed within the Camargo and Burleson soils is of the same nature. The writer believes that the relatively high free Na content helped to maintain a high pH, releasing more Si and Al for pozzolanic reaction. Overall behavior of the O→W and D→W groups suggests that some advantage in the field may be gained by wetting the soil after compaction, especially if a fairly high lime content is used. This additional water is primarily to stimulate lime reactivity. However, the improvement in these soils generally started from the outside surface and worked inward toward the core for a depth of 0.2 to 0.3 in., which suggests a confined weaker core surrounded by a hard crust. This phenomenon was noticed in many samples in which the surface had the appearance of a dry saline crust; the crust, however, was easy to scratch. If the crust thickness increases with time, the stress gain or loss with increased curing time should be very close to being a constant value. This is almost the

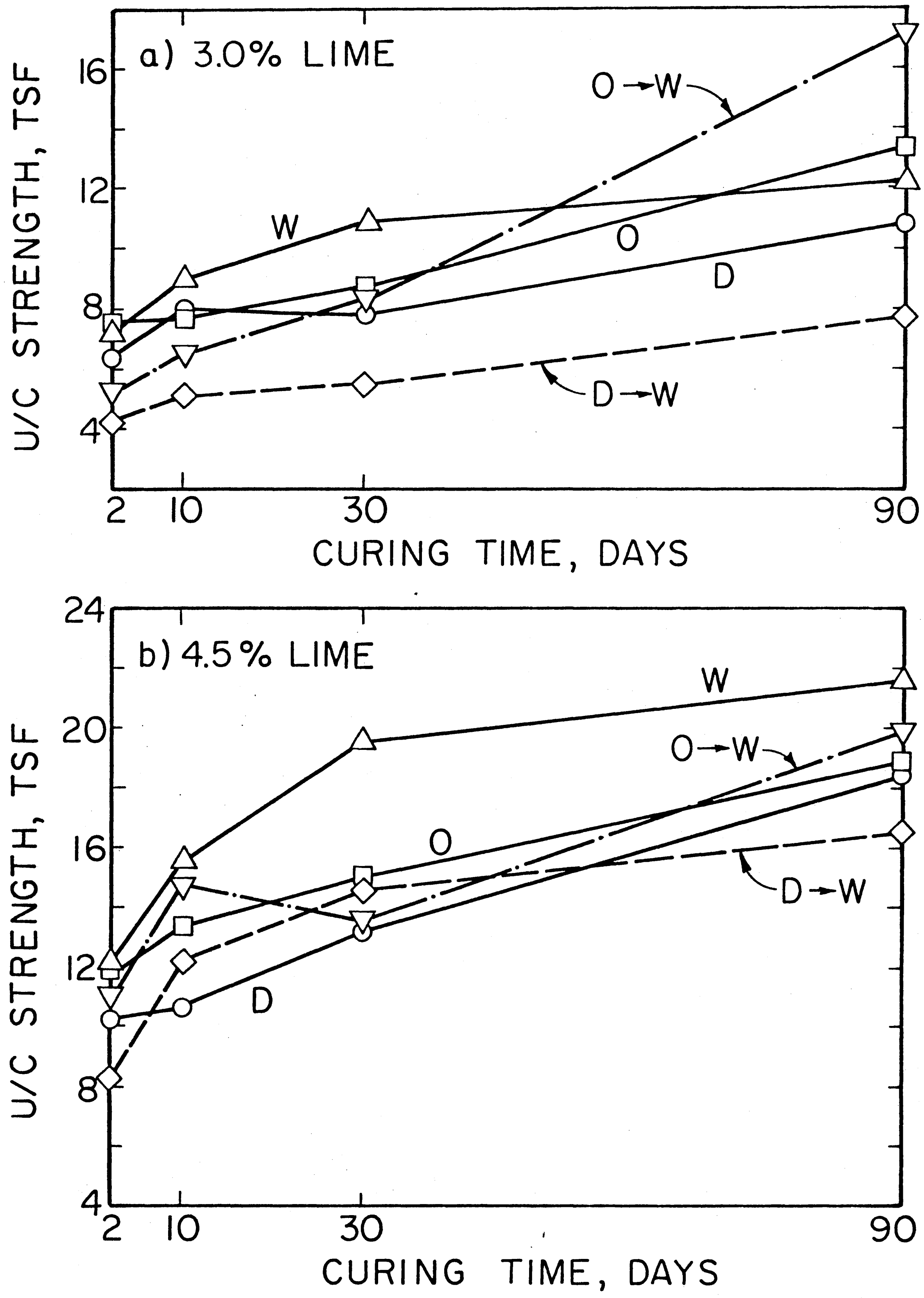


Figure 16. Variation of Strength with Curing Time for Burleson Soil

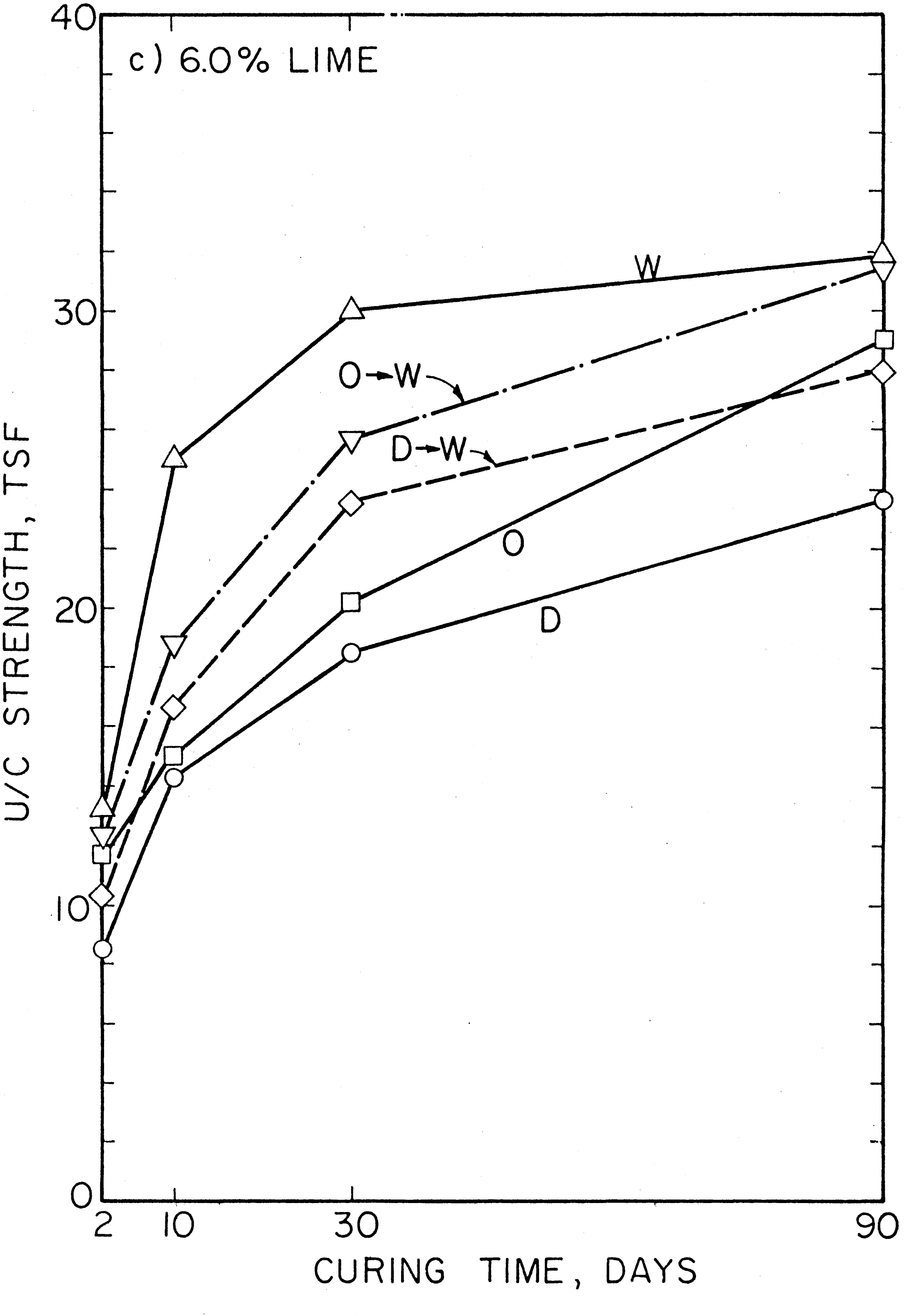


Figure 16. Continued

case for curves D and D→W in Figure 16(a), and D and D→W, O and O→W in Figure 16(c).

Union City Clay

Union City clay is quite different than those soils discussed previously because the clay fraction is entirely illite. It contains the highest clay content among the four tested soils, the lowest activity number, and the highest sulfur content. It is quite evident that Union City clay responds differently to lime than those described previously. It was found that O→W and D→W samples are the lowest in strength for all lime contents tested and at every curing time (Figure 17). The W samples possess no striking superiority over others during the early days of curing. However, the strength of the various groups after 90 days occupies a wide range from 8.7 to 39 tsf, and at that time the W group is clearly superior to the O group across a broad range of lime contents. The strength curves for the W and O samples cured for 90 days have similar trends which differ completely from those corresponding to these groups after 2 and 10 days curing time. The O→W and D→W samples treated at LMO show a higher strength after 2 and 30 days than those corresponding to other lime percentages. This was also the case for the O and D samples at 10 days of cure.

It is evident from examining Figure 18 that O, D, and W samples containing 2% lime have about the same strength after 90 days; however, O samples have continued to gain strength during the entire period, whereas the 90 day strength of W and D samples is less than their 30 day strength. This is not of great practical concern since field applications of lime are almost always in excess of 2%, and it is also

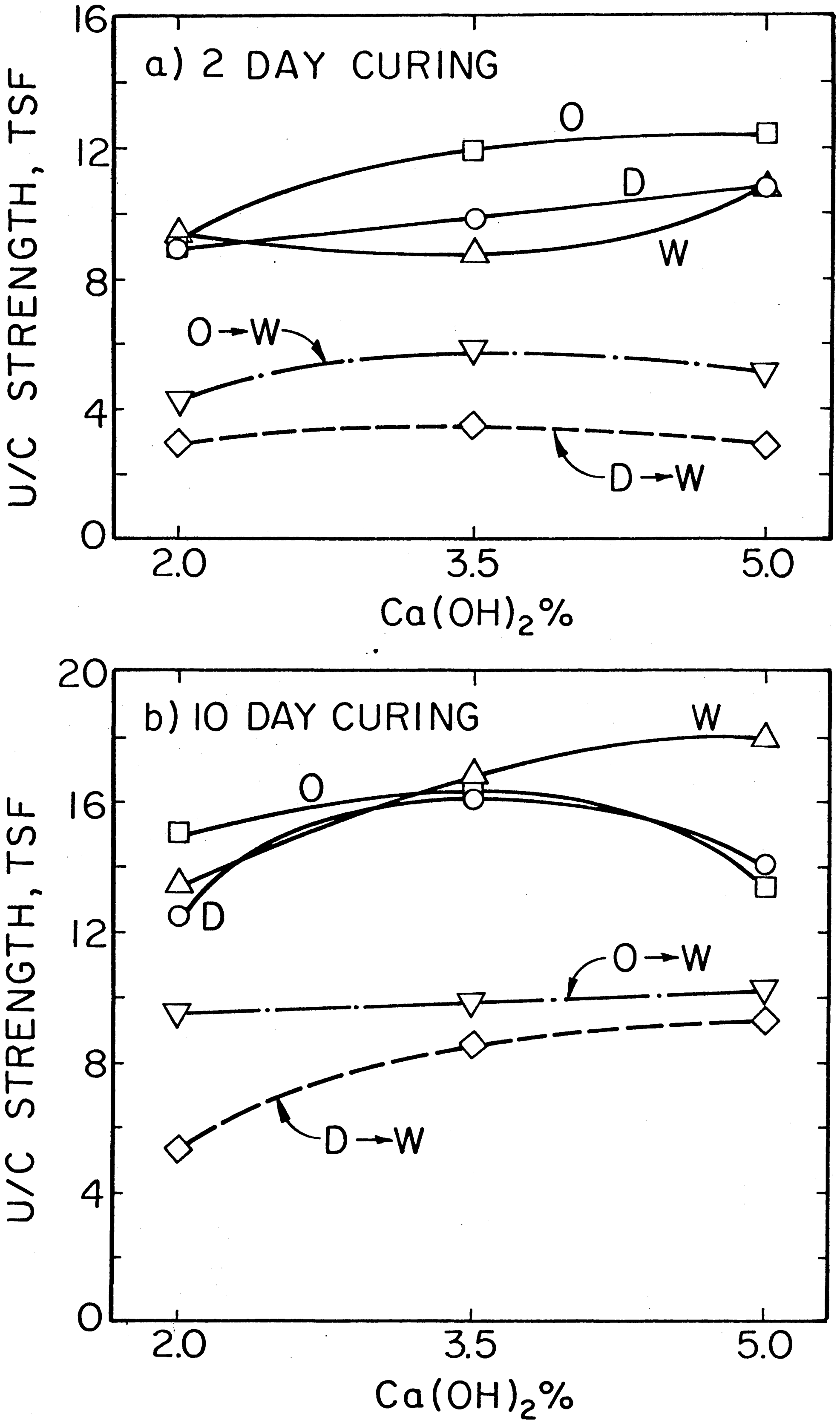


Figure 17. Variation of Strength with Lime Content for Union City Clay

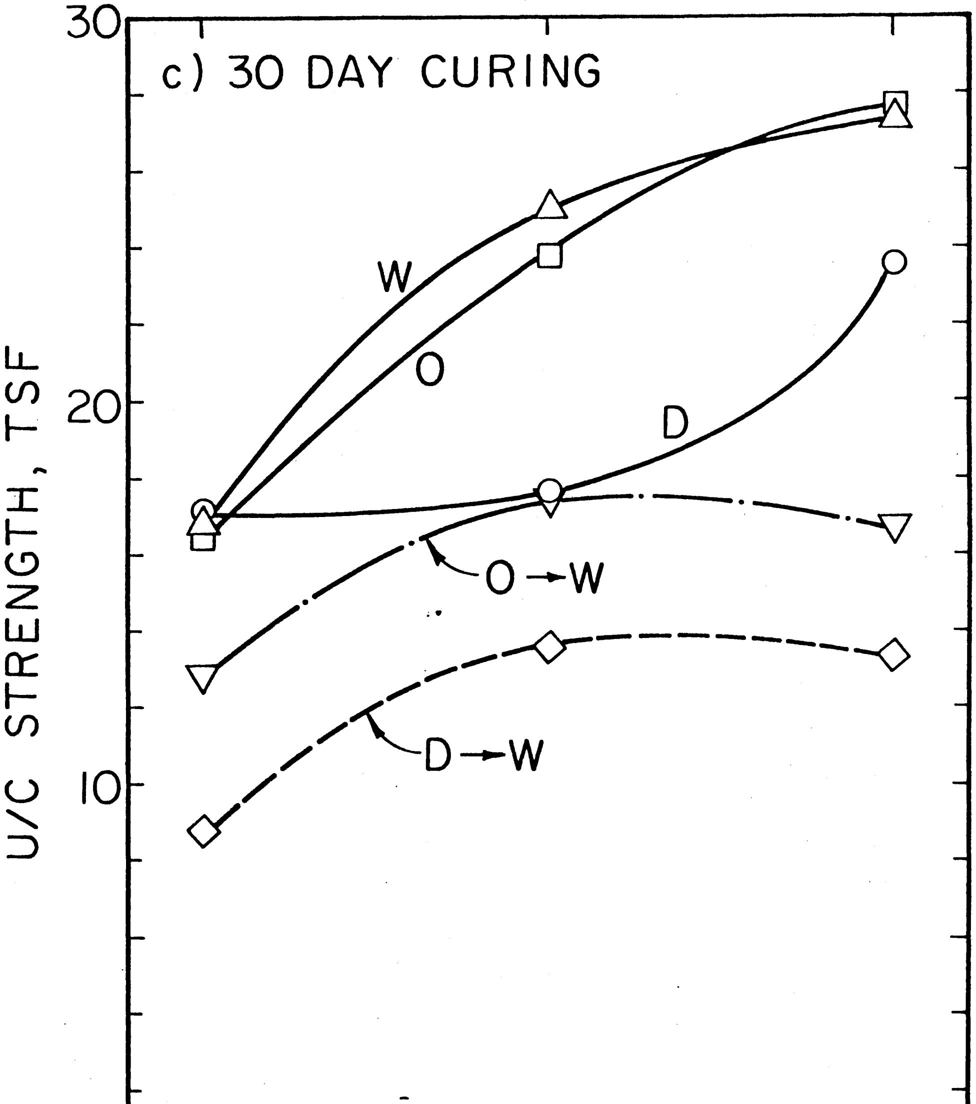


Figure 17. Continued

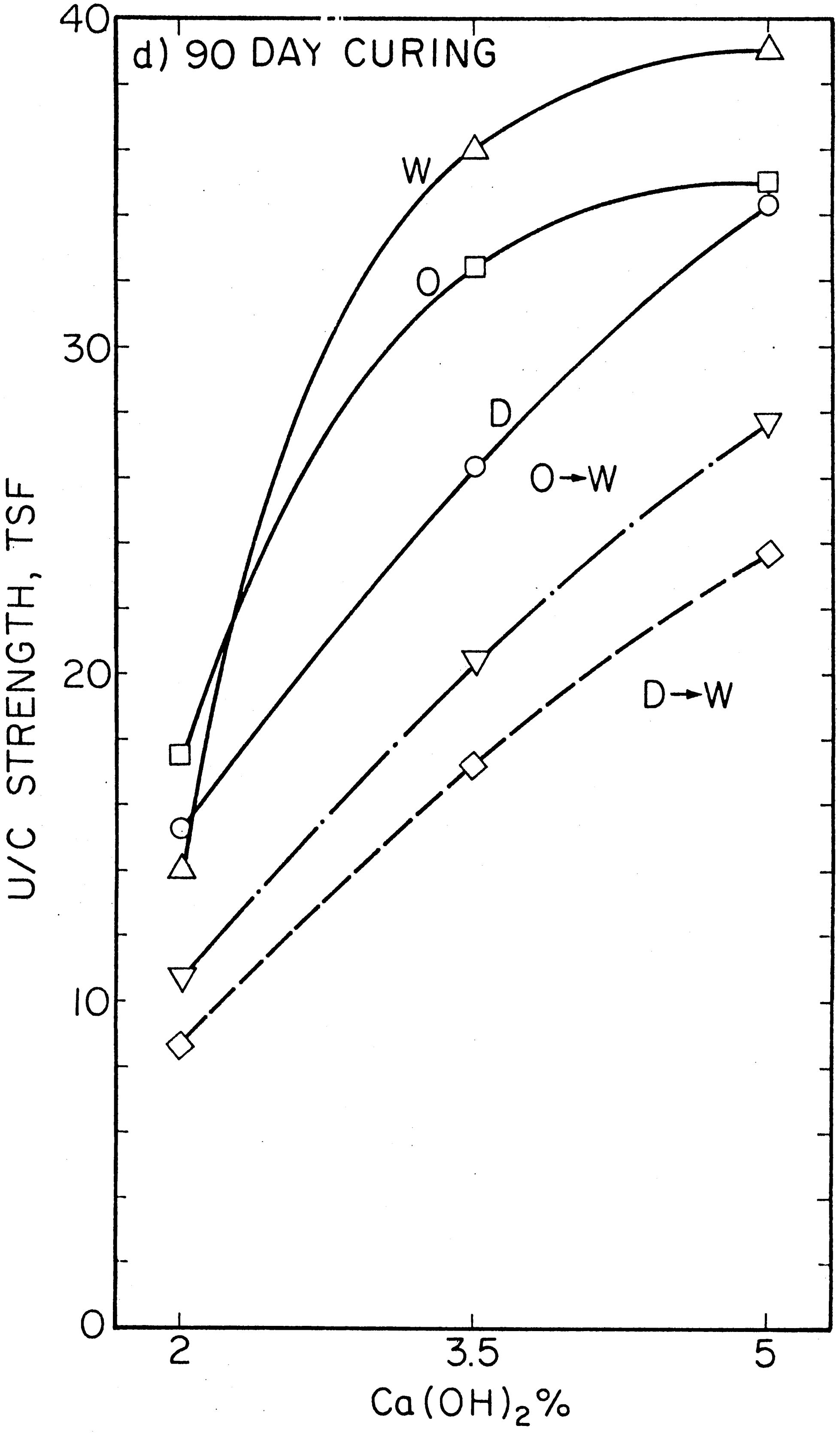


Figure 17. Continued

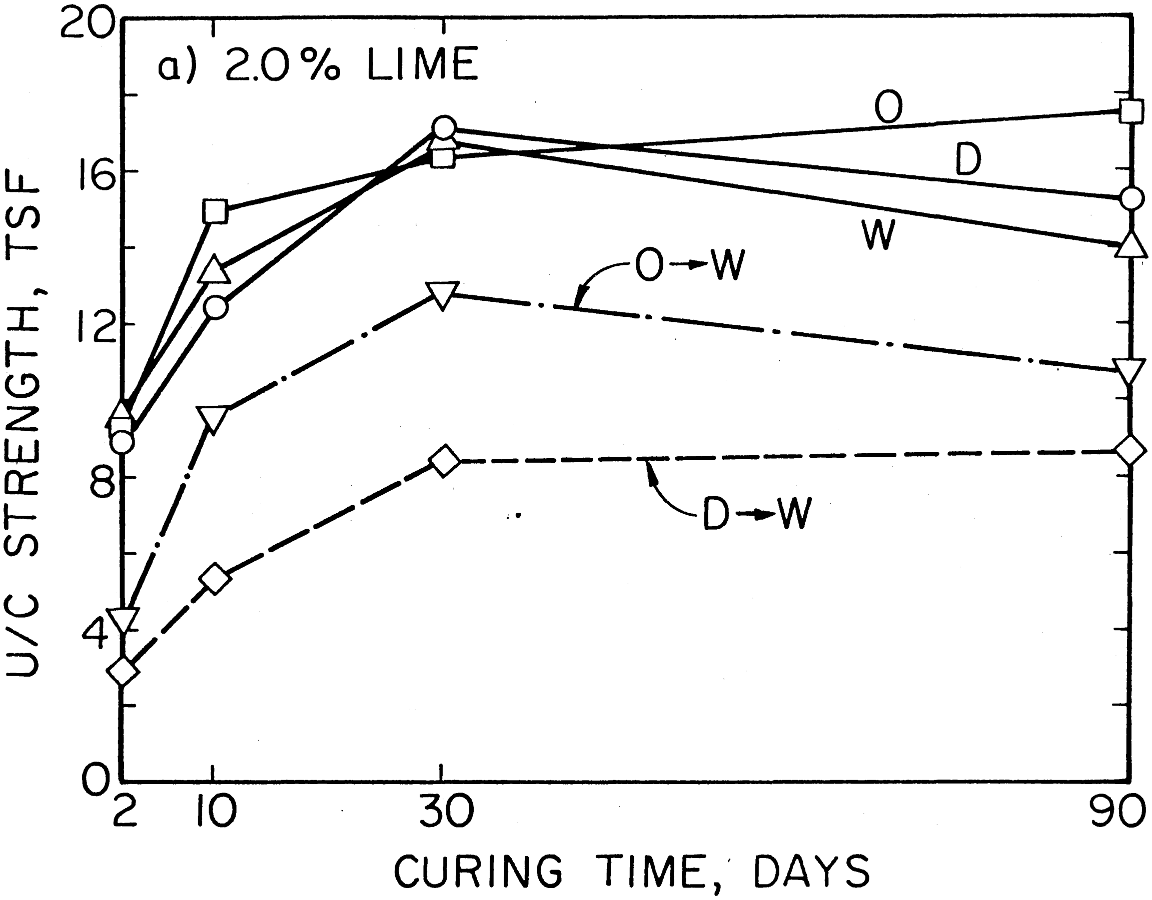


Figure 18. Variation of Strength with Curing Time for Union City Clay

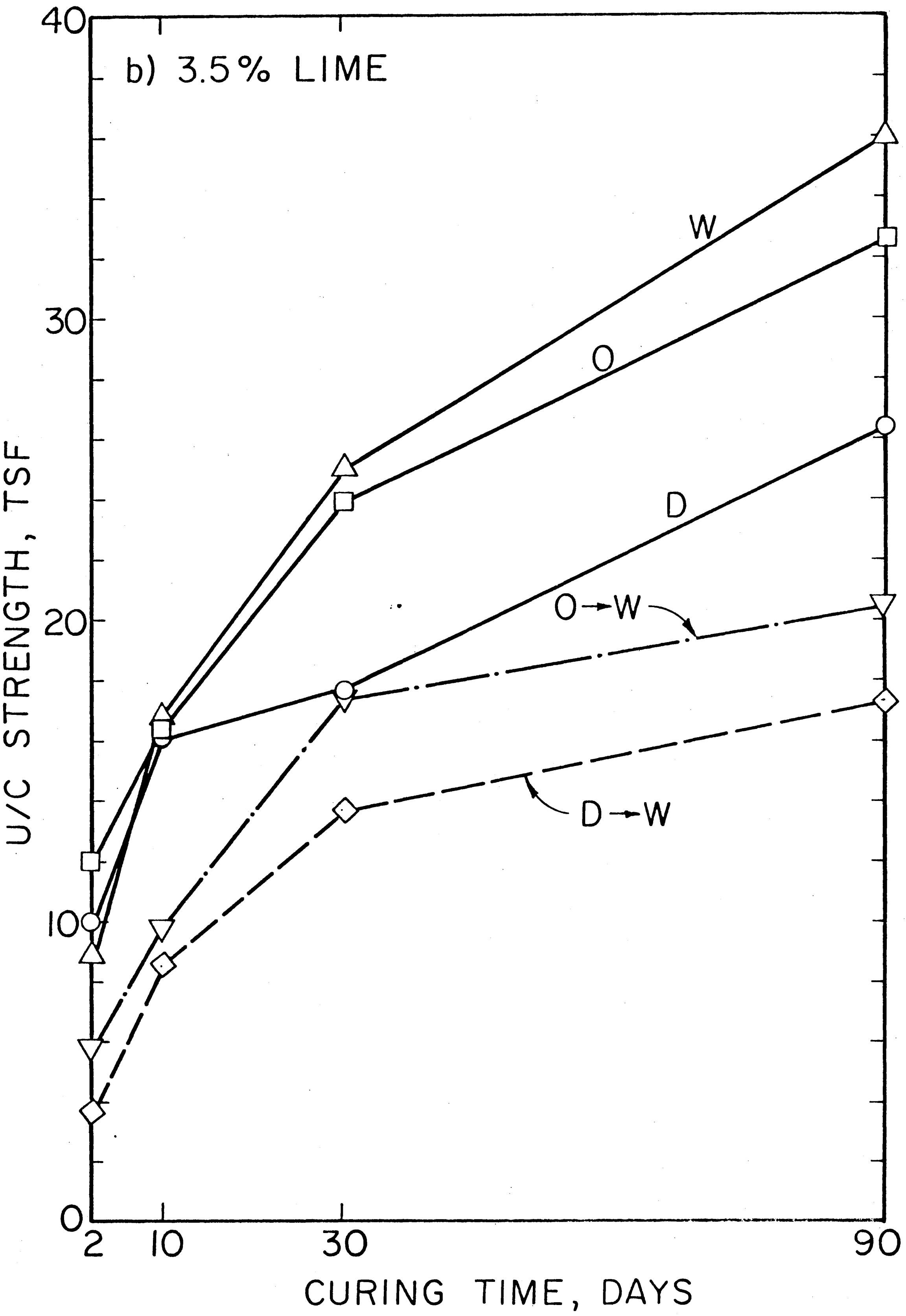


Figure 18. Continued

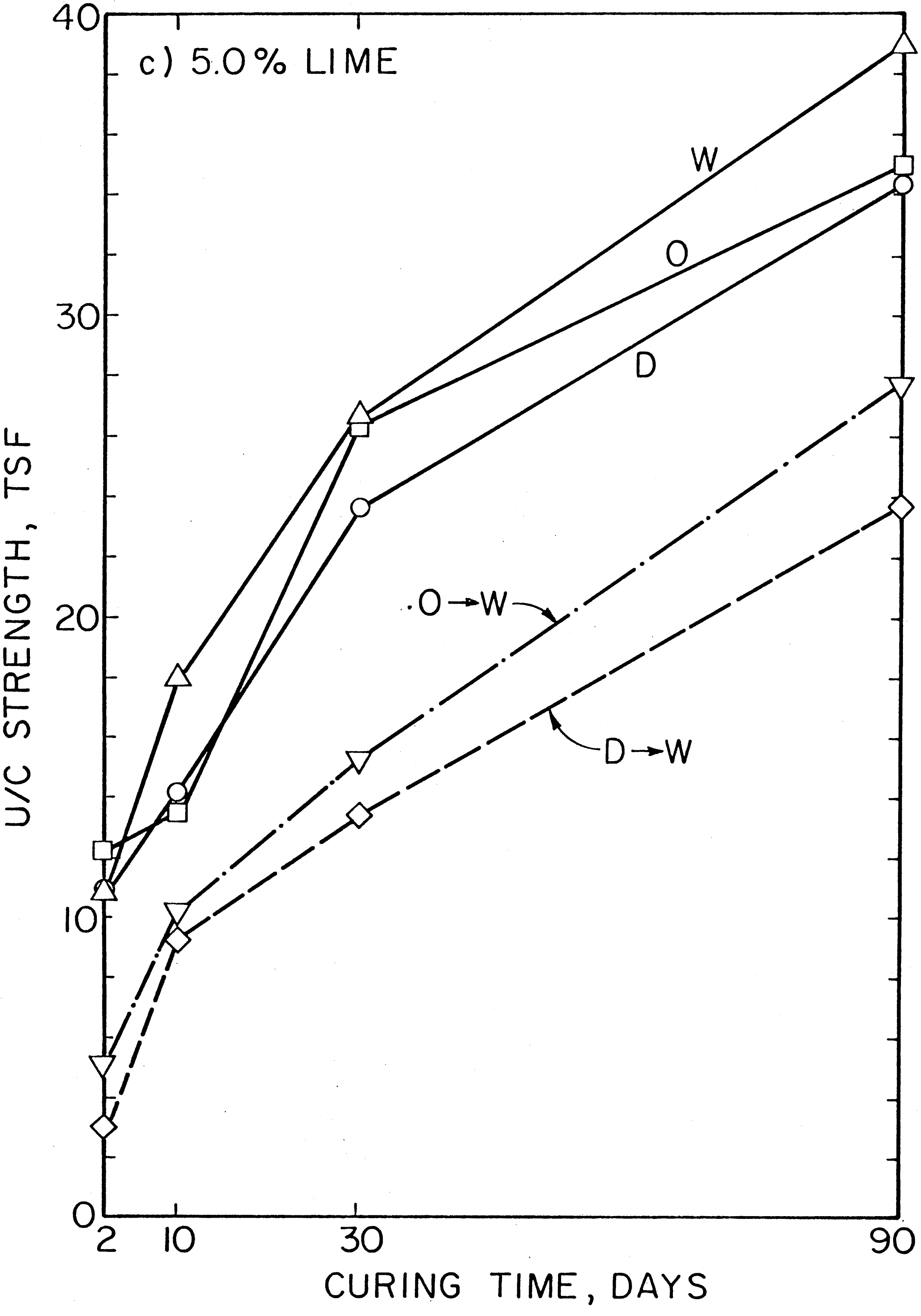


Figure 18. Continued

possible that strength losses may be recovered during still longer curing periods.

Samples compacted at LMO had a very high rate of strength increase. The rate is almost the same for W, O, and D samples in the time interval between 30 and 90 days, which indicates that at the LMO soil strength will continue to improve with time, beyond 90 days. This trend is even more evident for higher lime content. Figure 18 shows that for a lime content of 5.0% the O→W and D→W samples exhibit a rate of strength increase of 0.22 tsf/day, the highest of all.

In spite of the excellent rate of strength increase, the strength of O→W and D→W samples of Union City clay is consistently lower than that of O and D samples for curing periods of 90 days or less.

The general trend in the pattern of strength and strength development of treated Union City clay can perhaps be explained by the following hypothesis. In this clay a weak cementing material having a high degree of hydration (a low Ca/H_2O ratio) is rapidly formed. This phase is followed by the slow development of a stronger cementing agent with a low degree of hydration. If this postulation is correct, it follows that the outer shells of O→W and D→W specimens (where the excess water is applied) would contain a relatively great amount of the weaker cementing material, and that the water used in forming this material would not be easily released at normal temperature, since the degree of crystallinity is presumed to be high. This presumption is based on the high rate of strength increase observed, for all lime contents, during the first 10 days of curing.

