

THE INFLUENCE OF SESQUIOXIDES ON SOME
PHYSICO-CHEMICAL AND ENGINEERING
PROPERTIES OF A LATERITIC SOIL

By

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To my wife, Marvel, for her continuing encouragement and understanding during this study and to my parents, Wesley and Virginia, whose early guidance served as an inspiration.

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CHAPTER I

INTRODUCTION

General

"Laterite" and "lateritic soils" are terms used to describe red residual soils occurring throughout the tropical and subtropical regions of the world. This soil is formed by intense weathering of almost any rock type: basalt, granite, gneiss, breccia, volcanic ash, and conglomerate, to mention a few (5). Geomorphologically, laterization involves the leaching out or removal of the silica, alkali, and alkaline earths, and concentration of hydrated iron and aluminum oxides (sesquioxides). Generally the resulting soil possesses a granular structure due to the abundance of these sesquioxides, which coat the pore walls and knit the soil particles into tiny spherical aggregates (2).

The weathering process is highly complex and influenced by the geological and geographical factors of climate, elevation, vegetation, rainfall, water table fluctuations, parent rock, and age (52). Because of the diversity of these factors, laterites occur in a variety of forms ranging from hard concretionary deposits to friable soils. The multiplicity of forms has created a profusion of synonyms for laterites in the literature. Presently, the terms ferruginous and/or aluminous crusts or hardpans, plinthite, ferralitic soils, kaolisols, oxisols, latosols, and saprolitic clays all connote "laterite" in the broadest

sense (46).

The engineering characteristics of lateritic soils also differ extensively with geographic location. These differences have caused much confusion in the literature concerning the occurrence, properties, and the engineering characteristics of lateritic soils. While use of standard classification tests should enable soils engineers to predict adequately the engineering behavior of such materials regardless of their geographic origin, several investigators (54, 75, 79, 83) have reported considerable variations in the values obtained by using standard test procedures. These variations depended upon the treatment of the soil prior to and during testing. As a result, the use of standard tests for predicting the engineering behavior of these soils, as well as various test results reported in the literature, may be unreliable. Consequently, the engineering classification of tropical climate clays by systems oriented for temperate climate clays has been questioned (53).

Difficulties encountered during construction of Sasamua Dam in Kenya, Africa, illustrate some of the classification problems associated with a lateritic soil. Tests revealed that the material possessed a high plastic limit, but the overall plasticity index was somewhat lower than expected. Also, the Atterberg limit values varied considerably depending upon pretreatment of the soil and reproducible results were difficult to obtain. If the soil was oven dried to obtain the natural moisture content prior to testing, the plasticity index decreased compared to the values obtained for unheated soil. Grain size analyses also exhibited considerable variation depending upon mixing and the type of deflocculating agent used in the tests. Consequently,

engineering classification of this soil based upon these tests was difficult. The natural moisture content often exceeded the optimum moisture content. This condition hampered proper equipment utilization and good compaction unless the soil was allowed to dry prior to compaction and moisture contents were carefully controlled. The densities achieved were substantially lower than would be expected for normal temperate clays with similar plastic indices. The difficulties of handling such a material resulted in claims that the soil was "abnormal" and unsuitable as a construction material for this project. Subsequent investigations, however, revealed that although the soil possessed a higher permeability than expected from the classification tests, the shearing strength was quite acceptable. Later, chemical and mineralogical studies revealed that the clay consisted of almost 60 percent halloysite and 16 percent goethite (hydrated iron oxide), and that the structure of the clay fraction consisted of tiny spherical aggregates or clusters of clayey materials impregnated by the iron oxides (54, 75). From these analyses, it was possible to explain most of the abnormal properties associated with the soil.

Generally, the granular structure furnishes in situ laterites and lateritic soils with the favorable engineering characteristics of good internal drainage, moderate plasticity, and moderate to high bearing capacities. However, remolding by mechanical agents; i.e., heavy construction equipment, causes a breakdown of the granular structure with a subsequent increase in plasticity and a decrease in permeability and bearing capacity. This breakdown in structure is also partially responsible for the wide variation in values obtained for Atterberg limit tests and grain size analyses (54, 79).

Winterkorn (83) observed that mixing and compaction techniques which destroy the granular structure greatly alter the desirable engineering characteristics of lateritic soils. The use of the common stabilizing additives, lime and portland cement, to prevent or offset the effects of remolding offers a possible solution to this problem. However, the stabilizing susceptibility of laterites is varied and is influenced by remolding (78, 80). Remolding and stabilization involve respectively the destruction and formation of cementing agents. Therefore, it would appear that additional information on the role of the sesquioxides in forming and maintaining the granular structure, and of the physical, chemical, and mineralogical properties of the soil would permit more intelligent use of lateritic soils by civil engineers.

The problem is adequately summarized by Bawa (5) who stated, "a definite need exists for the standardization of terminology and a collection of data on the physical, chemical, mineralogical, and engineering properties of lateritic soils under different conditions."

Purpose and Scope of Investigation

Winterkorn (83) commented that "the presence of iron in lateritic soils is one of the most important factors which influences their engineering properties." Apparently, the sesquioxides and their relationship with the indigenous soil particles are key factors in understanding the unusual behavioral characteristics of lateritic soils. The present investigation was primarily directed toward studying the influence of the sesquioxides on the physico-chemical properties of a particular lateritic soil from the Panama Canal Zone. The objectives of the study were to understand more fully the causal effects of

remolding to determine the influence of the sesquioxides on engineering characteristics and to evaluate possible revisions of standard laboratory testing procedures. Concurrently, the properties exhibited by this soil were compared with those reported in the literature to ascertain if similar behavioral characteristics exist for other lateritic soils.

The chemical properties determined consisted of cation exchange capacity (CEC), sesquioxide and silica percentages, and the percentages of kaolinite and amorphous materials. The mineralogical properties were determined by x-ray diffraction and differential thermal analysis (DTA). The engineering properties which were examined consisted of Atterberg limits, grain size analysis, and unconfined compression and triaxial strength characteristics.

The influence of the sesquioxides on the Atterberg limits and grain size distribution was determined by chemically extracting these materials and then testing the "sesquioxide free" soil. To evaluate the effects of these iron and aluminum oxides on lime stabilization of the soil, the unconfined compressive strengths of lime treated specimens of the "sesquioxide free" soil were compared with the results of previous studies (80).

Source of Material

The lateritic soil used in this investigation was obtained from a borrow pit located in Curundu, Panama Canal Zone. The soil was excavated by hand from an exposed face at one end of the pit, with samples being taken at random depths varying from the surface to 17 feet.

The borrow pit from which the soil was obtained was used as a

source of fill material for construction of the Thatcher Ferry Bridge (Bridge of the Americas). Mr. R. H. Stewart,* geologist for the Panama Canal Company, described the geological profile as follows:

0-4 ft. Clay OH 3-4. Medium hard to hard, moderate strength, moderate to high plasticity, high dry strength, moderate water content, silt content increases with depth, consists of a residual saprolitic clay derived from agglomerate by normal weathering processes. Color: mottled bright reds and buff.

0-17.5 ft. Silt OH 3-4. Medium hard to hard, moderate strength, low plasticity, moderate to low dry strength, moderate water content, very clayey at top becoming sandy at base, consists of residual saprolitic clayey sandy silt derived from agglomerate by normal weathering processes. Color: mottled bright red and buff at top grading to mottled grays and browns at base.

17.5 - 18.5 ft. Top of sound rock Agglomerate RH 3. Hard, strong, massive jointing and bedding, consists of andesitic and basaltic pebbles ranging from 1/4 in. to 3.0 ft. in diameter in a fine grained sandy matrix of similar composition. Color: mottled reds, browns, blue-gray in a blue-gray matrix, oxidizes rapidly to dark gray and brown on exposure to air.

A narrow gray mottled zone mentioned by several authors (52, 55) as characteristic of lateritic soils, was observed at approximately 2-4 feet below the natural ground surface. Numerous small angular pebbles of quartz and chalcedony were encountered in the upper soil horizons. These pebbles were probably formed by chemical precipitation of the leached silica.

* Personal communication with Mr. R. H. Stewart, Fall 1967.

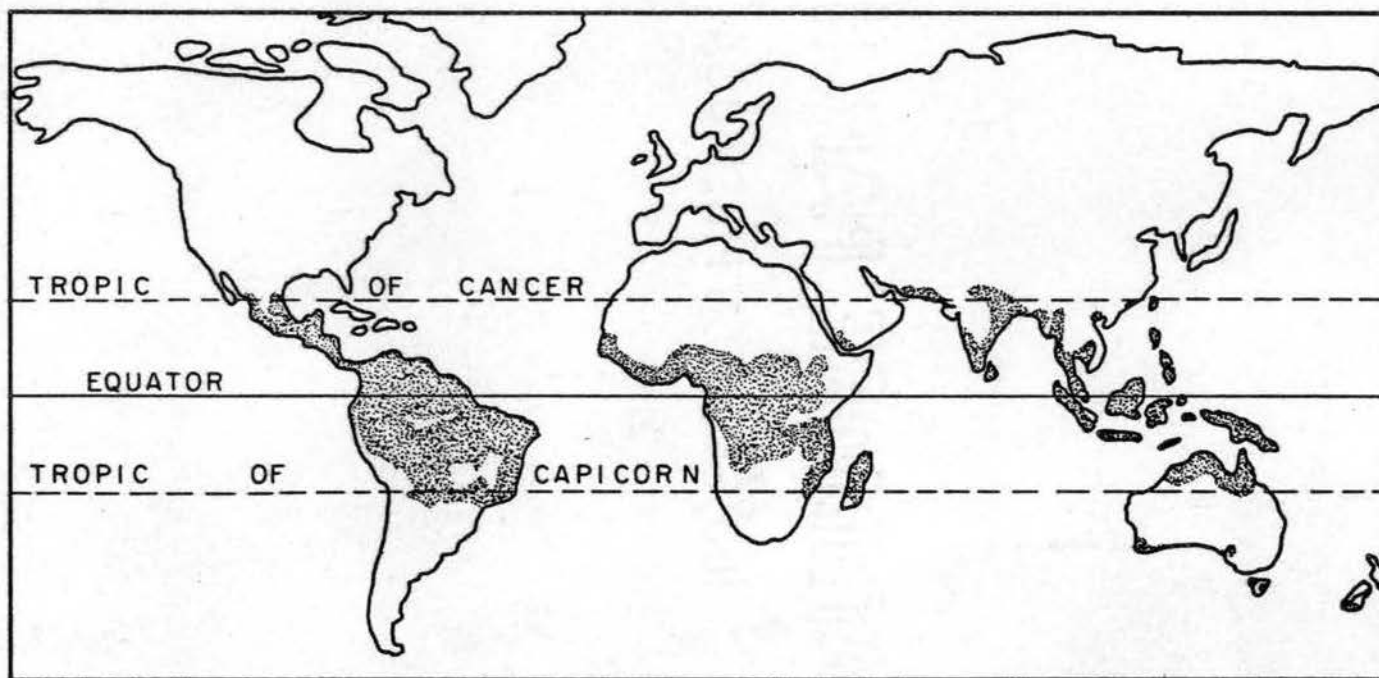
CHAPTER II

GEOLOGY OF LATERITE AND LATERITIC SOILS

Geographic Distribution

"Laterite" was first identified by Buchanan (9), who observed the natives of Angadipuram, India, cutting blocks of a soft ferruginous earth, and exposing them to the sun and air for drying. After drying, the blocks became hard as bricks and were used for building materials. The name "laterite" was derived from a translation of the Latin word, later, meaning brick. Although the term was originally applied to a particular morphology and weathering, laterite was soon associated with any red colored tropical soil.

Laterites and lateritic soils are widely distributed throughout the world, but they are predominately found between the Tropics of Cancer and Capricorn; in Africa, India, Southeast Asia, Australia, and Central and South America (see Figure 1). However, lateritic formations are not limited to intertropical regions, for during geological history favorable conditions for their development existed throughout various areas of the world. There are numerous red earths and red crusts covering large areas of the earth's surface which must be regarded as tropical, i.e., of noncontemporaneous origin (52). For example, latosols have been reported in southern USA, Germany, Belgium, and southern Russia. Even the Permian red sedimentary formations are suspected of being lateritic in nature (46).



From Goode's World Atlas

Figure 1. Geographical Distribution of Lateritic Soils (Dark Areas)

Evidence suggests that the Tertiary period presented favorable conditions for laterization. However, with the advance of Quaternary glaciers climatic conditions changed, and the glaciers obliterated and removed the lateritic influences (46). Since glaciation had no effect in the tropical regions, Tertiary laterites still exist in Indonesia, Lesser Antilles, Nigeria and Western Africa, India, and Australia (52).

Environmental Factors

The observed geographical distribution of laterite and lateritic soils associates them with tropical or subtropical environments. Such a climate is favorable for the alteration of rocks into lateritic soils by extreme weathering. Although the degree of laterization varies greatly, the environmental factors influential in the genesis of lateritic soils are: climate, vegetation, parent material, drainage, and topography (36, 46, 52, 69).

Climate

As far as climate is concerned, it is essential that it should be semihumid or humid tropical. Tropical climates provide; (a) alternating wet and dry seasons, and (b) warm temperatures, both of which favor laterization.

Alternating wet and dry seasons in which the wet season is warm, favor laterization far more than a continuously wet condition (34). The warm wet season is conducive to intense chemical weathering of the parent rock. The corresponding dry season permits desiccation of the iron hydrates and their crystallization into stable (immobile) forms.

Lateritic soils are found where temperatures are warm or are

believed to have been warm at the time of formation. The principal effect of temperature is to accelerate the rate of chemical weathering. However, it is possible that time, not temperature, is the controlling factor (57). If such is the case, temperate areas presently may be undergoing laterization, requiring only longer time intervals than in the tropics (69). Nevertheless, warm temperatures are more favorable to the formation of laterities.

Vegetation

Generally, indurated ferruginous crusts are more extensive under open grassy savannah rather than forest vegetation. In many cases the removal of tree cover results in the desiccation of the soil which leads to the formation of a hard indurated crust. With the development of incrustation, reforestation is restricted and the vegetation is replaced by herbaceous savannahs. This succession of events severely hampers agriculturists, since clearing a forested area for crops only creates lateritic crusts which are unsuitable for cultivation (2, 46, 52, 69). It appears that the role of tree cover is one of protection. The closeness of the tree crowns filter out the sun's rays protecting the soil from hardening, while the tree roots protect against erosion.

The evolution of organic matter under savannahs is far more rapid than under forests. The organic humic horizon which may develop is the source of humic acids which mobilize the hydrous iron oxides and lead to the formation of concretions. If drainage is sluggish, the mobilization and segregation of iron sometimes forms crusts at depth. Although concretions may be quite abundant under forests, hardpans are seldom encountered. It may be that forests are unable to mobilize

sufficiently the sesquioxides or that as crustation develops the trees are gradually replaced by savannahs. Although effects of vegetation on the pedogenesis of laterites are not yet completely understood, in general, lateritic soils correspond to forests, while laterite crusts correspond to savannahs (46).

Parent Material

With the possible exception of some particularly pure quartzites, lateritic soils are formed over all types of rocks. Any rock containing aluminosilicate minerals when subjected to the intense chemical weathering of the tropics can produce lateritic soils. The soils are more fully developed and widespread over basic rocks such as basalt, diabase, and gabbro, than over quartz rich acid rocks such as granite, gneiss and sericitic schists. The sedimentary rocks, shale, sandstone and limestone, have also been observed to underly lateritic soils (46, 69).

Generally, the thickness of the lateritic formation is related to the richness in ferromagnesian minerals of the underlying rock (2). However, iron enrichment introduced by ground water from adjacent formations may enhance lateritic development (49).

Drainage and Ground Water Movement

Laterites are not derived (in the strict sense of the word) from rocks, but rather are the result of a gradual removal of the greater portion of mineral matter from the original rock and the gradual deposition of lateritic constituents in their place (10, 61). This removal and deposition is caused by ground water movements; therefore,

good drainage is a necessary condition for laterization (52).

As rain water penetrates the soil, it acts as a disintegrating agent on the underlying rock, destroying it and forming new compounds. The water's chemical nature, pH, and ionic saturation, determines which minerals are leached or altered. For laterization, decomposition of the crystalline rock generally occurs below ground water level (10). Conditions here are anaerobic and iron can exist in the soluble divalent form. In this form, iron is susceptible to ground water movements. Fresh rain water is continually being added to the soil moisture producing saturation and downhill leaching. In time, this gradual movement removes the alkali and alkaline earths and much of the silica. Some small amounts of ferrous iron are also removed. However, generally the sesquioxides are concentrated because of their lower solubility and are not removed.

Above the ground water level, there exists a zone of oxidation. The upward movement of the sesquioxides may take place by capillarity or water table fluctuations. Exposure of the sesquioxides to the atmosphere causes oxidation into relatively insoluble hydroxides, e.g., ferrous to ferric, and thus they become immobilized. Continual enrichment with sesquioxides and their oxidation in the upper layers concentrates them into concretions and/or an indurated crust.

Common to the two zones is an intermediate zone clearly identified by its mottled gray color. It represents the extent of ground water fluctuations during wet and dry seasons. During the rainy season the water level rises to the top of this layer providing the overlying horizon with fresh sesquioxides. With the onset of the dry season the water table drops removing the source of sesquioxides and stimulating

desiccation. These continual fluctuations deplete this intermediate zone of the iron oxides and their characteristic red color.

In summary, the movement and chemical properties of the ground water are largely responsible for the type and profile of the laterite produced.

Topography

Laterite, referring to the ferruginous crust, has generally been associated with level or gently sloping surfaces; whereas, lateritic soils are seldom found in low, poorly drained areas. These are well drained soils, both internally and externally, and develop under conditions of high rainfall and forest cover. These factors are more common in hill topography with convex slopes which facilitate runoff. Accordingly, because of these topographical differences, a differentiation between "high level" and "low level" laterites is sometimes made (29, 46, 69).

High level laterites are residual soils formed by normal tropical weathering. They are formed on hillsides where wetting and drying conditions are optimal for oxidation, dehydration, and lateral leaching (29). Under certain conditions an aluminous indurated crust, primarily gibbsitic in nature, may form (46).

Low level laterites are formed by ferruginization of colluvial deposits. They are dependent upon; (a) the uplands for providing a source of iron hydroxides, and (b) ground water flow for mobilization and migration of the iron. As the ground water flows downslope, it transports iron and aluminum to the valley soils. Here, because of a change in gradient and soil texture, they become oxidized and fixed as

sesquioxides (29). The evolution of low level laterites is shown schematically in Figure 2. Low level laterites are predominately ferruginous rather than aluminous (46).

Genesis of Laterite

Origin of Lateritic Components

In tropical climates conditions are more suitable for microbial activities on decaying organic materials than in other areas of the world. Under these conditions, the immediate products, i.e., organic acids and humic salts, have little chance to react with the mineral fractions of the rocks. Hence, the leach waters are less acid, if not neutral to slightly alkaline (36). Moreover, the dissolution of the alkaline minerals in the rocks contributes to maintaining this near neutral condition, which develops a situation of alkaline hydrolysis. In such a chemical system, silica enters into solution more readily than do the sesquioxides. Recent studies (31, 57) have shown that even in moderately acid conditions silica is more soluble than the sesquioxides.

In the case of basic igneous rocks: basalts, gabbro, and syenites, laterization occurs more rapidly, as basic rocks are generally more permeable and possess less silica than acid igneous rocks. If the drainage is good, the rapid loss of silica transforms the feldspars into gibbsite (hydrated aluminum oxide). If drainage is only moderate, the aluminum hydroxides combine with amorphous silica to form kaolin (34, 52, 70). The ferromagnesian minerals: olivines, pyroxenes, and amphiboles, weather into various hydrated iron oxides grouped collectively as "limonite".

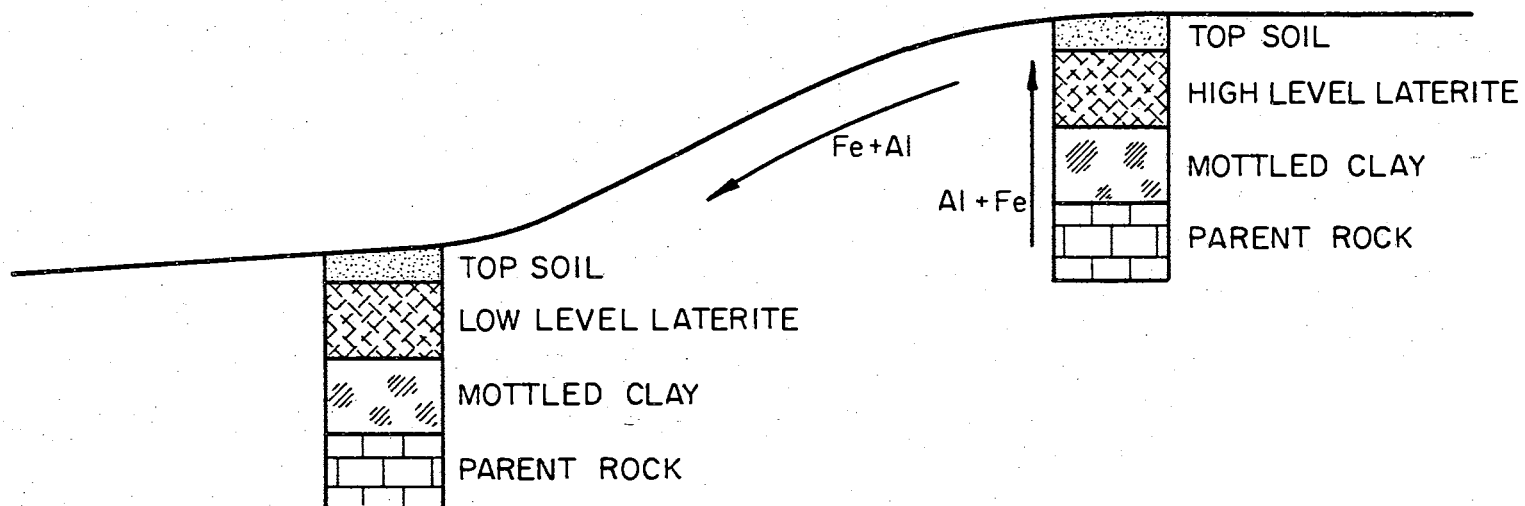


Figure 2. Schematic Representation of the Formation of High and Low Level Lateritic Soils

In the case of acid crystalline rocks: gneisses, granites, and mica schists, weathering occurs more gradually. Generally, more silica is present in the rocks than can be removed by the leach waters, so the feldspars weather directly to kaoline without passing through a gibbsitic stage. Again, as in the case of the basic rocks, the ferromagnesian minerals are the source of the iron hydroxides (46, 63).

In the case of both acid and basic rocks, kaoline may be formed. The mechanisms of kaolinite formation are complex and still not completely understood. However, one fact is certain: the structure of kaolin is quite unlike that of the silicate minerals such as feldspar, pyroxene and amphibole. Therefore, total destruction of the latter is essential and kaolin is probably formed from gels or amorphous compounds. It is quite possible that allophane, an amorphous mixture of hydrated alumina and silica, is a precursor of kaolin (2, 31). Characteristically, kaolin is formed under conditions of acid groundwaters and thorough leaching (63). The question of whether kaolin is an end product or merely an intermediate product of weathering is a controversial one.

Several investigations (30, 67, 70) believe kaolin weathers through loss of silica to hydrated aluminum oxide, gibbsite. As weathering continues, the supply of bases is depleted from the soil and the alkalinity of the ground waters is reduced. If the formation of kaolin has not greatly altered the internal drainage system and good drainage conditions persist, the acid leach waters attack the clay mineral. Acid destruction of the clay is a process of "de-aluminization," whereby the aluminum is mobilized into exchangeable positions. The resulting alumina saturated clay is not stable and the Al^{+3} ions

are displaced into solution. These aluminum ions then hydrolize to gibbsite, $\text{Al}(\text{OH})_3$ and generate H^+ ions. The generation of these H^+ ions maintains or increases the acidity of the ground water which contributes to the further destruction of the kaolin. If the conditions remain moist and the silica is rapidly removed, the destruction process continues until all the clay is decomposed, resulting in a bauxitic laterite. However, if silica removal is impeded, kaolin is reformed and the decomposition ceases (14, 65). However, this process seems to be restricted to certain environmental conditions (46, 57, 70).

The final components of laterites are: kaolin; hydrous aluminum oxides, principally gibbsite; hydrous iron oxides, particularly goethite and hematite; and a few resistant residual quartz grains.

Distribution of the Constituents in the

Lateritic Profile

In general, an examination of a "typical" lateritic profile appears in upward sequence to be (34):

- (1) Parent material, usually igneous bedrock.
- (2) A decomposed zone of bedrock having some remaining petrographic structure of the parent rock, and sometimes containing much free silica as in the case of an acid rock.
- (3) Lithomarge, a gray mottled zone, becoming brownish yellow at the bottom, perhaps containing some residual quartz. This layer is composed of kaolin with some small amounts of micaceous clay minerals.
- (4) Laterite proper, a zone which is rich in free sesquioxides with small iron nodules in the upper layers.

- (5) Cuirasse, a hard indurated crust which is very rich in iron.

The formation of each zone is interrelated by the process of mobilizing and segregating the lateritic components throughout the profile. In the case of "high level" laterites where no iron or aluminum enrichment occurs from outside sources, the sesquioxides must originate from the parent material. Their migration to the upper layers could only occur through the mechanism of aqueous solution, as the development of this type of profile apparently cannot be adequately explained by the mechanism of erosion (63). Gray mottled areas in soil profiles are generally associated with areas of continual wetness, and suggest a possible location of the water table. Many of the numerous hypotheses on laterite formation (2, 10, 46, 52, 61, 69) are based on the assumption of a fluctuating water table, at the top of which the sesquioxides are precipitated by oxidation. The alternating wet and dry seasons of the tropics provide the basis for this assumption.

Although the mechanism of migration for the iron and aluminum from parent material to the upper layers is still not clearly understood, there exists a variety of conditions by which the sesquioxides migrate and accumulate. The upward migration of the iron must occur in the ferrous state, because at the pH of tropical soils the ferric ion is quite insoluble. Later, above the water table, the ferrous ion will become oxidized to its ferric form and thus become immobilized. The iron and aluminum oxides produced by weathering are either amorphous or microcrystalline. Since they possess enormous specific surfaces in these forms, their solubility is much greater than the solubility of crystalline minerals. These sesquioxides can also move as sols or

gels (69). Fripiat and Gastuche (22) have shown that because of its electropositive charge, iron vigorously attaches itself to kaolin and can migrate with the clay. Under certain conditions, iron can migrate in the form of carbonates. The mobility of iron is greater than that of alumina, and there is some suggestion that alumina originates in the upper horizons only by weathering of kaolin (46).

The fact that the gray mottled zone is poor in iron, as noted by the lack of characteristic red color, substantiates the idea that this is a zone of separation and redistribution of the constituents. It is here that the material dissolved from the overlying layers would be detained with increased opportunity for adsorption or precipitation, and that material carried upward by a rising water table would meet conditions favorable for its deposition (2, 61).

Continual accumulation of iron oxides in the upper layers results in an iron coating of the pore walls and/or voids until a lattice-like network is formed. Occasionally the hydrous iron oxides orient themselves around certain nuclei where they dehydrate and become fixed. Continual deposition around these nuclei lead to the formation of concretions (34). The continual growth of these concretions along with the deposition of iron in the connecting pores forms a slag-like crust.

However, the mere presence of an accumulation of iron in the upper layers does not insure the development of an indurated crust. Favorable conditions must also exist for sesquioxide enrichment and alternate wetting and drying. Hardening is nearly always accompanied by a loss of kaolin. This observation suggests that the destruction of kaolin results in an increase in alumina and additional iron enrichment from the iron surface films originally on the kaolin (67). Crystallization

of these dilute amorphous iron and aluminum colloids into concretions or networks involves dehydration to goethite and hematite, or gibbsite. This process of dehydration plus concentration plus crystallization has been termed "siderization" (sideros, a Greek word meaning iron) (44). Obviously the wet and dry seasons provide conditions for weathering and destruction of the kaolin as well as a period of dehydration (2, 69).

The development of the crust formation is shown schematically in Figure 3 (46).

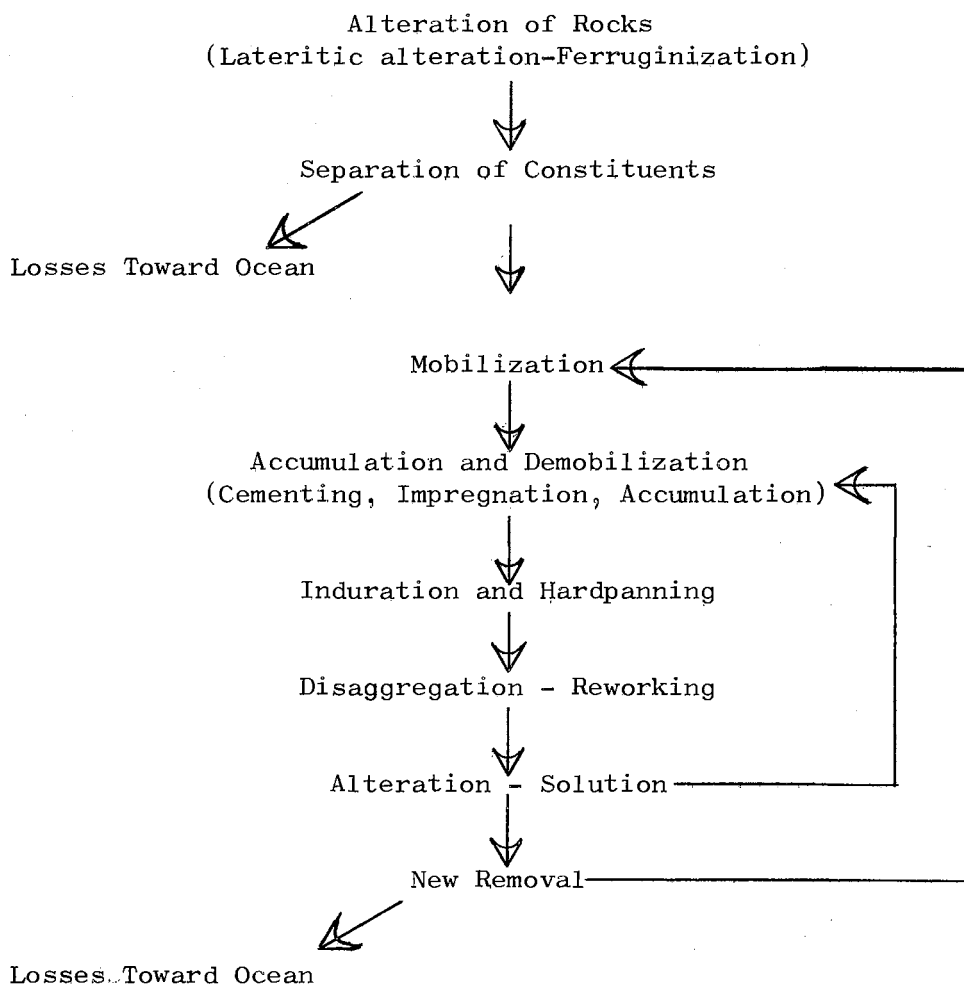


Figure 3. Schematic Summarization of the Development of Laterite

If sesquioxide liberation exceeds drainage losses to the oceans, then conditions for induration are increased. If mobilization and leaching effects predominate, there is no crust formation and old crusts disappear. If the gradient of the sesquioxide rich leach waters toward the ocean is impeded, "low level" laterites develop.

Microstructure of Laterites

According to Kubiena (39), lateritic soils are distinguishable from other tropical soils by their flocculant (erdig) structure. The fine elements have become organized into a porous spongy structure generally characterized by a bright red color. This flocculant structure is postulated to have been derived through flocculation of the colloidal constituents by dehydration.

Petrographic studies (2, 29, 34, 40, 62, 67, 69) have contributed additional evidence of the mechanisms entailing movement, segregation, and reorganization of the lateritic constituents. Some laterites conserve the original rock structure, including rough outlines of the original rock minerals preserved in a sesquioxidic matrix. The feldspar, pyroxene, and amphibole phenocrysts have been altered in situ as evidenced by pseudomorphs of gibbsite, kaolinite, and goethite. However, side by side with these in situ alterations, local movement of material or enrichment from outside sources is evident from the reorganization and segregation of the constituents.

The porous granular structure of in situ laterites is the result of a lattice-like network of oriented films of kaolin impregnated by iron. These clay films together with goethite (crystalline iron hydroxide) line and coat the pores and cavities in the soil. Such

behavior can only result from movement and segregation of the components. Commonly, minute goethite rich spherical aggregates of clayey material have assembled into more or less dense clusters resembling popcorn balls. The strength of this lattice-like structure is derived from the thin ferruginous films which are found inside the microjoints of the elementary clay particles (plasma) and as coatings over them (9). The friability of this type of structure is quite high.

The iron impregnation and coatings of the clay particles are formed by the absorption and immobilization of iron by kaolin (35). This absorption is enhanced by the extra amount of negative charge exhibited by clays in neutral pH environments. This extra charge is caused by dissociation of the SiOH groups to Si^- and H^+ , which increases the cation exchange capacity of the clay. The effect is more pronounced in 1:1 clays, than 2:1 clays. Thin sections commonly reveal that this immobilized iron may be set free upon destruction of the kaolin (2, 67).

The most frequently occurring percentage of iron oxide (Fe_2O_3) in the clay fraction of different lateritic soils is 10% to 12%, which corresponds to the oxide saturation of the clay surface. This saturation appears to be a characteristic of mature laterites as higher percentages lead to the formation of concretions (22). Microscopically these concretions are more or less rounded with concentric rhythmic bands of pure gibbsite, goethite, or hematite formed around some nucleus (quartz grains, rock debris). Some concretions, rather than consisting of crystalline materials, are formed of amorphous mixtures of clayey material stained with iron oxide (2). In time, these concretions may increase in size until they coalesce into a sesquioxide rich crust (34).

Conclusions derived by petrographic analysis can be used to explain certain engineering properties. The well developed internal drainage system can be attributed to the porous structure. The bearing capacity is a function of the lattice structure and strength of the iron films in the microjoints and pores. The observed water inertness of lateritic clays quite possibly is due to the iron coatings and staining which "neutralize" and protect the indigenous clay particles.

CHAPTER III

MINERALOGY OF LATERITES

Introduction

The reported mineralogical studies of laterite and lateritic soils (2, 34, 46, 56, 69) generally have been carried out in conjunction with chemical analyses. Quite often, chemical analyses alone are insufficient to reveal the true composition, nature and origin of laterites (10). Laterites having similar physical properties, may differ greatly in chemical composition and, conversely, laterites with similar chemical compositions may possess varying physical properties. For these reasons, mineralogical determinations are necessary and supplemental to chemical analyses. As discussed in Chapter II, the principal constituents of lateritic soils are the sesquioxides of iron and alumina, clays, and silica. The following forms of these constituents have been identified in lateritic soils.

Free alumina may be found in various forms, most commonly as gibbsite, $\gamma\text{Al}(\text{OH})_3$. Boehmite, $\gamma\text{AlO}(\text{OH})$, a monohydrate of aluminum is also occasionally reported. Amorphous forms of hydrated alumina, which are cryptocrystalline aggregates or possibly forms of allophane are known to exist in lateritic soils (46,69).

Iron likewise exists in varied forms, of which goethite, $\alpha\text{Fe}_2\text{O}(\text{OH})$, and hematite, Fe_2O_3 , are the most usual. In addition to these minerals which are derived during the evolution of laterites, residual iron

oxides such as magnetite, $\gamma\text{Fe}_3\text{O}_4$, may be encountered. As in the case of alumina, various amorphous oxides of iron exist as cryptocrystalline aggregates or unidentifiable coatings on other minerals. Commonly these amorphous oxides are believed to be forms of goethite containing variable amounts of water which account for the reddish yellow products classed as "limonite," $\text{Fe}_2\text{O}_3 \cdot n \text{H}_2\text{O}$.

The clay minerals encountered in lateritic soils are principally 1:1 minerals, of which kaolinite is by far the most common. Halloysites occur less frequently, but are known to exist in laterites. Although their presence in laterites is disputed, small amounts of illite and chlorite have been identified in lateritic soils where they occur as a stage in alteration near the parent rock (46, 56, 69). Montmorillonite and other 2:1 clay minerals generally are not associated with lateritic soils, but some traces of vermiculite have been reported.

Free silica often occurs as residual resistant quartz grains inherited from the parent rock. Various forms of chalcedony, opaline and secondary quartz, presumably precipitated from silica leached during lateritic alteration, have been observed in laterites. The presence of a colloidal and amorphous silica, allophane, has been suspected in laterites (46).

Investigative Procedures

Methods and Equipment

The methods utilized for mineralogical identification consisted of x-ray diffraction and differential thermal analyses. X-ray diffraction techniques utilize the principle of Bragg's Law, in which the distance

between crystal planes can be calculated if the angle of diffraction and the wave length of the x-ray beam are known. This distance between like planes is diagnostic for the determination of types of minerals. However, the use of x-ray diffraction is restricted to crystalline materials and unable to detect or identify the presence of amorphous compounds. Because of this limitation, differential thermal analyses were conducted in addition to x-ray studies. Differential thermal analysis, DTA, involves the continuous monitoring of the temperature difference between a sample (soil) and a thermally inert reference material (Al_2O_3) as both are heated to elevated temperatures at a controlled and known rate. A temperature differential will be measured between the sample and reference only when the sample undergoes a physical or chemical change. These thermal differences are plotted as curves depicting endothermic and exothermic reactions occurring within the sample and are indicative of the mineral composition of the sample.

X-ray diffraction patterns were obtained using a General Electric XRD 6 and SPG 2 spectrogoniometer with Ni filtered Cu K α radiation generated at 40 KV and 20ma. The slits used were: 1 $^\circ$ MR beam, HR soller, and 0.2 $^\circ$ detector slit. The time constant generally was 2.0 with a CPS range of 1000. The amplitude gains were set at 16 for coarse and 88 for fine. The takeoff angle was 3 or 4 degrees of 2θ . The goniometer scan rate corresponded to 1 degree of 2θ per minute with the chart rate set at $\frac{1}{2}$ inch per 1 degree of 2θ .

The differential thermal analysis curves were obtained using a Fisher Differential Thermolyzer Model 260 connected to a 1 mv strip chart recorder. The operating procedures involved heating the sample from ambient temperature to 1200 $^\circ\text{C}$ at a rate of 10 $^\circ\text{C}$ per minute.

A platinel thermocouple and a recorder speed of four inches per hour were used. The temperature associated with each endothermic or exothermic peak was obtained from a calibration curve prepared using a platinel calibration thermocouple in an ice bath.

Sample Preparation

Remolding of the soil, to simulate excess mechanical manipulation by heavy construction equipment, was accomplished by mixing the soil at moisture contents approximately equal to the liquid limit for two hours in a Hobart mixer. After being mixed, the remolded slurry was air dried and ground by a mechanical grinder to pass a U.S. Standard No. 10 sieve. Unremolded material was obtained by hand sieving of the soil.

The soil fractions utilized for determining the mineralogy of this lateritic soil were prepared in accordance with the following methods.

X-ray Diffraction: The soil fractions analyzed by this method were prepared utilizing the procedures of Jackson (35). The procedure involved separation, iron removal, ion saturation, glycerol solvation, and heating.

The preliminary preparation of the soil involved fractionation of the samples into silt (50-2 μ), coarse clay (2-0.2 μ), and fine clay (<0.2 μ) particle sizes in accordance with the procedures outlined in Appendix A. Due to the amount of mixing and centrifuging required during the soil fractionation procedures, the silt, coarse clay, and fine clay fractions are considered to be in the remolded condition. Therefore, no differentiation as to remolded or unremolded soil conditions was possible for these fractions. Fractionation of mineral species into simple mixtures provides for as few sets of diffraction

peaks as possible in each diffraction pattern, thus facilitating qualitative analysis. For example, montmorillonite minerals occur abundantly in the fine clay fraction, yet often are overlooked in the diffraction patterns of the whole soil. Because in latosols the crystals of secondary minerals often grow sufficiently large in size, the silt size fraction was analyzed as well as the clay fraction.

Subsamples of the silt, coarse and fine clay fractions were subjected to the sodium dithionite-citrate-bicarbonate treatment (51) for removal of the free iron oxides. This removal procedure is outlined in Appendix A. The removal of amorphous coatings and crystals of iron oxides is quite useful in mineral identification as these oxides tend to mask the characteristics of other minerals. X-ray diffraction procedures are much more sensitive to the "cleaned" clay minerals. The removal of these amorphous materials also facilitates concentration of the clays and greatly enhances parallel orientation, which subsequently increases the intensity of the diffraction patterns. Comparisons between samples with iron oxides and those in which the iron had been removed also assist in identification of these iron compounds.

X-ray diffraction analyses should include procedures for detecting expanding 2:1 clay minerals. Although 2:1 clays are not anticipated in this soil, calcium (Ca) saturated and glycerol solvated samples were prepared for diffraction. Solvation with glycerol increases the basal spacing of Ca saturated montmorillonitic clays to 18A (18 Angstroms), permitting differentiation from 14A chlorite or vermiculite whose basal spacings do not increase with solvation. In addition to Ca saturation and glycerol solvation, potassium (K) saturated and heat treated samples were also prepared for diffraction. K saturation

enhances the study of 10A clays better than Ca saturation. Heating of the K saturated fractions to various elevated temperatures causes decreases in the basal spacings or destruction of several minerals. For example, at 300°C some montmorillonites close from 14A to 10A, while gibbsite and goethite have been destroyed. At 400°C halloysite spacings shift from 7.6A to 6.2A, and at 550°C, halloysite and kaolinite are destroyed (35).

The respective soil fractions were suspended in various solutions, i.e., 1N CaCl_2 , 1N KCl , ten percent glycerol in ethanol, to achieve the appropriate ion saturation. The final phase of the pretreatment procedure was the mounting of the saturated specimen. A parallel orientation mount was prepared by pipetting the suspended soil fraction onto a glass microscope slide. The soil suspension on the slide was permitted to air dry for 24 hours prior to diffraction. The samples which were to be subjected to heat treatments of 300°C, 425°C and 550°C were mounted on pyrex slides to prevent undesirable warping of the slide during the four hour heating in a muffle furnace.

The flow chart in Figure 4 schematically summarizes the sample preparation procedure for x-ray diffraction.