While the weak cementing material is developing, the clay minerals are undergoing deterioration in the high pH environment, but the deficiency of Ca ions to react with the silica and alumina prevents the development of any stronger cementing materials. The strength will thus tend to become constant or to decrease (see Figure 18) for 2% lime content. In this figure it can be seen that most of the specimens gained about 4 tsf in strength during the first 8 days with no further gain during the next 2 weeks, indicating that the weaker cementing material is highly developed within a short period of time. However, in the case of 5% lime content, the additional lime maintains a high pH and also provides the Ca^{++} ions to react in time with Si and Al and form stronger cementing materials with greater crystallinity and stability. Initially, the high Ca/Si ratio (up to 3:1) tends to produce an abundance of gel-like cementing material; however, after some time the Ca/Si ratio will be reduced to about 1.5:1, an environment (still of high pH) conducive to the formation of well crystallized cementing materials (see Reference (74)). It appears that a high Ca/H_2O ratio may act to depress the formation of the weaker cementing materials while enhancing the development of stronger cementing compounds of more highly developed crystallinity.

Summit Soil

The Summit soil responds well to increasing lime content for all ages of cure. After 2 days of curing (Figure 19) the strength is mainly governed by the soil density and water content. Samples compacted at optimum were the highest in strength, followed by those dry of optimum and those wet of optimum. The O→W and D→W groups appear to have gained no strength in this short period of time. After 10 days of curing (Figure 19) some reaction has taken place as is shown by the behavior

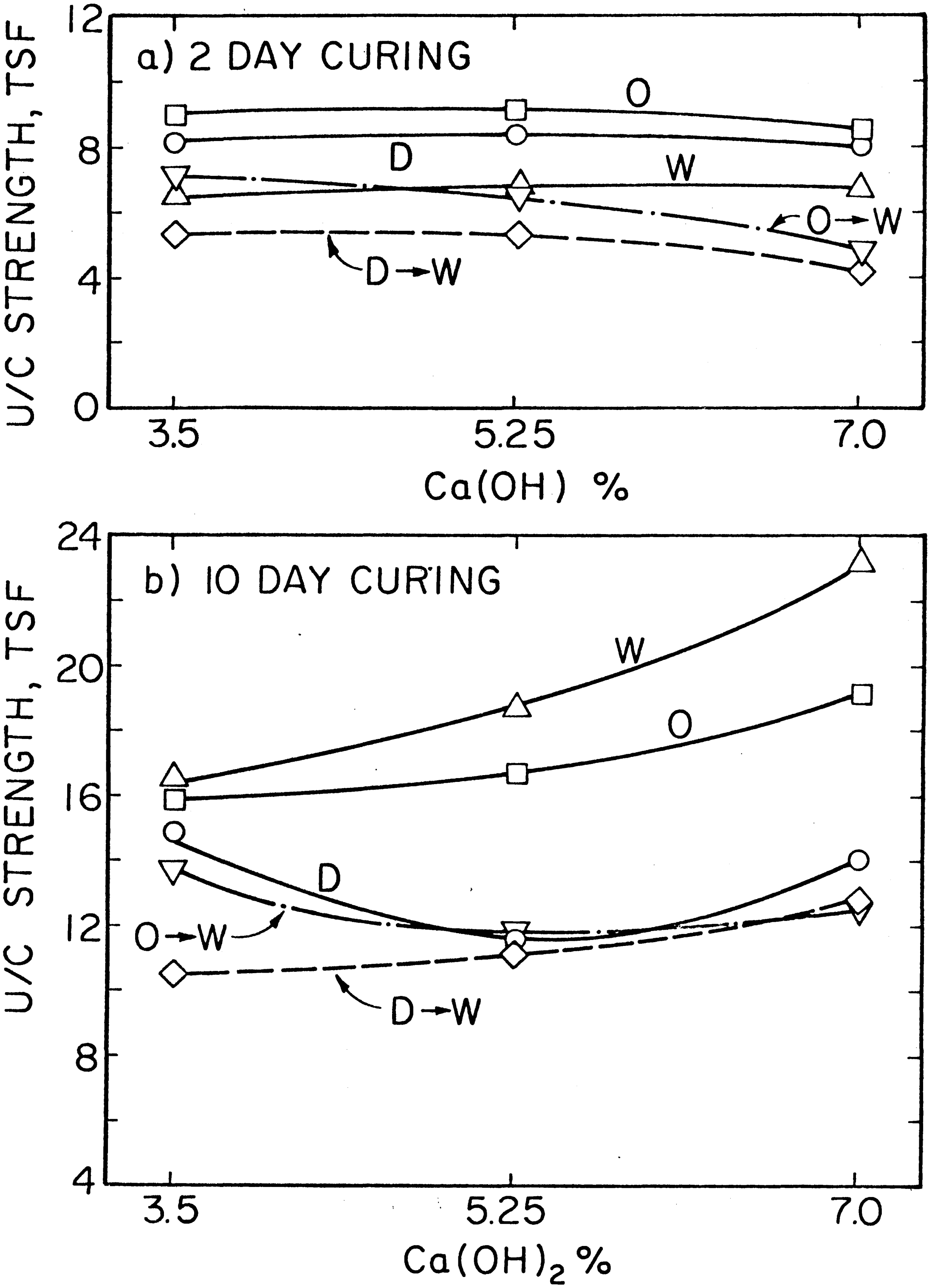


Figure 19. Variation of Strength with Lime Content for Summit Soil

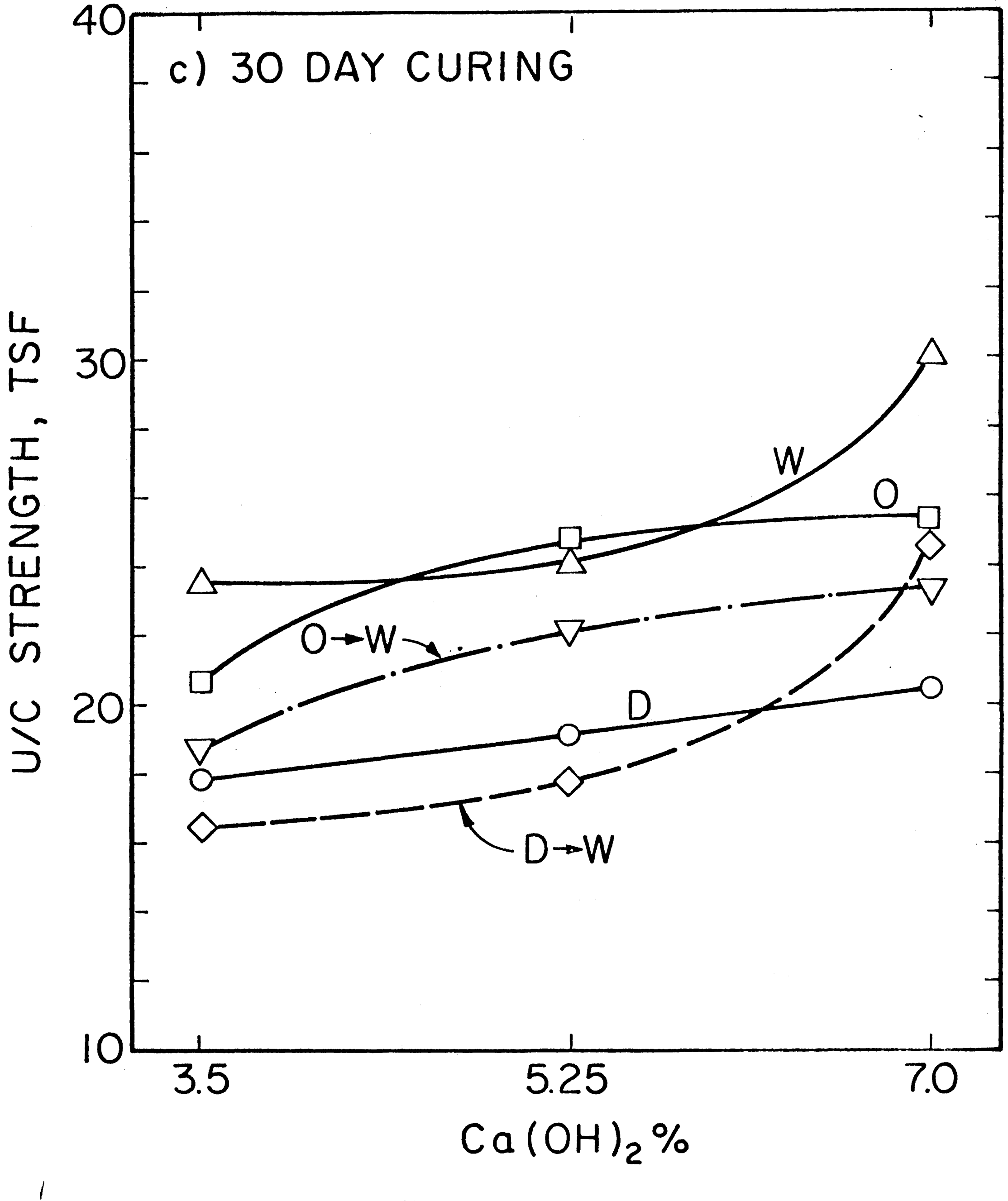


Figure 19. Continued

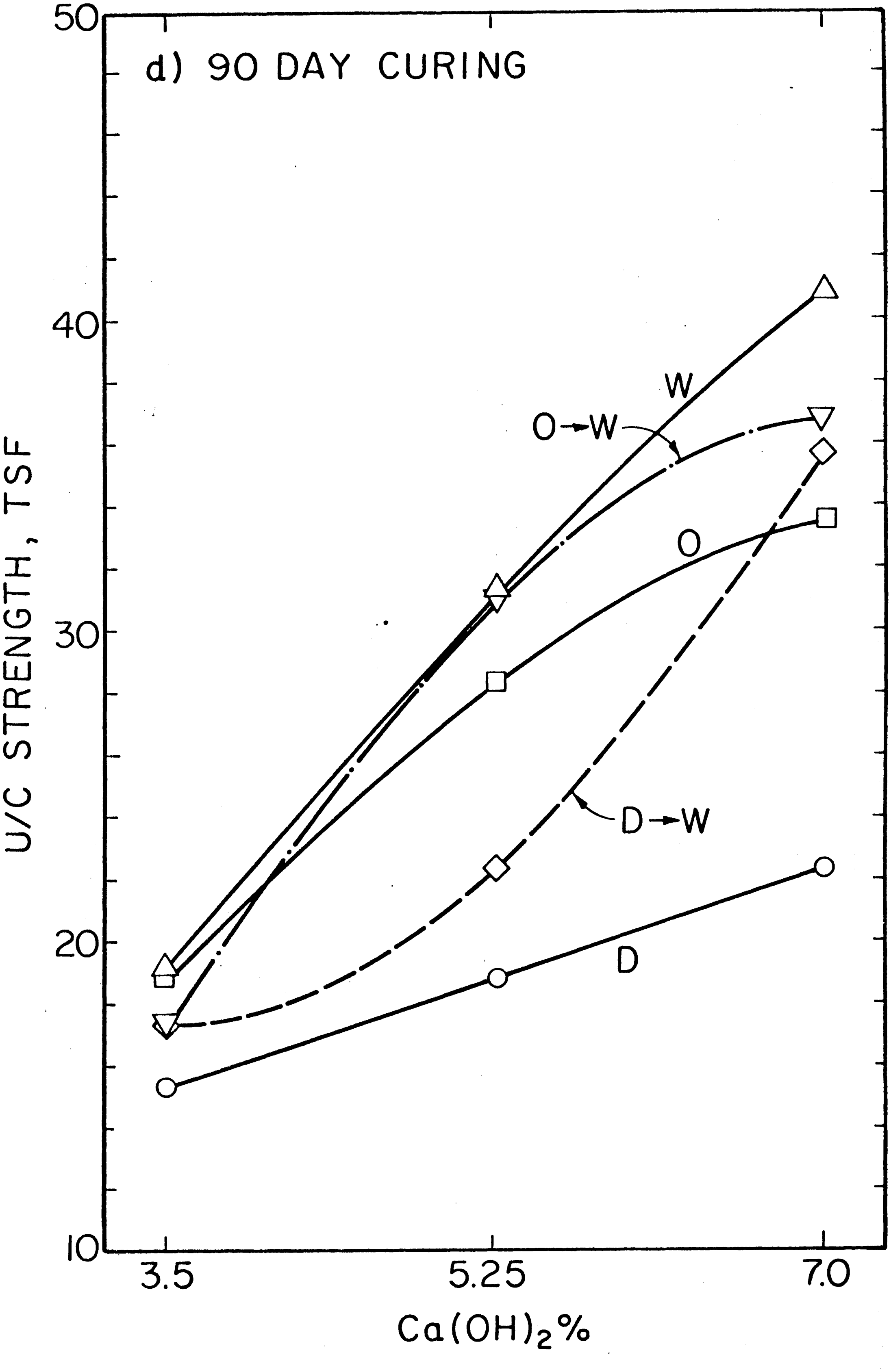


Figure 19. Continued

of the W samples. The O→W and D→W specimens have begun to pick up strength very rapidly. The D samples with a low lime content gained more strength than those of higher lime content. On the other hand, samples cured for 30 days exhibit strengths that are directly proportional to the lime content. Also, the O→W and D→W samples have attained strengths more nearly equal to those of the O and D specimens. After 90 days (Figure 19) the O→W and D→W specimens have gained strengths well above those of the corresponding O and D samples. It is significant that the O→W specimens attain almost the same strength as the W samples. The rate of strength gain with increasing lime content at 90 days remains positive for all groups of samples but the rate varies from one group to another. The D samples exhibit the lowest rate, while the W and D→W specimens display a very high rate of strength increase with increasing lime content. There was no consistent strength advantage in any one group during the entire curing period, which indicates that the cementation process occurs at different rates in the groups.

The curves in Figure 20 show the variation of strength with curing time at a given lime content. The general trend for specimens with low lime content is that they attain maximum strength at some intermediate time and then start decreasing. Specimens at the LMO exhibit a moderate strength increase after 30 days, while the greatest continuing improvement is associated with the group containing the most lime. The rate of strength gain varies inconsistently. It was found that, during the first 10 days, the group of samples compacted dry of optimum displayed its highest rate at the low lime content, whereas the W group had its high rate at the highest lime percent used in this study. It is clear

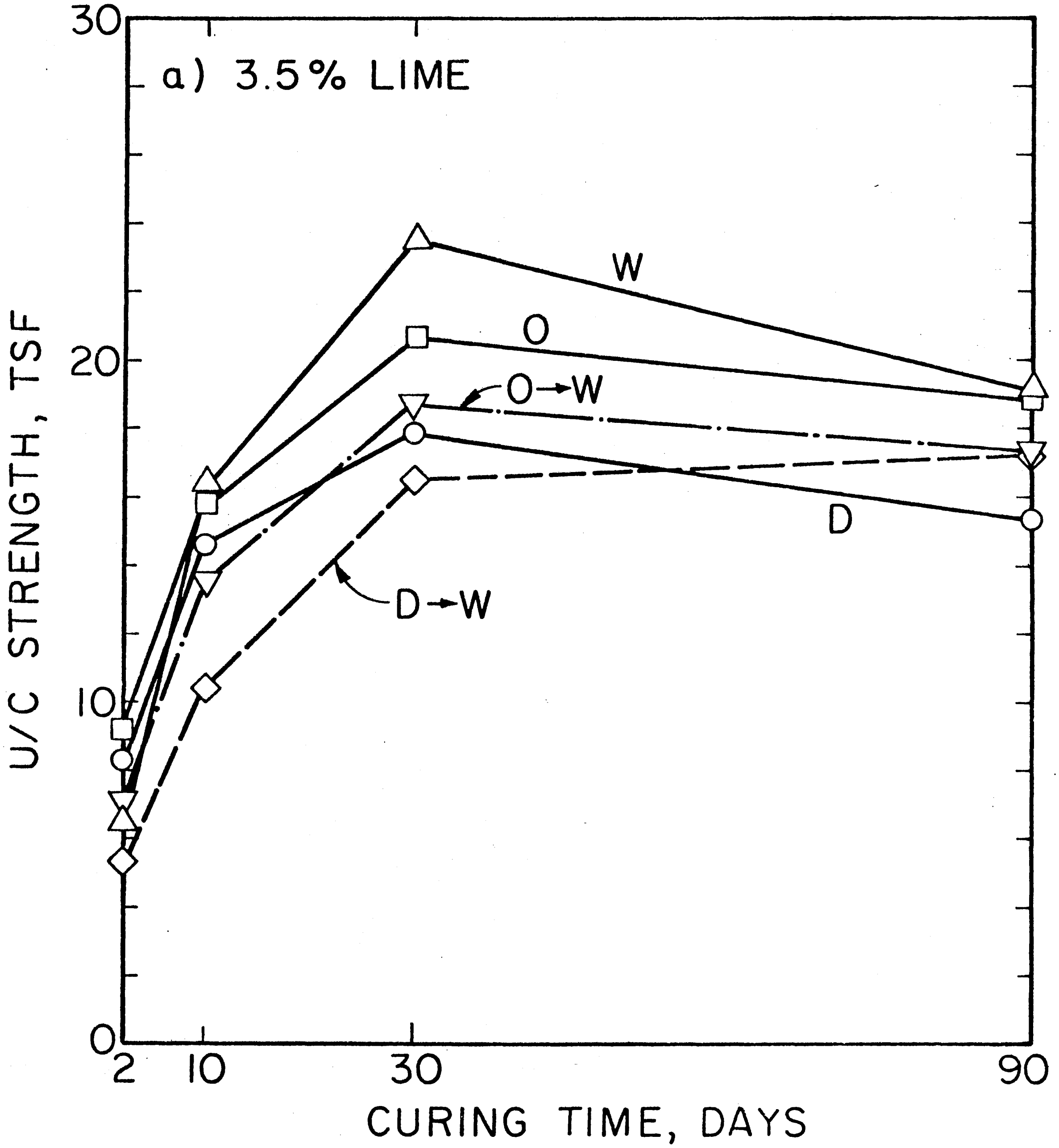


Figure 20. Variation of Strength with Curing Time for Summit Soil

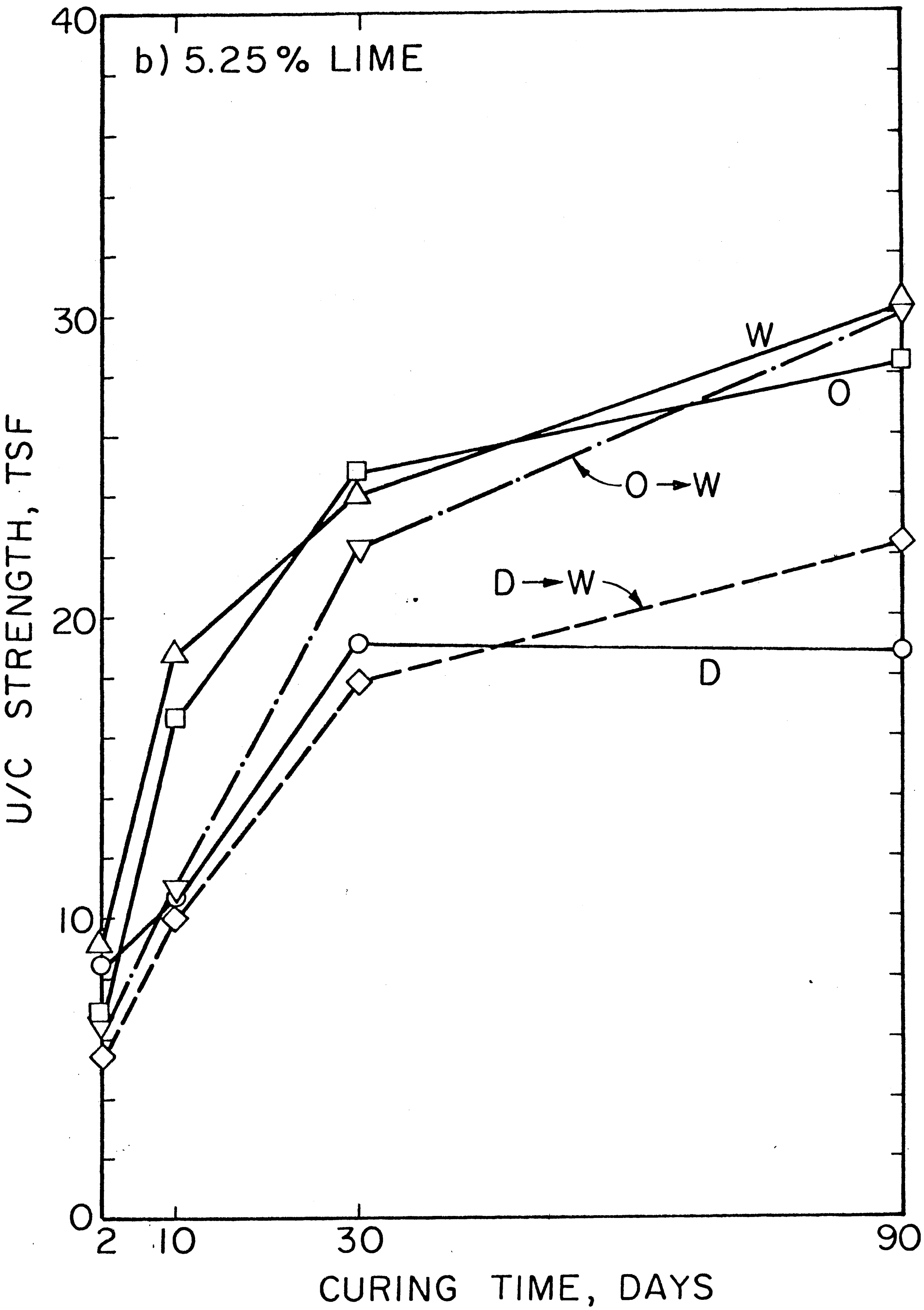


Figure 20. Continued

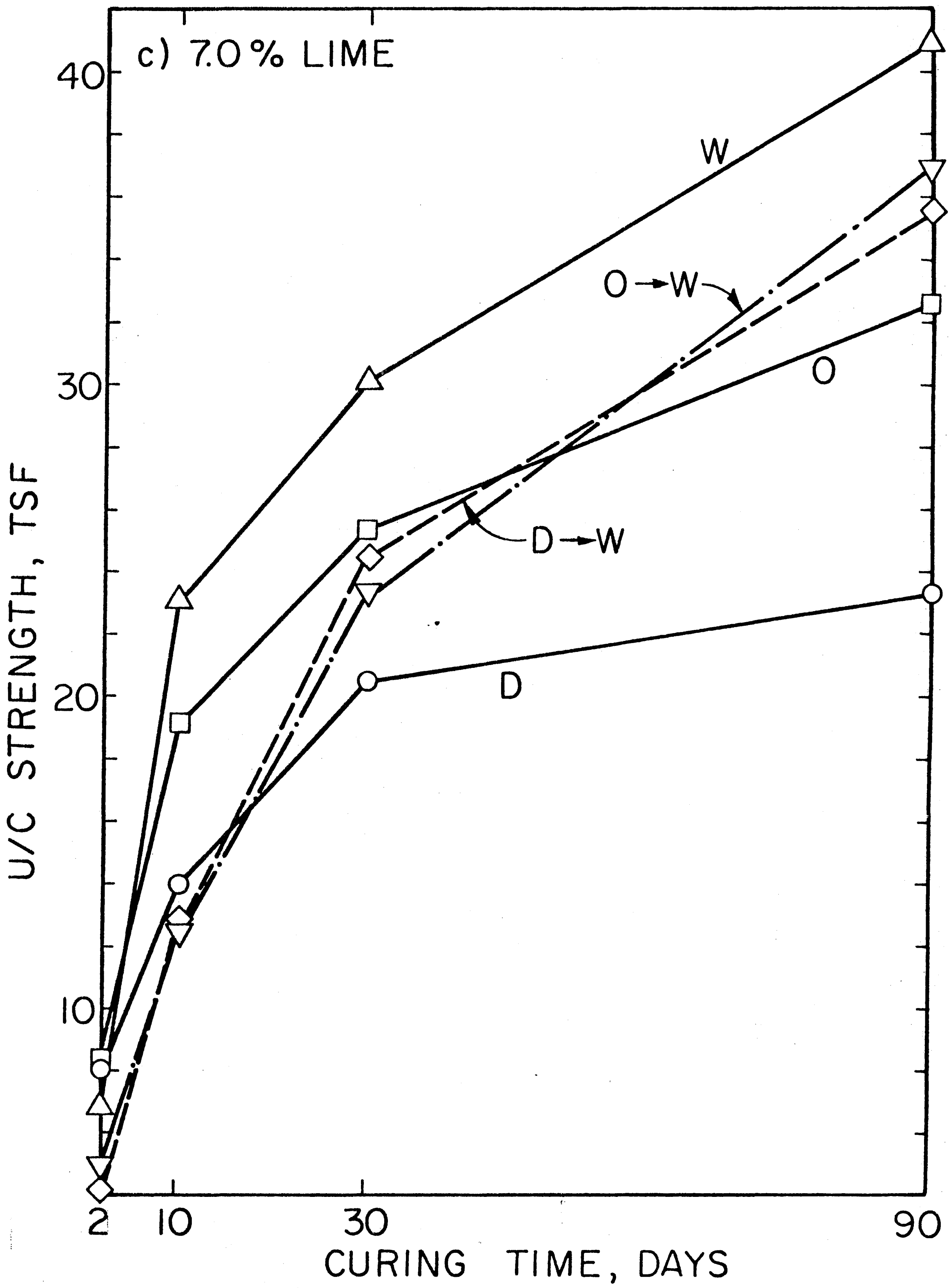


Figure 20. Continued

that O→W and D→W groups have an advantage over the O and D groups after 10 days of curing. It is quite remarkable that the D→W group, having initial strengths well below those of the corresponding D group, attained strengths about 50% greater than those of the D group after 90 days curing. Those groups treated with 3.5% lime and cured at their compaction moisture contents attain rather high strength after 10 days but begin to weaken after 90 days of curing. The W group, which is the strongest, loses the most strength. The strength of the O→W group lags that of the O group by a constant margin. The D→W group is the only one that maintains a strength increase and eventually surpasses that of the D group.

Samples compacted at the LMO behave differently than those at 30% below LMO. All sample groups had a positive rate of strength gain, with the exception of the D group at its final curing time. After 30 days of curing, the strength of the O group is not significantly greater than that of the W group. Samples at a lime content 30% above the LMO exhibit some very significant differences from those with less lime:

1. The greatest strength and most rapid strength gain among groups cured as compacted is found in the W groups followed in order by the O group and the D group.

2. The O→W and D→W groups display the most rapid strength gain, giving them a slight advantage over the W group with respect to anticipated future strengths (curing beyond 90 days).

These results are similar to those for the Union City clay because the O→W and D→W groups did not gain an immediate strength superiority over the D, O, and W groups; however, in the Summit soil it does appear that water applied to the surfaces of compacted specimens progresses slowly and effectively to the sample core. Moreover, it is clear that the Ca/H_2O ratio is of some importance although, in each case, the pore fluid has initially the highest possible pH (12.4). The point is, that for the reaction to proceed efficiently, this high pH must be maintained. Suppose, for instance, that lime contents of 5.25% and 7% both produce a pH of 12.4 in the fluid occupying the pores, and that a certain number of Si and Al ions are freed to react with the calcium ions. In the case of 7% lime content, the reaction may proceed without any reduction in pH, while the high pH environment continues to act on the clay mineral to produce a more or less continuing supply of Si and Al ions for combining with the plentiful Ca. Under this circumstance one may reasonably expect a strongly crystalline phase of CSH and/or CAH to be produced.

The same may not be true for the lower lime content, where the pH of the pore fluid may be slightly reduced by the consumption of Ca ions in the early stages of the reaction. The lowered pH is less efficient in providing the Si and Al ions needed to produce a highly crystalline phase, with the result that weaker, poorly crystallized phases may predominate. In the extreme case of very low lime contents and low degrees of saturation the predominant reaction product may be calcite rather than any phase of CAH and/or CSH. For a given lime content, a lower Ca/H_2O ratio provides for greater ionic mobility within the pore system, increases the degree of saturation, and helps to exclude CO_2 from the system.

Evidence supporting the above hypothesis lies in a comparison of the W groups of the Summit soil at lime contents of 5.25% and 7.0%, after 10 days of curing. The strength gain of the 7.0% group, between 2 days and 10 days of curing, is 16.5 tsf while that of the 5.25% group is only 9.6 tsf. It is also significant that during the interval between 10 and 30 days the D→W and O→W groups gained strength at a substantially higher rate than did the groups cured at their compaction moisture contents. This is believed to support the hypothesis that a more fluid medium is helpful in maintaining a uniformly high pH and in promoting the more efficient use of the lime.

Summary

The following tentative conclusions are believed to be warranted by the results of strength tests on the four soils studied:

1. Samples compacted wet of optimum moisture content are ultimately stronger than those compacted at or below optimum.
2. The strength of samples compacted at or below optimum moisture content is ultimately improved if additional water is made available during the curing process, provided that no compounds or minerals which inhibit the soil-lime reaction are present in the soil.
3. Eades and Grim's method for evaluating the Lime Modification Optimum is a useful tool for guidance in designing soil-lime mixes to meet stability requirements.
4. Cementing agents formed during the early stages of curing differ in general from those formed during later stages.
5. From the standpoint of the composition of the natural soil, the strength of lime-stabilized soils is governed primarily by the presence of extraneous minerals and organic compounds rather than by the type of clay mineral. However, the degree of alteration of the clay mineral itself may be a factor.

6. The prediction of soil reactivity on the basis of organic content and horizon of origin appears generally inapplicable.

7. The diffusion of lime in a compacted soil is a fairly rapid process that can be accelerated by the availability of water.

8. A high moisture content during curing promotes the maintenance of a uniformly high pH environment around the soil particles.

These conclusions are subject to confirmation by procedures unrelated to the determination of strength and are discussed again in Chapter V.

Durability Test

The four soils prepared at their LMO and cured for 30 days prior to testing were evaluated on the basis of their unconfined compressive strength before and after three cycles of freezing and thawing. The test results are shown in Table VI.

Strength losses in most of the samples were fairly high, with those compacted dry of optimum being the least affected by the freeze-thaw cycles. The Camargo and Burleson soils absorb water very readily after compaction and this capability, which enhanced the cured strength of these soils, has an opposite effect on their durability as it is evaluated in this study. The unconfined compressive strength of these soils diminished greatly as a result of cyclic freezing and thawing. Severe strength loss occurred in the O→W and D→W groups of the Camargo soils, probably because of the rather uniform distribution of absorbed water throughout the sample. In the W group, which had the highest initial strength and a uniform distribution of water, the strength loss was greatest of all. It is apparent that the water content was the

TABLE VI

RESULTS OF U/C STRENGTH TEST BEFORE AND AFTER THREE CYCLES OF FREEZE AND THAW

Samples	Camargo Clay			Burleson Soil		
	Before F & Th TSF	After F & Th TSF	Strength loss %	Before F & Th TSF	After F & Th TSF	Strength loss %
D	7.3	7.72	- 5.75	13.2	13.93	- 5.5
D→W	10.5	6.42	38.86	14.6	13.73	6.0
O	10.3	6.50	36.89	15.0	12.02	19.9
O→W	11.5	4.64	59.60	13.4	11.30	15.7
W	14.9	5.68	61.88	19.5	14.51	25.2

Samples	Union City Clay			Summit Soil		
	Before F & Th TSF	After F & Th TSF	Strength loss %	Before F & Th TSF	After F & Th TSF	Strength loss %
D	17.6	14.44	17.5	19.1	14.12	26.1
D→W	13.7	4.69	65.8	17.8	9.91	44.3
O	23.9	14.46	39.5	24.8	15.60	37.1
O→W	17.3	7.51	56.6	22.2	11.28	49.4
W	25.1	11.61	53.8	24.1	14.64	39.3

principal factor governing the response of D, O, and W groups to freezing and thawing; the strength loss was directly proportional to the water content.

The response of Union City clay and Summit soil differed from that described for the Camargo and Burleson soils in that the O→W and D→W groups were affected more severely than was the W group. These two soils absorb water very slowly and there is some doubt that water migrated efficiently within the sample. If the moisture was irregularly distributed in the D→W group of Union City clay, as previously described, the high concentration of water in an outer shell of the soil, coupled with the poor development of strength in the drier core, led to poorer performance than was noted in the O→W and W groups. The Summit soil, in which a similar distribution of moisture exists, is less affected than the Union City clay, probably because better quality cementing agents are formed during the curing period.

Because moisture content and the distribution of moisture within a sample so greatly affect the results of freeze-thaw tests, this test appears unreliable for evaluating durability. First, it has been shown that the addition of moisture following compaction and before curing procedures has quite different effects on the strength of different soils. Second, the temperature cycling must be done so rapidly that the effect may be quite different from that which would be observed under natural conditions, where an opportunity for healing and strength recovery processes exists between successive cycles.

In general, it is clear that very substantial strength reductions result from cyclic freezing and thawing in the laboratory. One surprising and anomalous result of this study is that specimens of the two montmorillonitic soils compacted dry of optimum were stronger after three freeze-thaw cycles than they were before. No explanation for this can be offered.

Permeability Test

It was desired to investigate the effect of both lime content and curing conditions on the soil permeability. For each of the four soils the 0 and 0→W groups were prepared at the LMO and cured for 30 days prior to testing. The coefficient of permeability of the untreated soil was determined for specimens compacted at optimum moisture content and tested immediately thereafter. The results of this study are given in Table VII, in which the reported k for untreated soil is the average value of several successive determinations made during the course of one week while flow was maintained continuously. In most cases no measurable flow was observed for the first two or three days. After continuous flow was established, the rate of flow consistently decreased during the course of the week, probably because of swelling associated with the adsorption of water by the clay minerals.

In each of the four soils studied the coefficient of permeability was increased by the addition of lime, even with mellowing periods of not more than one hour permitted prior to compaction. The observed increase in permeability is in accordance with the findings of Townsend and Klym (80); however, their conclusion that long mellowing periods are needed is not supported in this study. The latter is regarded as very significant and indicates that the immediate reaction modifies soil properties very significantly; that the longer mellowing periods (8 to 24 hours) often used for field compaction may not be necessary,

TABLE VII

PERMEABILITY TEST RESULTS

	Camargo Clay			Burleson Soil		
	Uncured-Untreated Samples	Cured Samples		Uncured-Untreated Samples	Cured Samples	
	K cm/sec	K cm/sec	Ca^{++} ppm	K cm/sec	K cm/sec	Ca^{++} ppm
0	9×10^{-9}	4.7×10^{-4}	90	3.8×10^{-9}	1.4×10^{-3}	170
0→W		5.5×10^{-5}	60		2.8×10^{-4}	163

	Union City Clay			Summit Soil		
	Uncured-Untreated Samples	Cured Samples		Uncured-Untreated Samples	Cured Samples	
	K cm/sec	K cm/sec	Ca^{++} ppm	K cm/sec	K cm/sec	Ca^{++} ppm
0	1.6×10^{-9}	1.3×10^{-5}	21	4.5×10^{-9}	6.8×10^{-6}	335
0→W		3.9×10^{-7}	3		*1.45×10^{-6}	180

*Results from one specimen only. Two other specimens yielded no measurable flow.

and that the formation of carbonate through atmospheric exposure during long periods of mellowing can be practicably avoided if that is found to be desirable.

The effect of lime treatment on the permeability of the montmorillonitic Camargo and Burleson soils is quite remarkable: the coefficient of permeability of the treated soils was found to be at least 10,000 times greater than that of the untreated soil. The permeability of the Union City and Summit soils was also increased by lime treatment, but the increase was modest in comparison with that of the first two soils.

The difference in permeability of the 0 and 0→W groups varied among these soils, but specimens of the 0→W group consistently exhibited the lower permeabilities. Measurable flows were established through the treated specimens within the first few hours of the test and, unlike the untreated soils, no reduction in permeability was observed during the one-week test period. The lesser permeability of the 0→W group is believed to result from the more efficient production of cementing minerals in those specimens during the 30-day curing period and the consequently greater obstruction to flow through the soil voids. Specimens of the 0→W group of the Summit soil exhibited especially low permeabilities. No measurable flow was observed in two of the specimens after one week under a 50 psi fluid pressure differential; however, the coefficient of permeability of the third specimen was 1.45 x 10^{-6} cm/sec and the flow was easily measured.

Whenever the flow was sufficient, an identical quantity of effluent from each specimen of the 0 and 0→W groups was collected and analyzed for its Ca content. The effluent of the 0→W group contained much less than was present in the effluent of the 0 group. This is believed to reflect a greater abundance of unreacted lime in the 0 group, and is regarded as a further indication that the soil-lime reaction proceeds more effectively when more water is available during the curing period.

These test results lead to the conclusion that most clay soils (perhaps all) experience an increase in permeability when they are treated with lime prior to compaction. Apparently, however, the permeability of lime-treated clays must be significantly reduced by spraying water over the surface of compacted layers and maintaining a moist curing environment. This procedure also reduces the susceptibility of the soil to loss of lime through leaching, both because of the reduced permeability and because of the enhanced soil-lime reaction during the curing process.

Variation of pH

The variation of the pH of compacted samples with time for various lime contents is shown in Table VIII. The method used to prepare the 20 g compacted specimens was described in the preceding chapter. It was observed generally that the soil pH decreases with time, indicating a reduction in the amount of free calcium. The pH of the Camargo and Burleson soils with low lime content decreases sharply during the first 2 days of curing; but these same soils (particularly the Burleson) exhibit an increase in pH after 2 days when the lime content is above the LMO. In no case did the pH of any of these soils fall below 10.9 during the 90-day curing period. The Camargo samples compacted with 6% lime and cured for 30 days had a slightly higher pH than those cured for 10 days. No logical explanation for this can be offered. The

TABLE VIII

VARIATIONS OF PH WITH CURING TIME

(a)

Camargo clay	4% lime = pH 12.28				6% lime = pH 12.37				8% lime = pH 12.37			
	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days
D	11.89	11.35	11.12	10.96	12.21	11.53	11.33	11.05	12.55	11.57	11.40	11.19
D→W	11.90	11.30	11.28	10.96	11.97	11.48	11.50	11.00	12.27	11.55	11.42	11.11
O	11.85	11.33	11.29	10.90	12.17	11.42	11.55	11.13	12.37	11.49	11.40	11.11
O→W	11.85	11.35	11.24	10.93	12.04	11.35	11.41	11.13	12.26	11.47	11.38	11.07
W	11.82	11.37	11.26	10.96	11.97	11.36	11.47	11.10	12.21	11.52	11.43	11.05

(b)

Union City clay	2% lime = pH 12.41				3.5% lime = pH 12.47				5% lime = pH 12.50			
	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days
D	12.35	11.91	11.43	10.93	12.77	12.58	11.95	11.31	12.82	12.62	12.62	11.70
D→W	12.27	11.95	11.53	10.95	12.78	12.47	11.99	11.23	12.83	12.65	12.59	11.60
O	12.27	11.96	11.54	10.93	12.73	12.48	12.00	11.27	12.84	12.72	12.59	11.74
O→W	12.26	11.95	11.53	10.94	12.75	12.43	11.99	11.22	12.82	12.79	12.54	11.69
W	12.34	11.97	11.40	10.92	12.76	12.25	11.97	11.32	12.81	12.73	12.63	11.62

TABLE VIII (Continued)

(c)

Bürleson soil	3% lime = pH 12.22				4.5% lime = pH 12.38				6% lime = pH 12.43			
	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days
D	11.77	11.49	11.20	11.02	12.37	11.82	11.58	11.26	12.66	12.21	11.85	11.45
D→W	11.72	11.50	11.36	11.02	12.36	11.82	11.55	11.26	12.61	12.21	11.81	11.34
O	11.96	11.75	11.30	11.06	12.18	11.82	11.57	11.23	12.63	12.12	11.79	11.36
O→W	11.92	11.51	11.25	11.00	12.18	11.75	11.55	11.19	12.57	12.07	11.76	11.29
W	11.92	11.51	11.31	10.96	12.04	11.80	11.51	11.24	12.62	12.00	11.79	11.32

(d)

Summit soil	3.5% lime = pH 12.27				5.25% lime = pH 12.41				7% lime = pH 12.44			
	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days	2 days	10 days	30 days	90 days
D	12.37	12.10	11.83	11.16	12.53	12.40	12.18	11.69	12.65	12.62	12.19	11.65
D→W	12.32	12.04	11.74	11.18	12.52	12.36	12.04	11.60	12.69	12.66	12.13	11.63
O	12.24	11.96	11.78	11.18	12.56	12.39	12.09	11.58	12.63	12.61	12.09	11.64
O→W	12.32	11.91	11.66	11.13	12.56	12.32	12.02	11.59	12.62	12.57	12.09	11.62
W	12.26	12.04	11.62	11.12	12.53	12.43	12.03	11.57	12.60	12.56	12.09	11.57

Union City clay and Summit soil, especially for lime content equal to or greater than LMO, exhibited an increased pH after 2 days of curing. In cases where the pH exceeds 12.5, the presence of Na and K ions may be indicated and it should be noted that Na_2O and K_2O were more abundant in the illitic Union City clay and the Summit soil than in the other two. Although the K ions may be initially bound in the clay mineral structure, they may be released during the corrosive reaction generated by the lime.

The pH test proved to be a good test for evaluating the soil-lime reaction process. However, since the maximum change in pH during the curing period between 2 days and 90 days was only 1.3, it appears impractical to use this test as a means of predicting differences in behavior of different soil types. In addition to the small range of the pH during the 90 days, the pattern of variation tends to be somewhat erratic.

CHAPTER V

MINERALOGY OF SOIL-LIME MIXTURES

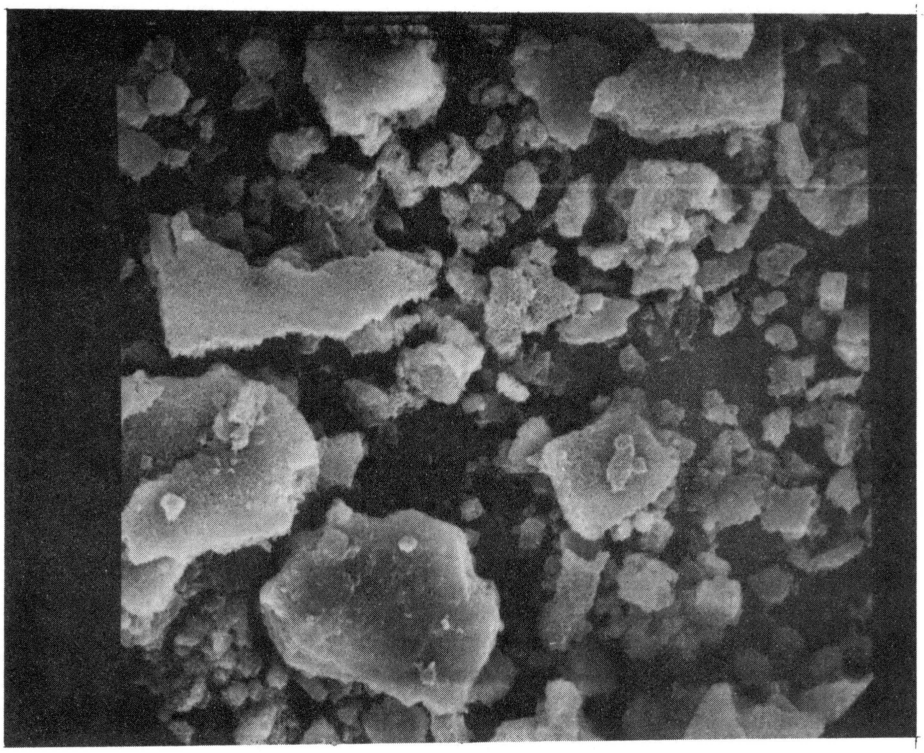
In this chapter morphological and chemical studies used to identify new minerals formed during the reaction of lime with soil are described, and the results analyzed. The procedures used to prepare test specimens for these investigations were described in Chapter III. Compacted discs of the various soil-lime mixtures were cured for periods of time ranging from 2 to 90 days. Most of these specimens, in an intact state, were subjected to X-ray diffractometry and some selected specimens were examined in a scanning electron microscope. These analytical tools, especially when used jointly, are capable of providing a good understanding of the chemistry and morphology of minerals and of facilitating their identification.

The scanning electron microscope (SEM) allows chemical, structural, and morphological data to be obtained from individual particles as small as 0.5μ. However, chemical data obtained by the SEM was minimized by certain limitations of the particular instrument used in this study. With the SEM differences in the appearance or form of a material on an exposed surface may be observed regardless of its abundance on the surface, while X-ray diffractometry is limited by its dependence on the degree of crystallinity of a mineral and by the relative abundance of that mineral. Coordinated studies permit the respective advantages of the two tools to be optimally combined. New minerals may be identified through the appearance of new peaks in the X-ray diffractogram. If no new peaks are observed, it indicates either that no minerals are present or that the new minerals are amorphous, or that the quantity of the new mineral is too low to be detected. There is one more possibility which is that the crystals are so randomly oriented that no enforcement of the diffracted signal can be detected.

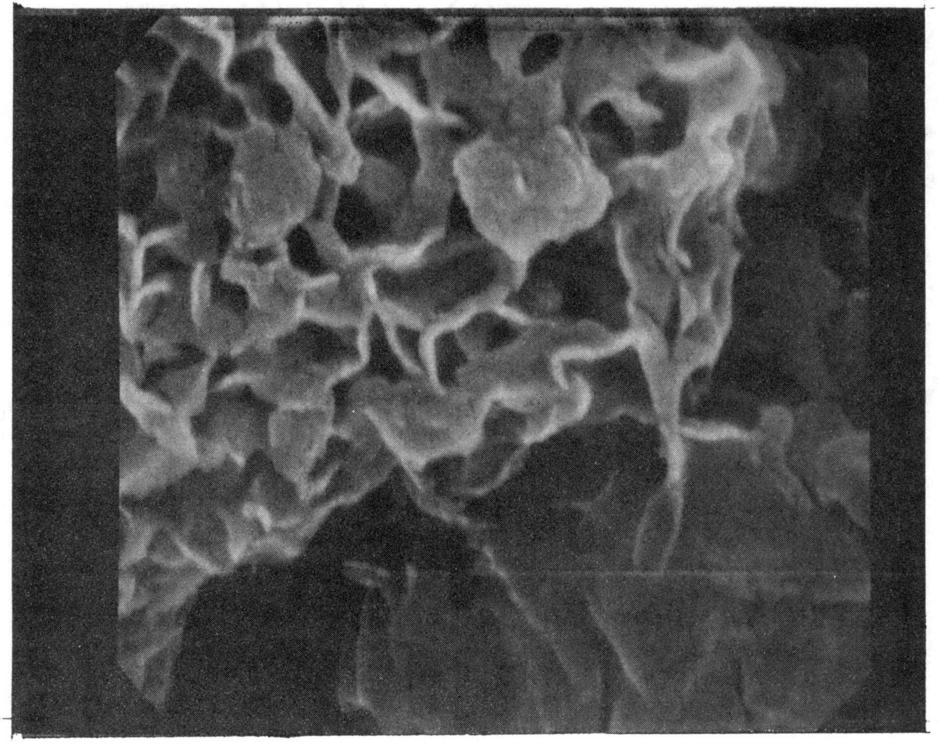
All soils were examined by X-ray diffraction and selected specimens were then studied in the SEM. Specific results and interpretations pertaining to the four different soils are described below.

Camargo Clay

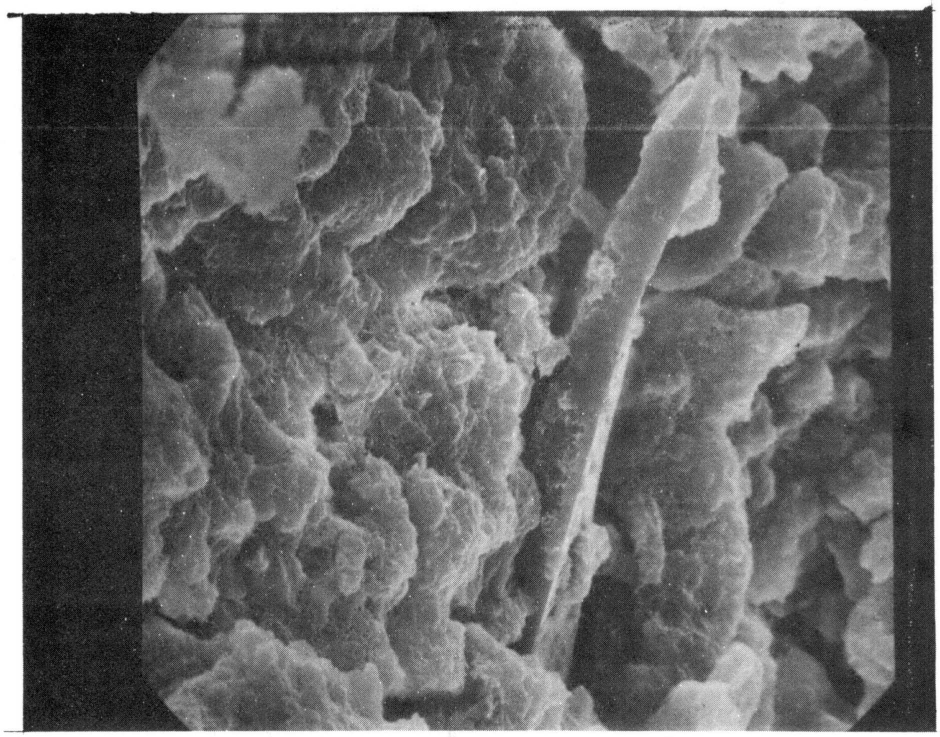
Photmicrographs 1 through 7 show both the raw soil and lime-treated compacted samples. It appears that this Ca-montmorillonite soil, seen in Photomicrograph 1, is composed of particles ranging in size from 1μ to 30μ. However, the magnification of a large particle (Photomicrograph 2) reveals that it is composed of many small irregular particles linked together. The spongy structure of this clay explains its high water absorptive capacity and poor compaction characteristics. Its distinctive structure arises from the relative strength of the attractive forces at the edges of particles and along their basal planes. Even when this structure is fully saturated, the mobility of the absorbed water is likely to be very low. Since no quartz particles could be detected, the silt and sand sizes present in the hydrometer analysis of the soil are apparently composed of tightly-bound clusters of clay particles that are unaffected by the dispersing agents used. Photomicrograph 3 and 4 show a D→W group specimen, treated with 8% lime and cured for two days. No crystal growth is visible in these



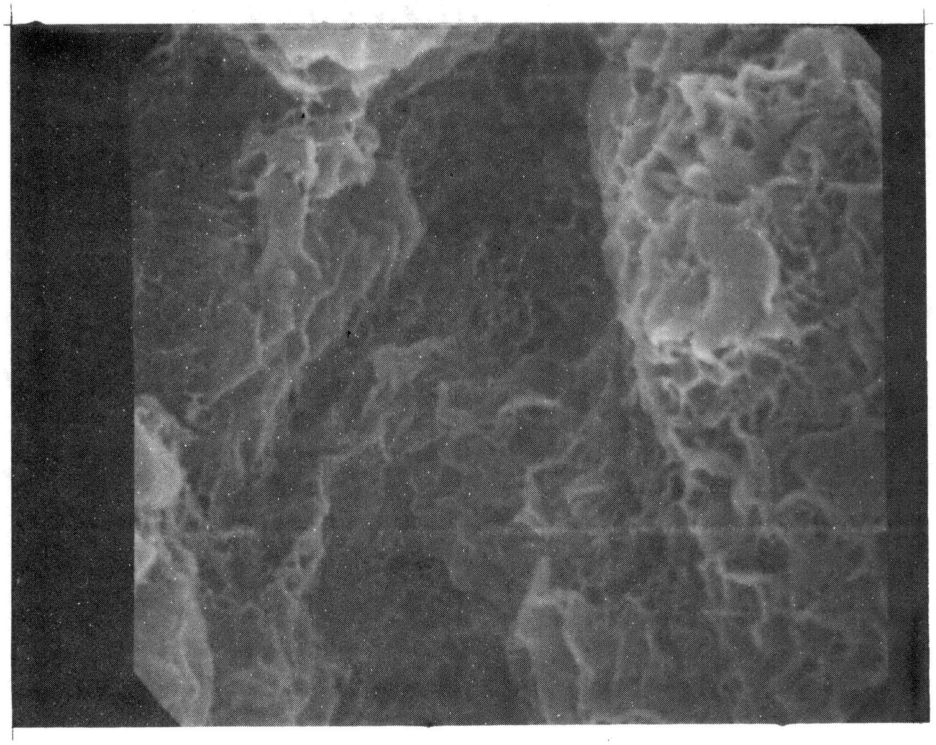
Photomicrograph 1. Camargo Clay 1100X-Untreated



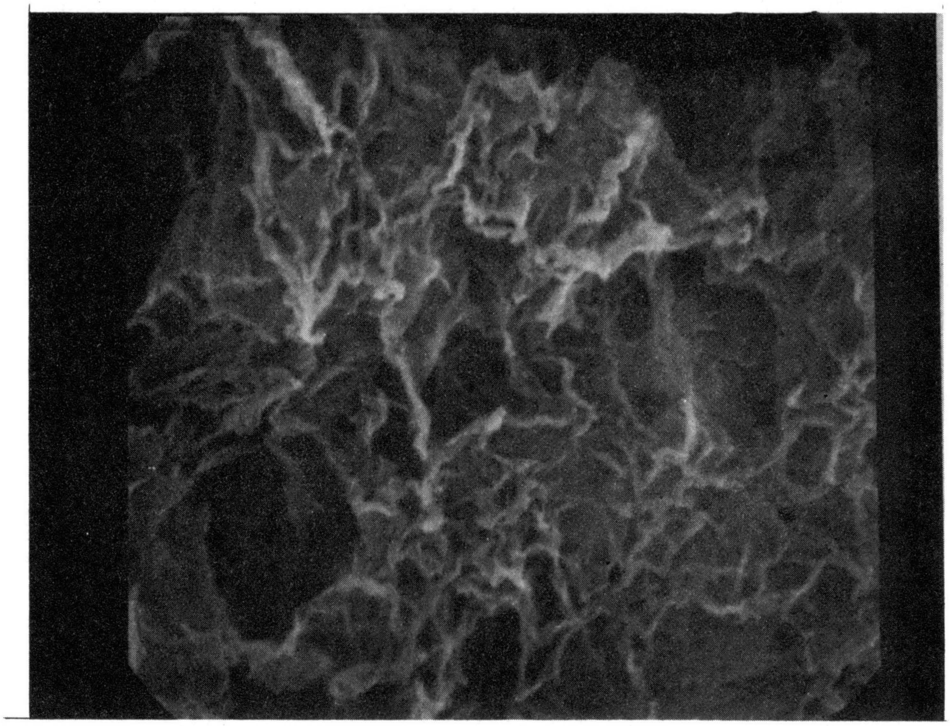
Photomicrograph 2. Camargo Clay 22000X-Untreated



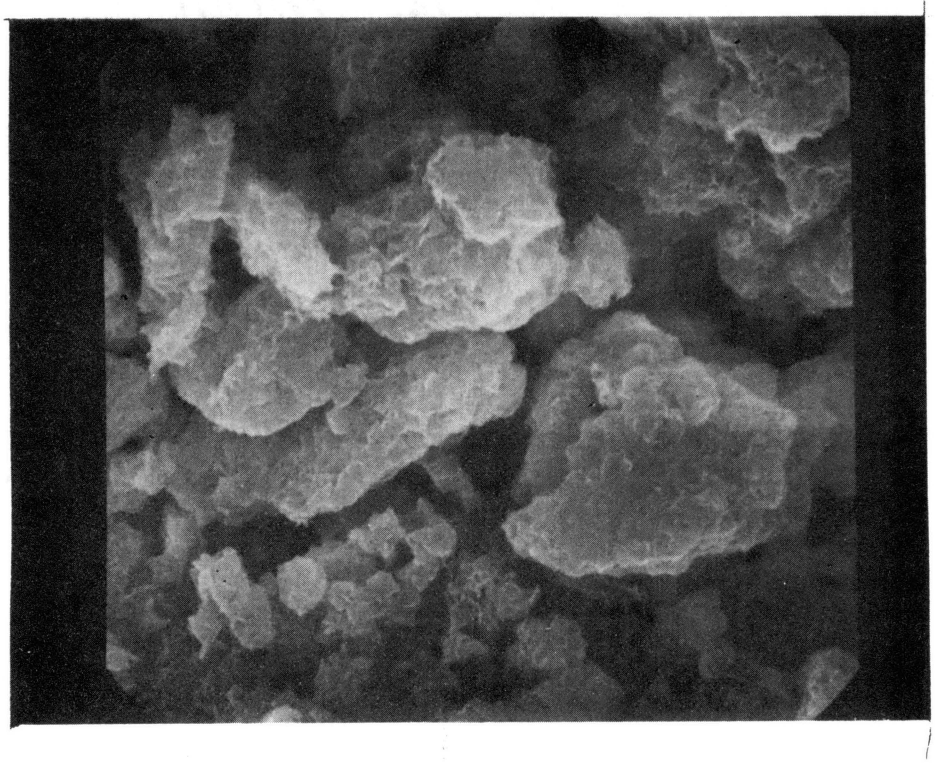
Photomicrograph 3. Camargo Clay 1600X-8% Lime-2 Day Cure-Group D→W



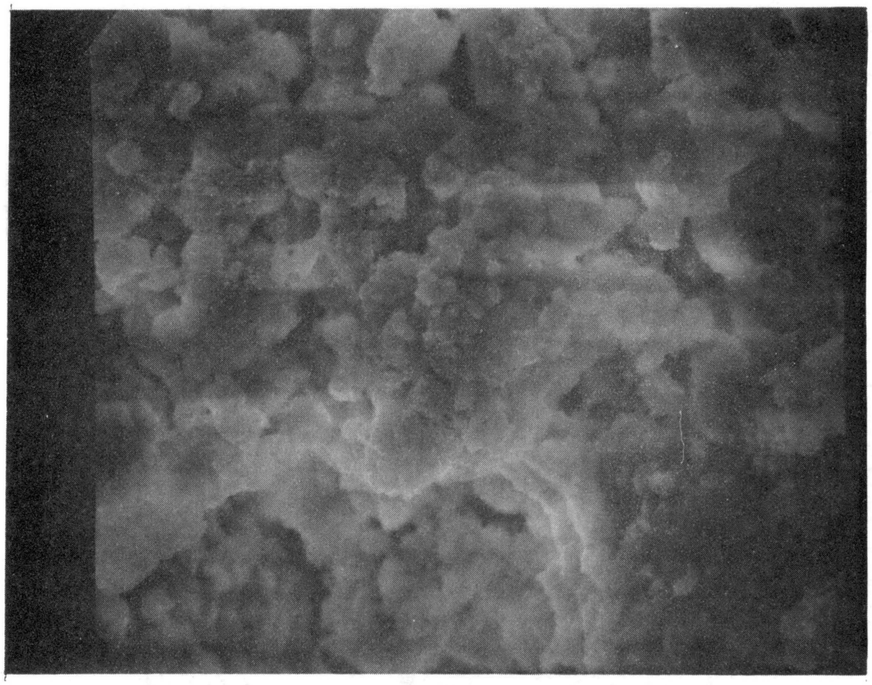
Photomicrograph 4. Camargo Clay 11000X-8% Lime-2 Day Cure-Group D→W



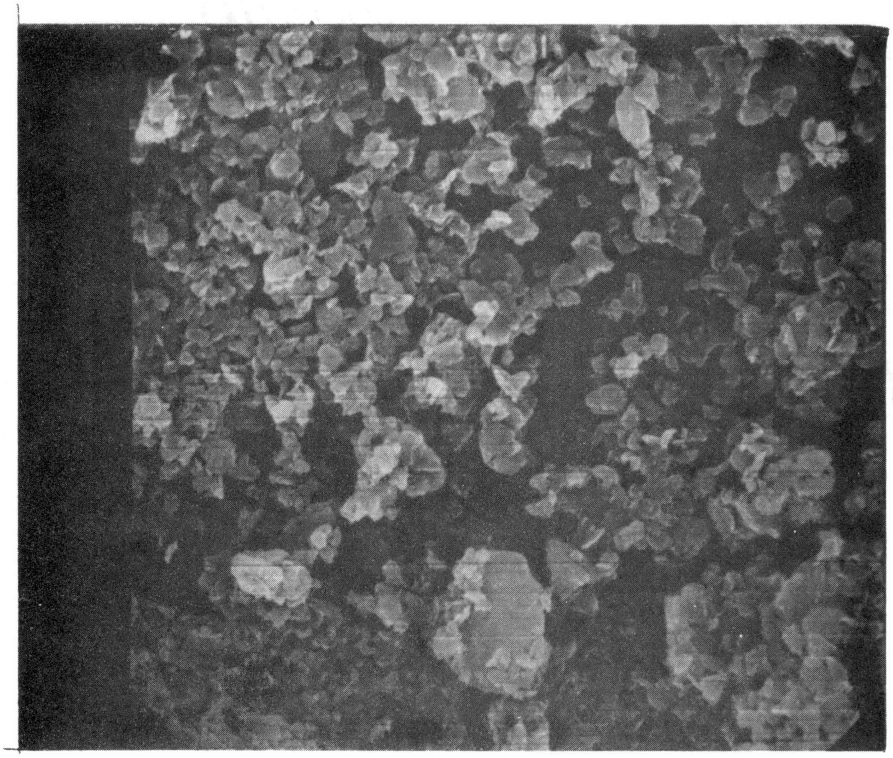
Photomicrograph 5. Camargo Clay 15750X-8% Lime-30 Day Cure-Group O→W



Photomicrograph 6. Camargo Clay 3150X-8% Lime-30 Day Cure-Group O→W



Photomicrograph 7. Camargo Clay 1210X-8% Lime-90 Day Cure-Group W



Photomicrograph 8. Burleson Soil 3300X-Untreated photographs; however, the exposed edges indicate that some chemical attack has taken place. The soil fragments are well-separated, forming fairly large voids which may reduce the change of forming any cementing material through dissolution of the clay. The general appearance of the sample indicates that a coating material has formed on the clay surface. A closer look at the surface (Photomicrograph 4) reveals that some ridges are protruding from that coating. These may be the edges of clay particles or the embryonic stages of a cementing product. Cementing material, if present, would probably be a form of CSH(gel) or CSH(I). The major difference in the two is that CSH(gel) has a Ca:Si ratio of 1.5 to 3.0, whereas the Ca:Si ratio of CSH(I) may be as low as 0.8. Both of these minerals can be formed at room temperature, and the possibility that CSH(I) is present must be considered. It may result either from the reaction of lime, silica, and water or from the reaction of a solution of calcium with silica in alkali environment. The first of these is a slow reaction, requiring weeks or months of shaking in the laboratory. The second type of reaction is rapid, but often gives a less well-crystallized product. Since the samples used in this study are in a compacted form, the second type of reaction is the most probable, and it would not be surprising to find CSH(I) in a poorly-crystallized phase. This is the same form of CSH(I) that might be found in the field.

Photomicrographs 5 and 6 show the progress of the reaction after 30 days. Photomicrograph 5 provides evidence that the ridges observed earlier are formed by a new mineral, while Photomicrograph 6 illustrates the large voids within the samples. The CSH(I) is highly impermeable, but has not bridged over the voids or plugged them. These two photographs suggest a reason for the high permeability noted in Chapter IV. The water can move readily through the large voids and the clay cannot swell to reduce the void size, since it has been wrapped and immobilized by the CSH(I). The gain in shearing resistance is mainly due to the formation of CSH(I) and to its confining and immobilizing effect on the clay surface. Once this binding material fails, the internal strength of the specimen will be lost.

The only conclusion that can be drawn from Photomicrograph 7 is that CSH(gel) and CSH(I) are increasing in quantity, with time, without any noticeable increase in quality.

Samples compacted dry of optimum or with a lime content of less than 8% were similar in appearance but not as strongly developed as those shown in Photomicrographs 3 through 7.

The X-ray diffractograms of the treated soil show some additional peaks belonging to the cementing material. Diffractograms for the raw soil are shown in Figure 6 and for the treated soil in Figure 21. Although the tracings were similar for all treated samples, that in Figure 21 exhibited the new peaks most distinctly. There was no noticeable difference among the O, D, O→W, and D→W groups. The major additional peaks occurred at 9.4 to 8.2, 6.66 to 5.80, 3.45, 3.21, 3.05, 3.03, 2.75, 2.56, and 2.53 Angstroms. Most of these peaks are weak and broad. It was reported by Carroll (11) that a cementing material such as calcite will not be identifiable in maximum intensity X-ray diffraction if it is present in amounts less than 5%.