Differential Thermal Analysis. The soil material analyzed consisted of unremolded, remolded and iron free soil samples which passed the U.S. Standard No. 80 sieve. This soil was then pretreated in accordance with the recommendations of Mackenzie (54). The samples were allowed to equilibrate for four days in a sealed chamber over a saturated solution of magnesium nitrate, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$. This pretreatment assures exposure of the samples to a standard of 56 percent relative humidity. The low temperature peak system of clay thermograms

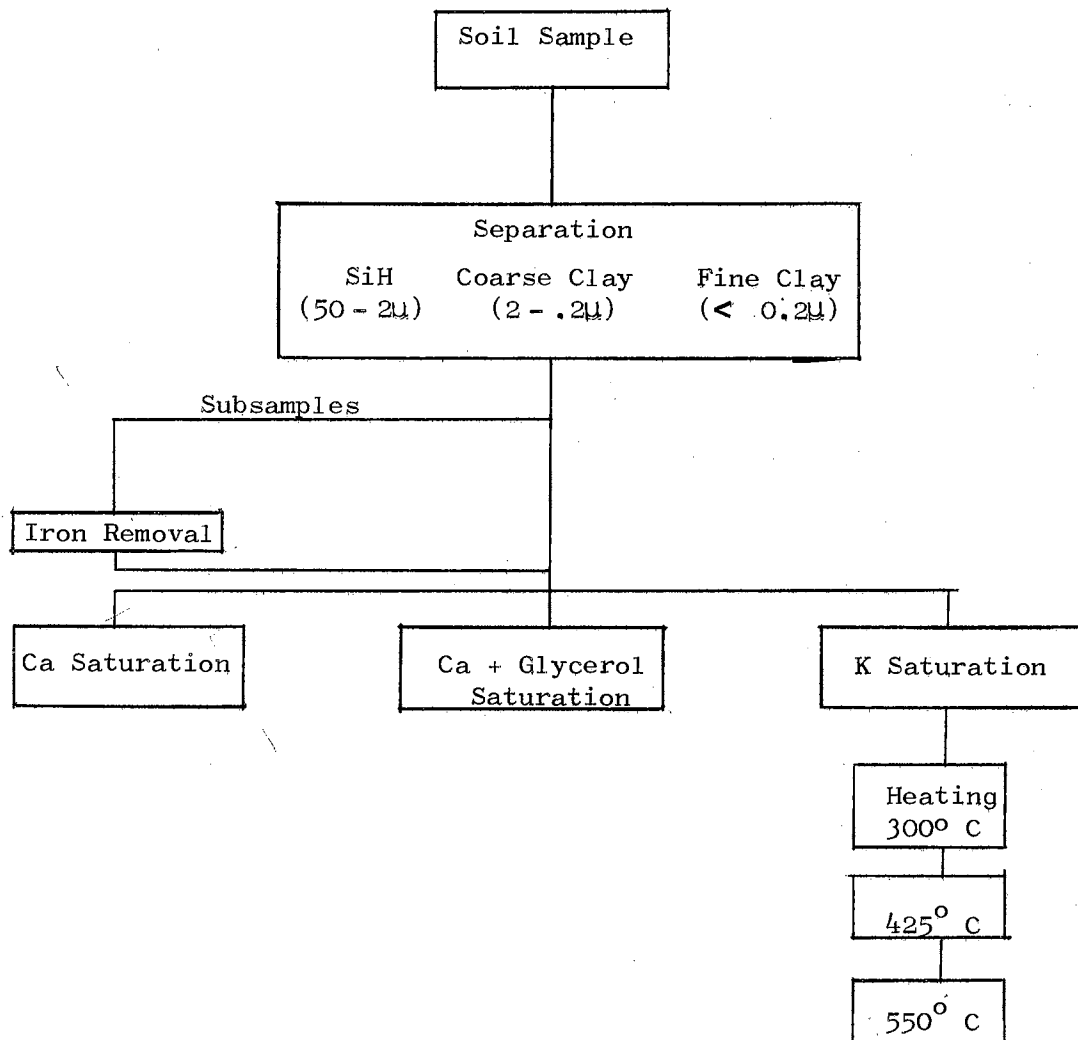


Figure 4. Flow Diagram of Sample Preparation Procedures for X-ray Diffraction Analysis

is dependent upon the clay moisture conditions immediately prior to testing. Since these moisture conditions are very sensitive to environmental variations, it is necessary to adopt a standard humidity in order to compare these peaks between various samples. All crucibles were carefully balanced utilizing 0.1 to 0.3 grams of soil and then lightly tamped prior to thermocouple placement.

Experimental Results and Discussion

X-Ray Diffraction

The x-ray diffraction patterns of the silt fraction, 50-20 μ , are shown in Figures 6 through 11. These patterns show that the identifiable crystalline minerals of the silt fraction are remnant quartz grains, a 1:1 clay mineral, and magnetite. The distinct peaks (4.27A and 3.35A) and lesser peaks (2.28A, 1.18A, and 1.54A) shown in Figure 6 identify the presence of well crystalline quartz. The broad peaks (7.30A, 4.45A and 3.57A) in Figure 6 are indicative of a mixture of poorly crystalline 1:1 kaolin minerals, predominantly kaolinite with some trace of halloysite. The dark black particles so frequently observed during laboratory testing of this soil were identified as magnetite from the 2.41A and 1.62 A peaks in Figure 6. The adherence of these particles to a magnet likewise confirmed this identification. A trace amount of secondary quartz as cristobalite (4.04A and 2.47A in Figure 6) may be present, but it apparently is in poorly crystalline form.

The x-ray diffractograms of the coarse clay fraction, 2-0.20 μ , are presented in Figures 12 through 16. The patterns of this component of the soil reveal the primary constituent is a mixture of pseudo-kaolin

minerals composed of kaolinite and trace amounts of halloysite. The absence of the previously observed kaolin peak (4.45A) in Figure 5 of the silt fraction establishes the presence of amorphous materials. The presence of iron coatings on the untreated soil particles is strongly indicated in Figures 12 and 13 by the occurrence of a previously absent quartz peak (3.35A) after removal of the iron oxides. The increased sharpness of the kaolin peaks (7.24A and 3.47A) in Figures 12 and 13 upon treatment of the soil for iron removal, also substantiates the presence of iron coatings.

The x-ray diffraction patterns for the fine clay fraction, <0.2 μ , are presented in Figures 17 through 21. The only identifiable mineral in these figures is a form of kaolin, predominately kaolinite mixed with some halloysite (7.31A and 3.57A). The presence of an amorphous material (allophane) is indicated by the absence of the 020 kaolin peak (4.45A). These clay minerals are coated by iron as indicated by the increased sharpness in the peaks of Figure 18 after the iron oxides have been removed from the soil.

These mineralogical data obtained from x-ray diffraction studies show that the soil has been extremely weathered, with only some resistant quartz grains, a poorly crystalline 1:1 pseudo-kaolin clay mineral, and magnetite remaining as the identifiable minerals. The presence of some secondary quartz as cristobalite is also suspected. Wissa (84), in his study of a Panamanian lateritic soil, identified quartz and kaolinite as the only identifiable mineralogical constituents. The kaolin was a mixture of kaolinite and dehydrated halloysite. Brown (8), in his studies, stated that Kinter identified kaolinite as the principal clay mineral found in Panamanian latosols. Kinter coined the

name "kaolinite intermediate" to identify a mixed layered mineral having basal spacings of 7 to 10A, but definitely of the kaolin group. Minor amounts of halloysite and cristobalite were also contained in these soils (8). The iron coatings inferred from comparisons of these patterns must exist as microcrystals or in amorphous forms which are undetectable by x-ray. No peaks were observed for goethite (4.15A, 2.69A and 2.44A) or hematite (2.69A, 2.51A and 1.69A) which are the most common iron oxide coatings.

Quartz. The most predominant peaks (4.27A and 3.35A) in Figures 5 and 6 of the silt fraction identify quartz as a primary constituent of this fraction. The repetition and sharpness of the peaks (4.27A, 3.35A, 2.28A, 1.81A and 1.54A) are indicative of a well crystalline mineral. On this basis, the quartz in the silt fraction probably exists as remnant grains which resisted weathering of the parent rock. Reprecipitated silica is also probably present in the silt fraction as suggested by the weak cristobalite peak (2.47A) in Figure 6. The presence of quartz in the coarse clay lessens and none is found in the fine clay fraction.

The existence of iron coatings on the quartz grains is substantiated by the appearance of Figures 12 and 13 of the quartz peak (3.35A) in samples which have been treated for iron removal; whereas, this peak often is masked in samples which have not been treated. This effect is quite evident in Figure 15 of the K saturated samples which have been heated to 425°C. The low intensity of this peak (3.35A) in the clay fractions as compared with that of the silt fraction indicates a reduction in the quartz crystallinity with decreasing grain size (see Figure 5).

Clay Minerals. The broadness of the clay mineral peaks (7.31A and 3.57A) in Figures 12, 13, 17 and 18 suggests that these minerals are not well crystallized. Because the 7A peak which is indicative of 1:1 clay minerals occurs at 7.31A instead of the usual 7.15A associated with kaolinite, the clay minerals are interpreted to be a mixture composed predominantly of kaolinite with some halloysite. The enhancement of this 7.31A peak with heating of the K saturated samples to 300°C and 425°C (see Figures 14, 15, 19 and 20) confirms the presence of partially hydrated halloysite which dehydrates at these temperatures (35).

The disappearance of the broad 020 kaolin peak, 4.45A, which was observed in the silt fraction is diagnostic of amorphous materials (see Figure 5). The presence of a high intensity diffraction peak at 4.45A establishes the dominance of crystalline clay minerals. However, absence of the 4.45A peak indicates the presence of amorphous materials (35). Counts of one hundred seconds in the 4.45A area failed to distinguish any noticeable peak in the coarse or fine clay fractions with and without the iron removal treatment.

The increased intensity and sharpness of the clay peaks (7.31A and 3.57A) in the patterns of those samples which have been treated for iron removal compared with those which have not been subjected to this treatment, readily confirms the existence of iron coatings on these clay minerals (see Figures 12, 13, and 18).

Generally 2:1 clay minerals (14A and 10A) are abundant in the fine clay fraction of soils containing such minerals and are not destroyed by heating to 550°C as are 1:1 clay minerals. Consequently, the lack of peaks in the 550°C heated samples of the fine clay as shown in

Figure 21 indicates the absence of any 2:1 minerals in the soil. The absence of any 18A or 14A peaks in the Ca saturated - glycerol solvated samples is likewise consistent with this finding (see Figure 5).

Differential Thermal Analysis (DTA)

The DTA thermograms of the unremolded, remolded, and iron free samples are presented in Figure 22. These patterns indicate the presence of an amorphous material which is undetectable by x-ray diffraction. The large endothermic peak at 100°C is an important feature of thermograms for halloysite and allophane, as kaolinite only exhibits a small peak (35, 45). Amorphous allophane which exhibits a substantial endothermic peak at 110°C often occurs as a co-precipitate of iron oxides in latosols (35). The marked decrease of this low temperature endotherm in the iron free sample indicates the presence of an allophanic material. Had this endotherm been attributed solely to halloysite, it would still exist at an appreciable magnitude after the iron removal treatment.

A large endotherm at 500°C to 600°C is characteristic of 1:1 kaolin minerals although kaolinite generally exhibits an endothermic peak at 600°C. Any mixture of halloysite or kaolin intermediary minerals lowers this peak to approximately 550°C to 570°C (45, 72). Thus, the main endotherm at 560°C indicates that the clay minerals are essentially a mixture of kaolinite and halloysite.

The small exotherm at 880°C is suggestive of a mixture of poorly crystalline 1:1 kaolin minerals. The kaolinite exotherm generally occurs in the neighborhood of 950°C to 980°C (35, 45). However, the presence of halloysite (exotherm, 890°C to 950°C) or allophane (830°C

or 950°C) lowers this kaolinite exothermic peak (35, 45, 72).

These data provide additional information concerning the characteristics of the clay minerals. Allophane, an amorphous hydrous aluminosilicate, occurs in rather highly weathered latosols and is believed to be a weathering relic (35) or precursor (2, 31) of halloysite and kaolinite (35). A previous study on Panamian latosols (8) detected the presence of halloysite and allophane in minor amounts. These DTA findings are consistent with those determined by x-ray diffraction, in that the clay fraction is found to consist of pseudo-kaolin minerals, probably kaolinite, with trace amounts of halloysite and allophane.

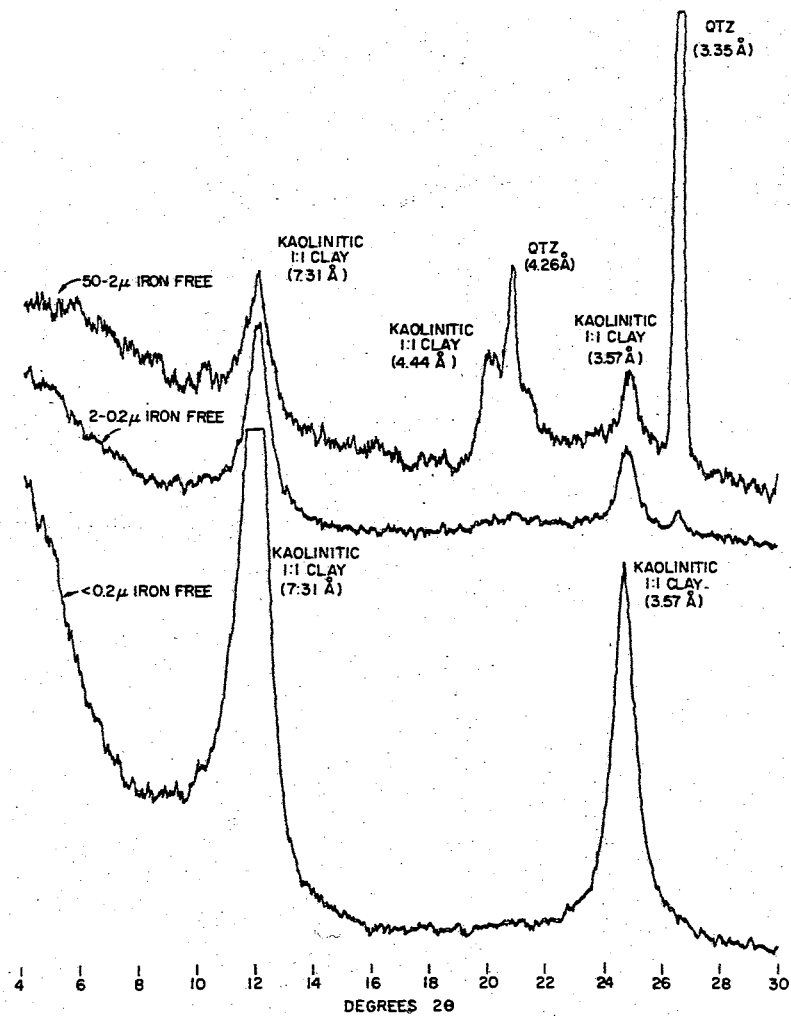


Figure 5. X-Ray Diffraction Patterns of Silt, Coarse and Fine Clay (Ca Saturated-Glycerol Solvated)

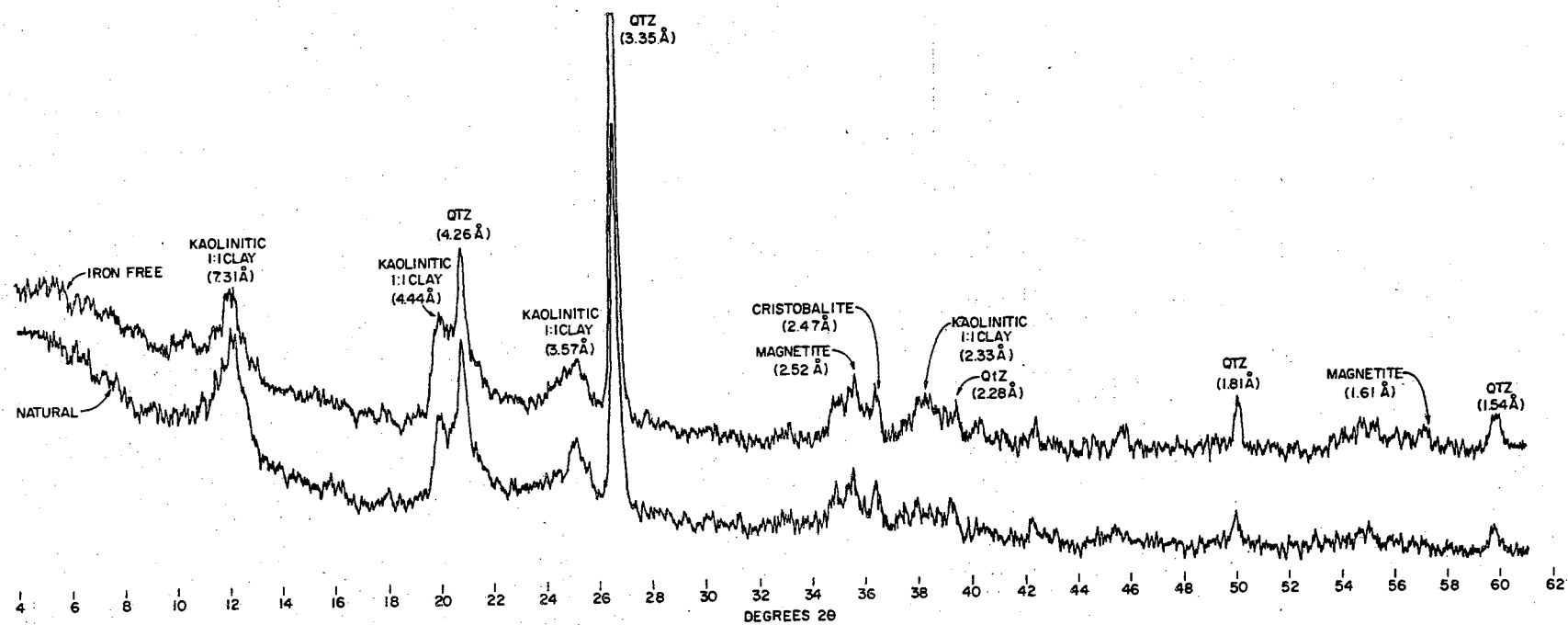


Figure 6. X-Ray Diffraction Patterns of the Silt Fraction (Ca Saturated)

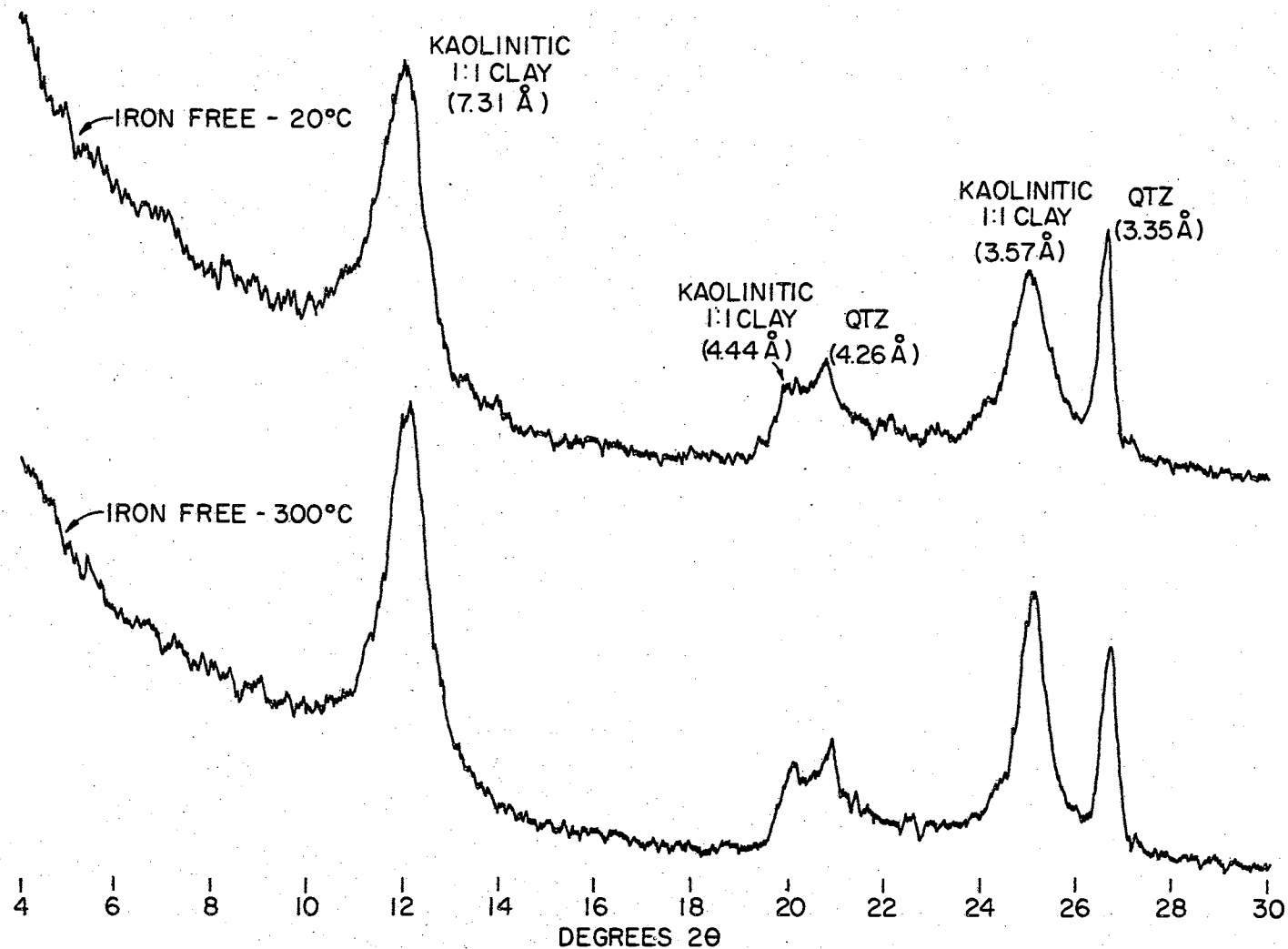


Figure 7. X-Ray Diffraction Patterns of the Heat Treated Silt Fraction (K Saturated, Iron Free, 20°C and 300°C)