X-ray diffraction data may be inconclusive for several reasons: (a) the amount of new material may be insufficient; (b) its crystallinity may be low grade; and (c) compacted specimens may not produce

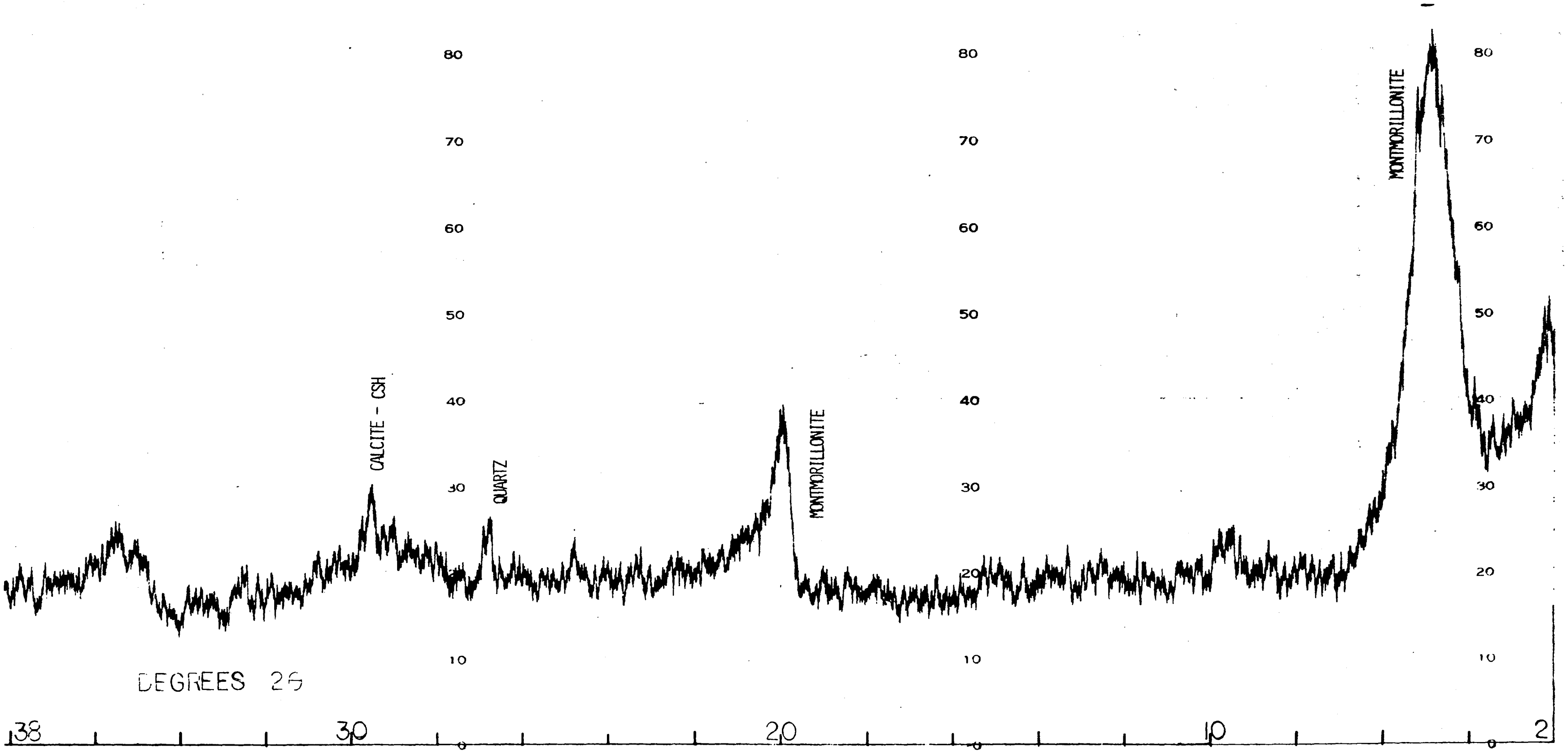


Figure 21. X-Ray Diffraction Pattern for Camargo Clay - 8% Lime - 90 Day Cure - O→W

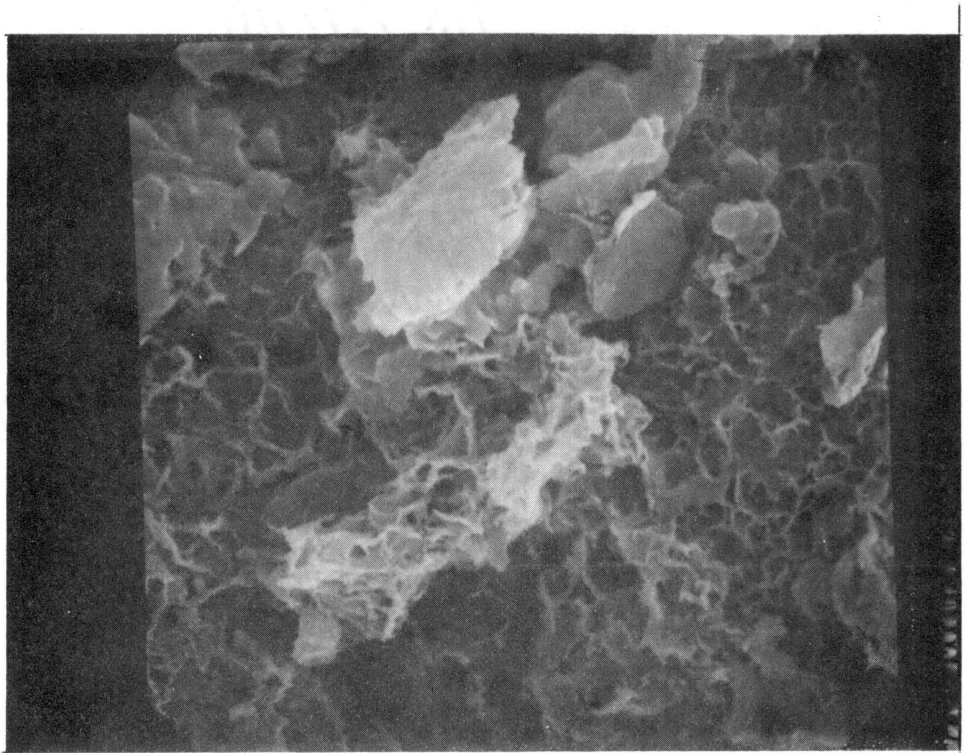
maximum intensity of diffracted peaks. To confirm that a given material is represented by these peaks, two or more of the peaks have to match the standard diffraction data for that material. Reference to the standard data in Appendix B will show that the matching job is not easy. Some of the observed new peaks coincide with more than one crystalline form, and not more than two are found in the standard data for any particular material. In some cases it is difficult to be sure that a new peak does in fact exist, or that there is not a slight error in the indicated value of 2 θ. Thus, it has not been possible to determine from the diffraction data precisely what the newly formed cementing materials are. The data do indicate, however, that it is probably made up of one or more of the following compounds: calcite, CSH(I), CSH(II), Afwillite, or $C_2SH(A)$.

Burleson Soil

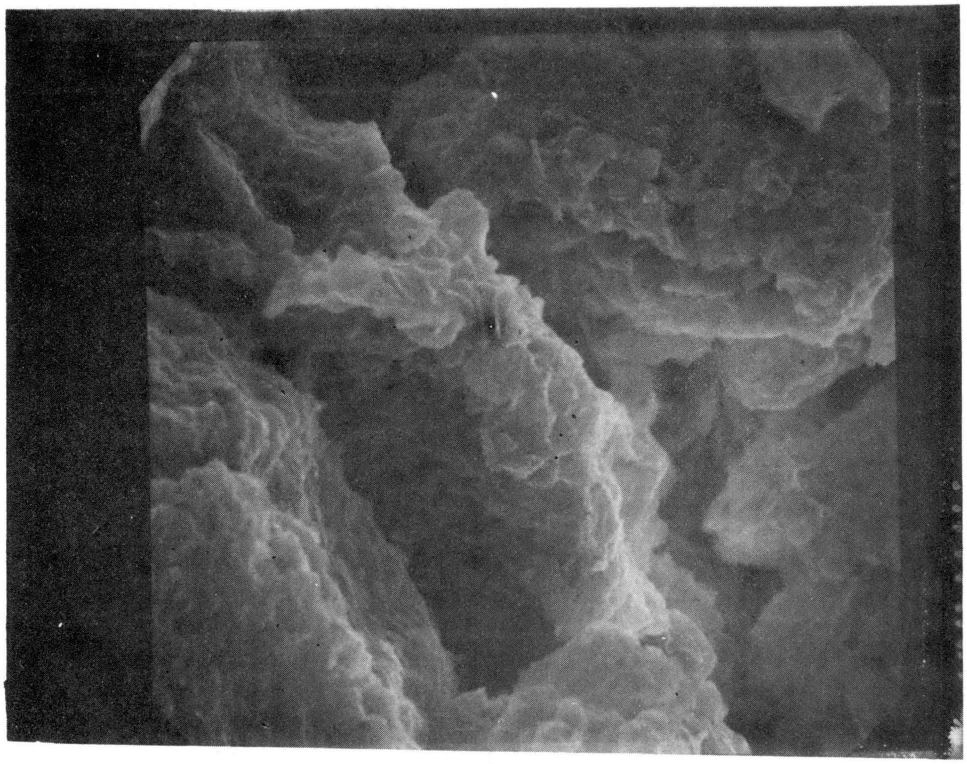
The clay fraction in the Burleson soil, as in the Camargo, is made up of Ca-montmorillonite, and the fairly low dry density of Burleson soil leads one to speculate that these two soils may produce the same type of cementing material. Photomicrograph 8 shows that this soil is composed of smaller particles than were found in the Camargo soil. The enlargement of a small area, where it appeared that a thin coating of organic matter was present, is shown in Photomicrograph 9, in which some staining of cracks may also reflect an organic content. Of principal interest is the fact that the organic material appears to be mainly associated with particle surfaces.

Photomicrographs 10 and 11 illustrate the difference between the O and O→W groups after 2 days of curing. It is evident that both

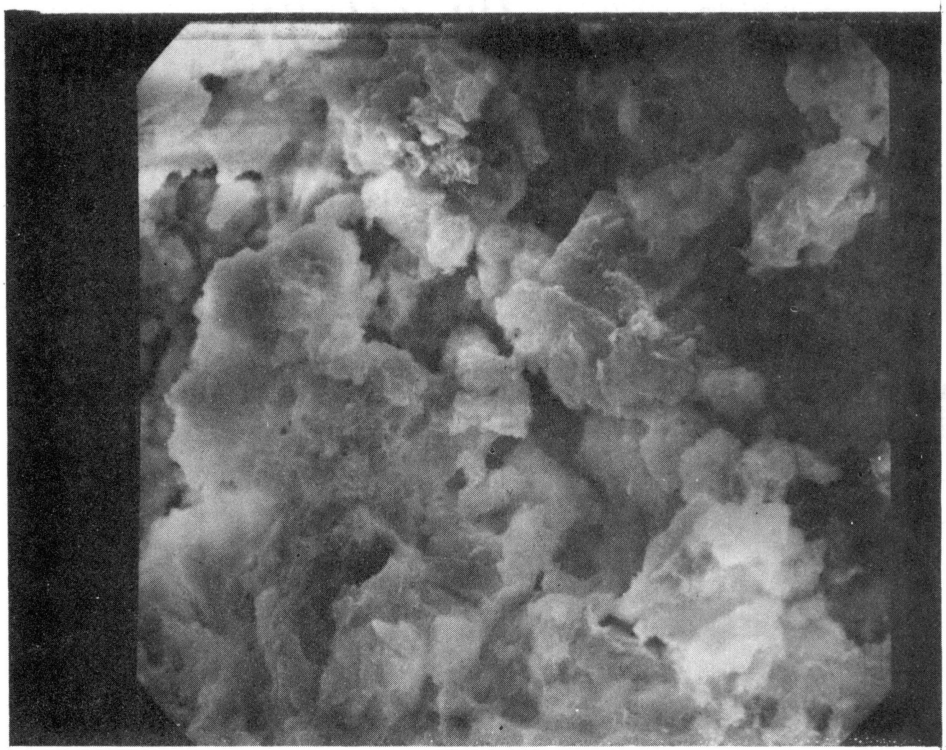
Photomicrograph 9. Burleson Soil 22000X-Untreated



Photomicrograph 10. Burleson Soil 7700X-6% Lime-2 Day Cure- Group O→W



Photomicrograph 11. Burleson Soil 2640X-6% Lime-2 Day Cure-Group O

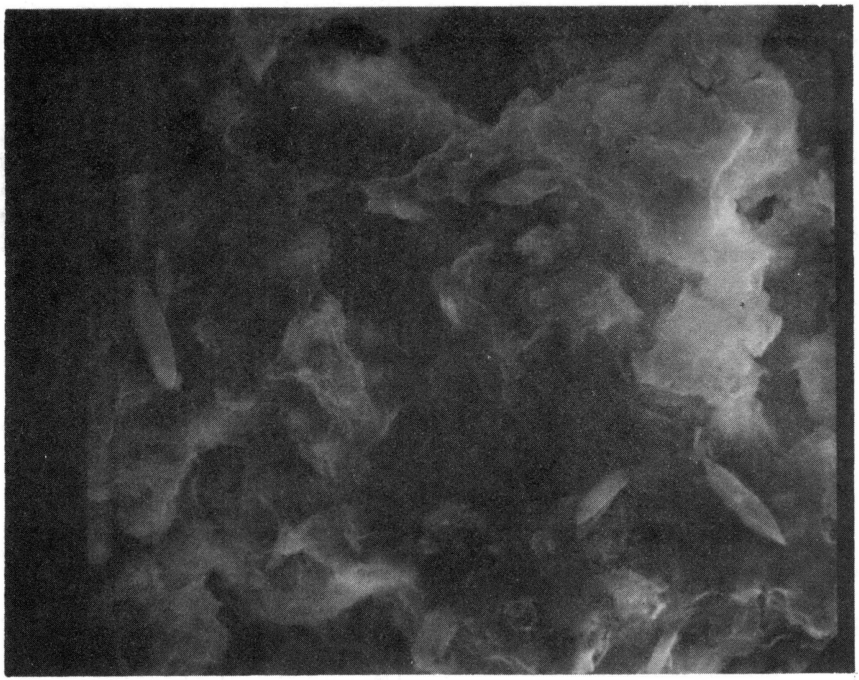


Photomicrograph 12. Burleson Soil 1100X-6% Lime-30 Day Cure-Group O→W materials were significantly attacked by the lime. However, Photomicrograph 10 shows the same kind of irregular edges on the coated surfaces as appeared in the Camargo soil after 30 days.

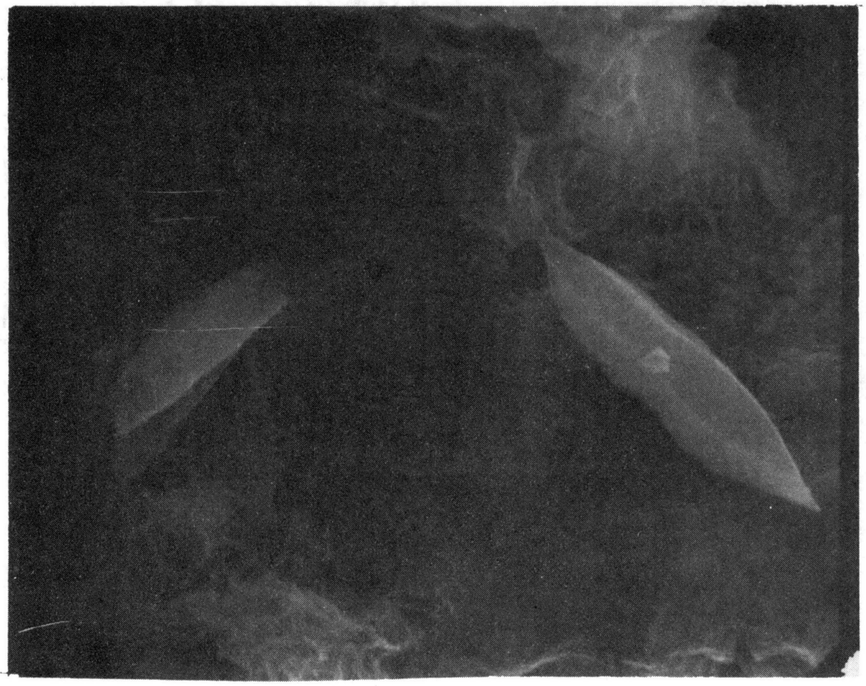
In Photomicrograph 11 the material appears to have been uniformly attacked, as no sharp edges may be seen in the closely packed particles. The cementing material in Photomicrograph 10 is probably CSH(I) whereas that in Photomicrograph 11 is probably CSH(gel). It may be reasoned that without dilution of the lime concentration in the O group the Ca:Si ratio is very high, which is a requirement for the formation of CSH(gel). In the O→W group the pH was not changed by the dilution but more clay surface area was exposed to the slurry, enhancing the Si release mechanism. Since the total Ca content did not change and the available Si increased, the Ca:Si ratio should have decreased to a point which is more favorable for the formation of CSH(I).

A sample of the O→W group treated with 6% lime and cured for 30 days is shown in Photomicrograph 12. The sample has experienced a very intense chemical attack and an abundance of CSH(gel) and CSH(I) are evident. The CSH(I) may be seen concentrated in the upper right-hand part of the photomicrograph, but there is as yet no good definition of crystallization.

Photomicrographs 13 and 14 show some evidence of strong crystallization after 90 days of curing. The few visible crystals appear to be growing from the soil base. The chemical components of this material were determined by the use of X-ray analysis (see Figure 22) and were found to be Ca, Si, and P. The Ca:Si ratio was found to be similar to that of CSH(II). While the existence of P casts some doubt as to whether the crystals are CSH(II), they cannot be CAH because no Al



Photomicrograph 13. Burleson Soil 1100X-6% Lime-90 Day Cure-Group O→W



Photomicrograph 14. Burleson Soil 3850X-6% Lime-90 Day Cure-Group O→W

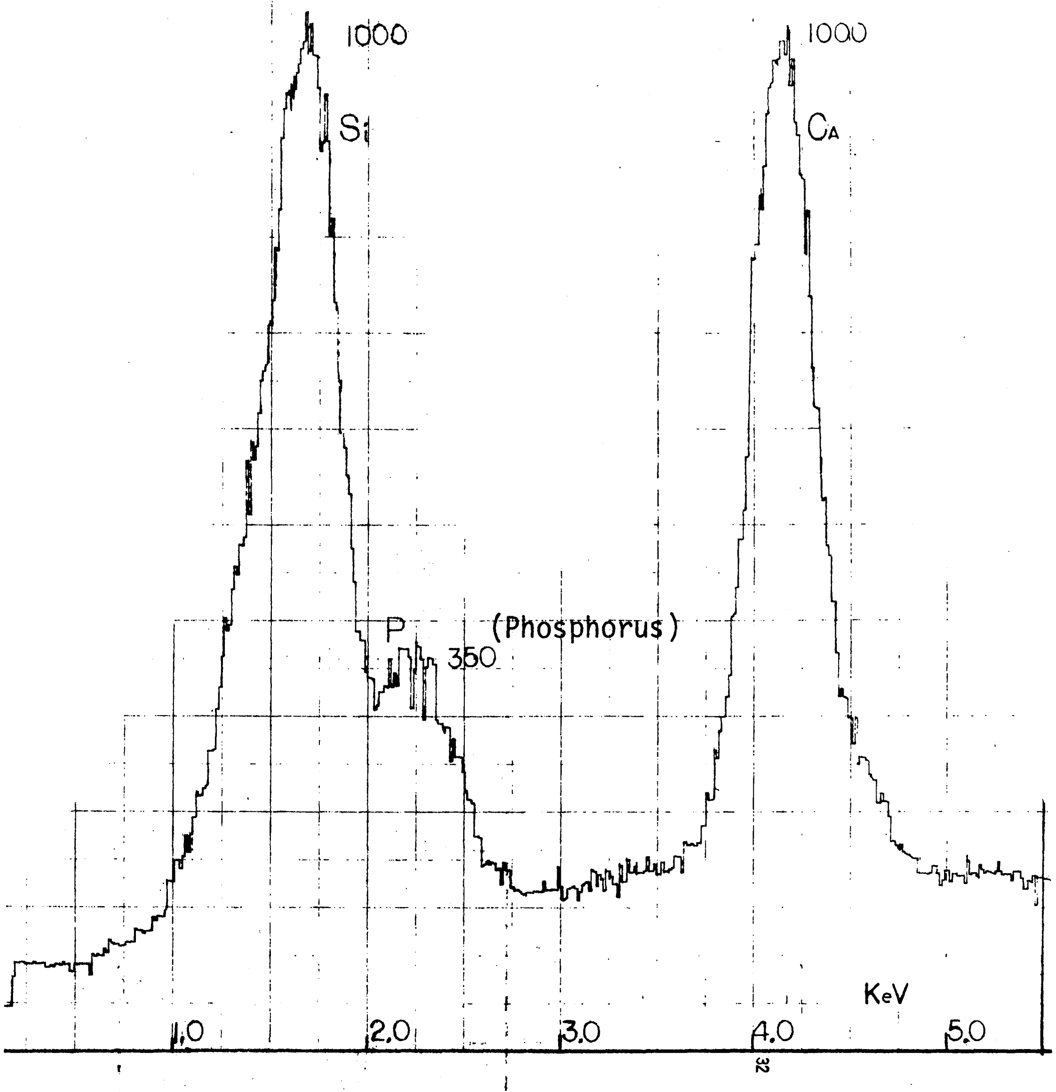


Figure 22. Electron Energy Emission Data - Burleson Soil - 6% Lime - 90 Day Cure - Group O→W (60 Second Scanning Time and Maximum County of 1000)

is present. Although no precise conclusion can be drawn as to their composition, the finding of such crystals in a compacted soil with a low lime content is itself significant because it increases the expectation for the development of strong crystalline minerals under long-term field conditions.

X-ray diffractograms are shown in Figure 7 for the raw Burleson soil and in Figure 23 for a sample from the O→W group treated with 6% lime. The latter is reasonably typical of those obtained for all lime-treated Burleson samples. The diffractogram for the treated soil is quite similar to that for the Camargo clay, in that no newly formed mineral peaks appear in the Burleson tracing that are not also present in the Camargo tracing. There is, however, one conspicuous difference in the two: the peaks identifying the original clay mineral in the diffractogram for the raw Burleson soil are not present in the diffractogram for the treated soil. This does not mean that the original soil has itself vanished, for not more than 5% of it could have been consumed in reacting with the lime. It simply means that the soil grains are thoroughly coated with the newly formed materials--a fact which is also evident in the SEM photographs.

The quartz peak is still present in the tracing for the treated soil but is reduced in size, possibly by the caustic attack but more probably because of the coating of new minerals enveloping the soil grains.

The new peaks that appear in the tracing for the treated soil correspond rather well with those of calcite (3.028, 2.462, and 2.098 Angstroms). Calcite, if present, probably results from the organic content of this soil rather than from CO_2 in the gaseous phase of the

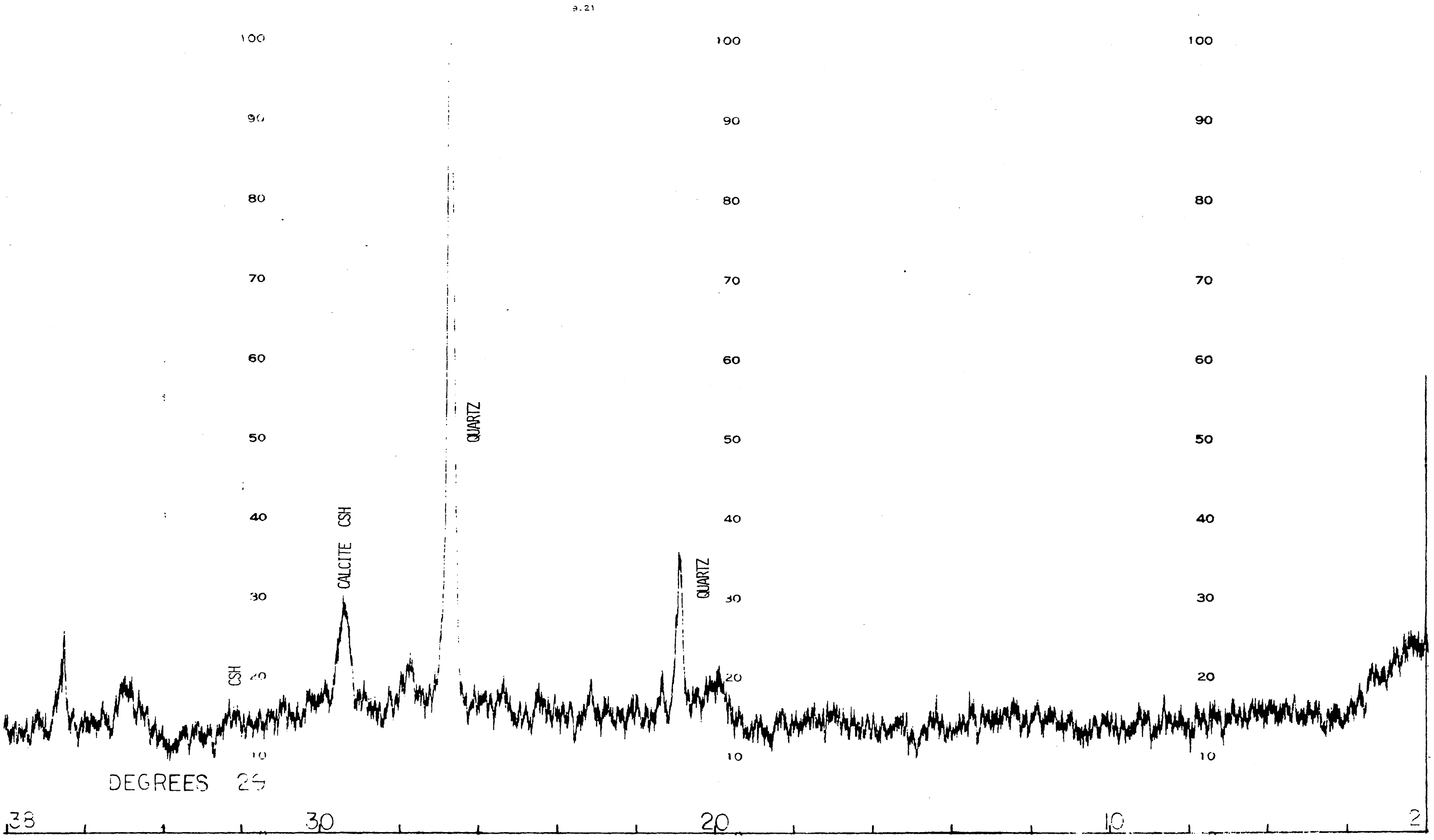


Figure 23. X-Ray Diffraction Pattern for Burleson Soil - 8% Lime - 90 Day Cure - O→W

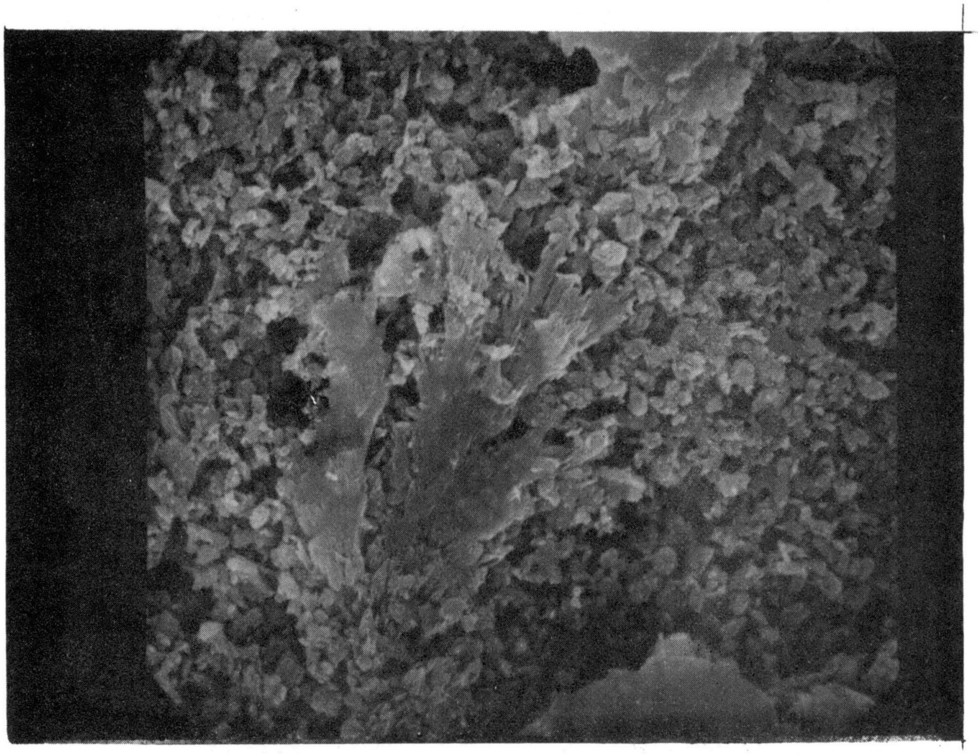
soil. The presence of calcite would not preclude a supposition that other cementing minerals are also present, even though they may not be identifiable in the diffractogram. It was found, for example, that the strong crystals previously described as being present after 90 days of curing are composed primarily of Ca and Si.

The strength tests showed that specimens of the O→W and D→W groups were superior to those of the O and D groups. In particular, there was a great difference in the behavior of the D→W and D groups; yet, there were no significant differences in the X-ray diffractograms for specimens from all the different groups. While it is discouraging to be unable to identify precisely the mineralogical reasons for differences in the behavior of the different groups, this fact emphasizes the shortcomings of the tools available for mineralogical studies when only minor differences are present in the minerals of greatest interest. However, these data, in conjunction with the SEM photomicrographs, strongly suggest that lime-treated soils undergo mineralogical alterations over long periods of time that are capable of influencing their stress-deformation characteristics.

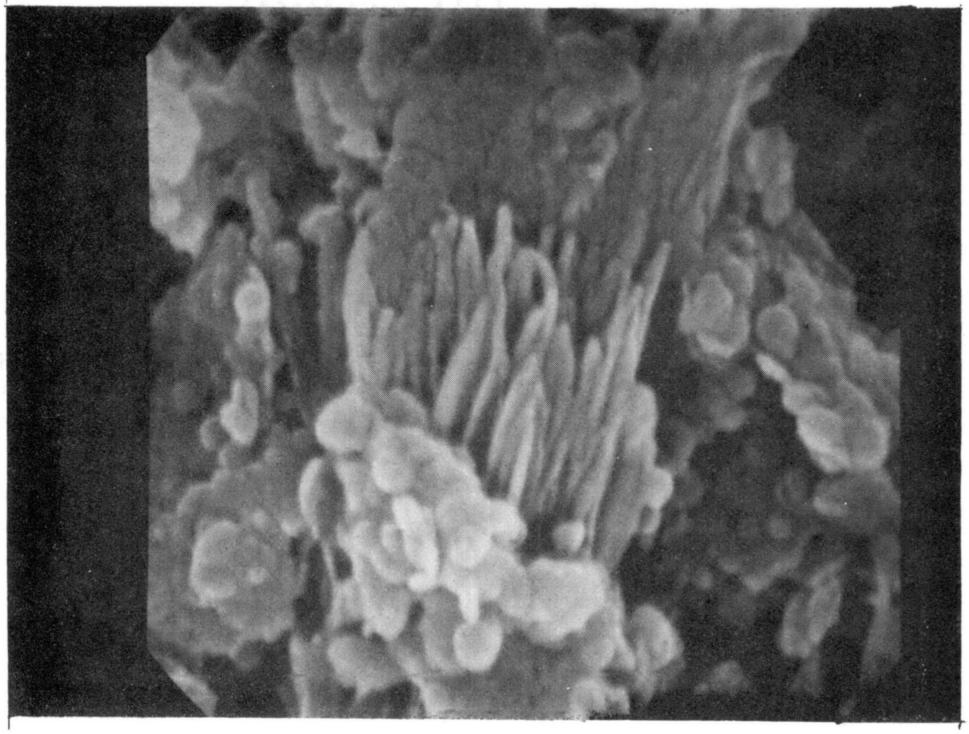
Union City Clay

Photomicrograph 15 shows that the raw Union City clay consists largely of particles having a fairly uniform size of less than 1.0μ diameter. There are also some much larger particles, one of which is shown highly magnified in Photomicrograph 16. This rather brush-form packing of illite structural units is highly vulnerable to lime attack.

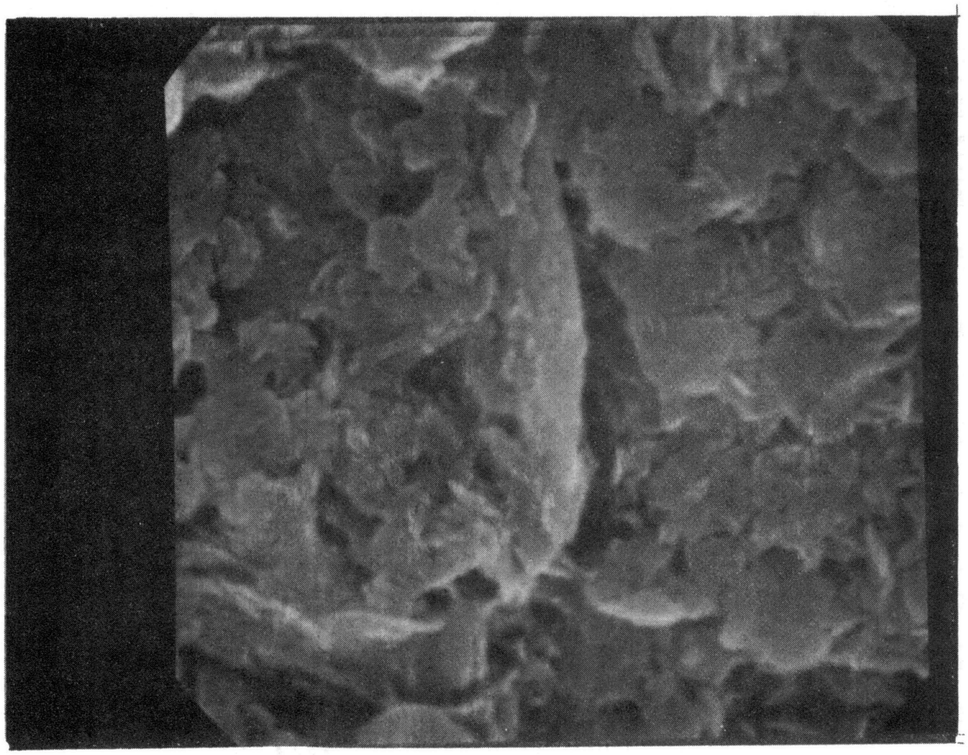
Photomicrographs 17 and 18 were taken of the same general area of a specimen from the W group 2 days after it was treated with 5% lime.



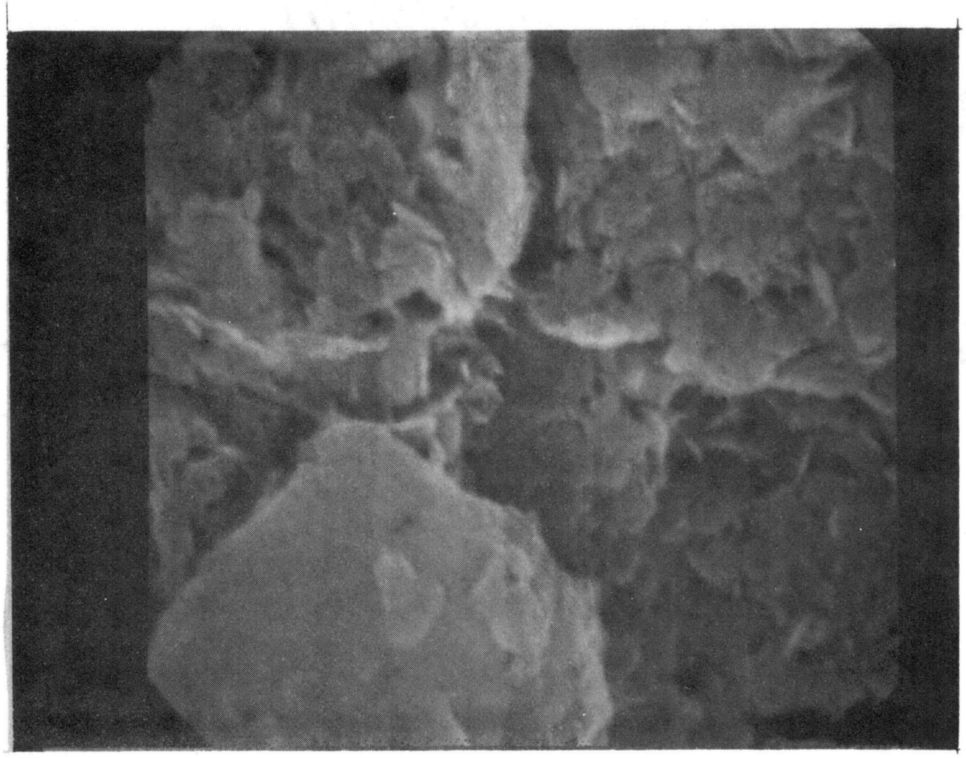
Photomicrograph 15. Union Clay 3300X-Untreated



Photomicrograph 16. Union Clay 27500X-Untreated



Photomicrograph 17. Union Clay 15000X-5% Lime-2 Day Cure-Group W



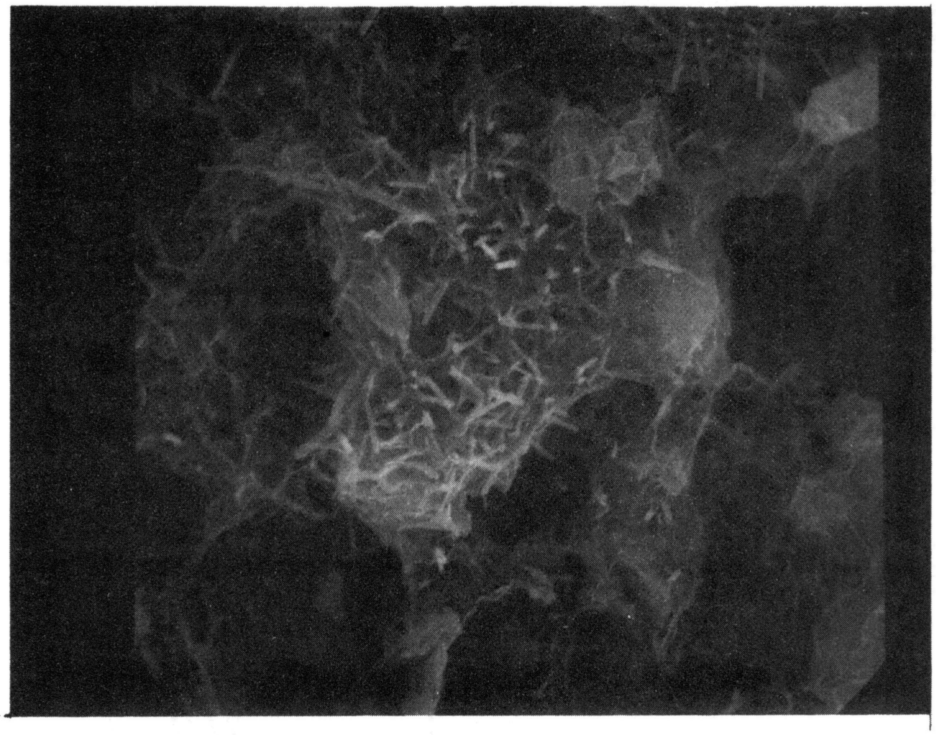
Photomicrograph 18. Union Clay 15000X-5% Lime-2 Day Cure-Group W

A sparse, scattered pattern of small, needle-like mineral crystals is already evident and it may be seen that there are no large voids in the soil structure. It should be noted that SEM studies were restricted to those groups containing 5% lime.

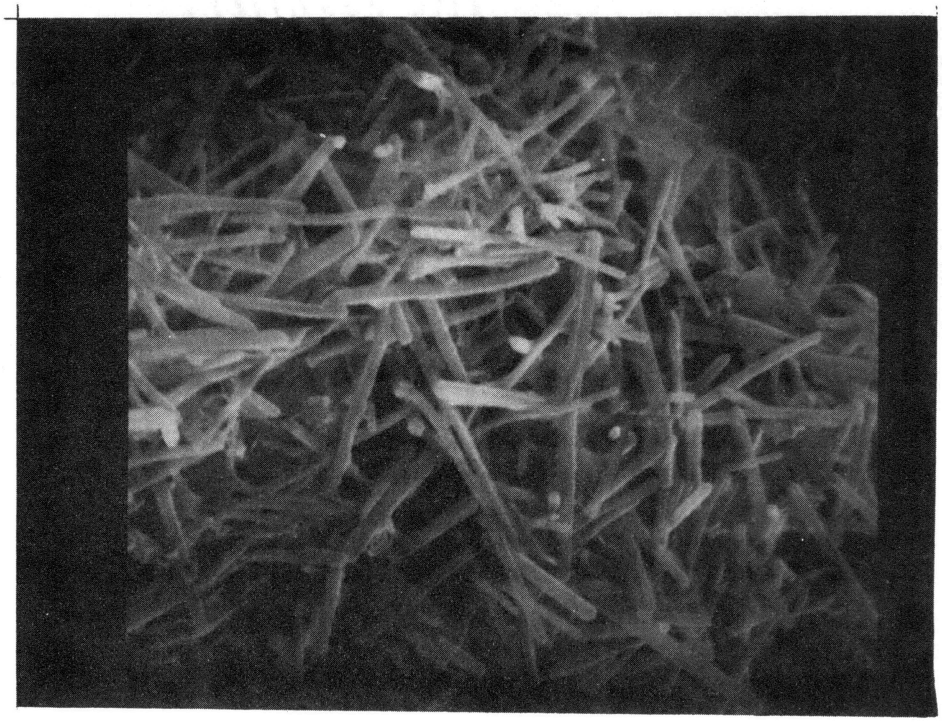
Photomicrographs 19 and 20 show that in the O→W group, after 30 days of curing, the crystalline growth observed in rudimentary form in the W sample after 2 days of curing had vastly proliferated. This profusion of crystals was also present in the D→W specimens but not in the O and D specimens. In Photomicrograph 19 it appears that the crystals are growing from the soil grains. A further magnified view of the same area is shown in Photomicrograph 20. The highly magnified close-up in Photomicrograph 21 shows that most of the crystals start from a soil grain and they are not cylindrical. The clear view of a cross section in Photomicrograph 22 indicates that the crystal is hexagonal uniaxial. The hexagonal angles are very nearly equal to 120° and the sides are about equal in length. This crystalline form is very similar to that of CSH(II), except that CSH(II) consists of semi-crystalline bundles of fibers.

Photomicrographs 23 and 24 are of an O→W specimen after 90 days of curing. The crystals have by this time become quite large and extensively distributed and in some places are quite densely packed, as shown in Photomicrograph 24.

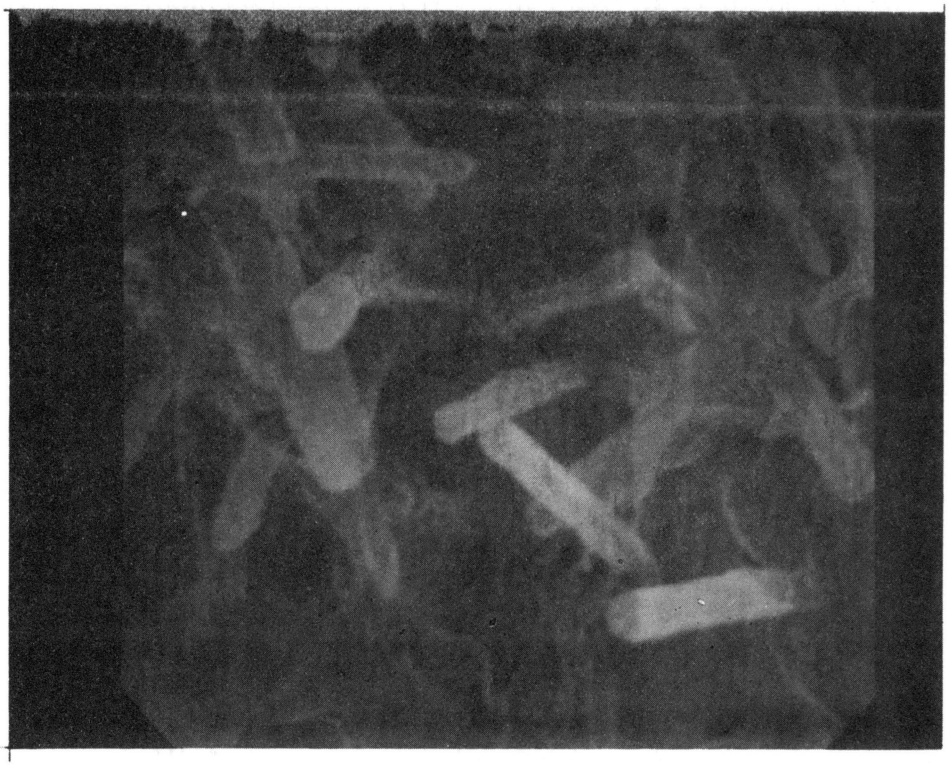
Through an X-ray examination of the electron energy emission of the crystals (Figure 24) the presence of Ca, S, and Al was disclosed. On the basis of a 60-second count, it was concluded that the Ca:S ratio in the crystals is 2.0, corresponding to the mineral Ettringite. This mineral is a highly hydrated ($32H_2O$) calcium aluminate trisulphate,



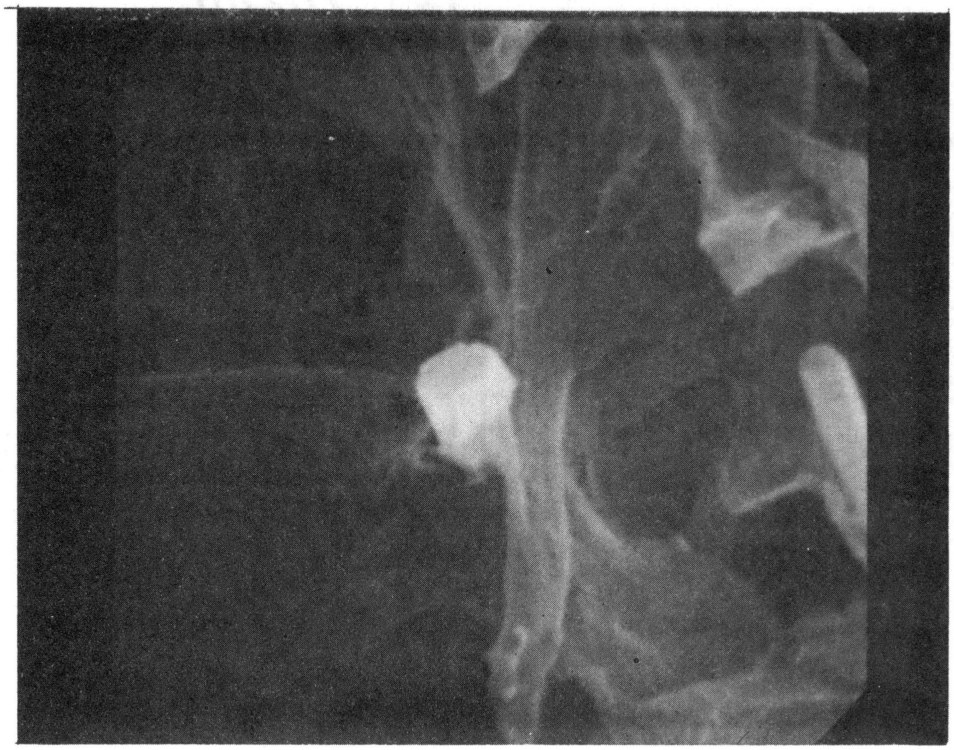
Photomicrograph 19. Union Clay 3300X-5% Lime-30 Day Cure-Group O→W



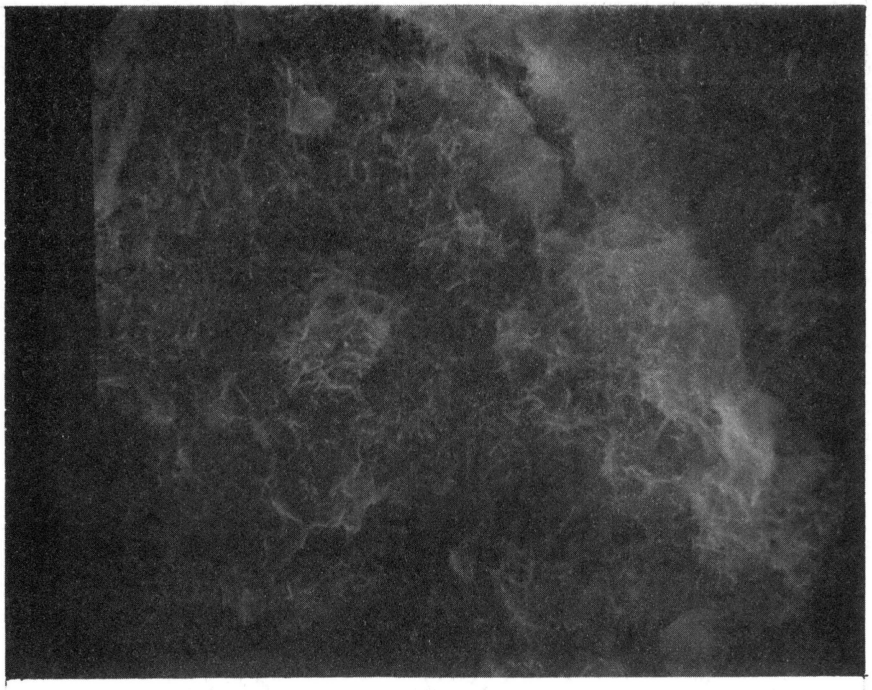
Photomicrograph 20. Union Clay 7700X-5% Lime-30 Day Cure-Group O→W



Photomicrograph 21. Union Clay 27500X-5% Lime-30 Day Cure-Group O→W



Photomicrograph 22. Union Clay 33000X-5% Lime-30 Day Cure-Group O→W



Photomicrograph 23. Union Clay 2420X-5% Lime-90 Day Cure-Group O→W

Photomicrograph 24. Union Clay 2420X-5% Lime-90 Day Cure-Group O→W

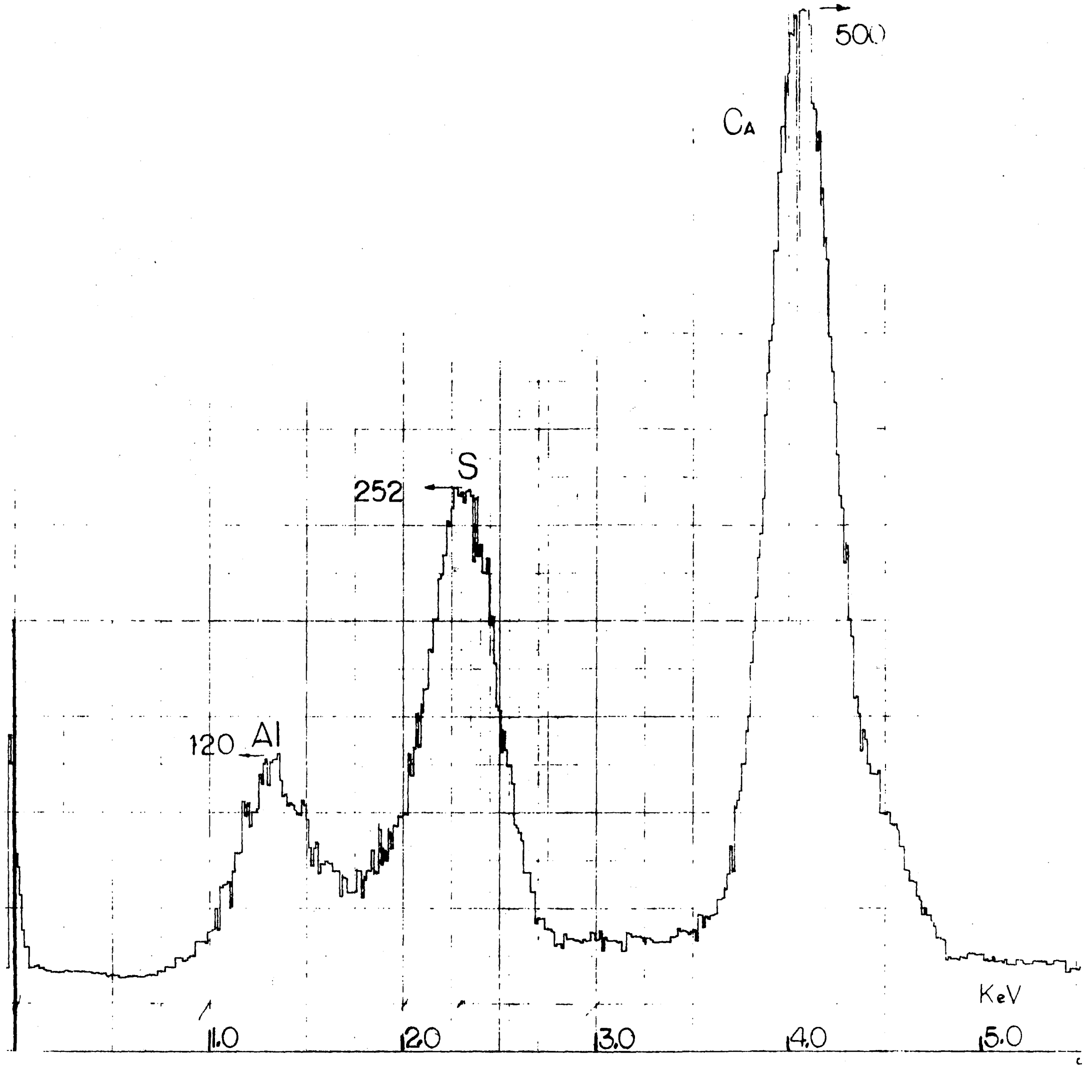


Figure 24. Electron Energy Emission Data - Union City Clay - 5% Lime - 90 Day Cure - Group O→W (60 Second Scanning Time and A Maximum Count of 500)

which probably accounts for the fact that it was found only in the $O \rightarrow W$ and $D \rightarrow W$ specimens to any significant extent. Ettringite forms quickly and is a very stable mineral despite its high degree of hydration. These characteristics would act to inhibit the uniform diffusion of calcium throughout the system, and the crystalline growth would produce cracks in the surrounding soil structure. Since Ettringite requires a relatively small amount of Al in comparison with the amount of calcium, it captures a great deal of the available calcium without the necessity for an extensive lime-soil reaction. For the above reasons, the formation of Ettringite in a lime-treated soil must be regarded as an unfavorable development, particularly when it occurs in the near proximity of compacted surfaces. It probably accounts for the difficulties encountered in stabilizing soils that are rich in sulphate. Ettringite might possibly contribute to stabilization if moisture conditions are such as to permit it to develop throughout the soil mass, but the high calcium requirement would have to be considered in designing the mix.

The X-ray diffraction patterns for two raw samples of the Union City clay--one wet and the other fairly dry--are shown in Figures 8 and 25. The additional peak that is present in the diffractogram of the dry soil disc is gypsum ($CaSO_4 \cdot 2H_2O$) which crystallized during the few hours in which the disc was allowed to dry.

The diffractograms of treated soil specimens did not reveal any substantial differences from one to another. It is apparent from the SEM photomicrograph that the Ettringite forms in a random fashion and with varying degrees of concentration, which may weaken its diffraction pattern. A typical diffractogram, shown in Figure 26, is for a sample treated with 5% lime and cured for 90 days. One of the peaks occurring

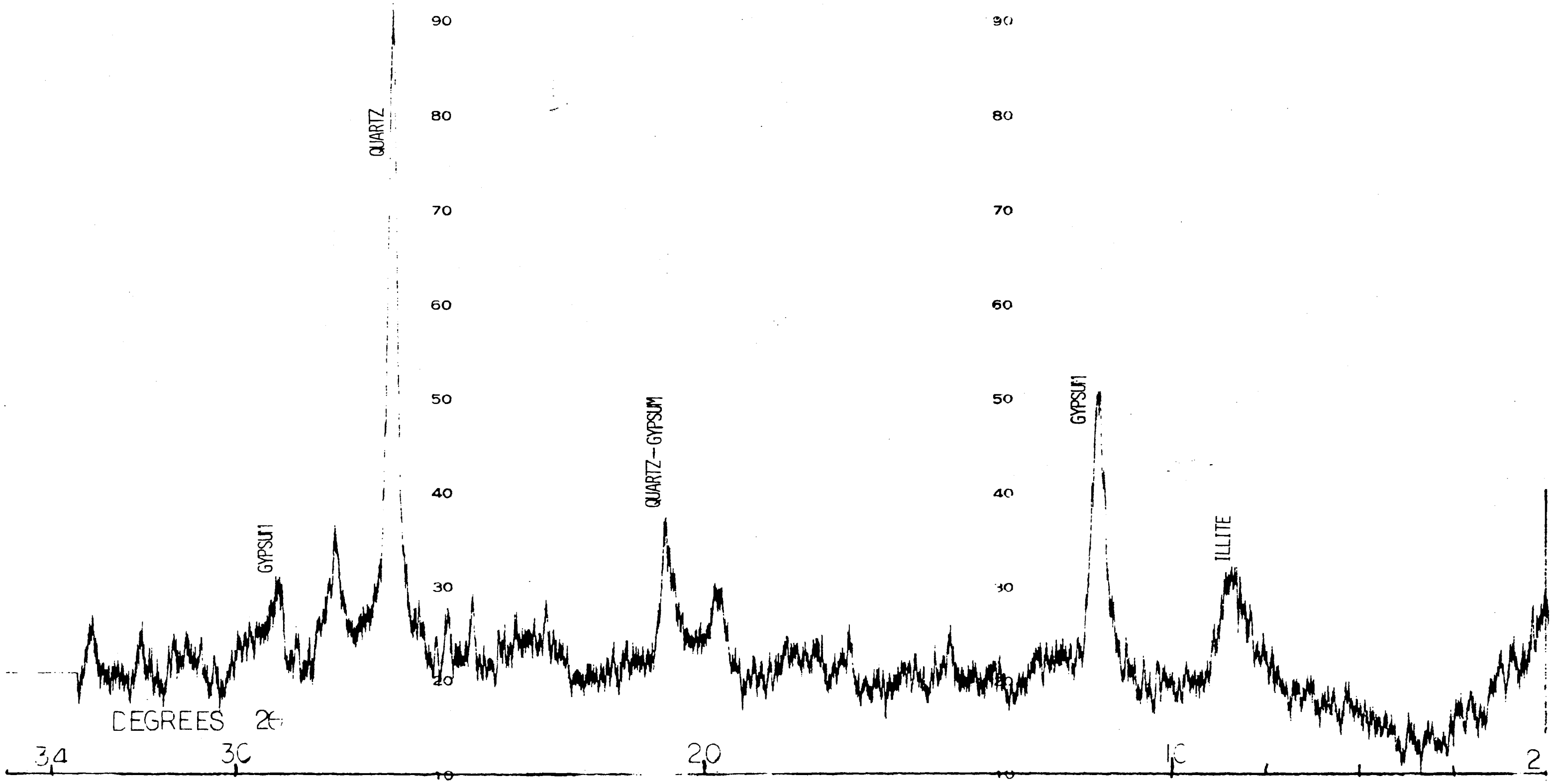


Figure 25. X-Ray Diffraction for Union City Clay - Untreated - Heated to 60^{0}C

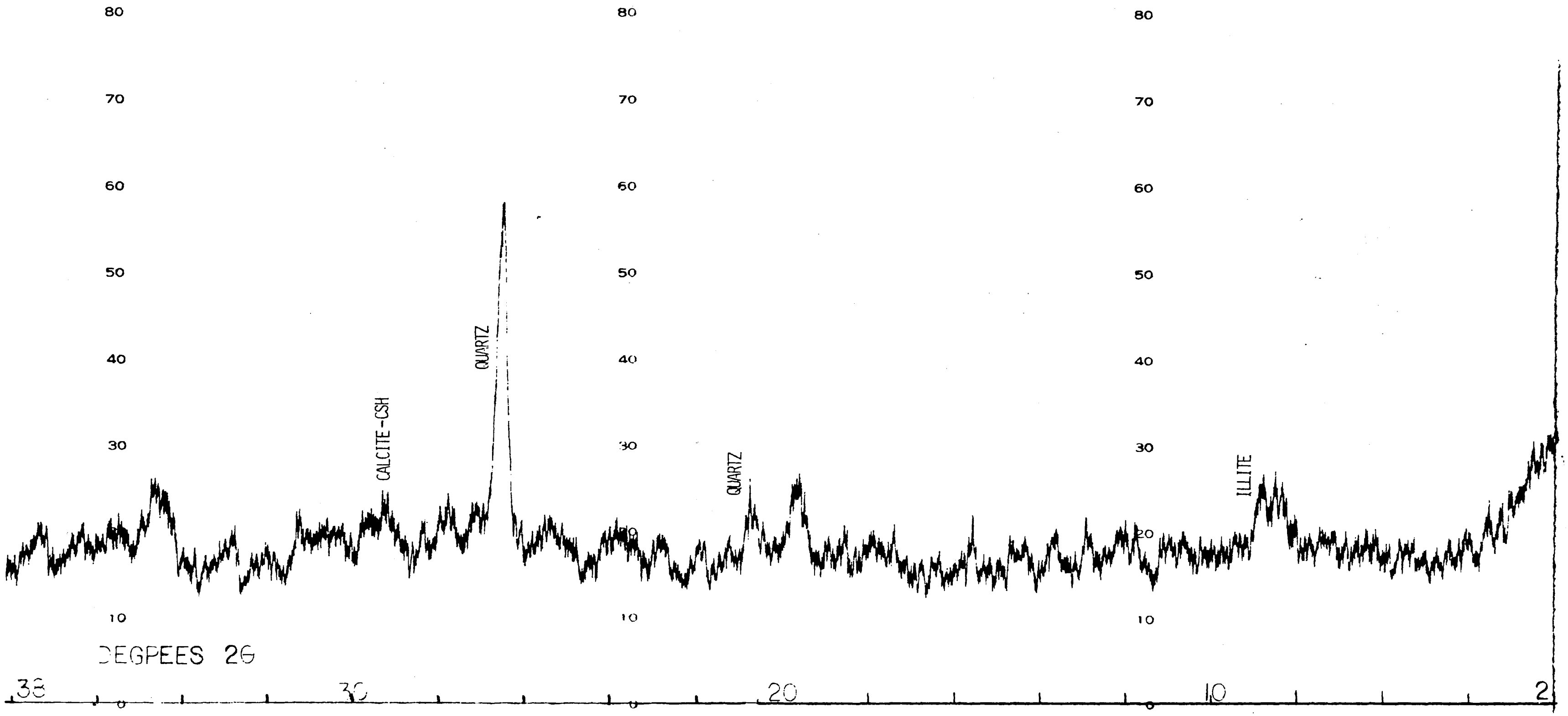


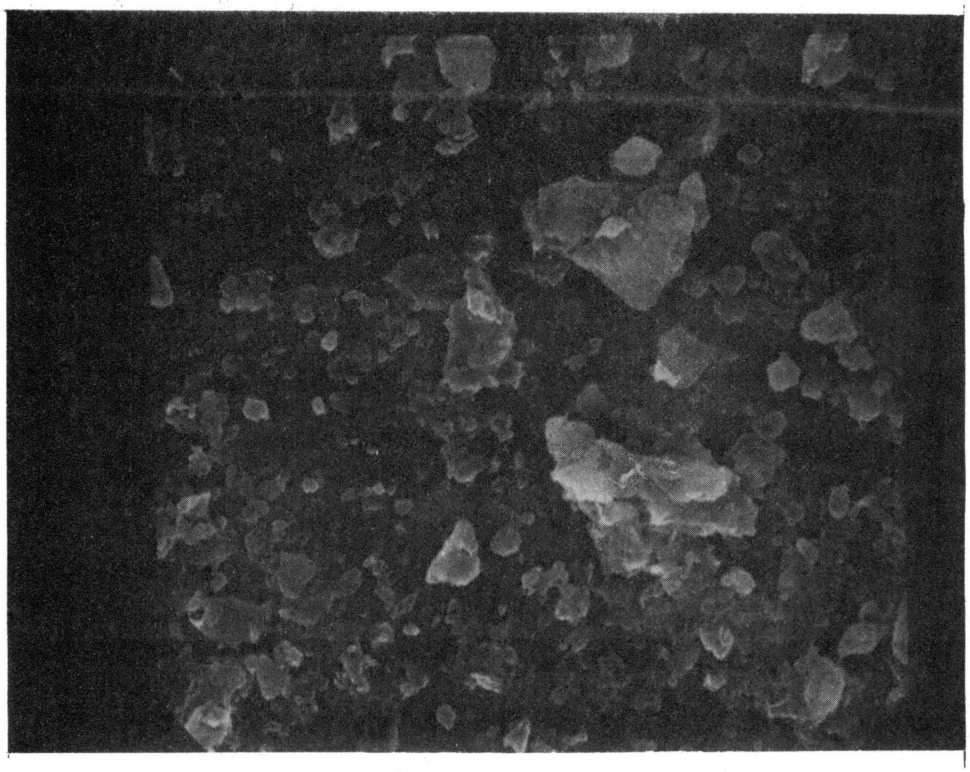
Figure 26. X-Ray Diffraction Pattern for Union City Clay - 5% Lime - 90 Day Cure - O→W

between 8^{0} and 9^{q} belongs to illite and the other to Ettringite. The remainder of the Ettringite peaks are quite small. The small peak at 29.4^{0} could be a calcite peak or a CSH compound.

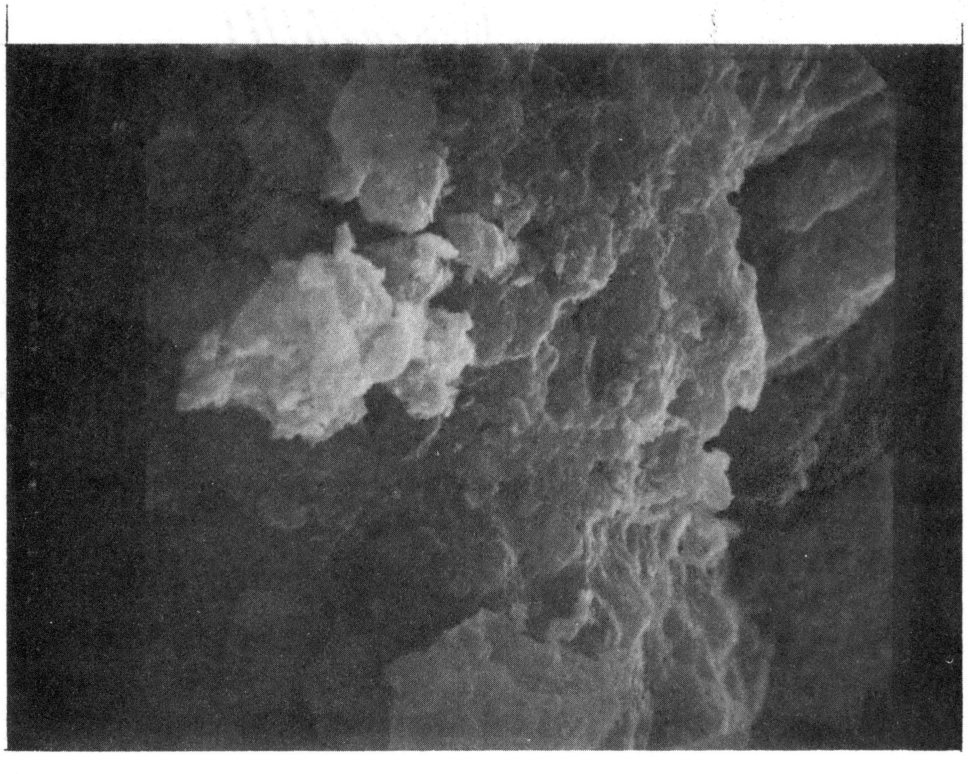
Summit Soil

Summit soil is a residual soil having a wide range of grain sizes. It contains mica and quartz as well as vermiculite as the dominant clay mineral. The soil also contains a noticeable amount of kaolinite. The distribution of particle size and mineral components is well illustrated in Photomicrograph 25, in which quartz, mica, and clay particles are all visible. Clearly, several sources of Si and Al are available in this soil. The intensity of the lime attack as well as the compactness of the soil is illustrated in Photomicrograph 26, where a kaolinite particle may be seen (upper left) still in its original hexagonal shape after 2 days of curing with a 7% lime content. It is also interesting to note in Photomicrographs 26 and 27 that the clay voids appear to be reduced in size by a lateral growth of cementing material and that the voids appear to be "dead end channels." These factors greatly affect the soil permeability.