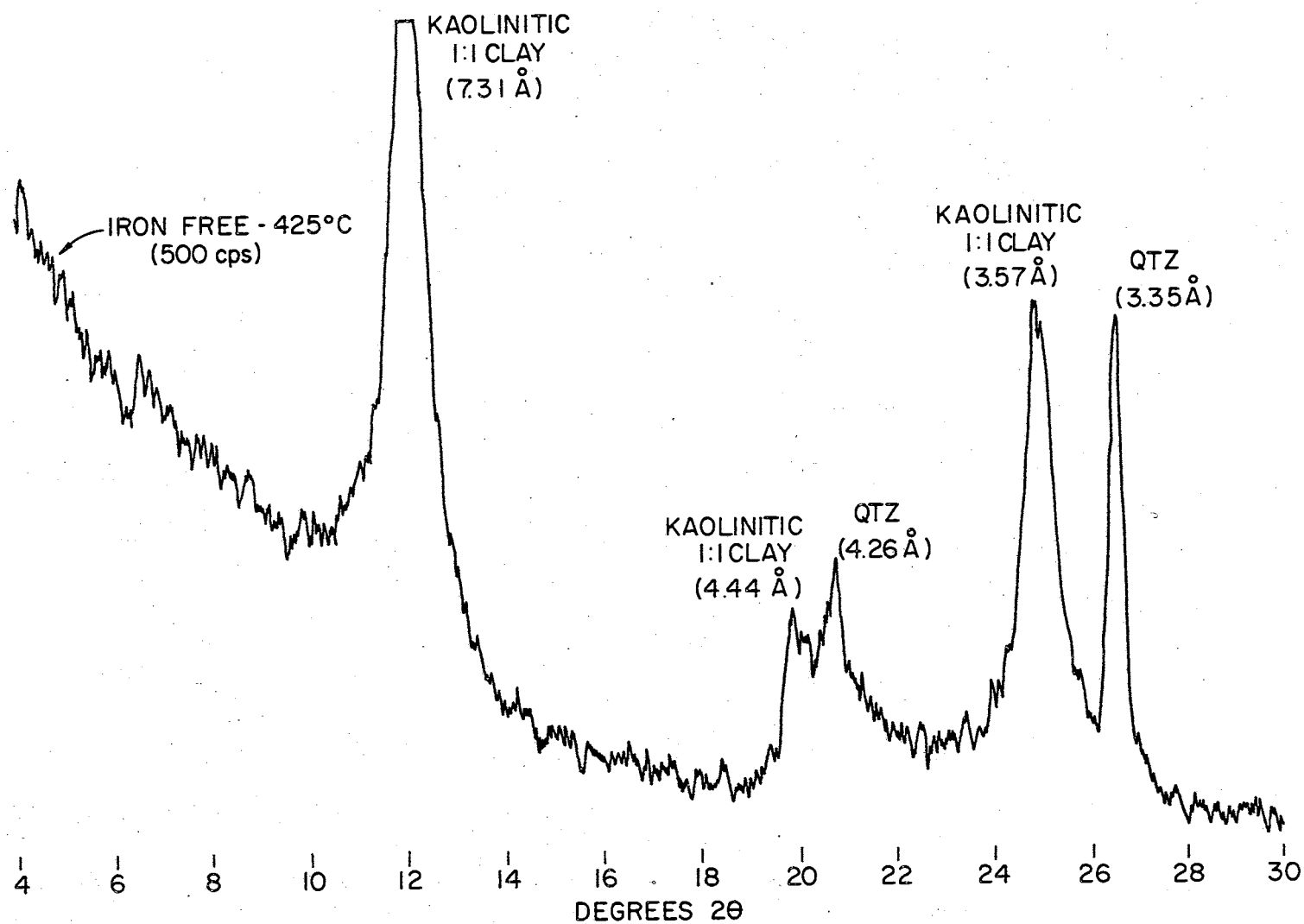


Figure 8. X-Ray Diffraction Patterns of the Heat Treated Silt Fraction (K Saturated, Iron Free, 425°C)

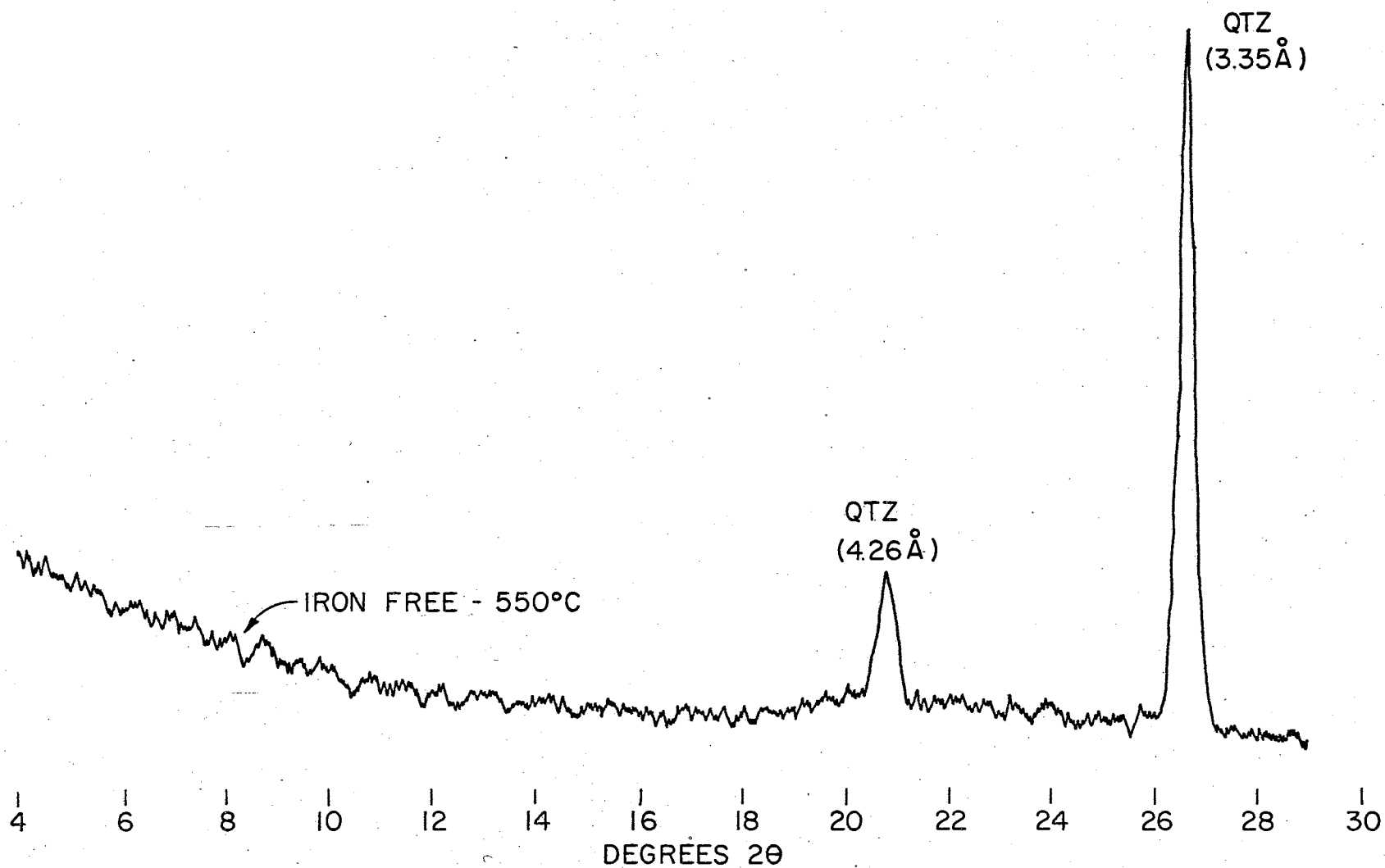


Figure 9. X-Ray Diffraction Patterns of the Heat Treated Silt Fraction (K Saturated, Iron Free, 550°C)

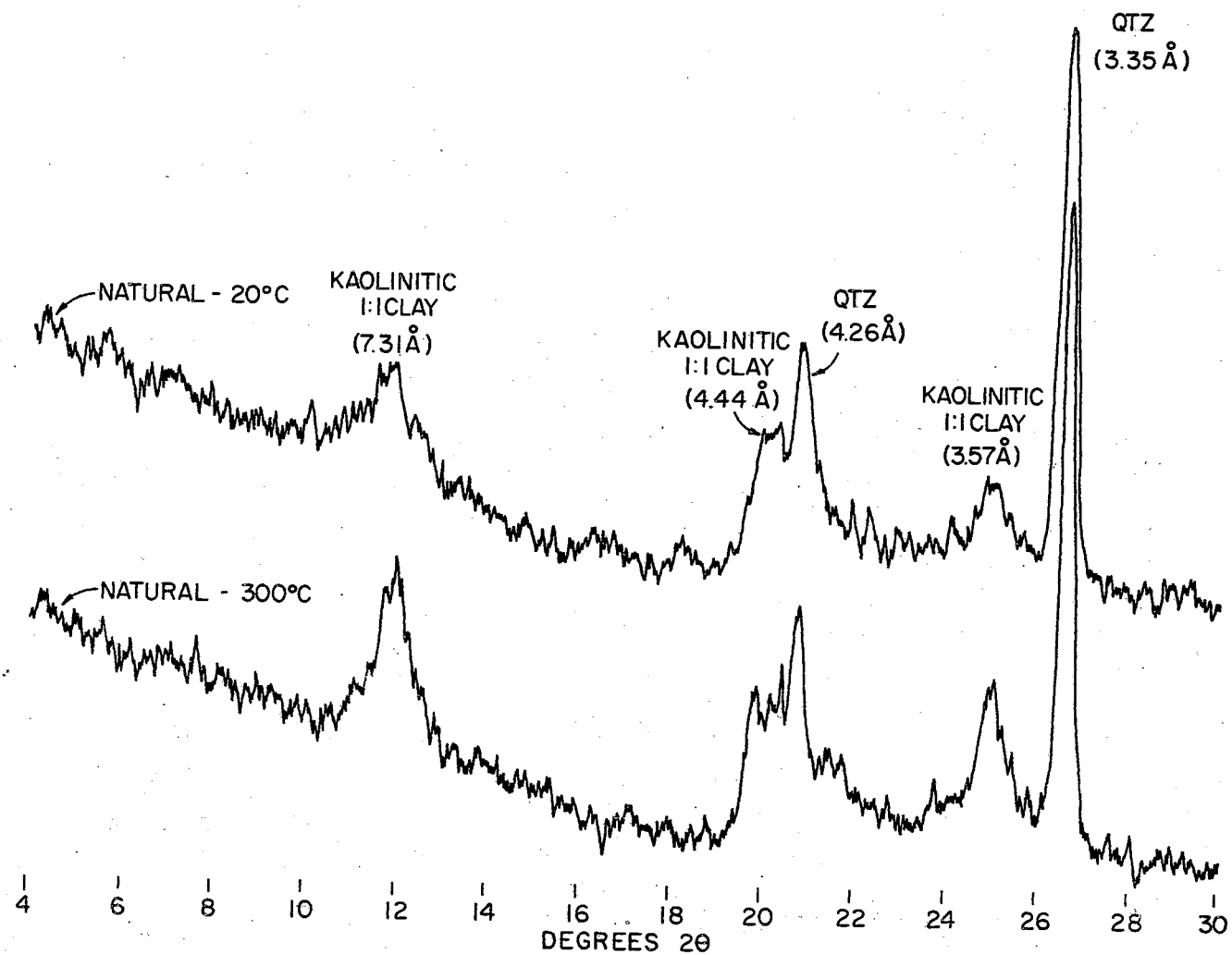


Figure 10. X-Ray Diffraction Patterns of the Heat Treated Silt Fraction (K Saturated, 20°C and 300°C)

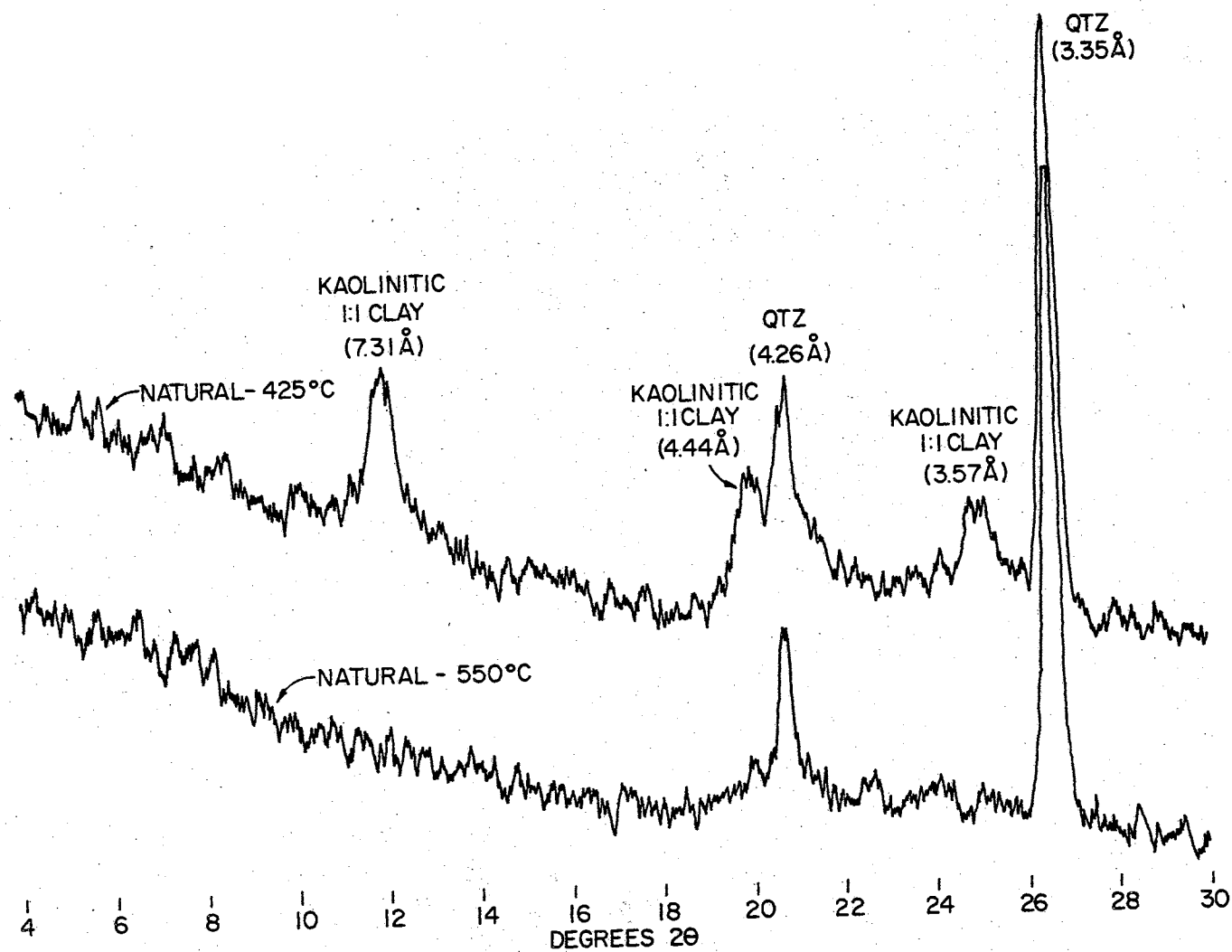


Figure 11. X-Ray Diffraction Patterns of the Heat Treated Silt Fraction (K Saturated, 425°C and 550°C)

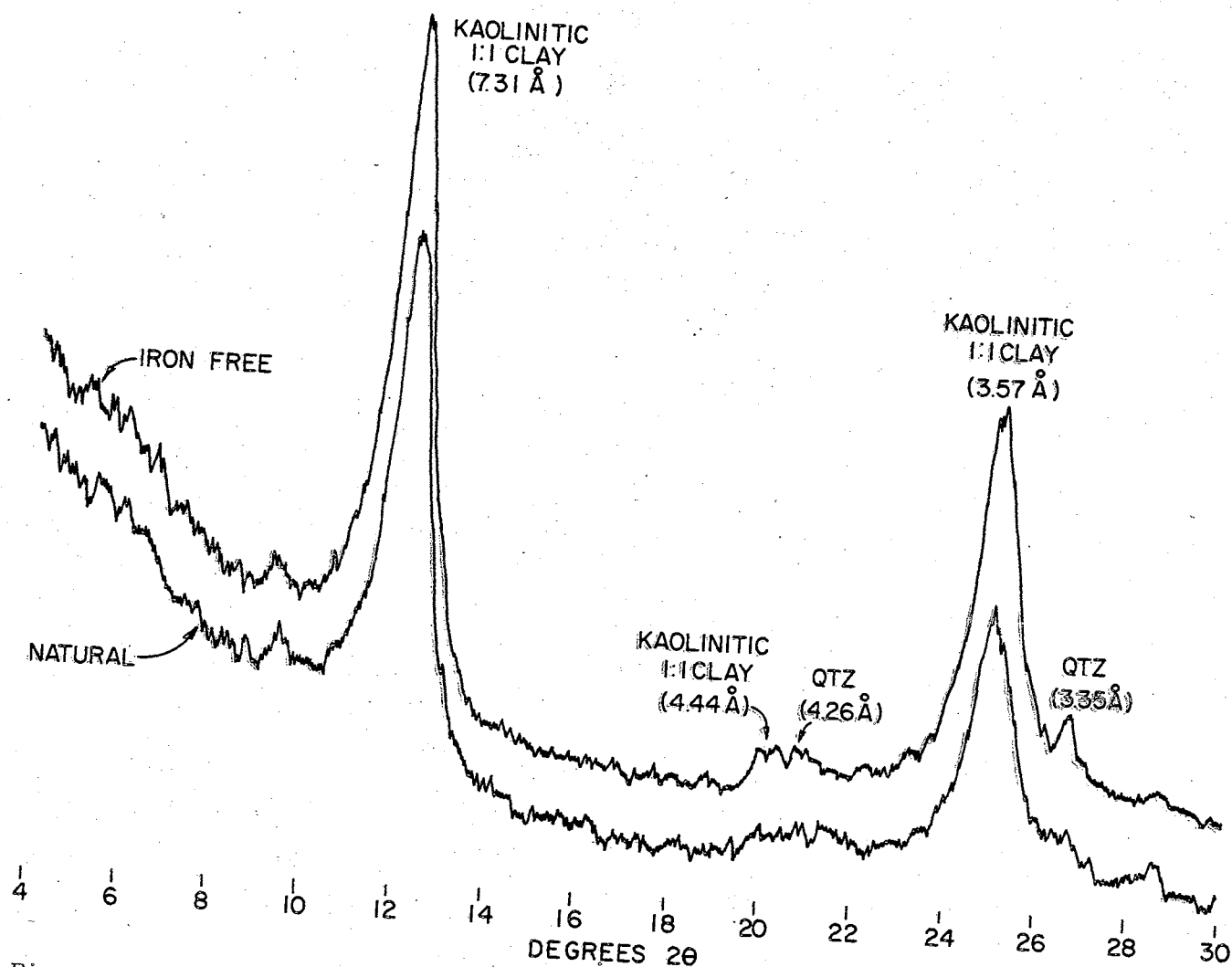


Figure 12. X-Ray Diffraction Patterns of the Coarse Clay Fraction (Ca Saturated)

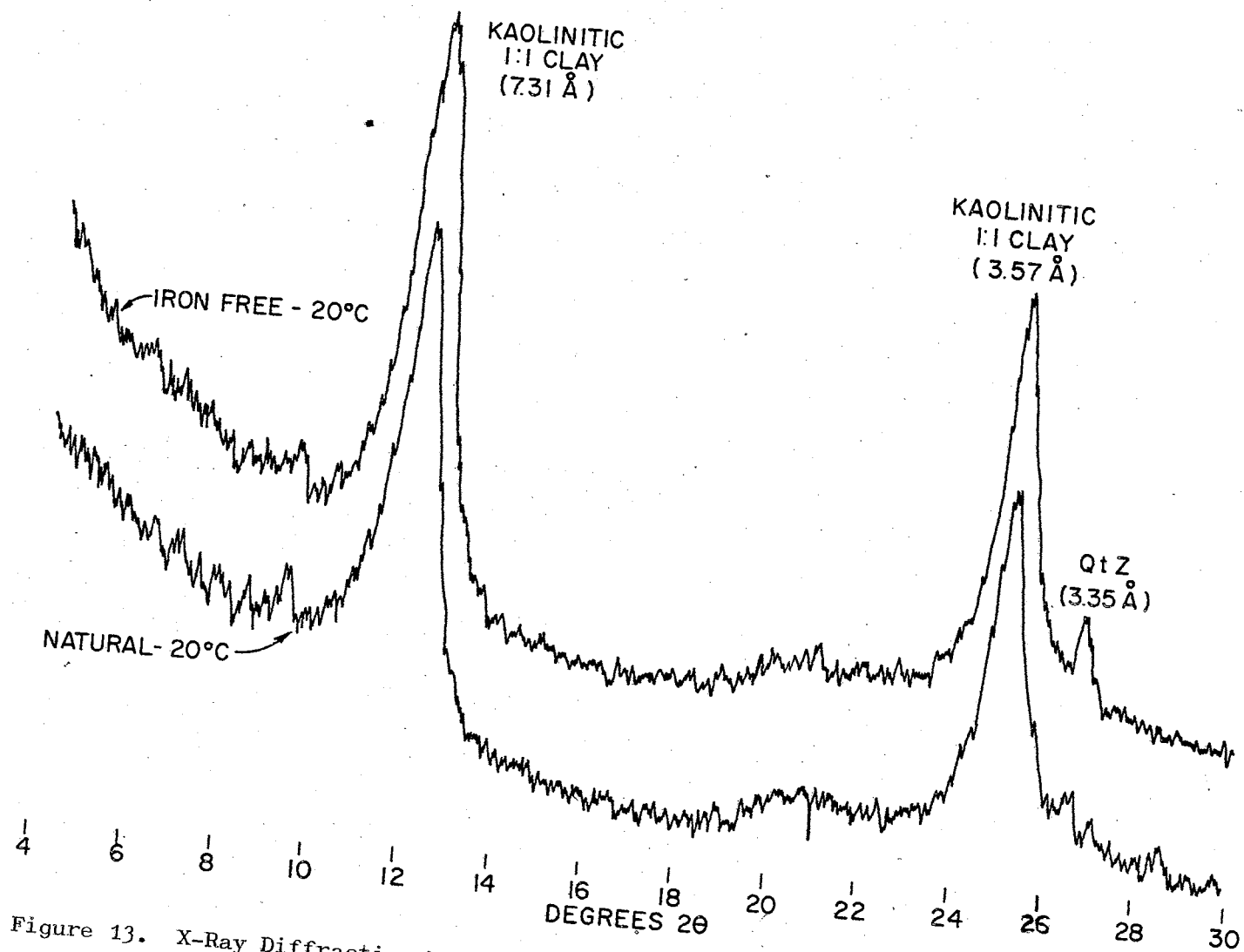


Figure 13. X-Ray Diffraction Patterns of the Coarse Clay Fraction (K Saturated)

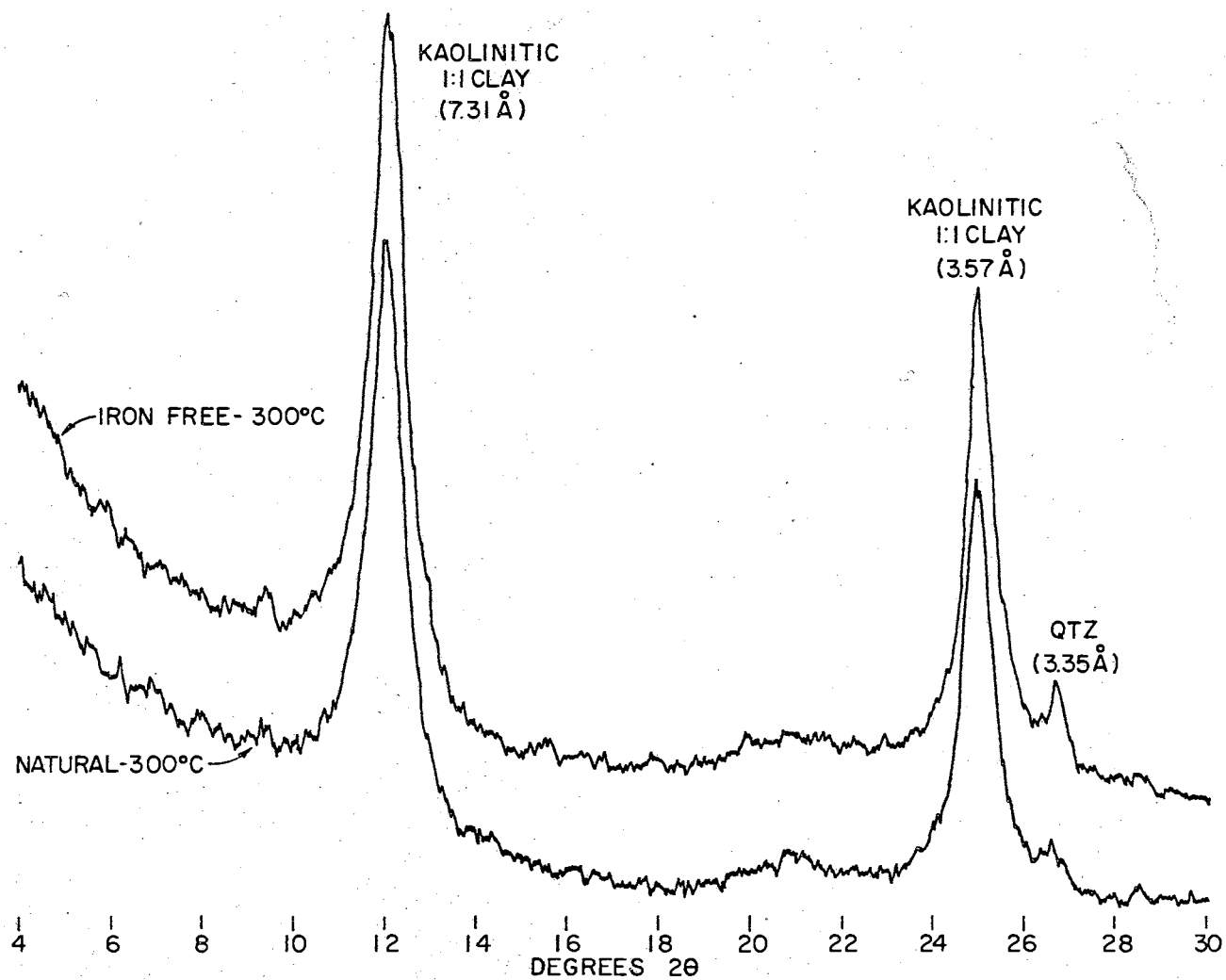


Figure 14. X-Ray Diffraction Patterns of the Heat Treated Coarse Clay Fraction
(K Saturated, 300°C)

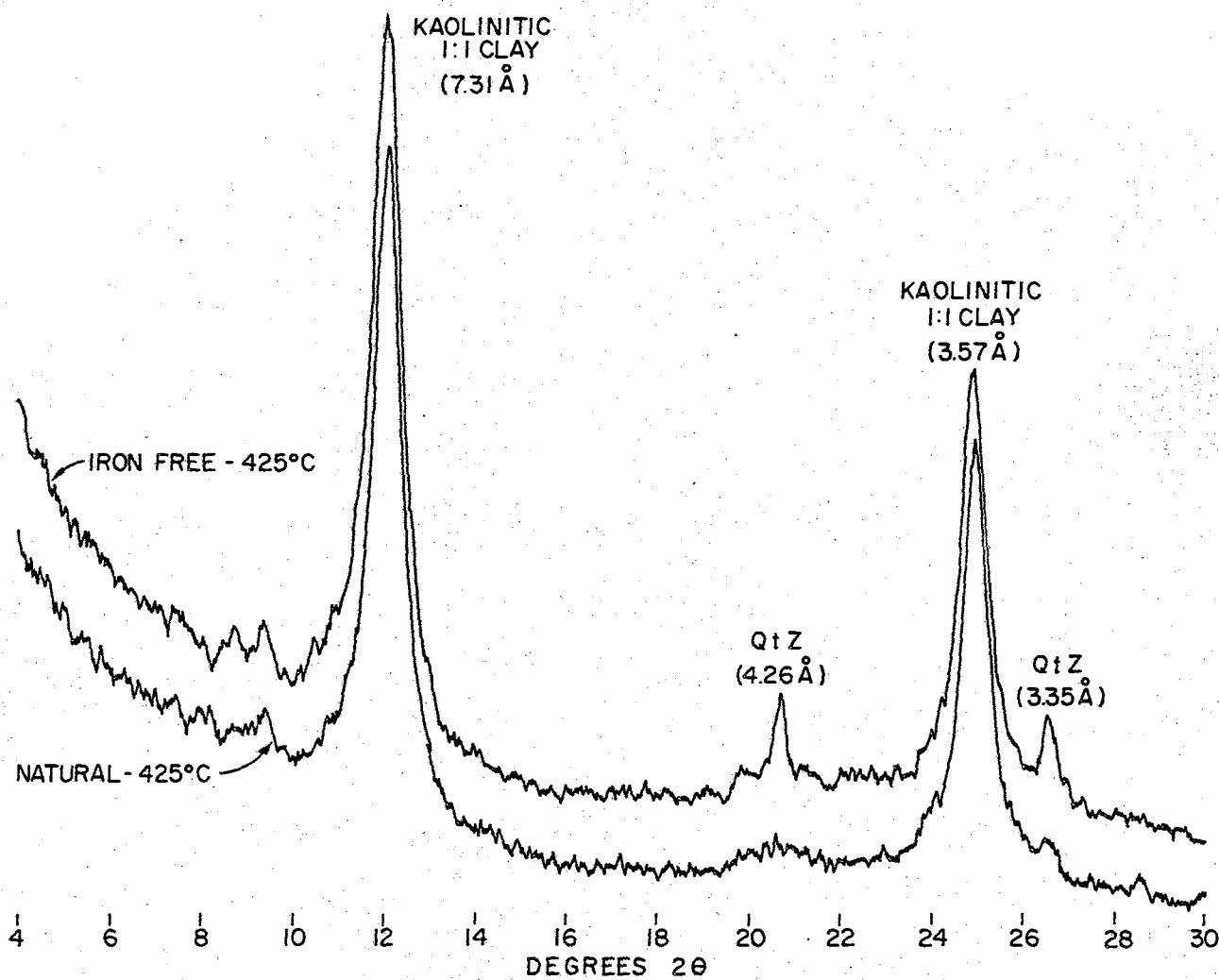


Figure 15. X-Ray Diffraction Patterns of the Heat Treated Coarse Clay Fraction
(K Saturated, 425°C)

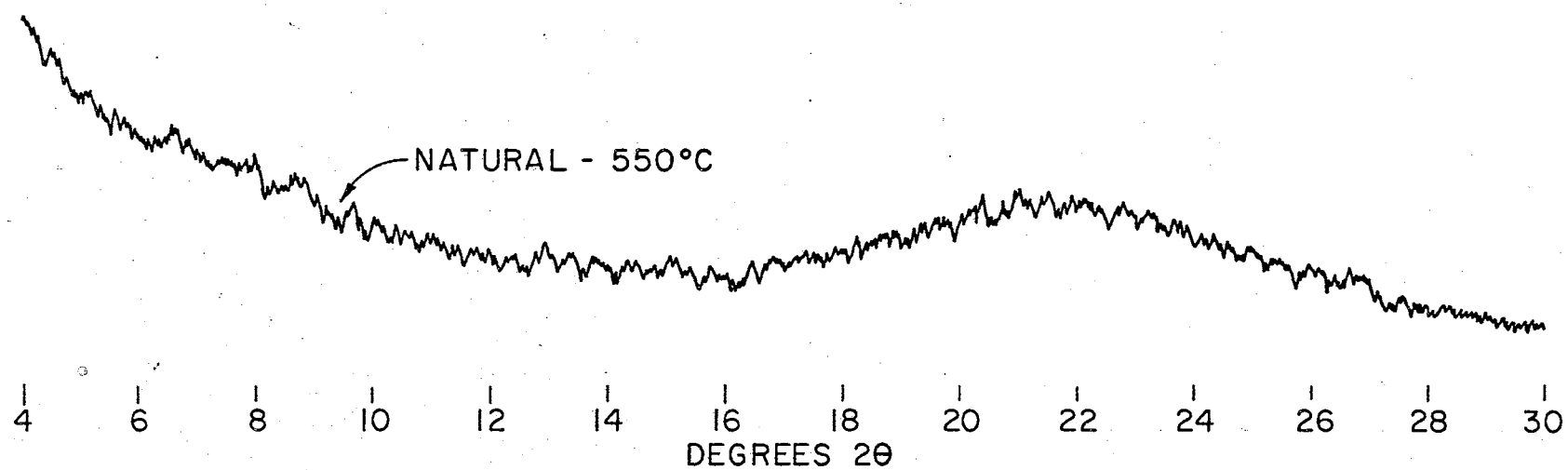


Figure 16. X-Ray Diffraction Patterns of the Heat Treated Coarse Clay Fraction
(K Saturated, 550°C)

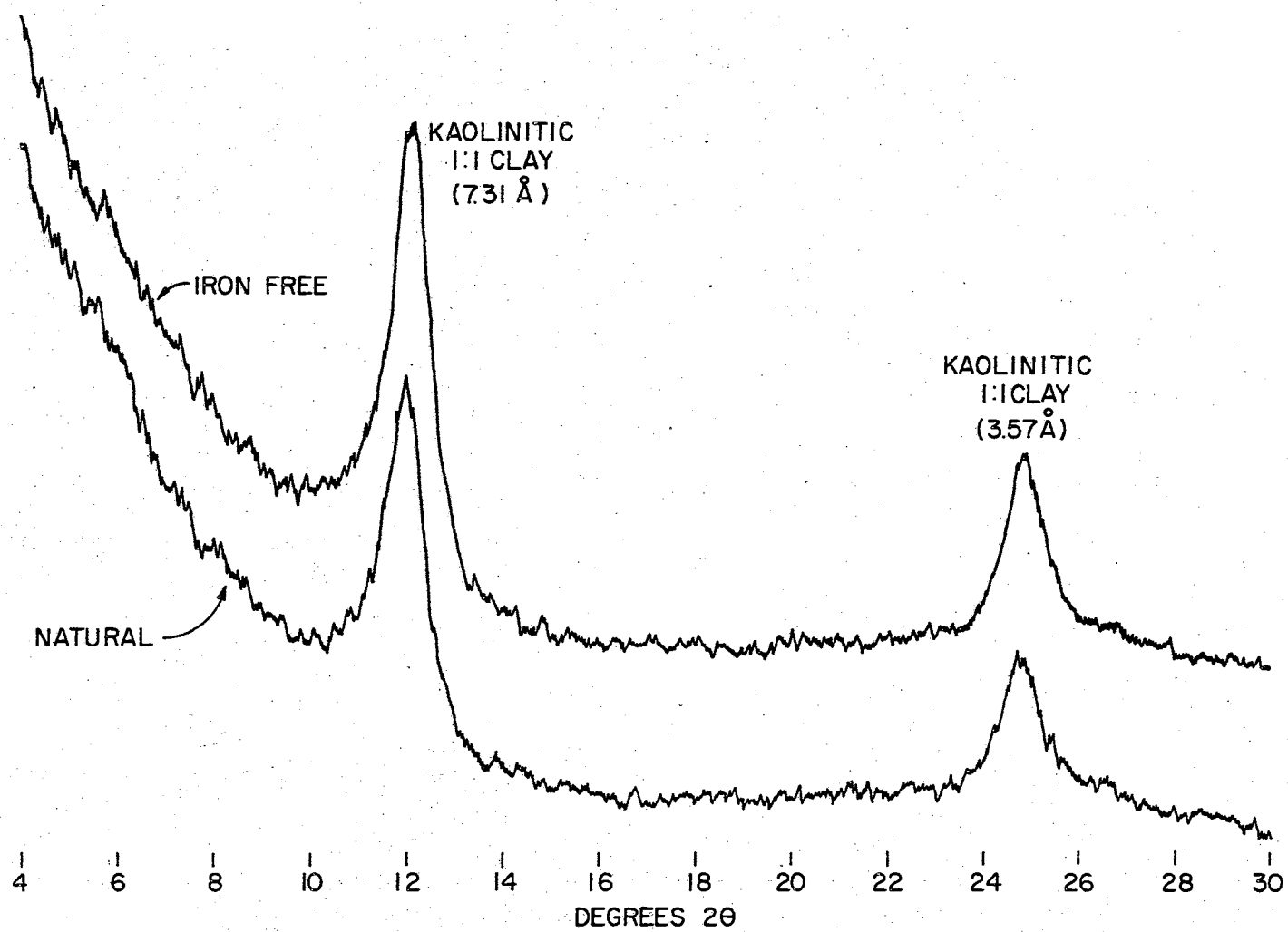


Figure 17. X-Ray Diffraction Patterns of the Fine Clay Fraction (Ca Saturated)

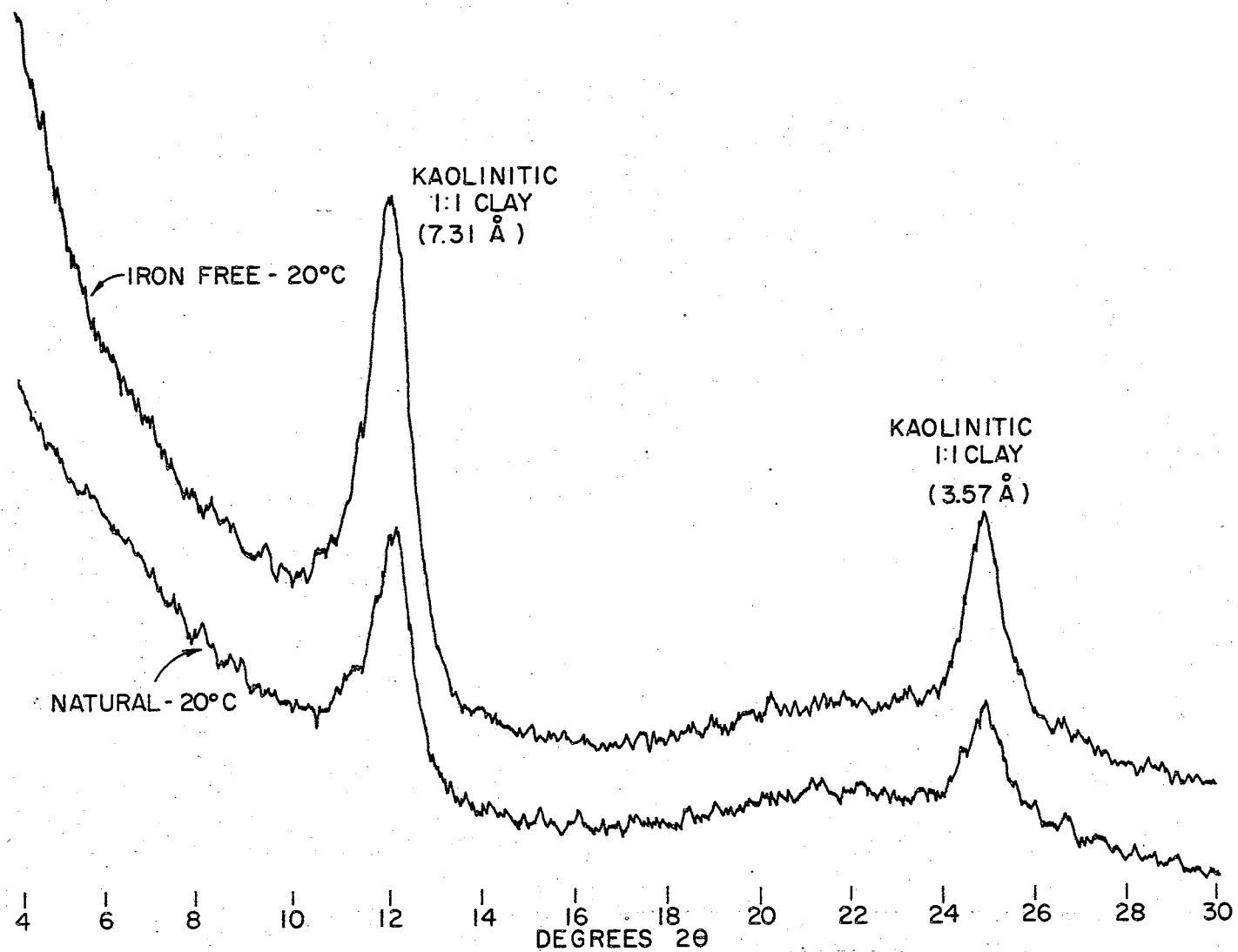


Figure 18. X-Ray Diffraction Patterns of the Fine Clay Fraction (K Saturated)