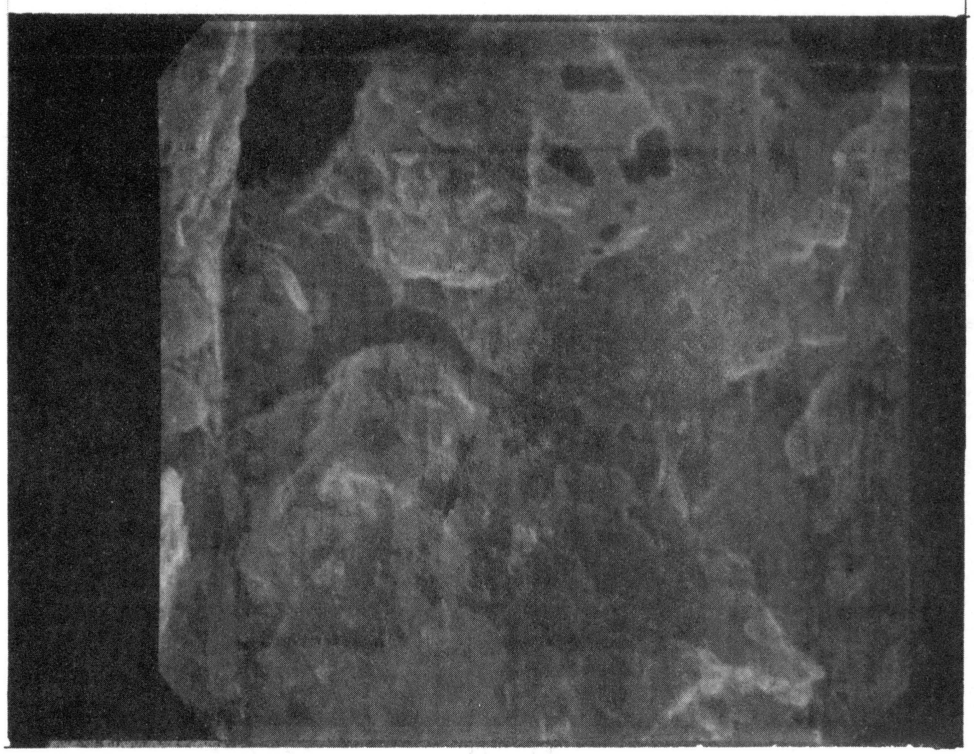
A sample from the O→W group cured for 2 days (Photomicrograph 28) displays a large number of spots and some sparsely scattered crystals. The spots are believed to be the initial stage in the development of a cementing material. Photomicrograph 29 illustrates an interesting and important phenomenon which occurred during the first 10 days of curing: a crystalline growth has spanned a void to provide a strong and secure structural link. In the central part of the photomicrograph it appears that void sizes have been reduced by mineral growth. Two of the three



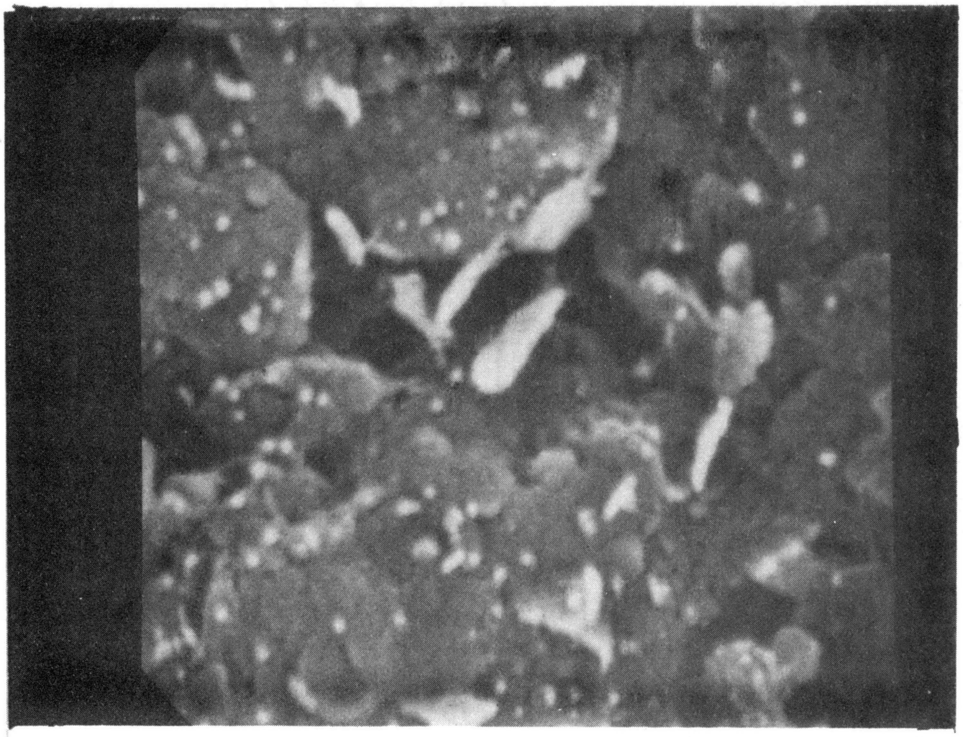
Photomicrograph 25. Summit Soil 3300X-Untreated



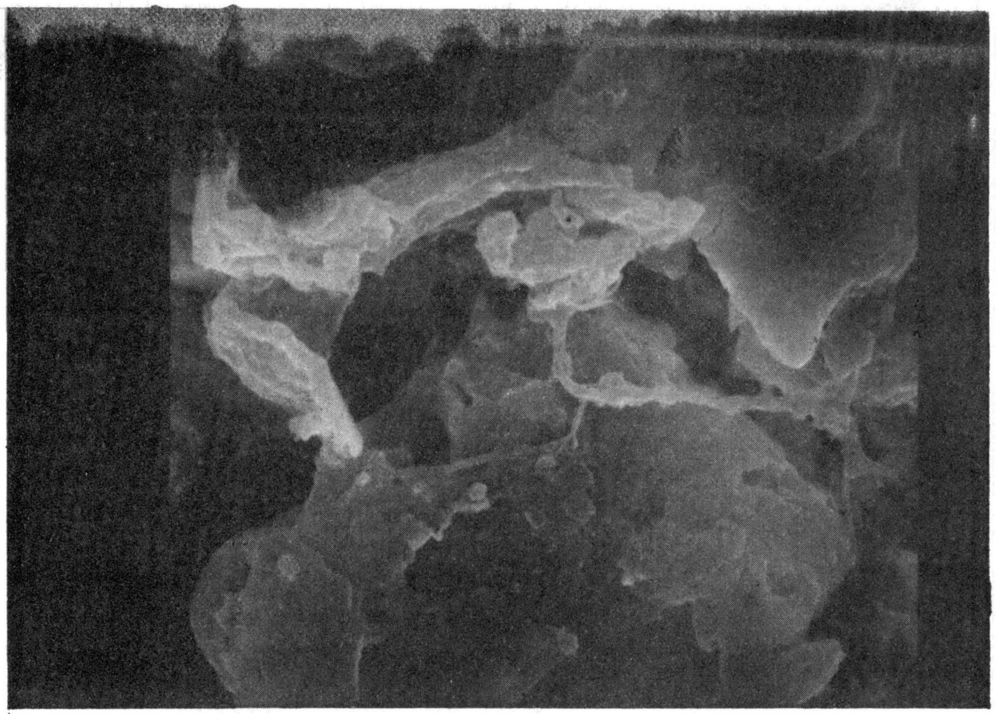
Photomicrograph 26. Summit Soil 3300X-7% Lime-2 Day Cure-Group W



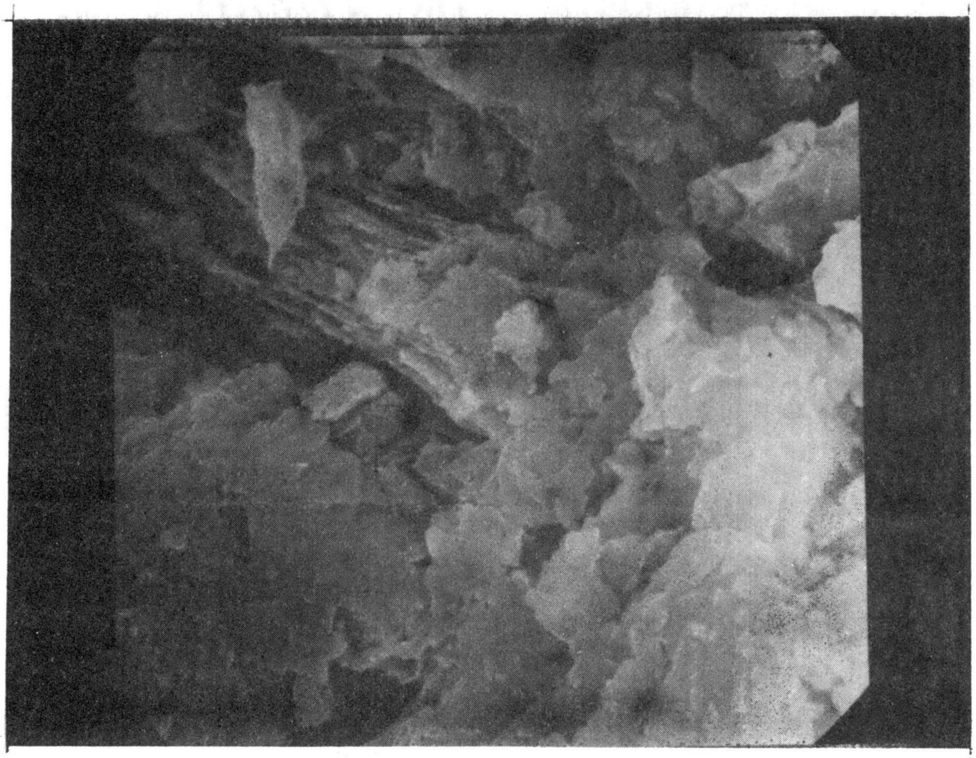
Photomicrograph 27. Summit Soil 11000X-7% Lime-2 Day Cure-Group W



Photomicrograph 28. Summit Soil 245000X-7% Lime-2 Day Cure-Group O→W

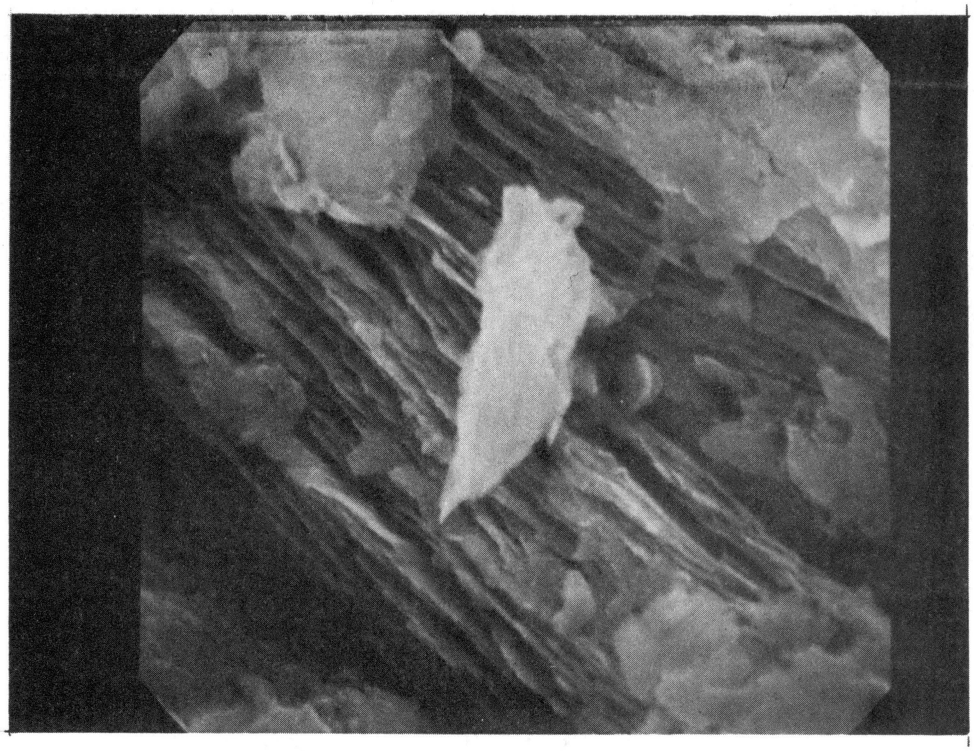


Photomicrograph 29. Summit Soil 2650X-7% Lime-10 Day Cure-Group O→W

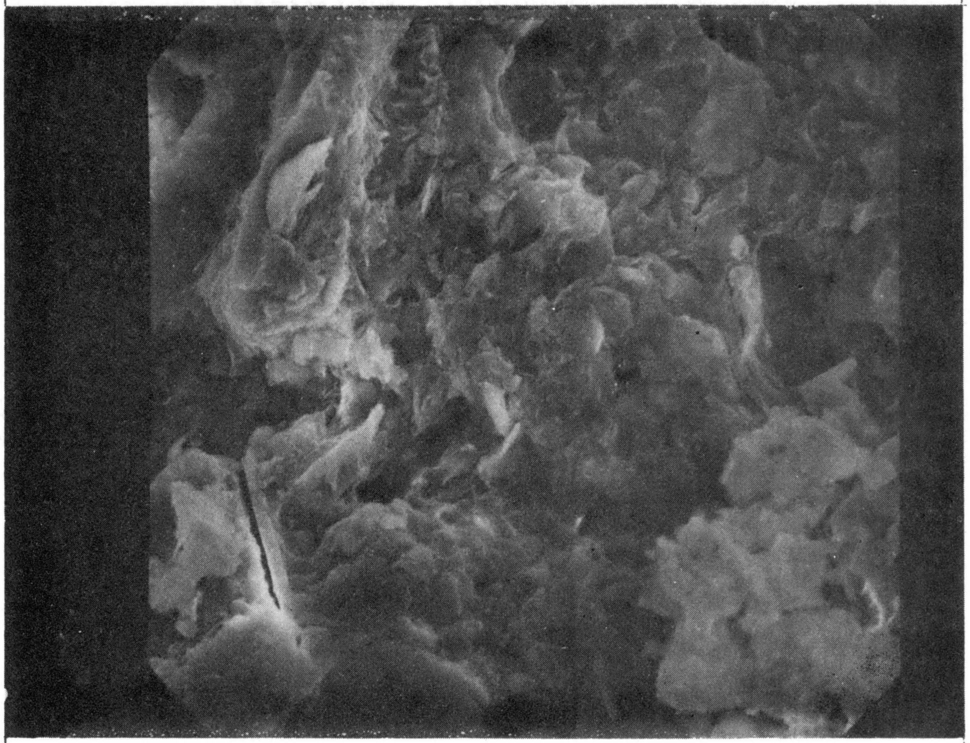


Photomicrograph 30. Summit Soil 4400X-7% Lime-90 Day Cure-Group D permeability test specimens of this soil failed to yield measurable flow in a 7-day period with a head of 50 psi. In Photomicrographs 30 and 31 a sample from the D group is shown after 90 days of curing. The lowest unconfined compressive strengths were associated with the D group. It is evident that a reaction has taken place, but some of the material appears never to have been attacked.

Photomicrograph 32 shows the broken surface of a D→sample after 90 days of curing. The dark gray areas in the bottom left-hand corner and on the right-hand side are the surfaces of voids that have been smoothed by the vigorous attack of the high pH pore fluid. This phenomenon does not appear in specimens of the D group (Photomicrograph 31) in which no additional water was added to the compacted clay. Another very interesting feature is that there was no indication of silica when the exposed surface was analyzed with X-ray energy emission. Ca and Al are the cominant ions. Photomicrograph 33 shows another part of the sample pictured in Photomicrograph 32. The cementing materials, which were probably bent and fractured during sample preparation, are quite conspicuous. Exposed surface areas of the same sample may be seen in Photomicrographs 34, 35, and 36. The full development of two types of bonding is evident in these photographs. There is a fine, lacy crystalline network spanning some of the voids, and groups of thin, strongly crystalline plates bridging over others. Based on their appearance it seems likely that these bonding materials are some form of either CSH or CAH. A few needle-like crystals that appear only in Photomicrograph 36 are probably calcite in the form of stalactites or stalagmites. The cementing material, in general, is completely different from that found in the Union City clay. The plate-like crystals are well oriented and spread out prolifically. The lacy network structure is similar to that

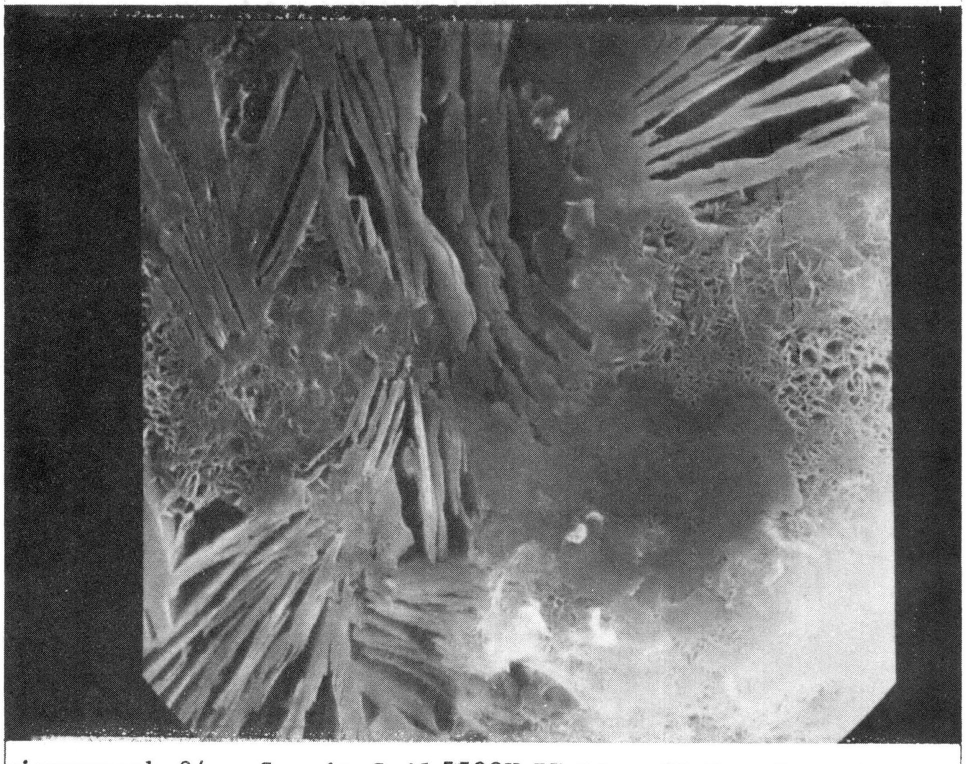


Photomicrograph 31. Summit Soil 7700X-7% Lime-90 Day Cure-Group D



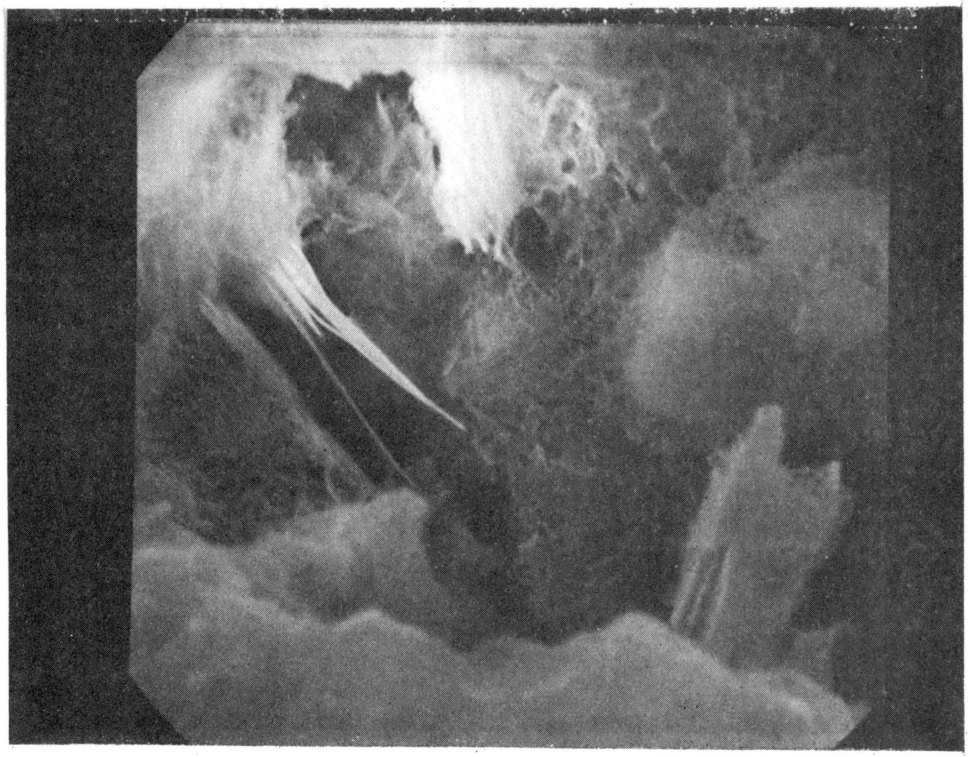
Photomicrograph 32. Summit Soil 1100X-7% Lime-90 Day Cure-Group D→W

Photomicrograph 33. Summit Soil 3300X-7% Lime-90 Day Cure-Group D→W



Photomicrograph 34. Summit Soil 5500X-7% Lime-90 Day Cure-Group D→W

Photomicrograph 35. Summit Soil 8800X-7% Lime-90 Day Cure-Group D→W



Photomicrograph 36. Summit Soil 4400X-7% Lime-90 Day Cure-Group D→W

found in the Camargo and Burleson soils but is more highly developed. The original small void size undoubtedly facilitated the bonding processes in the Summit soil, whereas the large voids in the Camargo and Burleson soils would be more difficult to bridge.

The X-ray studies of this soil supported various hypotheses advanced earlier in the discussions of compressive strength and pH variation. A diffractogram for untreated Summit soil is presented in Figure 9 which shows three very distinct peaks at 14.49, 9.83, and 7.10 Angstroms. Special treatments were used in an effort to determine the natural mineralogy. One specimen was glycolated for several hours before the X-ray examination, and another was heated in a furnace at 680°C for 8 hours prior to examination. The charts for these two treatments are designated (a) and (b), respectively, in Figure 9.

Glycolation produced a broad peak from 21.6 to 15.24 Angstroms and the heating collapsed the basal spacing of the clay structure to 10.1 Angstroms. This may be interpreted as an indication that the Summit soil is a mixed-layer clay. The original 9.83 Angstrom spacing indicates the existence of mica-like clay while the 14.49 Angstrom spacing may be indicative of vermiculite or chlorite or even Ca-montmorillonite. Since the clay mineral structure collapsed upon heating to a 10.0 Angstrom spacing, chlorite must be eliminated as a possibility. The broad peak after glycolation indicates that the clay most probably consists of vermiculite interlayered with montmorillonite. Another alternative is that this soil may be a mixture of vermiculite and kaolinite. Work performed on a soil from a nearby locality by the Agricultural Experiment Station of the Oklahoma State University (1) supports the first hypothesis but does not preclude the existence of kaolinite.

Diffractograms for six specimens of lime-treated Summit soil are shown in Figures 27 through 32. From Figures 27 and 28 it is very clear that the addition of water changes the mineralogical composition of the soil. The lime content of these specimens corresponded to the LMO. The diffractograms for two specimens, one from group O and one from group O→W with a lime content 30% above the LMO and cured for 30 days, are given in Figures 29 and 30; those for a D and D→W specimen having the same composition and cured for 90 days are shown in Figures 31 and 32. The X-ray data in these six figures indicate conclusively that when lime is present the addition of water causes major mineralogical changes in this soil. A determined effort was made to correlate these data with the standard X-ray spectra for a variety of possible minerals or compounds. Some of these compounds match in all major peaks with the observed diffraction data of the treated samples. For others only a few peaks could be matched, and for still others no correlation at all could be found. The addition of water had the effect of intensifying existing peaks and of generating new ones, both of which are indicative of the production of additional bonding materials. The quartz particles were corroded by lime attack, but a precipitation of silica was noted in the microscopic examination of treated samples. New forms of silicate minerals occur regardless of the water content, but additional water will help to corrode the quartz and may delay the silica precipitation.

The CAH compounds formed in differing amounts at one time or another in the Summit soil (see Appendix B) are: calcite; tetracalcium

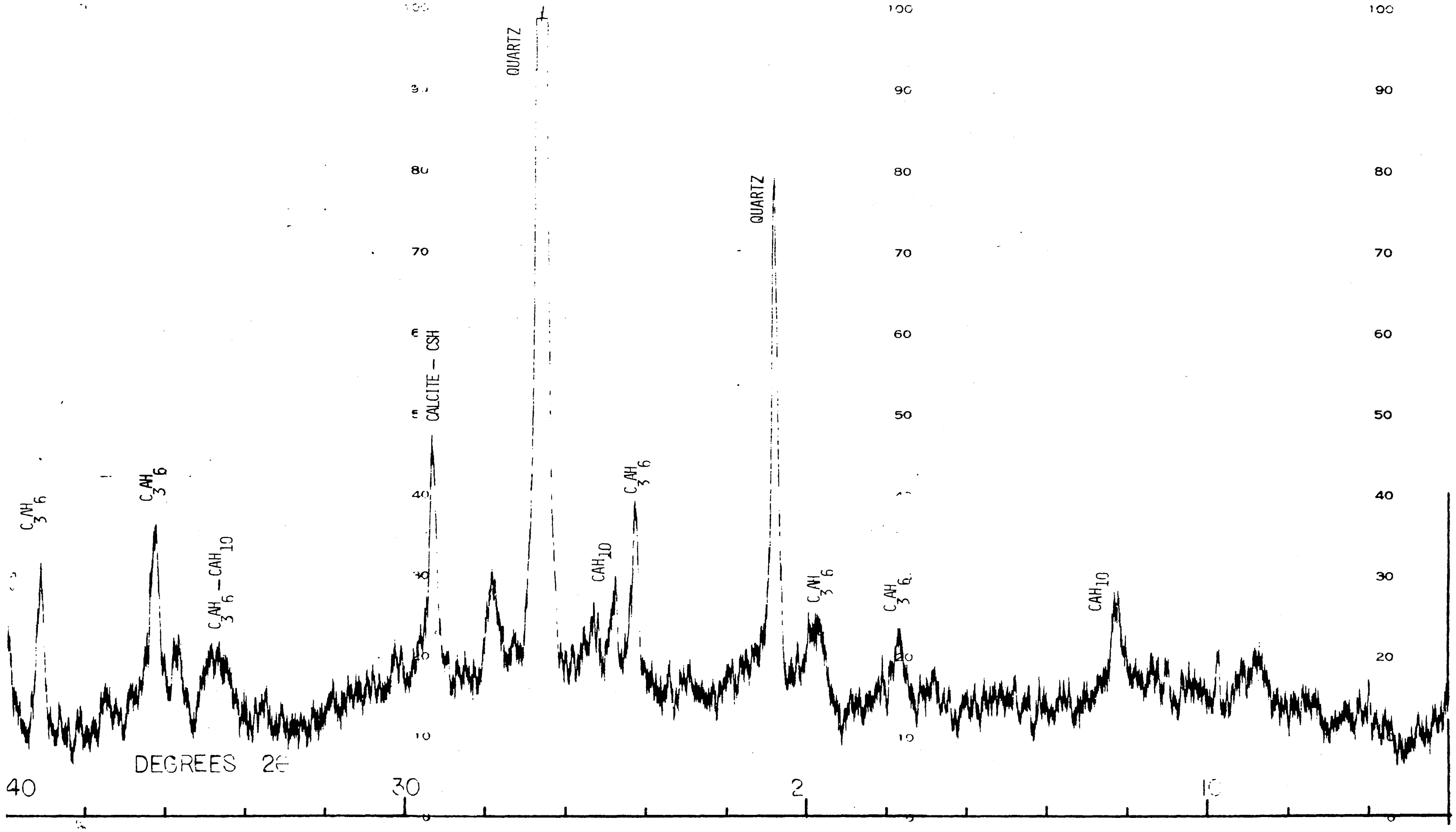


Figure 27. X-Ray Diffraction Pattern for Summit Soil - 5.25% Lime - 30 Day Cure - Group 0

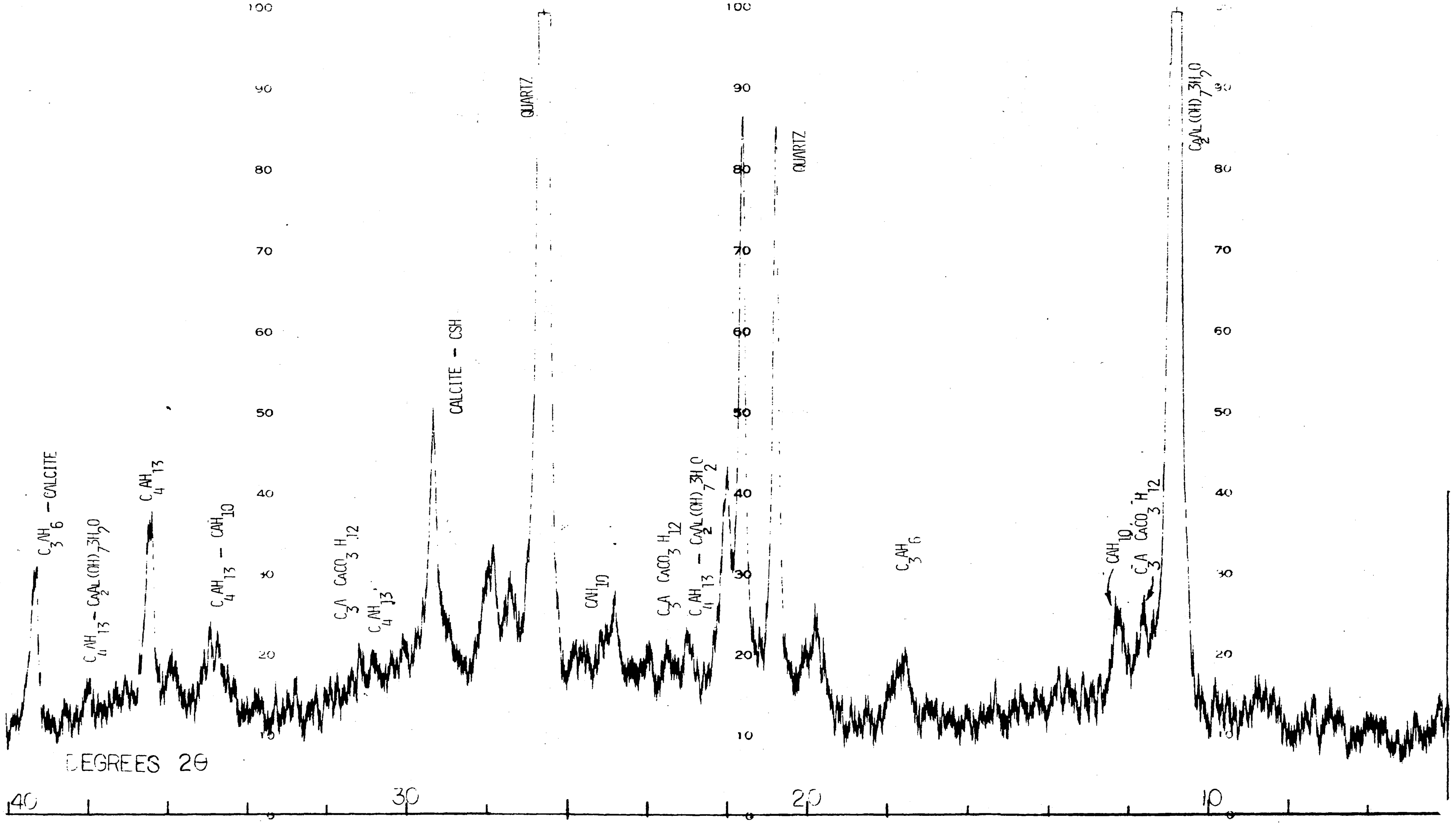


Figure 28. X-Ray Diffraction Pattern for Summit Soil - 5.25% Lime - 30 Day Cure - Group O→W