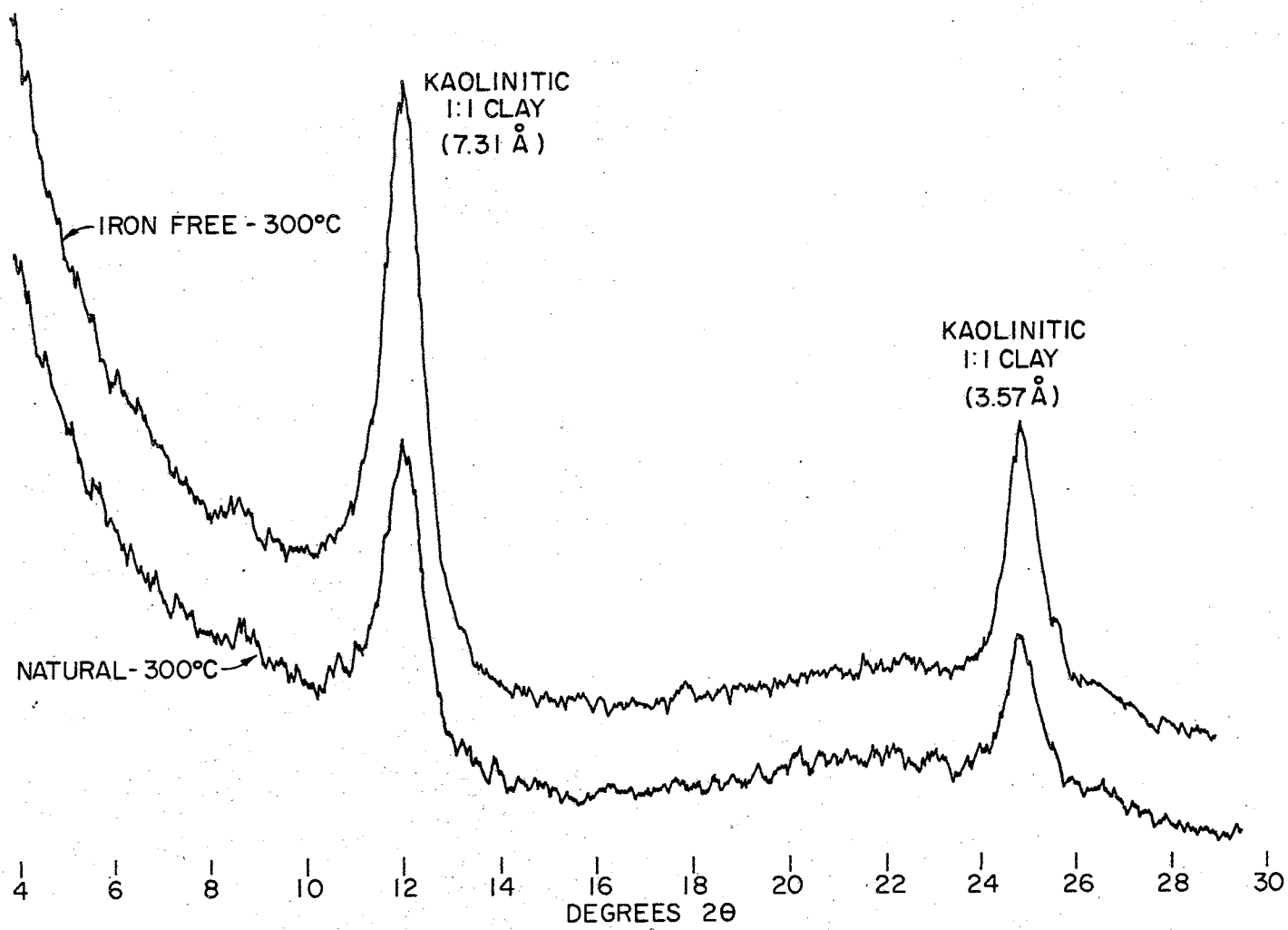


Figure 19. X-Ray Diffraction Patterns of the Heat Treated Fine Clay Fraction
(K Saturated, 300°C)

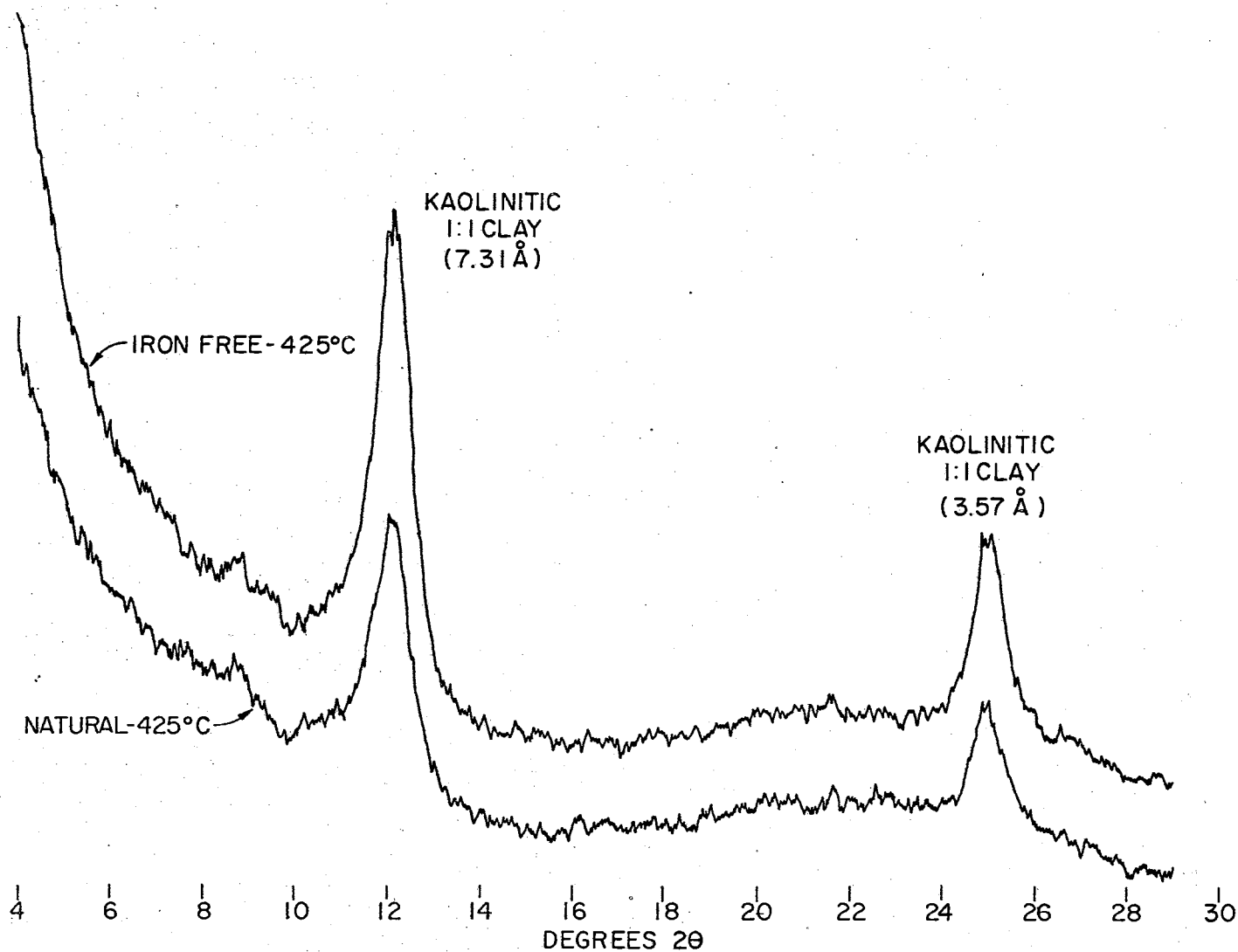


Figure 20. X-Ray Diffraction Patterns of the Heat Treated Fine Clay Fraction
(K Saturated, 425°C)

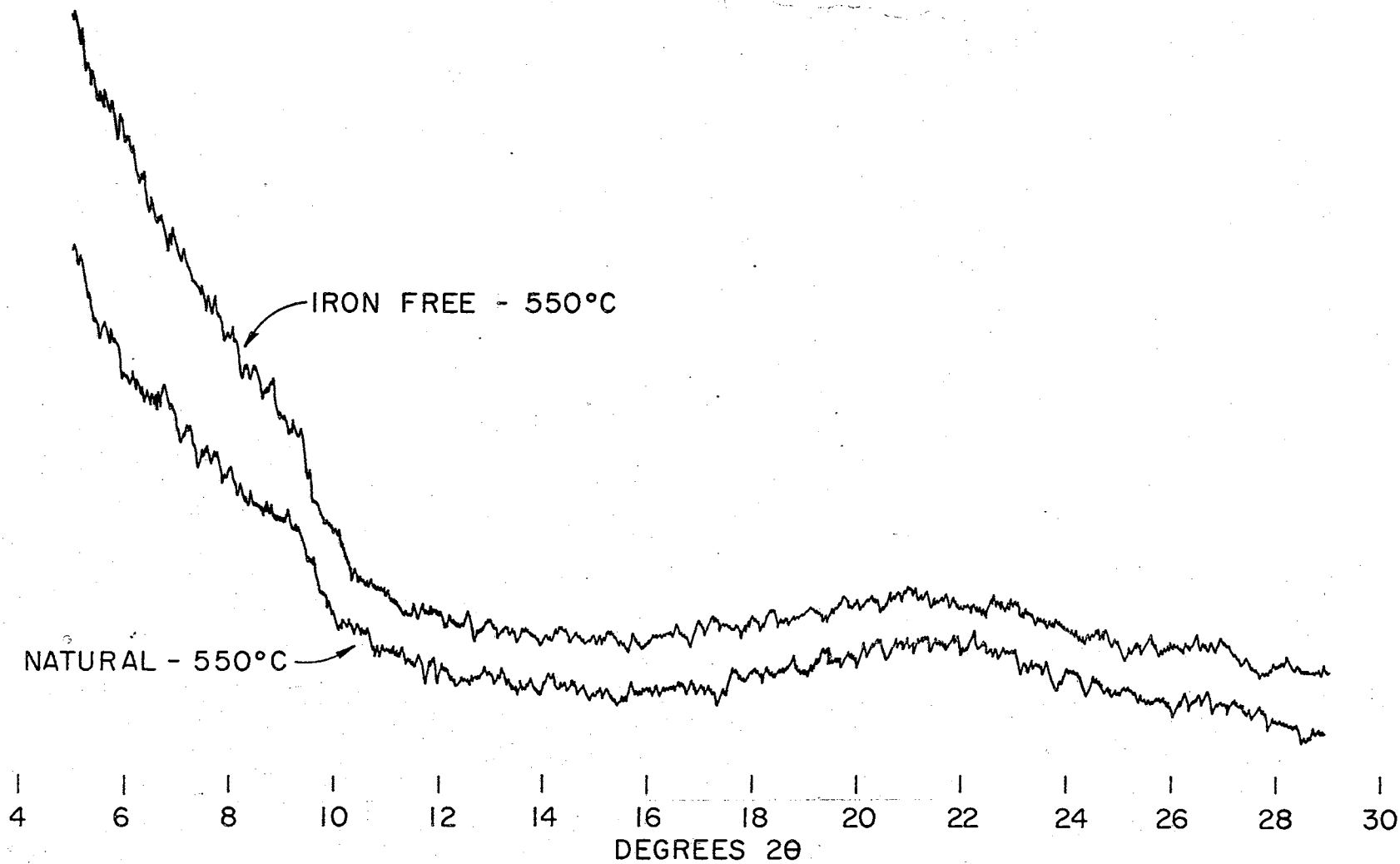


Figure 21. X-Ray Diffraction Patterns of the Heat Treated Fine Clay Fraction (K Saturated, 550°C)

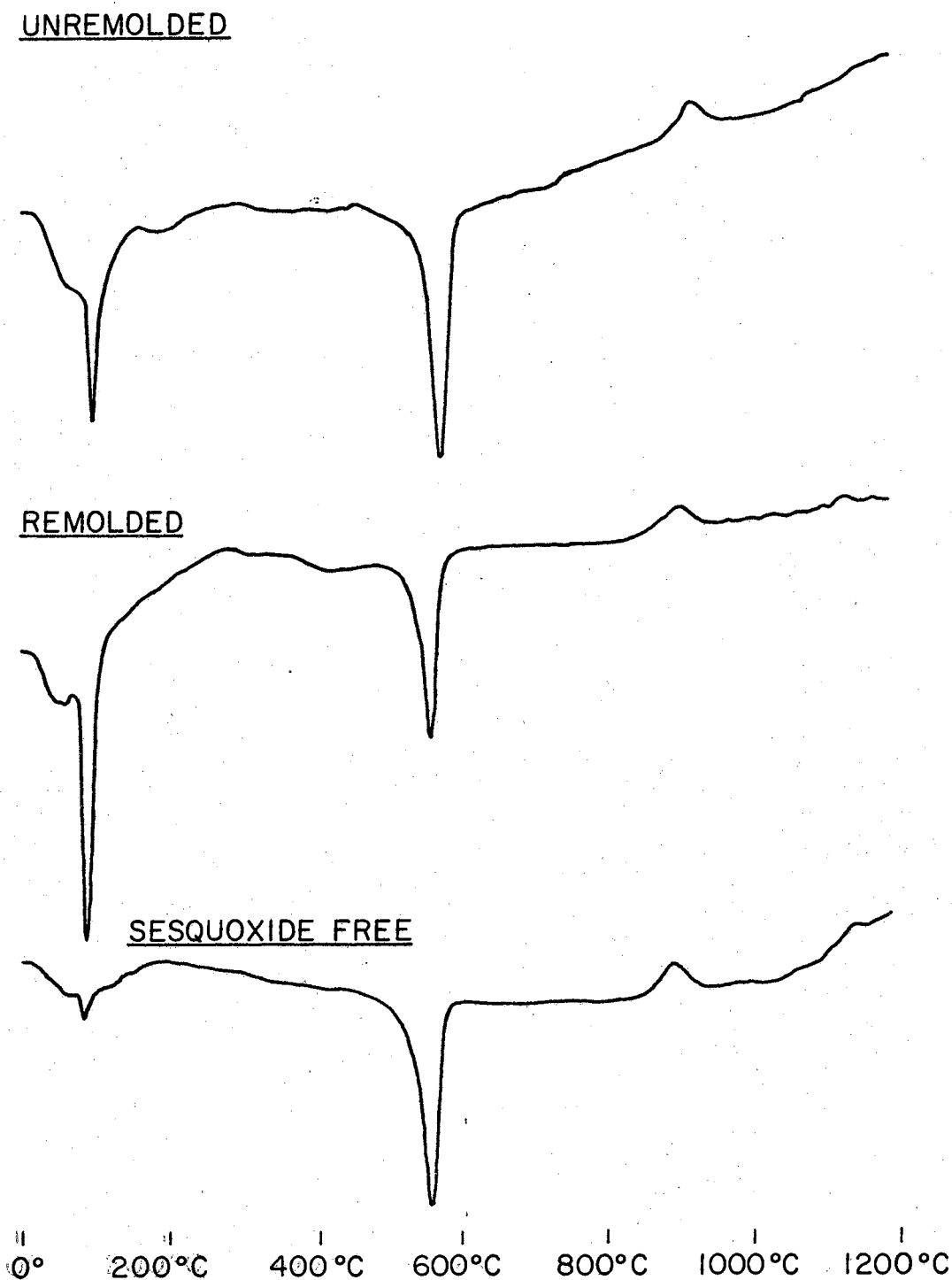


Figure 22. DTA Thermograms of Unremolded, Remolded, and Sesquioxide Free Lateritic Soil

CHAPTER IV

CHEMICAL PROPERTIES

Introduction

The salient chemical characteristics of laterite and lateritic soils are the presence of an abundant amount of the sesquioxides of iron and aluminum and the general absence of silica. These characteristics provided impetus to the use of silica:alumina and silica:sesquioxide ratios for classifying laterites and lateritic soils. The pioneer work of Martin and Doyne (47) introduced the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio as a basis of classification. Laterite was defined as a material whose clay fraction possessed a molecular $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio less than 1.33, while lateritic soils possessed ratios varying from 1.33 to 2.0. This initial definition was modified and became a silica:sesquioxide ($\text{SiO}_2/\text{R}_2\text{O}_3$) ratio, in which the iron oxides as well as the aluminum oxides were combined as the denominator of the fractional relationship. However, more recently the use of the $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio has diminished as an important criterion for classification, partly because of the change in viewpoint as to what constitutes a laterite and because of the increasing precision with which clay minerals can be identified. Notwithstanding, the $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio is still an important aspect of soils and has been shown to be indicative of the chemical and physical properties of colloidal materials (6, 48).

In previous chapters the existence of and mode of formation of iron coatings and the presence of amorphous material in the soil was established and discussed. The purpose of this phase of the investigation was to determine the effects of the sesquioxides on the cation exchange capacity (CEC) of the soil. It was anticipated that information concerning the relationship between the sesquioxides and the clay particles would result in a better understanding of the remolding phenomenon associated with lateritic soils.

Review of Literature

General agreement exists in the literature that the sesquioxides of iron and aluminum are absorbed on the surfaces of the clay minerals in lateritic soils. This absorption occurs through the interaction of the positively charged sesquioxides and the negatively charged clay particles. The mechanisms governing the absorption, however, are quite complex and dependent upon the surface characteristics of the iron and aluminum compounds, the charge characteristics of the clay minerals present, and the pH of the surrounding medium (27).

Early work by Mattson (48) demonstrated that the sesquioxides behave as electrical ampholytes. That is, above a pH of 7.0 they are electronegative, while below this pH they are electropositive. Because of this amphoteric nature and changes in surface charge with aging, the charge carried by the sesquioxides (principally the iron oxides) is difficult to predict (25). For example, in an acid medium the positively charged iron oxides may readily absorb anions, i.e., phosphate, sulphate, or organic compounds and exist as discrete negatively charged particles in the soil instead of attaching themselves to the clays (25,

26, 48). However, in the absence of these anions, the iron oxides vigorously attach themselves to the negatively charged clay particles and eventually form coatings. Under basic conditions, the situation is reversed, the sesquioxides become negatively charged and readily adsorb cations (48). In a basic medium repulsion rather than attraction is observed between the clay and iron oxides.

Adsorption of iron oxides on the surfaces of the clay minerals must occur at pH values below 7.0, where the sesquioxides are electropositive. Greenland, et al (26), obtained iron precipitates as surface coatings on kaolinite only at pH values of 5.0 or less, a value common to lateritic soils. Under acid conditions the adsorption of iron oxides by kaolinite is relatively unaffected by the presence of excess cations. However, the presence of anions can prevent the adsorption reaction of the iron by the clays (21).

The pH dependent amphoteric nature of the iron oxides and their ability to coat clay particles are clearly revealed by electron microscopy. Greenland, et al (26), observed that in some red soils the free iron oxides existed as discrete particles having little association with the clay minerals. In these observations, the iron hydroxides were partially associated with the clay platelets at the edges and not at the faces. Such an observation would indicate that continuous iron oxide coatings on the clay surfaces were very unlikely as the iron would probably be negatively charged and logically would not associate with the kaolin minerals except at the positively charged edges of the clay platelets. However, in the later work, Greenland, et al (27) observed that at low pH values iron hydroxides associated with the kaolinite surfaces and that some of these surfaces had been bound

together by the iron hydroxide. In these same experiments, at high pH values the iron hydroxides showed little association with the kaolinite surfaces and existed as a separate entity. Electron microscopy studies by Follett (21) indicated that colloidal iron hydroxide is adsorbed on only one basal surface of kaolinite flakes. Apparently only the silica tetrahedral surfaces possess sufficient charge to fix the iron particles. The nature of the alignment of the iron particles according to Follett (21) suggested some neutralization of the charge on the colloidal iron, resulting in aggregation of the clays. The tenacity with which the iron is adsorbed by the kaolinite is revealed, by the fact that prolonged washing or ultrasonic vibrations failed to dislodge the iron oxides from the clay surface. This colloidal iron was removed only by extraction with sodium dithionite.

The low cation exchange capacities (CEC) associated with lateritic soils generally have been attributed to the kaolin minerals and the neutralization of exchange sites on the clay surfaces by sesquioxide coatings. The kaolin minerals, which are the predominant clay mineral encountered in lateritic soils, by nature possess low cation exchange capacities. The adsorption of the positively charged iron hydroxides on the clay surfaces satisfies the negative sites and thus lowers the permanent CEC (21, 71, 76). However, due to their amphoteric nature, the presence in the soil of amorphous materials, i.e., allophane, colloidal hydrous iron or aluminum oxides, may provide additional pH dependent negative sites for replacing cations and increase the CEC measurement (31). It has been shown (3, 37, 57, 66) that these amorphous colloids can impart high pH dependent values to the CEC measurements.

Generally, removal of the sesquioxides exposes the negative sites on the basal surfaces of the clay minerals causing an increase in the CEC measurement of the soil (15, 51). It is possible, however, that the iron oxide coatings were formed in a basic medium, in which case only the positively charged clay edges and not the negatively charged basal surfaces would be blocked. Under such circumstances, removal of the iron oxides would cause a decrease in CEC measurements. Various hypothetical situations involving blockage of both the basal planes and the edges of the clay minerals by the sesquioxides can explain increases and/or decreases in the positive and negative charges following removal of the iron oxides (15).

Investigative Procedure

Sample Preparation

Remolded and unremolded samples of the soil (see Chapter III) passing the No. 40 U. S. Standard sieve (minus No. 40 material) were considered as representative of the range in soil conditions possible during construction operations. The various soil fractions, silt, coarse clay, and fine clay were considered to have been remolded during fractionation and analysis. No differentiation as to remolded or unremolded was made for comparisons of these fractions.

Methods and Equipment

The $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio was determined by the Rocky Mountain Geochemical Corporation, Salt Lake City, Utah, on the minus No. 80 sieve material in both the unremolded and remolded conditions. Their method of analysis (82) involved acid destruction of the soil and

subsequent determination of the Fe_2O_3 and Al_2O_3 percentages by atomic absorption, while the SiO_2 percentage was determined by colorimetric methods.