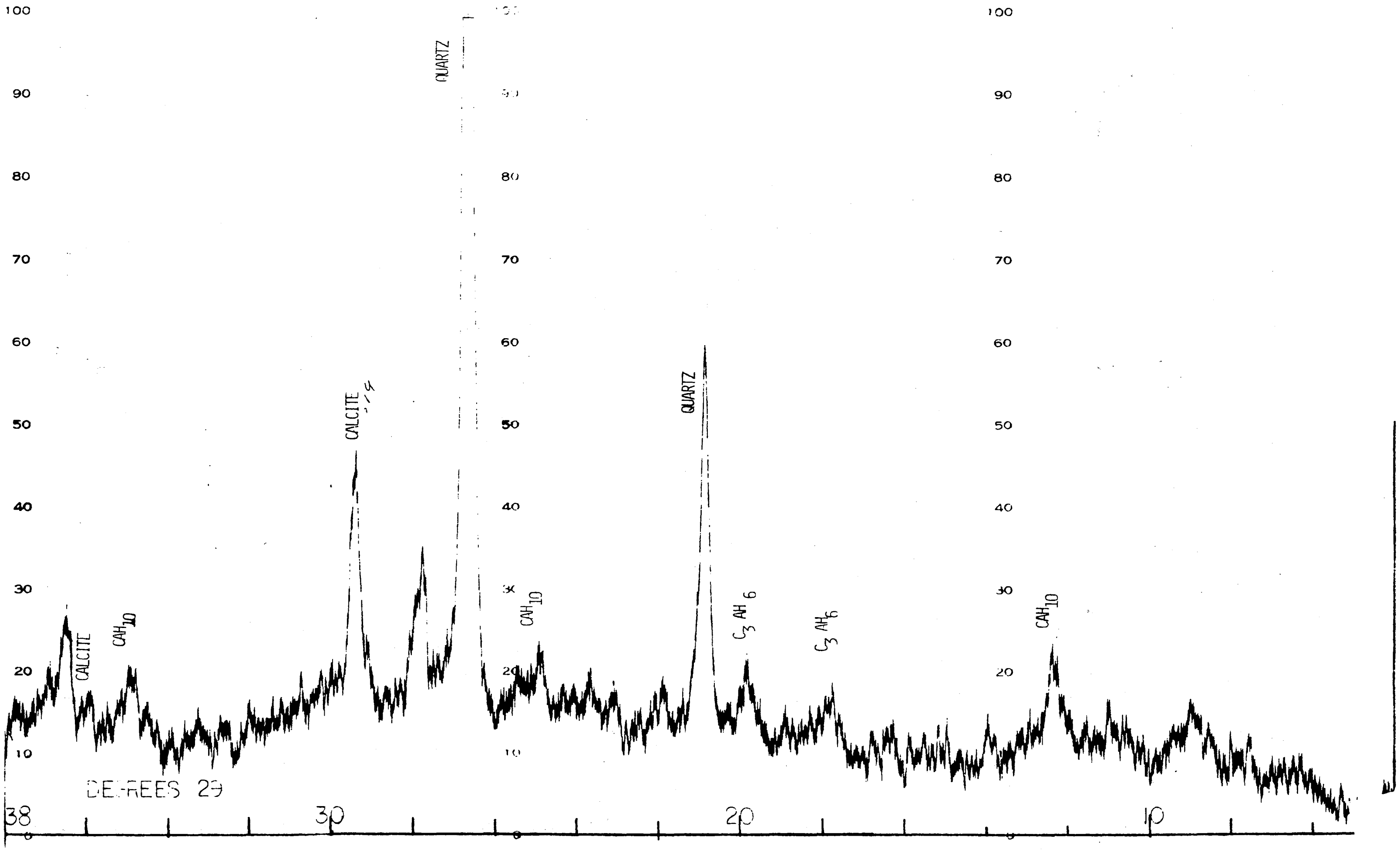


Figure 29. X-Ray Diffraction Pattern for Summit Soil - 7% Lime - 30 Day Cure - Group 0

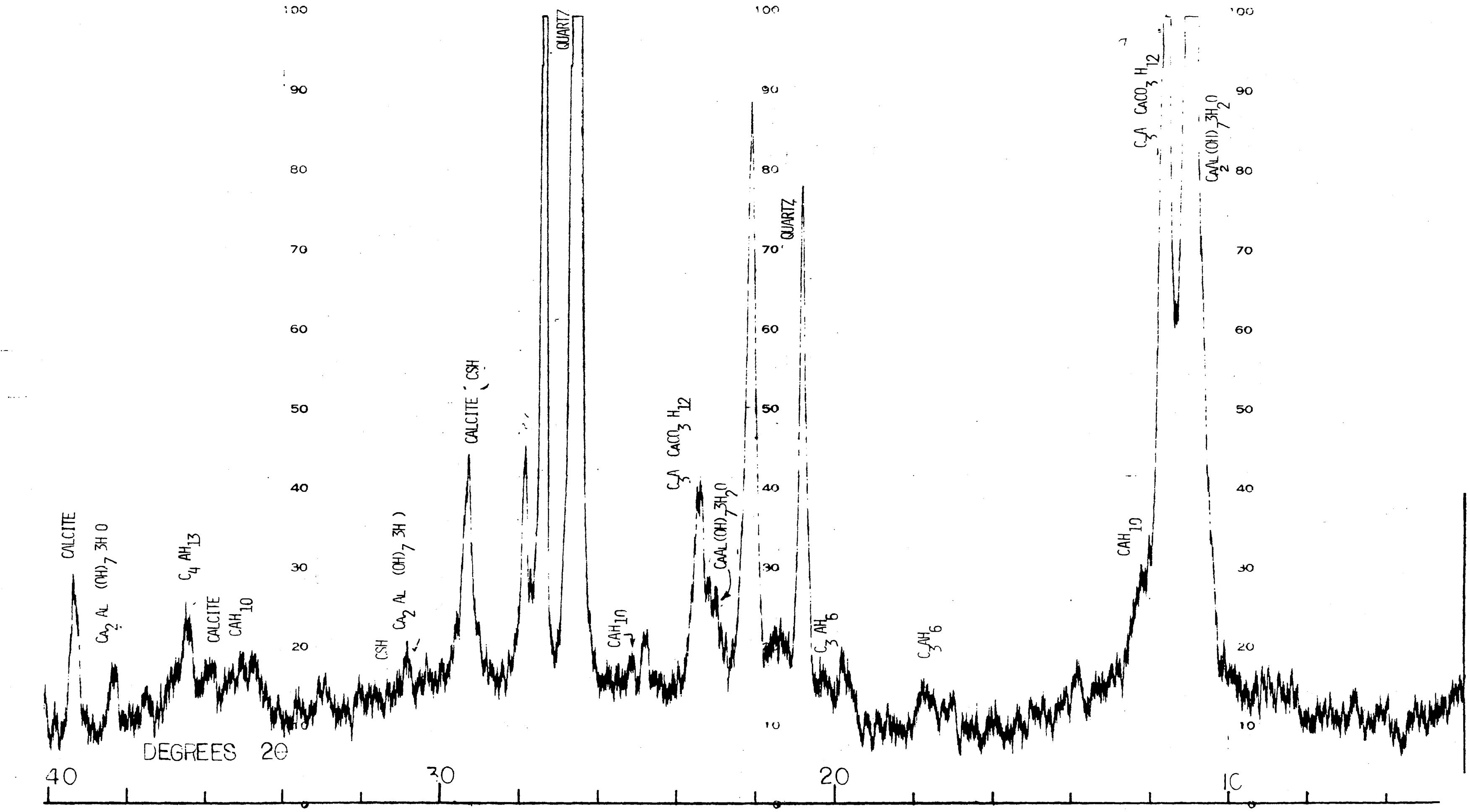


Figure 30. X-Ray Diffraction Pattern for Summit Soil - 7% Lime - 30 Day Cure - 0→W

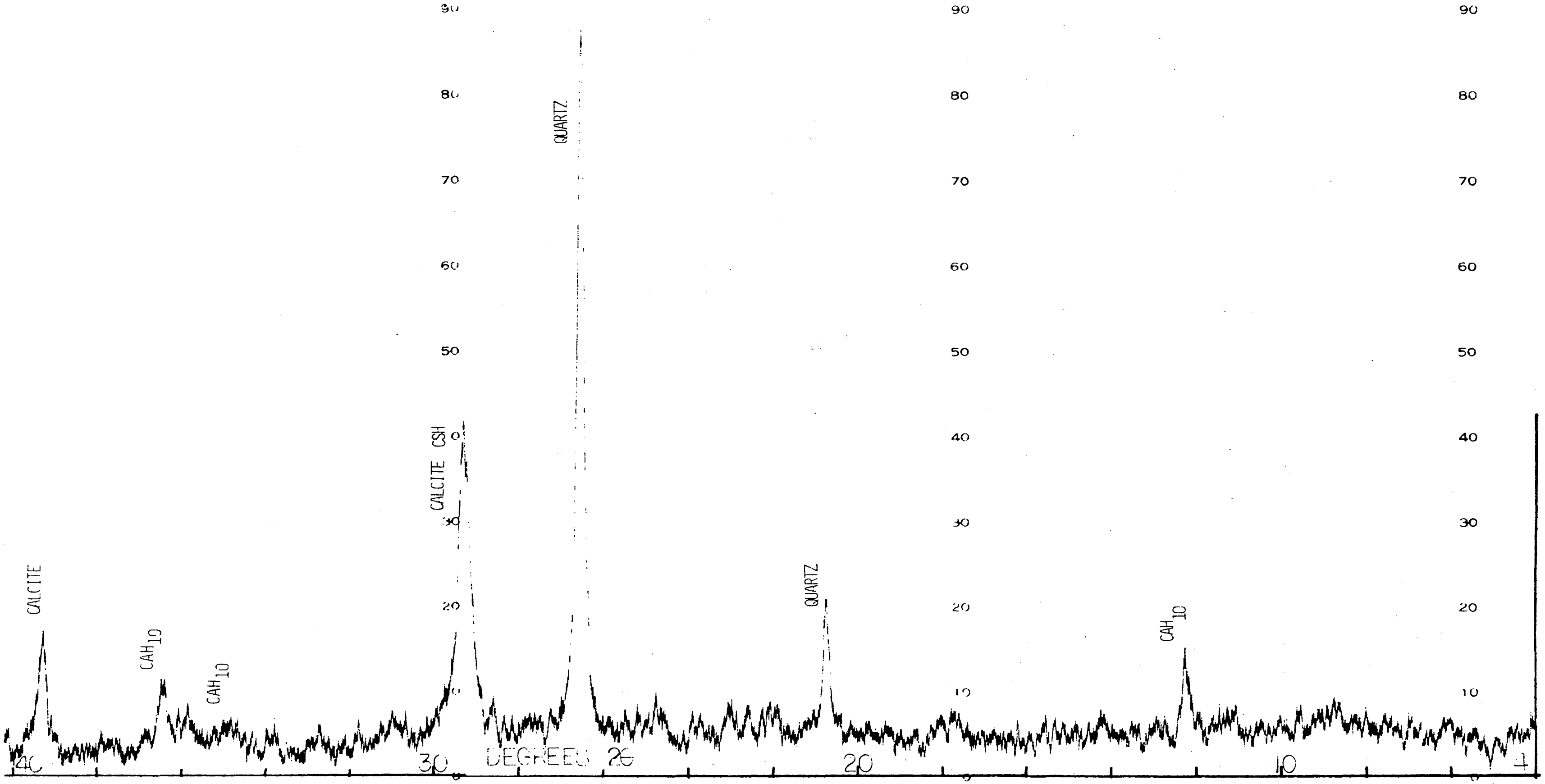


Figure 31. X-Ray Diffraction Pattern for Summit Soil - 7% Lime - 90 Day Cure - Group D

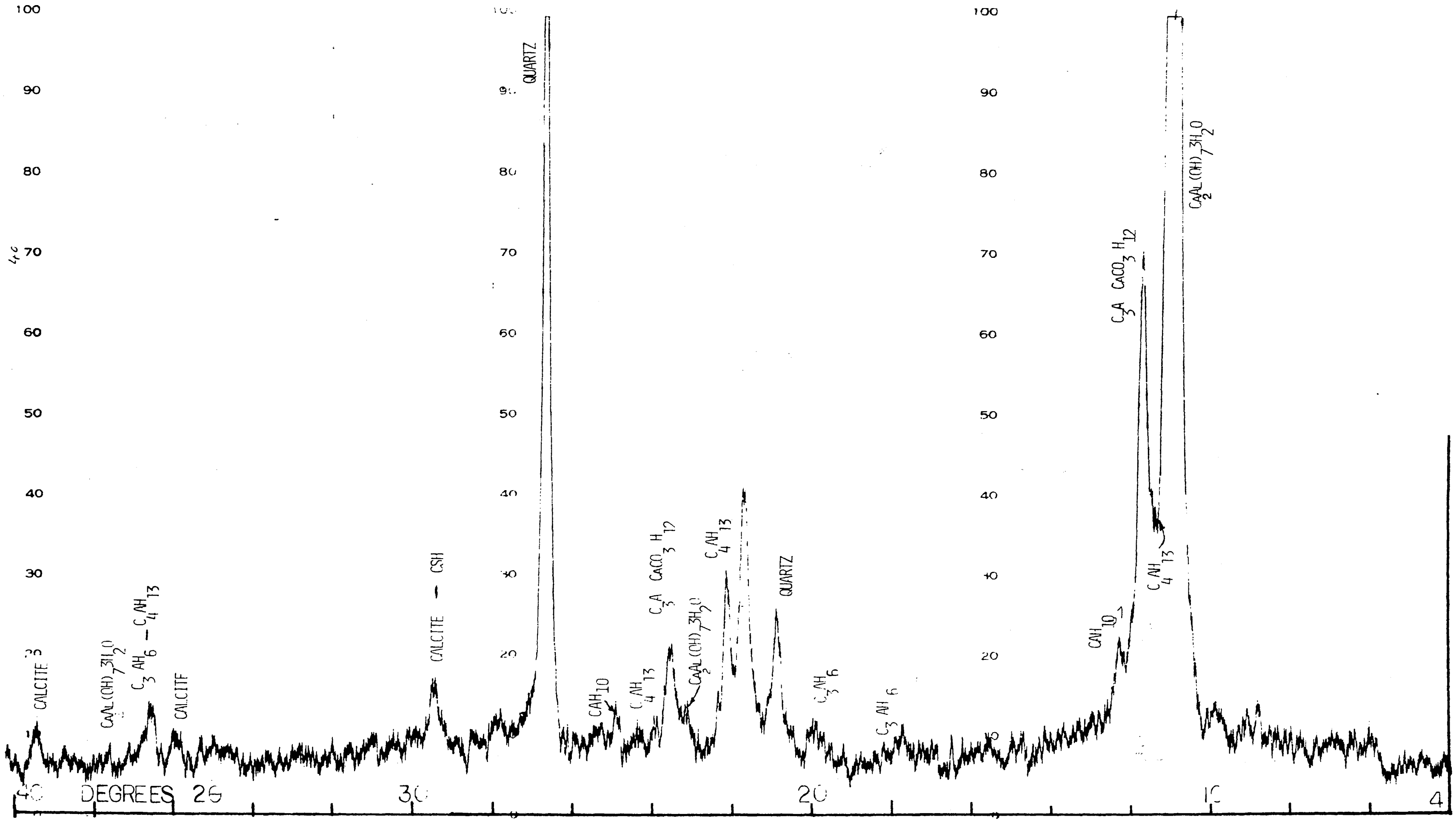


Figure 32. X-Ray Diffraction Pattern for Summit Soil - 7% Lime - 90 Day Cure - Group D→W

aluminate 13-hydrate ($Ca_2Al(OH_7)\cdot 3H_2O$, or C_4AH_{13}); α-tetracalcium aluminate 13-hydrate ($Ca_2Al(OH_7)\cdot 3H_2O$); tricalcium aluminate hexahydrate ($Ca_3Al_2(OH_{12})$ or C_3AH_6); monocalcium aluminate 10-hydrate ($CaAl_2(OH_8)\cdot 6H_2O$, or CAH_{10}); and tetracalcium aluminate carbonate 12-hydrate ($Ca_4Al_2(OH_{12})(CO_3)\cdot 6H_2O$, or $C_3A\cdot CaCO_3\cdot 12H_2O$). Note that the tetracalcium aluminate 13-hydrate is not the same as C_4AH_{13}. The one formed in this soil demands some essential CO_3^{2-} 1. The CSH(I) and CSH(II) were formed in lesser amounts than the CAH compounds, and the indications of their characteristic peaks were not distinct.

Samples cured for 30 days with a lime content of 7%, group O→W, exhibit very distinct peaks for $CaAl(OH_7)3H_2O$ and C_3A $CaCO_3\cdot 12H_2O$. These two compounds are very similar in structural form and unit cell dimensions, which implies that they will have no problem in stacking or organizing themselves in a limited space. The formation of these compounds may be illustrated by assuming that C_3AH_6 and CSH_{10} may be formed when the lime-water ratio is fairly high, as in O samples. The C_3AH_6 compound uses up more calcium and is very stable. When water is added, the clay sample will be surrounded by a more homogeneous environment and the trapped air will start to dissolve, introducing CO_2 to the chemical reaction. The CO_2 may also be continuously supplied by the existence of organic content. With time, the noncarbonated calcium compounds will be carbonated and will attain a higher stable condition. It is thus virtually assured that calcite will be formed in all lime-soil mixtures.

It should perhaps be presumed that calcite is a good cementing material. It is a strong crystal which is difficult to distinguish from any form of CSH, since the chemical and physical properties of the latter are still in doubt and one of the strong diffraction peaks of the two minerals practically coincides. On the other hand, silica is dissolved during the lime-soil reaction and is free to enter into any phase of CSH. The silica itself may also precipitate and recrystallize to form a very good cementing material.

In the Summit soil nothing new occurred during the final 60 days of curing, indicating either that stable compounds are formed early or that those which progress to a more stable form are continuously replaced by newly-generated compounds of the less stable form. It seems likely that the production of cementing material continues as long as the process can be supported in the semi-liquid environment surrounding the clay structure.

Interpretation of the X-ray diffraction data was aided by the experience gained earlier by the author in similar studies not herein reported. Diffractograms of the treated Summit soils exhibit new peaks at 4.114, 4.004, 3.250, and 3.210 Angstroms that have not been specifically discussed. Those peaks in the range of 4.10 Angstroms may indicate one of various forms of Si; however, the peaks at 3.21 and 3.25 Angstroms may match a form of calcium aluminum silicate: sodium (3.20_x, 3.21_9, 3.18_9 in the standard X-ray diffraction data). Another alternative is that all of these peaks are the diffraction pattern of $NaCa_{2.5}\ Al_6Si_{13}O_{32}.13.5H_2O$, For all practical purposes, the significance of these peaks may be of secondary importance.

Summary

The study of mineralogy of these soils has led to several conclusions, some based on the SEM disclosures and others on the diffraction data. The Camargo and Burleson soils did not develop any sizable crystals during the stabilization process. Evidence of the development of cementing material was contained in the observed initiation and intensification of a particle-coating material, which was found to be primarily a form of silica. This material was much in evidence in O→W and D→W groups, which indicates that the addition of water increases the production of cementing material. This was also quite evident from the results of strength tests. The study of the Union City clay introduced the important concept that the chemical reaction is not completely governed by the clay mineral, but may be retarded or accelerated by the presence of minor inorganic or organic impurities, depending on the nature of those impurities.

The Summit soil studies provided strong evidence, in both the SEM and the X-ray diffraction results, of the effect of adding water to compacted soils. Clearly, the formation of cementing materials depends on factors not previously appreciated, such as the void size but not the void ratio, and the lime-water ratio rather than just the lime content. Union City soil with 5% lime attained as much strength as the Summit soil with 7% lime after 90 days of curing. It must also be concluded that it is impossible to prevent the development of calcite in either laboratory or field-compacted soils; however, its production can apparently be reduced, especially during the early stages of curing. It is also apparent that no single chemical compound or bonding mineral is associated with a lime-treated clay, but rather that at least a modest spectrum of slightly differing compounds is formed. The SEM proved to be a powerful tool for examining soils and soil constituents, although considerable time is required to obtain the needed information from the instrument. Only five samples could be examined in a long working day.

CHAPTER VI

CONCLUSIONS, EVALUATION, AND RECOMMENDATIONS

In this chapter the significant accomplishments of the investigation are briefly reviewed, and consideration is given as to how the results may be practically applied to procedures used in construction activities. Principles and conclusions that appear to be justified by the data and general findings of this study are first enumerated, followed by an evaluation of the applicability of certain ones to the construction of lime-stabilized bases. Recommendations for future study will be directed toward clarifying areas of uncertainty in this study, extending the study to areas that were not investigated, and verifying major conclusions of the study under field conditions.

Conclusions

The conclusions listed below are either supported directly by analysis of the data obtained during the investigation or may be logically inferred from the data. The principal thrust of the study has been to determine the nature of mineralogical changes associated with lime stabilization and the manner in which these changes influence the behavior of the soil from an engineering viewpoint. Although four soils that differ significantly in character and mineralogy were used in the study, the conclusions reached are not necessarily valid for other soils; however, it is believed that they provide useful guidance with respect to a broad variety of soils.

1. Soils rich in montmorillonite and having a high activity number, such as the Camargo and Burleson soils used in this study, present the most troublesome problems in treatment, compaction, and stabilization processes.

2. The procedure for determining the pH of soil suspensions to evaluate the Lime Modification Optimum appears to be valid and to be in satisfactory agreement with methods based on soil plasticity.

3. The effects of lime in improving the workability and reducing the plasticity of clay soils are attained for lime contents well below the Lime Modification Optimum. Moreover, these effects are available almost immediately.

4. Soil compacted at a moisture content above the optimum attains, after a short curing period, strength superior to that associated with lower compaction moisture contents, probably because of the more uniform diffusion of lime and more nearly homogeneous curing environment.

5. If soils are compacted at or below optimum moisture content, their strength can usually be improved by adding water after compaction.

6. Soil strength is directly proportional to the lime content and, in a given soil, to the reduction in pH of the soil during curing.

7. Soil pH is an important factor controlling the cementation process, but its influence is enhanced (for a given lime content) when the water-lime ratio is fairly high.

8. The various forms of calcium silicate hydrate that are generated during the soil-lime reaction may be observed and studied more advantageously by means of a scanning electron microscope than by X-ray

diffraction, but analysis is facilitated by using the two methods jointly.

9. In the montmorillonitic Camargo and Burleson soils calcium silicate hydrates are the predominant cementing materials. The production of these materials is stimulated by the addition of water with a resulting improvement in strength of the soil.

10. Cementing materials formed in the Summit soil, a mixed-layer clay, are principally calcium aluminate hydrates, C_3AH_6 and CAH_{10}. When more water is available during curing, $Ca_2Al(OH)_7 \cdot 3H_2O$ and $C_3A \cdot CaCO_3 \cdot 12H_2O$ are also formed and are the predominant new minerals.

11. Since many of the cementing minerals formed during the soil-lime reaction contain three or more molecules of water of crystallization, water availability during the reaction is important.

12. Some calcite ($CaCO_3$) is always formed in lime-treated soils but the amount is affected by the lime content and the curing procedures. Its presence, while perhaps not harmful, is less beneficial than that of CSH or CAH which are strongly bonded to the clay minerals.

13. Soils such as the Union City clay which contain sulphur (in the form of gypsum) produce an abundance of $Ca_6Al_2(OH)_{12}(SO_4)_3 \cdot 26H_2O$ when lime is present. This appears to have an adverse effect on the development of soil strength and, especially, may negate the beneficial effects that would otherwise result from adding water to the soil after compaction. It is quite possible that other types of soluble minerals that may be present in a soil will also adversely affect the properties.

14. The existence of small voids in a lime-treated soil facilitates the formation of soil bonds through the growth of new crystals.

The void ratio itself appears to be of lesser importance.

15. Organic contents of less than 1.5% did not, in this study, prohibit the soil-lime reaction in mixes compacted wet of optimum. It appears that the pedological horizon from which a soil originates is a less significant factor in stabilization than is generally supposed.

16. The mineralogy of compacted specimens of lime-treated soils can be reliably investigated by X-ray diffraction and scanning electron microscopy. From both a theoretical and practical viewpoint, the procedure is preferable to that using soil suspensions.

17. The permeability of a lime-treated soil is greater than that of the similarly compacted untreated soil, but the permeability of treated soils may be reduced by as much as 300-fold by spraying water on the soil before it is cured.

18. High moisture content reduces the resistance of treated soils to weakening and deterioration under cyclic freezing and thawing; however, these tests differ from natural conditions in that no healing periods between cycles are available for recovery of strength.

Evaluation

The study focused on means for improving the effectiveness of lime as a stabilizer without sacrificing economy. In designing field stabilization procedures certain principles, either resulting from this study or verified by it, should be used for guidance.

1. Lime diffuses more rapidly and efficiently in a wet soil than it does in a dry one, regardless of the soil mineralogy. This suggests that slurried lime rather than dry lime should be mixed with the soil, and that soil should be compacted at a moisture content as far on the

wet side of optimum as is practicable. The use of slurry instead of powder is an important factor in reducing atmospheric carbonation of the lime.

2. All of the observed noncarbonate cementing compounds formed by the soil-lime reaction are hydrates. Adequate water is needed to promote the reaction and also to improve the distribution of the reaction products within the soil mass. The reaction is also accelerated or better sustained when the pH of the pore fluid is 12.0 or higher. It has been shown that the likelihood of maintaining a high pH improves as the degree of saturation increases. These facts also suggest that the moisture content used for constructing lime-stabilized bases should be as high as is practicable during the curing process.

3. The workability of a clay soil is vastly improved at lime contents well below the modification optimum (LMO). Moreover, it was found during this study that this improvement occurs, at least in the laboratory, during the first hour of "mellowing." It is also known that atmospheric exposure of the soil during a mellowing period prior to final mixing and compacting leads to carbonation of some of the lime. The $CaCO_3$ that forms during mellowing immobilizes some of the calcium that would otherwise be available for the soil reaction; and, having been prematurely formed, is of no value in stabilizing the soil. These facts suggest certain procedures that could be applied beneficially in construction: (a) apply only one-half the design lime content initially to the soil; (b) utilize mellowing periods of less than eight hours; (c) apply the remaining half of the lime just before the final mixing and compacting operation.

4. It is known that the soil density obtained by a given compaction procedure is related to its moisture content at the time of compaction, and that the maximum density is attained when the soil is at its so-called optimum moisture content. Yet, it was shown in this study that in some soils moisture contents greater than optimum lead to soil properties (strength and permeability) superior to those of soils compacted at or below optimum moisture content. It was also shown that water added to soils after they are compacted produces benefits comparable to or greater than those obtained by compacting the soils initially at a water content above optimum. It thus appears that in many situations it would be desirable to compact soils at or slightly below optimum moisture content and spray additional water over each compacted lift. In this manner both the benefits of increased density and of a high moisture content can be obtained. In addition, the construction and equipment difficulties associated with mixing and compacting an excessively wet material can be avoided.

Recommendations for Future Research

The following topics for investigation are suggested by problems encountered during the course of the research or by ideas generated during analysis of the data:

1. A controlled field study in the locality of one or more of the soils used in this investigation is suggested. Such a study could determine the practicability of implementing in the field some of the laboratory procedures that appeared beneficial. The real value of these benefits must be measured in terms of pavement performance. The long-term variation of factors such as moisture content and soil pH should be

monitored along with variation of the soil strength.

2. The effects of gypsum and other naturally occurring soil minerals should be further investigated.

3. A further specific study of the relationship between soil strength and lime-water ratio rather than lime-soil ratio could lead to more economical practices in soil stabilization. The influence of soil pH could be concurrently evaluated.

4. Long-term permeability tests should be performed as a means of evaluating the influence of continued leaching of the soil. Soil durability under more nearly natural cycles of freezing and thawing should also be investigated.

5. The influence of organic content should be examined more thoroughly, with particular regard to the differing results that may be obtained by slurry and powder applications of the lime.

Recommended Field Test Procedure

The field test should be designed to test the validity and practicability of the laboratory conclusions for use under full-scale construction conditions. The field test results must be capable of analysis in terms of both cost and benefits, and should permit comparison with results attained by current construction procedures.

Specific influences that should be investigated during the field test are

1. reduced mellowing time,
2. use of slurried lime as opposed to powder or pellets,
3. use of high moisture content during compaction, and
4. addition of water following compaction.

These influences should be evaluated from cores taken at intervals during the field test, from subjective evaluations of the roadway quality over an extended period of time, and from accounting records of the cost of maintenance during the life of the project.

It is recommended that a one-mile section of new highway in which a lime-stabilized subgrade is to be used be divided into four one-quarter mile control sections receiving the following different treatments:

1. One section constructed according to present procedures,

2. One section to be constructed using slurried lime, with all lime being applied at once, prior to mellowing; and with soil being mellowed and compacted at optimum moisture content.

3. One section to be constructed using slurried lime, with half the lime applied prior to mellowing and half just before final mixing and compacting. Mellowing and compacting are to be done at a moisture content of $1.3 \times w_{opt}$.

4. One section to be constructed as in (3) above, except that mellowing and compacting are to be done at a moisture content of $0.7 \times w_{opt}$, with additional water being added to the surface of each compacted lift to provide a total amount of moisture equal to $1.3 \times w_{opt}$.

For the field test, the lime content should be equal to the LMO plus 1%. The thickness of compacted lifts should not exceed 6 inches, and a membrane cover (probably sprayed asphalt) should be applied over the compacted surface as soon as possible, to retain the soil moisture. Detailed construction records should be kept to account for minor changes in procedure that may be necessitated by project conditions or that occur inadvertently during construction.

A systematic and detailed plan for evaluation must be proposed by the project director for the particular site conditions, and modified, if necessary, in consultation with Research and Development personnel of the Oklahoma Department of Transportation.

BIBLIOGRAPHY

(1) "Characteristics of Clayey Soils Associated With Cross Timbers in Central Oklahoma and Granitic Outcrops in South-Central Oklahoma." Miscellaneous Publication MP-90, March, 1973. Agricultural Experiment Station, Oklahoma State University.

(2) "Lime Base Solves Clay Problem." Rural and Urban Roads, Vol. 8 (Feb., 1970), pp. 18-20.

(3) "Lime Stabilization Construction Manual." National Lime Association Bulletin 326, 1972.

(4) "Largest Lime Stabilization Job is Geared to 500 Tons Daily." Roads and Streets (May, 1972).

(5) "State of the Art: Lime Stabilization Reaction, Properties, Design, Construction." TRB Circular No. 180, Sept., 1976.

(6) Alexander, M. L., R. E. Smith, and G. B. Sherman. "Relative Stabilizing Effect of Various Limes on Clayey Soils." HRB Record 381 (1972), pp. 27-33.

(7) Anday, M. C. "Curing Lime-Stabilized Soils." HRB Record 29 (1963), pp. 13-26.

(8) Anday, M. C. "Accelerated Curing for Lime-Stabilized Soil." HRB Bulletin 304 (1961), pp. 1-13.

(9) Arman, A. and G. A. Munfakh. "Lime Stabilization of Organic Soils." Louisiana State University, Engineering Research Bulletin No. 103, 1970.

(10) Brand, W. and W. Schoenberg. "Impact of Stabilization of Loess With Quicklime on Highway Construction." HRB Bulletin 231 (1959), pp. 18-23.

(11) Carroll, D. "Clay Minerals: A Guide to Their X-ray Identification." The Geological Society of America, Special Paper 126, 1970.

(12) Carroll, D. and H. C. Starkey. "Reactivity of Clay Minerals With Acids and Alkalines." Clays and Clay Minerals, Vol. 19 (1971), pp. 321-333.

(13) Davidson, L. K., T. Demirel, and R. L. Handy. "Soil Pulverization and Lime Migration in Soil Lime Stabilization." HRB Record 92 (1965), pp. 103-126.

(14) Davidson, D. T., M. Mateos, and R. K. Karri. "Activation of the Lime-Flyash Reaction by Trace Chemicals." HRB Bulletin 231 (1959), pp. 67-81.

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

(16) Dempsey, B. J. and M. R. Thompson. "Durability Properties of Lime-Soil Mixtures." HRB Record 235 (1968), pp. 61-75.

(17) Diamond, S. and E. B. Kinter. "Mechanisms of Soil-Lime Stabilization: An Interpretive Review." HRB Record 92 (1965), pp. 83-101.

(18) Diamond, S., J. L. White, and W. L. Dolch. "Transformation of Clay Minerals by Calcium Hydroxide Attack." Proceedings, 12th National Conference on Clays and Clay Minerals. New York: Pergamon Press, 1964, pp. 359-379.

(19) Diamond, S., J. F. Young, and F. V. Lawrence, Jr. "Scanning Electron Microscopy-Energy Dispersive X-ray Analysis of Cement Constituents--Some Cautions." Cement and Concrete Research, Vol. 4, No. 6 (Nov., 1974), pp. 899-914.

(20) Eades, J. L. and R. E. Grim. "A Quick Test to Determine Lime Requirements for Lime Stabilization." HRB Record 139 (1963), pp. 61-72.

(21) Eades, J. L. and R. E. Grim. "Reaction of Hydrated Lime With Pure Minerals in Soil Stabilization." HRB Bulletin 262 (1960), pp. 51-63.