The percentages of free amorphous alumina and silica expressed as SiO_2 and Al_2O_3 were colorimetrically determined after heating to 110°C by the selective dissolution procedure (35, 41) presented in Appendix A. The percentage of kaolinite was determined by the same procedure after destruction of the kaolin by heating to 560°C . These percentages were determined in the minus No. 40 remolded and unremolded soil, and the silt, coarse and fine clay fractions.

The percentage of Fe_2O_3 extracted from the minus No. 40 remolded material by the sodium citrate-dithionite-bicarbonate method was determined by atomic absorption utilizing a model 303 Perkin-Elmer Spectrophotometer.

The cation exchange capacity (CEC) of the natural soil was measured by calcium saturation and displacement with sodium in accordance with the procedure given in Appendix A. After these CEC values had been measured, the iron and aluminum oxides were extracted by the sodium citrate-dithionite-bicarbonate treatment (51) and the CEC of the sesquioxide free soil was measured for comparative purposes. These measurements were determined for the minus No. 40 remolded and unremolded soil, and the silt, coarse and fine clay fractions.

Statistical techniques employed to analyze data were coefficient of variation and t tests. These tests and their results are presented in Appendix B.

Experimental Results and Discussion

Silica and Sesquioxide Composition

The total percentages of silica, alumina, and iron in the natural soil are presented in Table I. These percentages include contributions of silica and alumina from the tetrahedral and octahedral layers of clay minerals, quartz silica, and magnetite iron. Because the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios are for the minus No. 80 sieve fraction instead of the clay fraction, the classification scheme of Martin and Doyne (47) is not justified. The presence of quartz silica in this lateritic soil fraction raises the $\text{SiO}_2/\text{Al}_2\text{O}_3$ above 2.0. Only a very slight difference exists between the silica and sesquioxide composition of the unremolded and remolded soils. Evidently, remolding has very little effect, as might be expected, on the chemical composition.

The percentage values of free silica and alumina presented in Table I represent the average values of duplicate tests. The coefficients of variance for these values ranged from 13 percent to 26 percent with an average coefficient of 24.5 percent (see Appendix B). These percentages indicate that considerable quantities of amorphous material, perhaps sesquioxidic allophane, exist in this soil. This finding is consistent with that determined by mineralogical analyses reported in Chapter III. Generally, lateritic soils are considered to be low in free silica and relatively inactive chemically. However, amorphous aluminosilicates have been reported in Panamanian soils (8) and in soils derived by weathering of andesite (31), the parent rock of this particular laterite. The presence of these amorphous colloids undoubtedly contributes greatly to the chemical behavior of this soil.

TABLE I
SILICA AND SESQUIOXIDE COMPOSITION

Soil Fraction	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SiO ₂ /Al ₂ O ₃ (molecular)	SiO ₂ /R ₂ O ₃ (molecular)
Total SiO ₂ , Al ₂ O ₃ , and Fe ₂ O ₃					
Unremolded (minus No. 80)	40.3%	21.6%	11.4%	3.16	2.36
Remolded (minus No. 80)	41.1%	22.6%	12.5%	3.08	2.28
Free SiO ₂ , Al ₂ O ₃					
Unremolded (minus No. 40)	19.9%	6.6%	--	5.11	--
Remolded (minus No. 40)	22.9%	3.9%	6.2%	9.95	4.94
Silt (50-2μ)	8.6%	3.5%	--	4.17	--
Coarse Clay (2-0.2μ)	17.0%	6.3%	--	4.58	--
Fine Clay (< 0.2μ)	12.6%	4.0%	--	5.33	--

High pH dependent exchange capacities, high water retention capabilities, and large specific surfaces are characteristic of these amorphous colloids.

Apparently, remolding the soil has little effect on the amount of free silica and alumina present in the minus No. 40 fraction. However, the free $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio is higher for the remolded soil, which might indicate a greater activity than for the unremolded soil. Likewise, the free $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio increases inversely with grain size, which suggests that a greater activity may be imparted to the finer fractions by the amorphous materials.

Kaolinite Composition

The approximate percentages of kaolinite present in the various fractions of the soil are given in Table II. These values are the results of duplicate tests for silica and alumina after heating the soil to 560°C . The kaolinite percentages are calculated on the basis of 56.5 percent SiO_2 or 39.5 percent Al_2O_3 , which are the respective theoretical formula weight percentages for kaolinite. Such calculations are not entirely correct for this lateritic soil, since the mineralogical analyses (see Chapter III) revealed that the clay is a mixture of poorly crystalline kaolinite and possibly halloysite. The formula weight of this mixture of kaolin minerals probably is not equal to that of the well crystalline kaolinite utilized for these calculations.

Chemical analysis indicates that remolding has little effect on the percentages of kaolin present in the minus No. 40 fraction. Although grain size analyses in previous work (79) showed that

TABLE II
KAOLINITE CONTENT OF SOIL FRACTIONS DETERMINED
BY SILICA AND ALUMINA COMPOSITIONS

Soil Fraction	Based Upon Si Composition	Based Upon Al Composition	Average
Unremolded (minus No. 40)	35.5%	32.9%	34.2%
Remolded (minus No. 40)	32.2%	34.9%	33.6%
Silt (50-2 μ)	41.6%	38.6%	40.1%
Coarse Clay (2-0.2 μ)	48.4%	47.5%	47.9%
Fine Clay (< 0.2 μ)	54.5%	42.7%	48.6%

remolding increases the percentage of clay size particles due to a breakdown of the natural cementing agents, the actual amounts of kaolin in the unremolded and remolded fractions are approximately equal. These observations indicate that the coarser particles of the unremolded soil essentially consist of cemented kaolinite, and that removal of these cementing agents, i.e., free silica and alumina, disaggregates these coarse particles in the unremolded soil. Thus, the removal of these cementing agents by the chemical procedure utilized for kaolin analysis creates a condition similar to that created by remolding. A similar condition apparently exists in the silt fraction, where the high percentage of kaolinite, 40 percent, suggests that cementation by the sesquioxides agglomerates the kaolin into silt sized particles.

As expected, the percentage of kaolinite increases as the soil fractions become finer. The wide variation between the kaolinite percentages based on the Si or the Al composition of the fine clay fraction indicates that these colloids are poorly crystalline and, therefore, inconsistent with the assumed formula weight. By a similar analogy, the close agreement between the reported percentages of kaolinite in the coarse clay fraction indicates a more crystalline material. As discussed in Chapter III, the greater peak intensities of the coarse clay x-ray patterns compared with those of the fine clay also indicate a greater crystallinity in the coarse clay.

The consistently higher percentages of silica than alumina suggest an acid weathering condition whereby the alumina is removed and silica remains. Such conditions are consistent with the findings of cristobalite x-ray peaks and chalcedony pebbles in the soil.

Cation Exchange Capacity

The cation exchange capacities (CEC) of the various soil fractions are listed in Table III. These results represent the average values of duplicate tests. The coefficients of variation ranged from 22.4 percent to 0.13 percent with the pooled coefficient of variation being 7.56 percent (see Appendix B).

Considering that this soil is of lateritic origin, the exchange values are somewhat higher than expected. Ferralitic soils with $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios slightly less than two and composed of 1:1 kaolin clays generally exhibit CEC values greater than 5.0 me/100gms but less than 10 me/100 gms (11). Kaolinite generally exhibits a range of CEC values of 3 to 15 me/100 gms, while halloysite has values ranging from 5 to 10 me/100 gms. Therefore, the higher values exhibited by the clay fractions of this soil confirm the presence of amorphous material. Other studies on lateritic soils containing amorphous colloids have shown CEC values of 10 to 25 me/100 gms and 36 me/100 gms for a Panamanian latosol (8), which are consistent with the values given.

Remolding of the soil appears to have little effect on the cation exchange capacity of the minus No. 40 fraction; likewise, similar results (not reported) also were observed in the silt fractions of unremolded and remolded soil.

The possibility exists that during the various analytical procedures the unremolded soil undergoes a certain amount of remolding. However, disregarding this possibility, hydrometer analyses show that the remolded soil contains a greater percentage of fines than the unremolded soil. Therefore, because the CEC values are essentially equal for the unremolded and remolded soil, the clay minerals must be

TABLE III
CATION EXCHANGE CAPACITIES OF THE NATURAL
AND "SESQUIOXIDE FREE" SOIL FRACTIONS

Soil Fraction	Natural (me/100 gms)	Sesquioxide Free (me/100 gms)
Unremolded (minus No. 40)	10.49	15.93
Remolded (minus No. 40)	10.79	16.91
Silt (50-2 μ)	11.21	13.09
Coarse Clay (2-0.2 μ)	16.43	19.15
Fine Clay (< 0.2 μ)	25.06	21.01

neutralized and contribute little to the exchange capacity. For had the exchange values been higher in the remolded soil it would appear logical that the greater percentage of fines would have been responsible. The fact that no such difference was noted indicates that the increased amount of fines had no contributing effect on the exchange capacity. This condition could only exist if the exchange sites on the clay particles were blocked, presumably by the sesquioxides. Therefore, the exchange capacity exhibited by the soil must largely be due to the amorphous materials. In Table I, these amorphous materials are shown to exist in equal amounts in the unremolded and remolded fractions.

As expected, the exchange capacities increase as the fractions become finer. The high CEC value measured in the silt fraction, which ordinarily exhibits negligible exchange capacities, confirms that the free silica and alumina are responsible for the major portions of the exchange capacity. Although the percentage of kaolinite is essentially the same for the coarse and fine clay fractions, the significant difference ($\alpha = .05$) in their exchange capacities indicates that poor crystallinity and fineness are more influential on the exchange capacity than is the amount of kaolinite.

The percentages of free silica and alumina are greater in the coarse clay than in the fine clay (see Table I); yet the fine clay exhibits a higher exchange capacity. This higher exchange capacity is probably caused by the more disordered structure in the fine clay due to isomorphic substitution, which contributes more exchange sites.

Removal of the sesquioxides from the soil by sodium citrate-dithionite-bicarbonate extraction would be expected to (a) increase the overall number of individual clay particles because of a breakdown

of the aggregated clay clusters through loss of the natural cementing agents and (b) remove iron and aluminum oxides from neutralizing positions on the basal surfaces of the kaolin particles. Both of these actions would contribute to an increase in the exchange capacity of the soil. However, the substantial influence of the amorphous colloids on the exchange capacity cannot be overlooked. The extraction procedure also removes the amorphous iron and alumina, which alters the properties of the allophanic materials. Consequently, the extraction of the free iron and aluminum oxides can either enhance or diminish the exchange capacity depending upon the nature of the clay minerals involved, the amount of blockage and cementing by the sesquioxides, and the percentage of amorphous colloids present.

An examination of the exchange capacity of the silt fraction reveals that no significant difference ($\alpha = 0.05$) in the CEC measurement is produced after sesquioxide extraction. Apparently the presence of 40.1 percent (see Table II) of "cleaned" kaolinite to the silt fraction through the removal of blocking and cementing agents is insufficient to counteract the loss of amorphous colloids and produce an increase in exchange capacity. In the case of the fine clay fraction, sesquioxide extraction can contribute little to disaggregation of cemented clay clusters, and the primary contribution to exchange capacity would be that of removing iron and alumina from neutralizing positions on the basal surfaces of the clays. However, this contribution is insufficient to offset the loss of amorphous materials, which results in a significant ($\alpha = 0.05$) decrease in the exchange capacity of the soil. Similarly, the increase in the exchange capacity upon sesquioxide removal in the minus No. 40 and coarse clay fractions is

interpreted as being due to greater percentages of "unblocked" clay particles offsetting decreases due to the loss of amorphous colloids.

CHAPTER V

PHYSICAL PROPERTIES

Introduction

Physical properties of soils are of particular interest to soils engineers, in that these properties are considered to be indicative of the engineering characteristics of a soil. The combination of the plasticity and textural characteristics have been used extensively and quite successfully for the engineering classification of cohesive soils in temperate areas. However, correlations between the index properties and the engineering behavior of lateritic soils have often resulted in erroneous conclusions. Beginning with Fruhauf (24) in 1946, numerous authors (54, 55, 64, 75, 80, 83) have mentioned the difficulties in applying temperate zone classification systems to lateritic soils. The primary objection is that considerable variation in standard test results is possible depending upon the amount of remolding and the treatment of the soil prior to testing. The most general conclusion is that a need exists for modification of the standard index tests if such tests are to be used to evaluate the physical properties of lateritic soils. Desirably such revisions would result in more consistent and better evaluations of test results and more accurate correlations between laboratory values and field behavior.

There is a general agreement in the literature that the high contents of iron and aluminum oxides and their relationship with the

clayey material in the soil are largely responsible for the observed physical properties of lateritic soils. The $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio appears to have a considerable influence on the friability and plasticity of soils (6).

The purpose of this phase of the investigation was to examine the influence of the sesquioxides on the Atterberg limits, grain size, and specific gravity of the soil. Concurrently, possible revisions of standard laboratory test procedures were evaluated in order to overcome the reported inconsistencies in test results due to remolding.

Review of Literature

Although higher limits have been reported, in general, lateritic soils exhibit liquid limit values ranging from 40 percent to 70 percent, and plastic limits ranging from 25 percent to 50 percent (5). The wide range of these results confirm the variable nature of this soil group. For temperate clays liquid limits in excess of 50 percent indicate the probability of the presence of montmorillonite in the soil (50). While lateritic soils seldom contain montmorillonitic clays, they often exhibit liquid limits greater than 50 percent. Terzaghi (75) attributed the unusually high liquid limits of lateritic soils to the micro-aggregate clayey clusters associated with this type of soil. He stated that while the soil had the plasticity index and engineering properties of a relatively coarse grained soil with a low plasticity index, the liquid limit was equal to that of a temperate zone clay with a high liquid limit. He concluded that this was due to the evaporation (during drying) of water located between the clusters as well as water contained in the voids of the porous grains.

Remolding of the soil during testing has been recognized as a handicap in the accurate determination of Atterberg limits for lateritic soils. However, the literature contains only sparse data concerning this problem. Some of the available results on Atterberg limit determinations on natural and remolded lateritic soil are presented in Table IV. There is general agreement among the authors listed in Table IV that the increase in the liquid limit with remolding is due to a breakdown of the clay particle aggregations. The breakdown of the clayey clusters increases the liquid limit by: (a) providing more clay particles to absorb water, and (b) exposing more surface area. These effects thereby increase the liquid limit. There has been no adequate explanation for the relatively minor effects that remolding has on the plastic limit.

In addition to the effects of remolding, wide variations in the liquid limit values have been ascribed to the drying procedures used prior to testing. Newill (54) reported a liquid limit decrease from 104 percent to 77 percent for one soil and a decrease from 76 percent to 74 percent for another resulting from air drying at room temperatures. This decrease was attributed to the irreversible conversion of hydrated halloysite to metahalloysite by the loss of two molecules of water. Frost (23), in his studies involving tropical soils containing allophanic colloids, described a decrease in the liquid limit from 207 percent to 85 percent due to air drying. Apparently, air drying can cause irreversible dehydration of halloysite and/or allophanic clays and may result in considerable variation in Atterberg limits for the same soil in the natural and air dried conditions.

These objectionable effects of remolding and drying on the

TABLE IV
 ATTERBERG LIMITS OF NATURAL AND REMOLDED LATERITIC SOILS

Type and Location	Liquid Limit		Plastic Limit		Plasticity Index		Source
	Natural	Remolded	Natural	Remolded	Natural	Remolded	
Red Clay - Kenya	74	84	38	39	36	45	Newill (54)
Red Clay - Kenya	77	91	61	59	16	32	Newill (54)
Lateritic Soil - Cuba	46	53	31	31	15	22	Winterkorn and Chandrasekharan (83)
Lateritic Soil - Panama	60	70	39	40	21	30	Townsend (80)

accurate determination of the liquid limit make it difficult to correlate laboratory values and field behavior. Fruhauf (24), Pollitt (59) and Townsend (80) have suggested a "differential" plasticity test, in which the Atterberg limits would be determined on natural soil in a relatively undisturbed state using fresh soil for each point on the curve. Then, if remolding of the soil was suspected during construction activities the limits would be evaluated for the soil in the remolded condition. Nixon and Skipp (55) proposed the use of a standard crushing test to evaluate whether remolding of the soil during construction is likely to occur or not. Then if remolding was anticipated, the soil would be tested in the remolded condition.

Lateritic soils generally possess grain size distributions containing all size fractions from gravel to clay. However, as in Atterberg limit determinations, the friable nature of the soil allows a ready breakdown of the soil aggregates into finer sizes. Because of this friable nature particle size determinations depend upon the amount of disaggregation prior to testing, and correlations between different methods and operators are not strictly justified (5, 24, 54, 55, 64, 79).

Drying, also, exerts considerable influence on grain size analyses of lateritic soils. This fact is not surprising considering that Buchanan (9) coined the term "laterite" upon observing the transformation of a tropical red friable soil into hardened bricks after air-drying. Newill (54) observed a decrease in the clay percentage of a lateritic soil from 79 percent to 47 percent for one soil and a similar decrease from 82 percent to 68 percent for another soil upon oven drying. This decrease strongly suggests that increased cementation

occurs between soil aggregates due to dehydration of the hydrous cementing agents.

The specific gravity of lateritic soils usually ranges from 2.70 to 3.50 (5). These values are generally higher than nonlateritic soils, whose specific gravities ordinarily range from 2.65 to 2.75. The higher specific gravities of lateritic soils reflect the high percentages of iron oxides, which are characteristic of these soils (5, 54, 56).

Although the role of the sesquioxides in aggregation is not fully understood, their influence is generally ascribed to one or all of the following: (a) cementation due to precipitation of a hydrated iron or aluminum gel and a subsequent irreversible dehydration of these materials, (b) the presence of iron in solution which prevents deflocculation, and (c) the formation of organic mineral compounds of humic acids with free sesquioxides (49). In general, removal of these iron and aluminum oxides destroys the natural soil aggregates increasing the amounts of finer particles (4, 12, 38, 43). This increase indicates that aggregation of the soil is due to the physical binding forces of the sesquioxides (49).

Bennett (6) observed that the physical properties of clay colloids are influenced by the $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio. Those soils possessing low $\text{SiO}_2/\text{R}_2\text{O}_3$ ratios exhibited low plasticity, friability, good internal drainage and permeability, and little volume change characteristics. He concluded that on the basis of the $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio a differentiation might be possible between friable and nonplastic soils and nonfriable and plastic soils. Such a differentiation would be a considerable

asset to highway engineers in determining the quality of a doubtful clay soil for a road subgrade.

Sample Preparation

Atterberg limits were determined for the soil in the unremolded, remolded, and sesquioxide free conditions. New material was used for the determination of each point on the flow curve of the unremolded soil in order to minimize spatula manipulations and subsequent remolding of the soil. The used unremolded soil was then remolded (see Chapter III) and the limits were redetermined by standard procedures. The sesquioxides were extracted from a similar soil sample by the procedure presented in Appendix A and the limits redetermined for the sesquioxide free soil. Four flow curves were plotted for both the remolded and unremolded soil and a least squares regression analysis was used to combine the curves into one average curve for each of the soil conditions (see Appendix B).

The hydrometer method of analysis was used to obtain the grain size distribution curves of the portion passing U. S. Standard No. 100 sieve. The unremolded soil was dispersed by gentle rotation of the graduated cylinder while the remolded soil was dispersed in an electric mixing cup according to the usual procedure. The sesquioxides were then extracted from the soil samples used for the unremolded and remolded analyses and then the grain size distributions of the iron free soils were determined. Two mls of a 4 percent solution of Calgon was used as the deflocculating agent for all hydrometer tests.

Experimental Results and Discussion

Atterberg Limits

The results of the Atterberg limits tests listed in Table V show that remolding increased the liquid limit of the soil from 57.8 percent to 69.0 percent while the plastic limit remained essentially constant. These results are consistent with those previously reported in the literature and presented in Table IV. The increase in the liquid is attributed to the breakdown of the granular structure by remolding which increases the amount of fines and exposes more surface area for the absorption of moisture.

Since conventional methods for liquid limit determinations involve remolding of the soil at high moisture contents, the One-Point Liquid Limit test procedure offered a means of evaluating the liquid limit for the unremolded soil. The Waterways Experiment Station (WES), Vicksburg, Mississippi, proposed the following equation based upon their test results (58).

$$LL = W_n (N/25)^{\tan \beta}$$

where

LL = liquid limit of the soil,

W_n = moisture content at N blows,

N = number of blows, and

$\tan \beta$ = slope of the flow curve on a log-log plot of N vs W.

The average value of $\tan \beta$, determined by WES experiments, was 0.121, and this value was adopted in ASTM Test D 423-59. If a substantial difference existed between the values of $\tan \beta$ for the unremolded and remolded soil, it might be expected that the One-Point Liquid Limit

determination would be unreliable. It was found from a least squares regression analysis that the $\tan \beta$ for the unremolded soil was 0.05, while $\tan \beta$ for the remolded soil was 0.16. Such differences in $\tan \beta$ would appear to prohibit the use of this method. However, if the range of N is restricted to 20 to 30 blows, the error induced by using $\tan \beta = 0.121$ is 1.55 percent for the unremolded soil and 1.16 percent for the remolded soil. Since these errors do not exceed those normally associated with standard liquid limit determinations, the one-point procedure appears practicable for this type of soil.