(22) Eades, J. L., F. P. Nichols, Jr., and R. E. Grim. "Formation of New Minerals With Lime-Stabilization as Proven by Field Experiments in Virginia." HRB Bulletin 335 (1962), pp. 31-39.

(23) Flaherty, C. A. and M. N. Gray. "The Influence of Alkali-Metal Compounds on the Compaction and Early Strength Properties of Lime-Soil Mixtures." Australian Road Research, Vol. 5, No. 5 (June, 1974), pp. 4-15.

(24) Fossberg, P. E. "Some Fundamental Engineering Properties of Lime-Stabilized Clay." HRB Record 263 (1969), pp. 19-26.

(25) Glenn, G. R. "Differential Thermal and Thermogravimetric Analysis of Reacted Bentonite-Lime-Water Mixtures." HRB Record 315 (1970), pp. 122-132.

(26) Glenn, G. R. and R. L. Handy. "Lime Clay Mineral Reaction Products." HRB Record 29 (1963), pp. 70-82.

(27) Goldberg, I. and A. Klein. "Some Effects of Treating Expansive Clays With Calcium Hydroxide." ASTM Special Technical Report No. 142, 1952.

(28) Gray, F. and H. M. Galloway. "Soils of Oklahoma." Miscellaneous Publication MP-56, July, 1956. Agricultural Experiment Station, Oklahoma State University.

(29) Grim, R. E. Applied Clay Mineralogy. New York: McGraw-Hill Book Co., Inc., 1962.

(30) Grim, R. E. Clay Mineralogy. 2nd ed. New York: McGraw-Hill Book Co., Inc., 1968.

(31) Harty, J. R. and M. R. Thompson. "Lime Reactivity of Tropical and Subtropical Soils." HRB Record 442 (1973), pp. 102-112.

(32) Hayden, M. L. "Chemical Stabilization of Dispersive Clay Soils." (Unpub. Master's thesis, Oklahoma State University, 1975.)

(33) Herrin, M. and H. Mitchell. "Lime-Soil Mixtures." HRB Bulletin 304 (1961), pp. 99-138.

(34) Hilt, G. H. and D. T. Davidson. "Lime Fixation in Clayey Soils." HRB Bulletin 262 (1960), pp. 20-32.

(35) Hilt, G. H. and D. T. Davidson. "Isolation and Investigation of a Lime-Montmorillonite Crystalline Reaction Product." HRB Bulletin 304 (1961), pp. 51-64.

(36) Hoskins, E. R., D. W. Hammerquist, and D. Irby. "Investigation of Several Additives for Controlling the Expansion of Pierre Shale." HRB Record 381 (1972), pp. 34-36.

(37) Ingles, O. G. and J. B. Metcalf. Soil Stabilization Principles and Practice. New York: John Wiley & Sons, 1973.

(38) Ireland, H. O. "Soil Stabilization." American Railway Engineering Association Bulletin, Vol. 628 (1970), pp. 846-855.

(39) Jackson, M. L. Soil Chemical Analysis--Advanced Course. 2nd ed. Madison, Wis.: Dept. of Soil Science, University of Wisconsin, 1973.

(40) Kobashi, T. "Reaction of Calcium Hydroxide With Allophane-Kaolinite Clay Minerals." Clay Science, Vol. 3, Nos. 1-2 (1967), pp. 11-36.

(41) Krauskopf, K. B. Introduction to Geochemistry. New York: McGraw-Hill Book Co., 1967.

(42) Kröenert, W. and K. Wetzel. "Reactions of Alkaline Earth Hydroxides With Clay Minerals." Ziegelindustrie, Vol. 25 (1972), pp. 562-567.

(43) Kröenert, W., and K. Wetzel. "Reactions of Alkaline Earty Hydroxides With Clay Minerals." Ziegelindustrie, Vol. 25 (1972), pp. 524-527.

(44) Kröenert, W., and K. Wetzel. "Reactions of Alkaline Earty Hydroxides With Clay Minerals." Ziegelindustrie, Vol. 25 (1972), pp. 468-471.

(45) Kröenert, W., and K. Wetzel. "Reactions of Alkaline Earth Hydroxides With Clay Minerals." Ziegelindustrie, Vol. 26 (1973), pp. 62-67.

(46) Ladd, C. C., Z. C. Moh, and T. W. Lambe. "Recent Soil-Lime Research at the Massachusetts Institute of Technology." HRB Bulletin 262 (1960), pp. 64-85.

(47) Lytton, R. L., and K. T. Moyer. "Stiffened Mats on Expansive Clay." ASCE Journal of the Soil Mechanics and Foundation Division SMT. July, 1971, pp. 999-1019.

(48) Lea, F. M. The Chemistry of Cement and Concrete (3rd ed). Great Britain: Edward Arnold (Publishers) Ltd., 1970.

(49) Leonard, R. J., and P. I. Davidson. "Pozzolanic Reactivity Study of Flyash." HRB Bulletin 231 (1959), pp. 1-17.

(50) Lund, O. L., and W. J. Ramsey. "Experimental Lime Stabilization in Nebraska." HRB Bulletin 231 (1959), pp. 24-59.

(51) MacMurdo, F. D., and E. J. B-renberg. "Determination of Realistic Cutoff Dates for Late-Season Construction with Lime-Fly Ash and Lime-Cement-Fly Mixtures." HRB Record 442 (1973), pp. 92-99.

(52) Marks, B. D., and T. A. Haliburton. "Acceleration of Lime Clay Reaction With Salt." Journal of the Soil Mechanics and Foundation Division, ASCE, Vol. 98, No. SM4, Proceedings Paper 8827 (April, 1972), pp. 327-339.

(53) Marks, D. B., and T. A. Haliburton. "Effect of Sodium Chloride and Sodium Chloride-Lime Admixtures on Cohesive Oklahoma Soils." HRB Record 315 (1970), pp. 102-111.

(54) Mateos, M., and D. T. Davidson. "Further Evaluation of Promising Chemical Additives for Accelerating Hardening of Soil Lime Fly Ash Mixtures." HRB Bulletin 304 (1961), pp. 32-50.

(55) Matsuo, S., and Y. K. Sung. "Fabric of Lime Stabilized Soils by X-ray and Electron Microscopy." Doboku Gakkai Ronbunshu (Japenese Socity of Civil Engineers), No. 193 (Sept., 1971), pp. 59-68.

(56) McDowell, C. "Evaluation of Soil Lime Stabilization Mixtures." HRB Record 139 (1966), pp. 15-24.

(57) McDowell, C. "Stabilization of Soils With Lime, Lime-Flyash, and Other Lime Reactive Materials." HRB Bulletin 231 (1959), pp. 60-66.

(58) McDowell, C. "Flexible Pavement Design Guide." Bulletin No. 327. The National Lime Association, Washington, D.C.,

(59) Mitchell, J. K. and D. R. Hooper. "Influence of Time Between Mixing and Compaction on Properties of a Lime-Stabilized Expansive Clay." HRB Bulletin 304 (1961), pp. 14-31.

(60) Mitchell, J. K. "Soil Stabilization Methods and Their Applications in Highway Construction." Fourth Annual Nevada Street and Highway Conference, Reno and Las Vegas, Nevada, March 5-6, 1969, pp. 1-26.

(61) Moore, J. C. and R. L. Jones. "Effect of Soil Surface Area and Extractable Silica, Alumina, and Iron on Lime Stabilization Characteristics of Illinois Soils." HRB Record 351 (1971), pp. 87-92.

(62) Neubauer, C. H., Jr., and M. R. Thompson. "Stability Properties of Uncured Lime-Treated Fine Grained Soils." HRB Record 381 (1972), pp. 20-26.

(63) O'Flaherty, C. A. and M. N. Gray. "The Influence of Alkali-Metal Compounds on the Compaction and Early Strength Properties of Lime-Soil Mixtures." Australian Road Research (Board), Vol. 5, No. 5 (June, 1974), pp. 4-15.

(64) Ormsby, W. C. and L. H. Bolz. "Microtexture and Composition of Reaction Products in the System Kaolin-Lime-Water." Journal of the American Ceramic Society, Vol. 49, No. 7 (1966), pp. 364-366.

(65) Patrick, D. M. "Mineralogy and Geochemistry of Volcanic Ash and Bentonite in the Ogallala Formation (Tertiary) of Western Oklahoma." (Unpub. Ph.D. dissertation, University of Oklahoma, Norman, Oklahoma, 1972.)

(66) Petry, T. M. "Identification of Dispersive Clay Soils by a Physical Test." (Unpub. Ph.D. dissertation, Oklahoma State University, Stillwater, Oklahoma, 1974.)

(67) Rosen, W. G. and B. D. Marks. "Cold Weather Lime Stabilization." HRB Record 501 (1974), pp. 35-41.

(68) Ruff, C. G. and Clara Ho. "Time-Temperature Strength-Reaction Product Relationships in Lime-Bentonite-Water Mixtures." HRB Record 139 (1966), pp. 42-60.

(69) Sabry, M. A. "Soil Lime Reaction." Unpublished report, December, 1974.

(70) Shen, C. K. and S. K. Li. "Lime Stabilization of Clay-Sand Mixtures." HRB Record 315 (1970), pp. 91-101.

(71) Slone, R. L. "Early Reaction in the Kaolinite-Hydrated Lime-Water System." Paper presented at 1st Annual Meeting, Clay Minerals Society, Madison, Wis., 1964, pp. 121-125.

(72) Stocker, P. T. "Diffusion and Diffuse Cementation in Lime and Cement Stabilized Clayey Soils--Studies of Plasticity and Aggregation." Australian Road Research, Vol. 5, No. 6 (1974), pp. 51-75.

(73) Stocker, P. T. "Diffusion and Diffuse Cementation in Lime and Cement Stabilized Clayey Soils--Chemical Aspects." Australian Road Research, Vol. 5, No. 9 (1975), pp. 6-47.

(74) Taylor, H. F. W. The Chemistry of Cements. Vols. 1 and 2. New York: Academic Press, 1964.

(75) Thompson, M. R. "Lime Reactivity of Illinois Soils." Journal of the Soil Mechanics and Foundations Division, ASCE, Vol. 92 No. SM5 (Sept., 1966), pp. 67-92.

(76) Thompson, M. R. "Split-Tensile Strength of Lime-Stabilized Soils." HRB Record 92 (1965), pp. 75-86.

(77) Thompson, M. R. "Shear Strength and Elastic Properties of Lime-Soil Mixtures." HRB Record 139 (1966), pp. 1-15.

(78) Thompson, M. R. and B. J. Dempsey. "Autogenous Healing of Lime Soil Mixture." HRB Record 263 (1969), pp. 1-7.

(79) Torrance, J. K. "A Laboratory Investigation of the Effect of Leaching on the Compressibility and Shear Strength of Norwegian Marine Clays." Geotechnique, Vol. 24, No. 2 (1974), pp. 155-173.

(80) Townsend, D. L. and T. W. Klym. "Durability of Lime Stabilized Soils." HRB Record 139 (1966), pp. 25-41.

(81) Tulloch, W. S., II, W. R. Hudson, and T. W. Kennedy. "Evaluation and Prediction of the Tensile Properties of Lime Treated Materials." Research Report 98-5. Center for Highway Research, The University of Texas at Austin, June, 1970.

(82) Van Ganse, R. F. "Immediate Amelioration of Wet Cohesive Soils by Quicklime." TRB Record 501 (1974), pp. 42-50.

(83) Walker, R. D. and C. Karabulut. "Effect of Freezing and Thawing on Unconfined Compressive Strength of Lime-Stabilized Soils." HRB Record 92 (1965), pp. 1-11.

(84) Weaver, C. E. and L. Pollard. "Developments in Sedimentology 15." The Chemistry of Clay Minerals. Amsterdam: Elsevier Scientific Publishing Company, 1973.

(85) Wright, P. J. "Lime Slurry Pressure Injection Tames Expansive Clays." Civil Engineering--ASCE (Oct., 1973), pp. 25-28.

APPENDIX A

TEST PROCEDURE FOR THE LIME MODIFICATION OPTIMUM, EADES AND GRIM METHOD (20)

SOIL MECHANICS LABORATORY
School of Civil Engineering
Oklahoma State University
Stillwater, Oklahoma

pH TEST FOR LIME MODIFICATION OPTIMUM

Tested By____________________Tested for____________________Sheet___of___

Date_______________Sample No.______Test No.______Lime Type: CaO $Ca(OH)_2$ $CaCl_2$

Description of Sample____

Percent** Lime In Solution	pH of Solution

Summary of Test Procedure

1. For each lime percentage to be tested weigh out 20g of air dry (-) U.S. No. 40 Sieve soil and pour into plastic 250 ml centrifuge bottle.
2. Add desired percentage of lime to each bottle.
3. Add 100 ml deionized distilled water to each bottle, shake for at least 30 sec or until all dry material is in suspension.
4. Shake each bottle for 30 sec every 10 min until 1 hr has elapsed.*
5. Then transfer part of the slurry to a 100 ml beaker and record pH of same.
6. The pH should increase to approx. 12.4 then it should remain constant for higher lime percentages. If pH does not exceed 12.3 try higher lime percentages. The lowest lime percentage required to develop constant pH conditions is the lime modification optimum.

Remarks____

---

* Alternate Procedure is to shake bottles continuously for 1 hr in bottle shaker on high speed.

** Percent is based on weight of dry soil.

APPENDIX B

TABULATED CRYSTALLOGRAPHIC AND X-RAY DATA

Calcium hydroxide ($Ca(OH)_2$)

Crystal form

Hexagonal flakes, (0001) cleavage

Unit cell

Trigonal *a* 3·593 *c* 4·909 Å

Space group	*Z*	*D*
$P\bar{3}m1$	1	2·241

Refractive indices

ω 1·573 ε 1·545

Other optical properties

Uniaxial –

Main reference [5].

X-Ray Powder Data [5]

d	I	*hkil*	*d*	I	*hkil*
4·90	vs	0001	1·1432	w	21·1, 10·4
3·112	m	10·0	1·1275	vvw	20·3
2·628	vvs	10·1	1·0599	w	21·2
2·447	vvw	0002	1·0366	vw	30·0
1·927	ms	10·2	1·0143	vw	30·1, 11·4
1·796	ms	11·0	0·9551	vvw	30·2, 21·3
1·687	m	11·1	0·9369	vvw	10·5
1·634	vvw	0003	0·8979	vvw	22·0
1·557	vvw	20·0	0·8838	vvw	22·1
1·484	w	20·1	0·8760	vvw	30·3
1·449	w	11·2, 10·3	0·8623	vvw	31·0, 11·5, 22·2
1·314	vw	20·2	0·8495	vw	31·1, 21·4
1·228	vvw	0004	0·8140	vw	0006
1·211	vvw	11·3			
1·1762	vvw	21·0			

Quartz (low-quartz, SiO_2)

Crystal form	Trisms, length *c*	
Unit cell	Trigonal a 4·913 c 5·405 Å	
Space group $P3_121(P3_221)$	Z 3	D 2·6485
Refractive indices	ω 1·5443 ϵ 1·5534	
Other optical properties	Uniaxial +	

Main references [*3*, *4*].

X-Ray Powder Data [*3*]

d	I	*hkl*	*d*	I	*hkl*
4·26	**s**	10·0	1·228	vvw	22·0
3·343	**vvs**	10·1	1·1997	vw	21·3
2·458	mw	11·0	1·1973	vvw	22·1
2·282	mw	10·2	1·1838	vw	11·4
2·237	w	11·1	1·1802	vw	31·0
2·128	w	20.0	1·1530	vvw	31·1
1·980	w	20·1	1·1408	vvw	20·4
1·817	**ms**	11·2	1·1144	vvw	30·3
1·801	vvw	0003	1·0816	vw	31·2
1·672	w	20·2	1·0636	vvw	40·0
1·659	vw	10·3	1·0477	vvw	10·5
1·608	vvw	21·0	1·0437	vvw	40·1
1·541	**m**	21·1	1·0346	vvw	21·4
1·453	vw	11·3	1·0149	vvw	22·3
1·418	vvw	30·0	0·9896	vvw	40·2, 11·5
1·382	w	21·2	0·9872	vvw	31·3
1·375	mw	20·3	0·9781	vvw	30·4
1·372	w	30·1	0·9762	vvw	32·0
1·288	vw	10·4	0·9607	vvw	32·1
1·256	vw	30·2	0·9285	vvw	41·0

Gypsum ($CaSO_4.2H_2O$)

Crystal form

Tablets, (010) cleavage; may be elongated || *c*.

Unit cell

Monoclinic *a* 5·68 *b* 15·18 *c* 6·29 Å; *β* 113·8°*

Space group	*Z*	*D*
*A*2/a	4	2·32

Refractive indices

α 1·520 *β* 1·524 *γ* 1·530

Other optical properties

Biaxial +, 2V = 58°. Y = *b*

Main references [*4, 10*].

* The unit cell may be defined in various ways; see reference [*4*] Volume 5, p. 202. The powder data (below) are referred to a body-centred cell with *a* 5·68 *b* 15·18 *c* 6·51 Å, *β* 118·4°.

X-Ray Powder Data [*10*]

d	I	*hkl*	*d*	I	*hkl*
7·56	vs	020	1·879	mw	143
4·27	s	$12\bar{1}$	1·864	w	$31\bar{2}$
3·79	m	031, 040	1·843	vvw	231
3·163	vw	$11\bar{2}$	1·812	mw	$26\bar{2}$
3·059	s	$14\bar{1}$	1·796	w	$32\bar{1}$
2·867	ms	002	1·778	mw	260
2·786	w	$21\bar{1}$	1·711	vvw	253
2·679	ms	022, 051	1·684	vvw	$32\bar{3}$
2·591	w	150, $20\bar{2}$	1·664	w	$34\bar{1}$
2·530	vvw	060	1·645	vw	163
2·495	w	200	1·621	w	204, 181, 053
2·450	w	$22\bar{2}$	1·599	vvw	352, 190
2·400	w	141	1·584	vvw	$22\bar{4}$, etc.
2·216	w	$15\bar{2}$	1·532	vvw	282
2·139	vvw	$24\bar{2}$	1·522	vvw	222, 134
2·080	mw	$12\bar{3}$			
2·073	w	112, $25\bar{1}$			
1·990	w	170			
1·953	vw	211			
1·898	mw	080, 062			

Calcite ($CaCO_3$)

Crystal form

Rhombs, $\{10\bar{1}4\}$ cleavage

Unit cell*

Hex. axes: a_H 4·989 c_H 17·062 Å; Rhomb. axes: a_R 6·37 Å, α 46·0°

Space group	Z	D
$R\bar{3}c$	(Rh.) 2	2·711

Refractive indices

ω 1·659 ϵ 1·487

Other optical properties

Uniaxial –

Main references [*4*, *8*].

* For other choices of axes, see reference [*4*], Volume 5, p. 229.

X-RAY POWDER DATA [*8*]

d	I	*hkil*	*d*	I	*hkil*
3·86	mw	10·2	1·297	vvw	21·8
3·035	**vvs**	10·4	1·284	vvw	30·6
2·845	vw	0006	1·247	vvw	22·0
2·495	**m**	11·0	1·235	vvw	11·12
2·285	**s**	11·3	1·1795	vw	21·10
2·095	**s**	20·2	1·1538	vw	31·4
1·927	w	20·4	1·1425	vvw	22·6
1·913	**ms**	10·8	1·1244	vvw	21·11
1·875	**ms**	11·6	1·0613	vvw	20·14
1·626	vw	21·1	1·0473	vw	40·4
1·604	w	21·2	1·0447	vw	13·8
1·587	vvw	10·10	1·0352	vvw	01·16, 11·15
1·525	w	21·4	1·0234	vvw	12·13
1·518	vw	20·8	1·0118	vvw	30·12
1·510	vw	11·9	0·9895	vvw	23·1
1·473	vvw	21·5			
1·440	w	30·0	0·9846	vvw	32·2
1·422	vw	00012	0·9782	vvw	10·17
1·356	vvw	21·7	0·9767	vw	21·14
1·339	vvw	20·10	0·9655	vvw	23·4

C-S-H(I) ($0{\cdot}8$–$1{\cdot}5$ $CaO.SiO_2.0{\cdot}5$–$2{\cdot}5$ H_2O)*

Crystal form

Crumpled foils (semi-crystalline)

Unit cell

Geometrically orthorhombic *a* 11·2 *b* 7·3 *c* 9–14 Å

Space group	*Z*	*D*
—	10 Ca (?)	2·0–2·2

Refractive indices

1·494–1·530 (mean)

Other optical properties

Main references [*13*, *18*].

* Calcium silicate hydrate (I). Natural *plombierite* [*18*] is closely similar but gives no basal X-ray reflection. Density and refractive index given above relate to natural plombierite of approx. composition $0{\cdot}8CaO.SiO_2.2H_2O$. Basal spacing of C-S-H (I) varies with both water content and Ca/Si ratio; see Volume 1, Chapter 5.

X-RAY POWDER DATA [*13*]

d	I	*hkl*	*d*	I	*hkl*
12·5	**vs**	001	1·23	vw	(?)
5·3	vvw	(?)	1·17	vw	260
3·07	**vs**	220	1·11	w	840
2·80	**s**	400	1·07	vw	10·20
2·4	w/d	(?)			
2·1	w/d	(?)			
1·83	**s**	040			
1·67	mw	620			
1·53	vw	440			
1·40	w	800			

C-S-H(II) (1·5–2·0 $CaO.SiO_2.1–4\ H_2O$)*

Crystal form

Fibre bundles (semi-crystalline)

Unit cell

Related to C-S-H (I); *c* 10 Å approx.

Space group	*Z*	*D*
—	(?)	(?)

Refractive indices

(?)

Other optical properties

Main reference [*13*].

* Calcium silicate hydrate (II). Several varieties exist: see Volume 1, Chapter 5. Powder data below relate to fibre bundles. Some other varieties give powder patterns close to that of C-S-H (I). The basal spacing, if it occurs at all, is always about 10 Å. The reflections at 3·07, 2·80 and 1·83 Å are also given by tobermorite gel (see Volume 1, Chapter 5).

X-Ray Powder Data [*13*]

d	I	*hkl*	*d*	I	*hkl*
9·8	vs		1·72	vvw	
4·9	w		1·62	vvw	
3·07	vvs		1·56	m	
2·85	m		1·40	mw	
2·80	vs		1·225	w	
2·40	m		1·165	w	
2·20	vvw		1·1	vvw/d	
2·10	vvw		1·045	vw	
2·00	ms		1·025	vvw	
1·83	vs		1·000	vw	

Ettringite (calcium aluminate trisulphate hydrate, $Ca_6Al_2(OH)_{12}(SO_4)_3 . 26H_2O$ or $C_3A . 3CaSO_4 . 32H_2O$)

Crystal form

Hexagonal prisms or needles with length *c*

Unit cell

Hexagonal, *a* 11·23 *c* 21·44 Å

Space group	*Z*	*D*
$P6_3/mmc$	2	1·73–1·79

Refractive indices

ω 1·4655 ϵ 1·4618

Other optical properties

Uniaxial –

Main references [*39–40*].

X-Ray Powder Data [*39*]

d	I	*hkil*	*d*	I	*hkil*
9·73	vvs	10·0	2·616	m	31·2
8·86	mw	10·1	**2·564**	s	21·6
5·61	vs	11·0	2·524	vw	31·3
4·98	m	11·2	2·487	vw	22·4
4·86	w	20·0	2·434	vw	40·0
4·69	ms	10·4	2·422	vw	11·8
4·41	vw	31·4	2·401	w	30·6
4·02	w	20·3	2·347	vw	20·8
3·88	s	11·4	2·230	m	32·0
3·67	w	21·0	**2·209**	s	22·6
3·60	mw	20·4	2·185	w	32·2
3·48	m	21·2	2·154	m	31·6
3·27	vw	21·3	2·130	vw	32·3
3·240	m	30·0	2·124	vw	41·0
3·016	w	11·6	2·081	vw	41·2
2·806	w	22·0	2·062	vw	32·4
2·773	ms	30·4	2·033	vvw	41·3
2·714	w	22·2	2·027	vvw	31·7
2·697	mw	31·0	1·979	vw	32·5
2·680	w	0008			

Tricalcium aluminate hexahydrate ($Ca_3Al_2(OH)_{12}$ or C_3AH_6)

Crystal form

Trapezohedra and other cubic forms

Unit cell

Cubic *a* 12·576 Å

Space group	*Z*	*D*
*I*a3d	8	2·522

Refractive indices

n 1·604

Other optical properties

Isotropic

Main reference [*36*].

X-Ray Powder Data [*36*]

(Mo Radiation)

d	I	*hkl*	*d*	I	*hkl*
5·13	s	211	1·595	m	651, 732
4·45	s	220	1·572	m	800
3·36	s	321	1·484	w	660, 822
3·14	s	400	1·407	s	840
2·814	**vs**	420	1·372	m	842
2·68	vw	332	1·342	m	664
2·57	w	422	1·298	w	763, 932
2·47	m	510, 431	1·283	w	844
2·30	**vs**	521	1·244	w	772
2·04	**vs**	611	1·199	m	952
1·99	vw	620	1·167	m	10·40
1·815	w	444	1·148	m	10·42
1·742	s	640	1·120	m	963
1·710	w	552, 633, 721	1·086	w	972
1·679	s	642	1·048	w	884, 12·00
			1·020	m	10·64, 12·22

Reflections have also been reported at 2·21 Å (440), 1·354 Å (921), and 1·268 Å (941) [*37*]

Monocalcium aluminate 10-hydrate ($CaAl_2(OH)_8.6H_2O$ or CAH_{10})

Crystal form

Very finely divided. Crumpled or rolled foils.† Hexagonal prisms

Unit cell

Unknown

Space group	Z	D
(?)	(?)	(?)

Refractive indices

1·471 (mean)

Other optical properties

Main reference [*31*].

† See Chapter 21, Plate 41.

X-Ray Powder Data [*30*]

d	I	*hkl*	*d*	I	*hkl*
14·3	**vs**		1·94	m	
7·16	**vs**		1·87	vvw	
5·39	mw		1·83	w	
4·75	mw		1·79	mw	
4·52	w		1·75	vvw	
4·16	w		1·71	w	
3·93	vvw		1·64	m	
3·72	m		1·60	ms	
3·56	**s**		1·56	vvw	
3·26	ms		1·52	vw	
3·10	m		1·47	w	
2·88	ms		1·40	vw	
2·69	m		1·38	mw	
2·55	**s**		1·27	vvw	
2·47	m		1·24	vw	
2·36	ms		1·18	vw	
2·26	ms		1·07	vw	
2·18	ms				
2·11	mw				
2·06	mw				

Tetracalcium aluminate carbonate 12-hydrate
($Ca_4Al_2(OH)_{12}(CO_3).6H_2O$ or $C_3A.CaCO_3.12\ H_2O$)

Crystal form

Hexagonal plates, (0001) cleavage

Unit cell

Pseudo cell: hexagonal with *a* 5·7 *c* 7·6 Å*

Space group	*Z*	*D*
(?)	½	(?)

Refractive indices

ω 1·554 ϵ 1·532

Other optical properties

Uniaxial –

Main references [*34*, *38*], see also Volume 1, Chapter 6

* The true value of *a* is possibly (2 × 5·7) Å.

X-Ray Powder Data [*34*]

d	I	*hkil*	*d*	I	*hkil*
7·6	vvs	0001	2·17	vw	
4·93	vvw/b		2·11	vw	
4·41	vvw/b		2·01	vw/b	
3·97	vvw/b		1·94	vw	
3·80	ms	0002	1·88	vw	
3·65	vvw		1·86	vw	
3·46	vvw		1·83	w	
3·30	vvw		1·69	vvw	
3·17	vvw		**1·66**	m	
3·04	vvw		1·64	vw	
2·86	m		1·60	vw	
2·78	vw		1·58	vvw/b	
2·73	vw		1·55	vvw	
2·53	w	0003	1·51	vw/b	
2·49	mw				
2·45	vw/b				
2·42	vw/b				
2·34	vw/b				
2·29	vw				
2·24	vw				

"α-Tetracalcium aluminate 13-hydrate"($Ca_2Al(OH)_7.3H_2O$ with some essential CO_3^{2-}1

Crystal form	Hexagonal plates with (0001) cleavage
Unit cell	Structural element: hexagonal, *a* 5·7 *c* 8·2 Å

Space group	*Z*	*D*
(?)	1	2·0 approx.

Refractive indices	ω 1·536 ε 1·520 (approx.)
Other optical properties	Uniaxial –

Main references [*32–34*], see also Volume 1, Chapter 6.

X-Ray Powder Data [*34*]

d	I	*hkil*	*d*	I	*hkil*
8·2	vvs	0001	1·99	mw	
4·63	vw		1·94	w	
4·11	mw	0002	1·88	vvw	
3·88	ms		1·86	w	
3·63	vvw		1·85	vw	
3·51	vw		1·82	vvw	
3·02	vw		1·80	vvw	
2·88	vs		1·78	vvw	
2·83	vw		1·76	vw	
2·72	m		1·74	vvw	
2·62	vw		1·72	vvw	
2·59	vvw		1·69	vvw	
2·55	m		1·68	vvw	
2·49	vvw		**1·66**	m	
2·45	vs		**1·63**	m	
2·36	m		1·61	vw	
2·34	vvw		1·54	vw	
2·31	mw		1·53	vvw	
2·27	vvw		1·51	vw	
2·22	w		1·50	vw	
2·21	vw				
2·19	vvw				
2·17	vw				
2·07	vvw				
2·04	w				

Tetracalcium aluminate 13-hydrate ($Ca_2Al(OH)_7.3H_2O$ or C_4AH_{13})

Crystal form

Hexagonal plates, (0001) cleavage

Unit cell*

Hexagonal, *a* 5·74 *c* 7·92 Å

Space group	*Z*	*D*
(?)	1	2·02

Refractive indices

ω 1·534–1·539 ε 1·514–1·524

Other optical properties

Uniaxial –

Main references [*32–35*].

* Structural element. True *c*-axis is either a high multiple of 7·92 Å or indefinite (O–D structure). Details of powder pattern are probably variable due to polytypism.

X-Ray Powder Data [*34*]

d	I	*hkil*	*d*	I	*hkil*
7·9	**vvs**	0001	2·73	vvw	
4·95	vvw		**2·69**	**ms**	
4·75	vvw		2·63	w	0003
4·62	vvw		2·49	w	
4·54	vw		**2·47**	**ms**	
4·48	w		**2·45**	**vs**	
3·95	**m**	0002	2·38	w	
3·88	w		2·31	vw	
3·76	vvw		2·27	vvw	
3·62	w		2·24	mw	
3·31	vw		2·21	vvw	
2·88	**s**		2·17	vw	
2·86	**vs**		2·16	vvw	
2·83	vvw		2·11	vw	
2·79	**m**		2·05	vw	

X-RAY DATA

Hillebrandite 9 (Synthetic)($C_2SH(B)$)		Afwillite 9		$2CaO:SiO_2$ x hydrate ($C_2SH(A)$	
d	I	d	I	d	I
12.0?	1	6.45	8	5.35	3
8.10	3	5.74	8	4.63	1
5.70	3	5.08	5	4.22	9
4.74	9	4.73	8	3.90	8
4.03	4	4.15	5	3.54	8
3.51	6	3.91	5	3.27	10
3.32	6	3.75	5	3.04	3
3.00	7	3.28	5	2.87	8
2.90	10	3.19	10	2.80	8
2.80	2	3.05	5	2.77	3
2.75	9	2.84	10	2.71	3
2.67	4	2.74	10	2.69	2
2.62	4	2.67	5	2.65	6
2.44	3	2.59	5	2.60	8
2.36	7	2.44	4	2.56	3
2.23	9	2.35	6	2.52	6
2.05	6	2.31	5	2.47	1
1.95	6	2.21	5	2.41	9
		1.145	8	2.31	2
		2.064	4	2.27	2
		2.-17	4	2.24	3
				2.18	5
				2.16	3
				2.10	2
				2.08	3

VITA

Mohamed Mamdouh Aly Sabry

Candidate for the Degree of

Doctor of Philosophy

Thesis: MINERALOGICAL AND ENGINEERING PROPERTIES OF COMPACTED SOIL-LIME MIXES

Major Field: Civil Engineering

Biographical:

Personal Data: Born in Alexandria, Egypt, May 8, 1947, the son of Mr. and Mrs. Aly A. Sabry.

Education: Graduated from Cairo University at Cairo, Egypt, with the degree of Bachelor of Science in Civil Engineering in September, 1969; attended graduate school at Cairo University, Cairo, Egypt, between 1970-1973; received the Master of Science degree with a major in Civil Engineering at Oklahoma State University in July, 1974; completed the requirements for the Doctor of Philosophy degree in May, 1977.

Professional Experience: Junior Engineer for Sabry and Saeid Engineering Firm, October 1969 to December 1972; teaching assistant at Cairo University 1970-1972; teaching assistant at Oklahoma State University, January 1973 to March 1976; research assistant at Oklahoma State University, March 1976 to May 1977.

Professional Societies: Member of the Egyptian Engineering Association; member of the Institutions of Engineering Professions, Egypt; associate member of the American Society of Civil Engineers; member of the Transportation Research Board; EIT No. 3749 state of Oklahoma.

Honor Societies: Member of Phi Kappa Phi and Chi Epsilon.