Removal of iron and aluminum oxides would be expected to increase plasticity of the soil due to (a) increased amounts of fine particles released from previously cemented clay clusters and (b) removal of plasticity suppressing sesquioxide coatings from the surfaces of the clay minerals. Newill (54) obtained an increase in liquid limit from 77 percent to 93 percent after removal of the iron oxides from his lateritic soil sample. However, the results presented in Table V show that removal of the sesquioxides caused a significant decrease in the liquid limit and plastic limit values of the soil. Apparently, removal of the sesquioxides, i.e., the amorphous allophanic materials, lowers the water retention capabilities of the soil as shown by a significant loss in peak intensity of the 100°C peak of the DTA curves in Figure 22 of Chapter III. Concurrently, removal of sesquioxide cementing agents destroys the porous microaggregates, thereby eliminating the microvoids and clayey clusters which Terzaghi (75) described as the cause of the high liquid limits associated with lateritic soils.

TABLE V
PHYSICAL PROPERTIES OF UNREMOLDED, REMOLDED
AND "SESQUIOXIDE FREE" LATERITIC SOIL

Property	Unremolded	Remolded	Sesquioxide Free
Atterberg Limits			
Liquid Limit	57.8%	69.0%	51.3%
Plastic Limit	39.5%	40.1%	32.1%
Plasticity Index	18.3%	28.9%	19.2%
Specific Gravity	2.80	2.80	2.67

Grain Size Analysis

Grain size distribution curves in Figure 23 show that remolded soil has a greater percentage of fine particles than does unremolded soil. This greater percentage of fines indicates that remolding by mechanical agents causes disaggregation of the friable granular soil aggregates. In a separate study it was found that mixing of the unremolded soil for 15 minutes in an electric mixing cup as is normally specified produced a grain size curve analogous to that of the remolded soil. This result emphasizes the friability of these soils and the need for test modifications.

A comparison between the grain size curves for the unremolded soil in the natural and sesquioxide free conditions shows a considerable increase in finer sizes after removal of the sesquioxides. This increase confirms that the soil in its natural state consists of clay sized particles bonded into microaggregates by sesquioxide cementing agents. Similar results have been reported by Winterkorn (83) and Pearing (56), who showed increases in clay size fractions and corresponding decreases in sand and silt size fractions after removal of the sesquioxides from various lateritic soils.

A comparison of the grain size curves for remolded soil in the natural and sesquioxide free conditions reveals that removal of the sesquioxides further increases the amount of fines in the soil. This increase suggests that the iron and aluminum oxides continue to coat and to bond the soil particles. Thus, the remolding phenomenon merely produces smaller microaggregates of iron impregnated clays.

Specific gravity determinations which were required in order to evaluate the hydrometer analyses are presented in Table V.

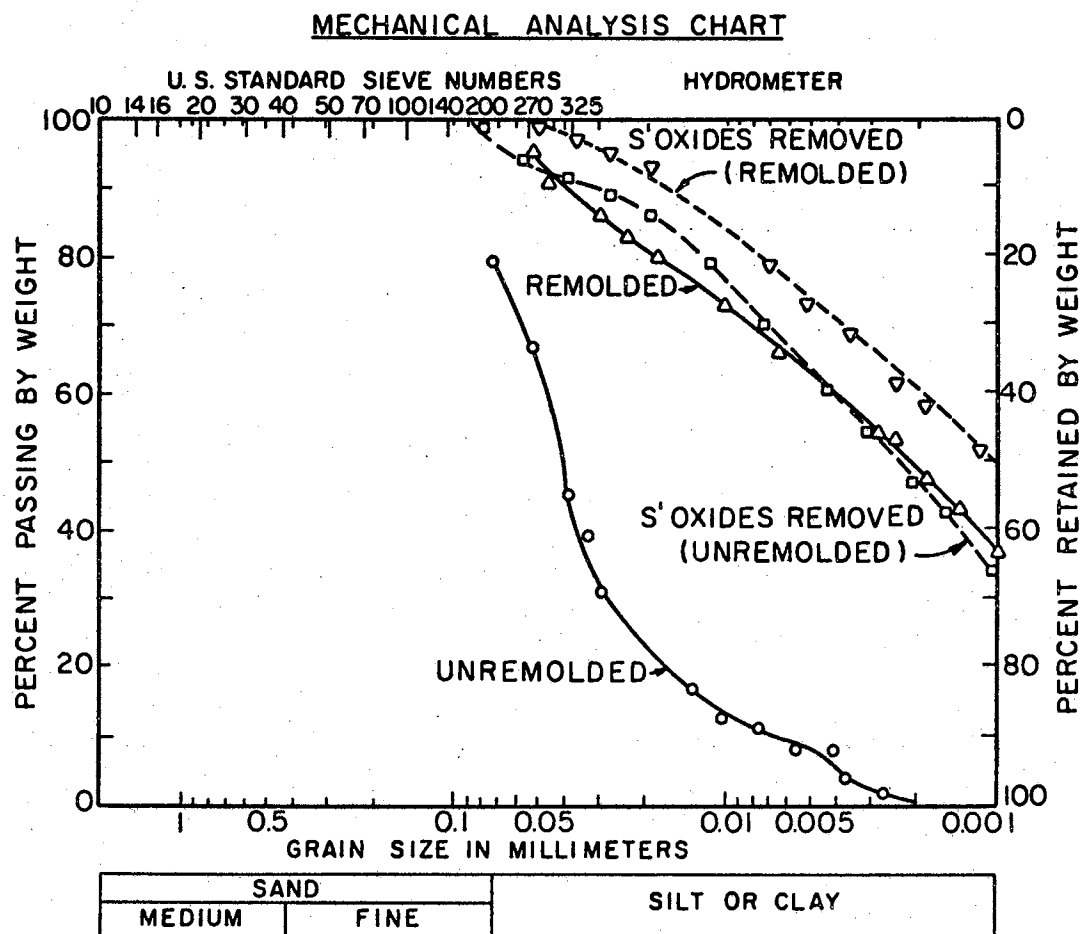


Figure 23. Effect of Sesquioxide Removal on the Grain Size of Lateritic Soil

The results show that removal of the heavy sesquioxides lowers the specific gravity of the soil.

Suggested Test Procedures

Due to the effects of remolding and heating on the index properties of lateritic soils, the following procedures are suggested as a means for providing a more reliable indication of the physical properties of these soils.

Atterberg Limits. Preliminary studies indicate that comparisons between the liquid limits of (a) air-dried unremolded, (b) air-dried remolded, and (c) oven-dried unremolded samples provide an acceptable means of evaluating the susceptibility of the soil to remolding and/or establishing the presence of temperature sensitive colloids. Because the liquid limit is more sensitive to changes in the soil than is the plastic limit, comparisons involving only the liquid limit rather than the plasticity index are recommended. However, the variability of the liquid limit due to remolding hampers the achievement of consistent determinations of this value. To offset this effect the One-Point Liquid Limit method is suggested. A comparison between the liquid limits of natural relatively undisturbed soil (unremolded) and soil which has been compacted to standard Proctor density at optimum moisture content (remolded) should furnish some insight as to the amount of granular breakdown of the soil anticipated under field conditions. A significant increase in the liquid limit with compaction would indicate that the friable microaggregates of the soil are unable to withstand the rigors of remolding by construction equipment. This increase

would indicate that evaluation of the Atterberg limits of the soil in a more finely divided state is justified.

The presence of halloysite and/or allophanic materials can be determined by comparing the liquid limit of air-dried unremolded soil with that of oven-dried soil. A significant decrease in liquid limit suggests the presence of these temperature sensitive colloids.

Grain Size Analysis. The adoption of a standardized mixing time, e.g., 5 minutes, in an electric mixer should provide some continuity to results reported in the literature, and provide a basis for comparing the friability and water stability of the soil aggregates. The proper selection of dispersing agent is also critical, as varying results have been reported (75) dependent upon the chemical used. A sodium salt, i.e., sodium metahexaphosphate (Calgon), or Na_2CO_3 , adjusted to a pH of eight to nine is suggested.

CHAPTER VI

ENGINEERING PROPERTIES

Introduction

Generally, lateritic soils possess physical properties indicative of a clayey material, yet because of their granular structure they often do not behave as clays. Frequently, judgement based upon standard index properties and classification schemes results in the mistaken conclusion that lateritic soils are troublesome and should be avoided as construction materials. Such a generalization is incorrect as in situ laterites, because of their granular structure, possess high internal permeabilities, moderate bearing capacities, medium plasticities, and low volume change characteristics. However, in the presence of moisture the pulverizing action of heavy construction equipment causes these soils to undergo a transformation. Remolding breaks down the friable granular structure of the soil transforming it into a highly plastic material with low bearing capacities and low permeabilities.

The purpose of this phase of the investigation was to evaluate the effects of remolding and sesquioxide removal on the shear strength parameters and the lime susceptibility of the soil. Although remolding by construction equipment has been recognized as a troublesome aspect of these soils, little or no information was available in the literature concerning this effect on shear strength of these soils. The

sesquioxides were removed as an alternative means of breaking down the granular structure of the soil, i.e., chemical instead of mechanical remolding. It was anticipated that this alternative procedure would provide additional information concerning the effects of remolding on the shear strength of the soil. In previous studies (78, 79) it was shown that remolding affected the response of the soil to lime stabilization. At that time, it was suggested that the sesquioxide coatings on the clay minerals prevented the clays from entering into the stabilizing reactions. Removal of the sesquioxides should provide additional data concerning this aspect of selecting favorable additives for stabilization of these soils.

Review of the Literature

Compaction Characteristics

One of the principal problems associated with lateritic soils is the remolding phenomenon that occurs during manipulation by heavy construction equipment. Experience has shown that primitive manual compaction, which minimizes remolding of the material, has resulted in more stable bases and subgrades for airfield runways than compaction of the same soil by heavy equipment (83). Experience has also shown that, during the rainy season, the natural moisture content of lateritic soils often exists slightly above the plastic limit. For this reason, moisture contents of these soils must be carefully controlled during compaction or increased plasticity produced by remolding will severely restrict proper equipment utilization.

Bawa (5) stated that, in general, relatively high compacted densities could be expected in these soils due to the high specific gravity

of the solids. However, as shown in Table VI standard Proctor densities of lateritic soils can be quite low in spite of the high specific gravities of the soil components. This abnormality is attributed to the clay microaggregates which provide a granular nature to the soil and thus lower its compacted densities.

Shear Strength Parameters, ϕ and C

The characteristic granular structure of lateritic soils is likely to have a two-fold influence in increasing the shear strength of these soils. First, the porous structure of the soil under most conditions will retain some air between and within the clayey clusters. Because of these air pockets, the undrained rate of increase of the pore water pressure with increase in load will be small. This circumstance is quite different from that of saturated clays where the increase in undrained pore pressure may equal the increase in applied stress. Because an increase in pore pressure reduces the effective stress, the influence of aggregation would be that of reducing these pressures and increasing the strength of the soil (13). Secondly, the aggregated structure provides greater mechanical interlocking of the soil particles than would be produced by a platy structure.

Example shear strength parameters are presented in Table VII. Considerable difficulty was experienced in extrapolating the data presented in Table VII from the literature. In addition to the usual problems associated with the nomenclature used to describe lateritic soils, the specifics as to the exact type and conditions of testing for evaluating ϕ and C were generally absent in the reference material. Nevertheless, the high friction angles (ϕ' values of 30° - 40°) reflect

TABLE VI
DENSITY AND MOISTURE CONTENTS OF LATERITIC SOILS

Soil Type and Location	Gs	Dry Density	Opt M.C.	PL	Source
Lateritic Soil Matanzas, Cuba	2.90	88 pcf	30%	31.2%	Winterkorn (83)
Lateritic Soil Sasamua, Kenya	2.83	79 pcf	50%	54%	Terzaghi (75)
Residual Red Clay Brazil	2.68	81-90 pcf	30%	31%	Grizienski (28)
Lateritic Soil Panama Canal Zone	2.80	84.5 pcf	35%	39.5%	Townsend (80)

TABLE VII
SHEAR STRENGTH PARAMETERS, ϕ AND C, FOR
COMPACTED LATERITIC SOILS

Soil Type and Location	ϕ	C	Source
Unconsolidated/Undrained - Q Test			
Pisolitic Lateritic Soil Uganda, Africa	23°	3.25 kg/cm ²	Nixon & Skipp (55)
Gneissic Residual Clay Limoeiro, Brazil	15°	0.6 kg/cm ²	Teixeira da Cruz (74)
Basaltic Residual Clay Bariri, Brazil	19°	1.6 kg/cm ²	Teixeira da Cruz (74)
Basaltic Residual Clay Jupia, Brazil	20°	0.8 kg/cm ²	Teixeira da Cruz (74)
Consolidated/Undrained - R Test (Saturated)			
Granitic Residual Clay Brazil	17.5°	0.6 kg/cm ²	Vargas (81)
Red Lateritic Soil Alaotra, Madagascar	20° - 27°	0.1 - 0.2 kg/cm ²	Agnes and Soeiro (1)
Consolidated/Drained - S Test or Consolidated/Undrained - \bar{R} Test			
	ϕ'	C'	
Hydrated Lithomarge Sasamua, Kenya	34°	0.27 - 0.31 kg/cm ²	Terzaghi (75)
Granitic Residual Clay Brazil	31°	0	Vargas (81)
Weathered Halloysitic Clayey Gabbro Guma, Sierra Leone	32° - 39°	0.34 - 0.50 kg/cm ²	Dixon (17)
Tropically Weathered Soils Hong Kong & Malaya	30° - 39°	0.20 - 0.30 kg/cm ²	Little (42)
Gneissic Residual Clay Euclides da Cunha, Brazil	26°	0.40 kg/cm ²	Teixeira da Cruz (74)
Basaltic Residual Clay Bariri, Brazil	29°	0.40 kg/cm ²	Teixeira da Cruz (74)

the granular nature of lateritic soils. For purposes of comparison, silty clays exhibit ϕ' values around 28° , and ϕ' values for silty sands may range from 30° to 37° (50).

Stabilization

Lateritic soils have been stabilized by using various additives, e.g., lime, portland cement, asphalt, various chemicals, and sand. Because of the widely varying nature of lateritic soils, none of these additives are universally successful. Therefore, laboratory investigations are essential prior to any extensive stabilization project (55).

Results obtained from the use of lime as a stabilizing additive are quite inconsistent as shown in Table VIII. Some lateritic soils respond favorably to the addition of lime by showing strength gains and reductions in plasticity; whereas, other lateritic soils cannot be effectively stabilized with lime. Winterkorn (83) reported complete failures with high percentages of lime, 8-18 percent, yet Schofield (68) describes a successful lime stabilization project of a lateritic soil in Nyasaland where 3 percent lime reduced the plasticity, and 5 percent lime was used as a stabilizer. Biszewski (7) also used lime as an effective means of increasing the strength of a lateritic soil. He found that for maximum strength gains the soil-lime mixture should be compacted within 48 hours after addition of the lime. In previous work (78, 79) it was revealed that remolding and method of compaction can produce significant variations in achieved strength values.

In general, the beneficial effects of adding lime to a soil include a reduction in plasticity and substantial strength increases with time. The reduction in plasticity, which improves the overall

TABLE VIII

EFFECT OF LIME STABILIZATION ON THE UNCONFINED COMPRESSIVE
STRENGTHS OF LATERITIC SOILS

Soil Type and Location	% Lime	Curing Age (Days)	U/C Strength (psi)	Source
Red Clay Soil Kabete, Kenya	5%	7 Days	130	Newill (54)
		28 Days	130	
		21 Days Curing + 7 Days Soak	70	
	10%	7 Days	205	
		28 Days	340	
		21 Days Curing + 7 Days Soak	255	
Red Clay Soil Sasamua, Kenya	5%	7 Days	75	
		28 Days	80	
		21 Days Curing + 7 Days Soak	50	
	10%	7 Days	195	
		28 Days	220	
Bluefields Clay (Lateritic) Panama Canal Zone	0%	2 Days	12	Wissa (84)
	8%	2 Days	35	
		7 Days (Soak)	27	
	8% CaO + 1% MgSO ₄	2 Days	121	

TABLE VIII (Continued)

Soil Type and Location	% Lime	Curing Age (Days)	U/C Strength (psi)	Source
Red Granulated Lateritic Soil Matanzas, Cuba	8 - 18%	wet and dry	Slaked and Failed	Winterkorn (83)
Lateritic Soil Panama	5%	12 Days 28 Days 60 Days	38 75 80	Townsend (80)
	10%	7 Days 28 Days 60 Days	27 100 195	Toukan (78)
	20%	7 Days 28 Days 60 Days	28 56 100	
Lateritic Clayey-Sand Bien Hoa, Vietnam	4%	7 Days 28 Days	14 9	Post (60)
Lateritic Clay Nakhon Phanom, Vietnam	6%	7 Days 28 Days	24 16	
Lateritic Clay Phan Rang, Vietnam	4%	7 Days 28 Days	3 5	
Lateritic Clayey Gravel Uganda	3% 5%	2 Days 2 Days	85 110	Biszewski (7)

workability of the soil, is termed lime modification, while the substantial strength increases are termed lime stabilization. These improvements to the soil are generally attributed to four basic reactions: (a) cation exchange, (b) flocculation and agglomeration, (c) carbonation, and (d) pozzolanic reactions.

The reactions of cation exchange and flocculation and agglomeration, particularly the latter, are primarily responsible for the reductions in plasticity and increased workability of the soil. The quantity of lime, generally a small amount, required to achieve these changes and reduce the plasticity to zero has been termed the "lime fixation percentage" (32). Although controversial, it has been shown that additional lime in excess of the lime fixation percentage must be incorporated with the soil to provide any significant strength gains. These stabilizing strength gains are primarily attributed to pozzolanic reactions. Lime carbonation to form calcium carbonate (limestone), a weak cementing agent, may also contribute minor amounts to the development of increased strength.

As lime, water, and soil are combined in a mixture, the pH of the water increases rapidly due to the partial dissociation of calcium hydroxide from the lime until a pH of 12.3 is reached. In this highly basic medium, silica increases in solubility (400 ppm at pH 10 to 5000 ppm at pH 11). The negative charge possessed by the clay particles also increases greatly in this alkaline medium (18). The opposing charges cause the highly electropositive calcium ions to be readily adsorbed by the clay particles. This action neutralizes the clays permitting closer contact until flocculation occurs (33). These flocs may be further held together by limited chemical reaction products at

the points of contact between the clays. This flocculation of the soil colloids rapidly reduces the plasticity of the soil. Concurrently, provided a high pH and the presence of sufficient calcium is maintained, pozzolanic reactions slowly occur. These reactions involve the formation of calcium silicate hydrates (CSH) and/or calcium aluminate hydrates (CAH). The severe attack on the silica tetrahedral by the highly alkaline water partially decomposes the clay mineral structure liberating silica and alumina for the formation of these compounds. Initially, the CSH and CAH products exist as gel-like insoluble cementing agents. However, following compaction which forces the soil aggregates closer together, these reaction products slowly gain in crystallinity and eventually interaggregate cementation stabilizes the mass.

Investigative Procedures

Sample Preparation

Unremolded, remolded (see Chapter III), and sesquioxide free (see Appendix A) soil which passed the No. 10 U. S. Standard sieve was used for compaction, shear strength, and lime stabilization testing.

Sufficient moisture to achieve the optimum moisture content was added to the various soil samples prior to compaction of the test specimens. The moist soil was placed in pans, sealed in a plastic bag, and allowed to sit undisturbed for 24 hours. This procedure provided a uniform distribution of the added moisture throughout the soil. The unremolded and remolded soils exhibited water inert tendencies, and a fairly uniform moisture content was obtained for both soils after

24 hours without any mixing. However, the sesquioxide free soil readily adsorbed the added moisture, forming "clay balls," and considerable mixing was required to achieve a uniform moisture content.

Since lime stabilization requires large quantities of water to serve as a medium for cationic exchange and clay-lime reactions, the following mixing procedure was used for preparing the soil-lime mixtures. The lime used for these treatments was a pelletized quicklime (97.5% CaO).

- (1) The soil and lime were mixed by hand and spread at a depth of 2 to 3 inches in a pan.
- (2) A predetermined amount of water, which was sufficient to raise the moisture content to 50 percent (approximately midway between the plastic and liquid limits), was sprinkled over the mixture.
- (3) The mixture was then allowed to air-dry for four days until the moisture content was below the optimum value.
- (4) The dry mixture was gently stirred to break up any large soil-lime agglomerations and sieved through a No. 10 U. S. Standard sieve.
- (5) The mixture was then brought to the optimum moisture content as described in the previous paragraph.

All test specimens were compacted in a Harvard Miniature compaction mold. Approximately 110 grams of moist soil were compacted in three layers by a drop hammer weighing 0.825 pounds with a face diameter of 0.70 inches. By using a drop height of 6 inches, this equipment approximates Standard Proctor compactive effort (91). Twenty-five blows per layer were used to compact all triaxial test

specimens. To compare objectively the strength values of various soil-lime mixtures, it was necessary that all specimens be compacted to the same density and possess relatively the same particle orientations. Based upon previous studies (80) of compactive effort versus density, the soil-lime specimens were compacted to a density of 82.5 pcf. After compaction, the specimens were wrapped in Saran Wrap to prevent any moisture loss. The wrapped lime stabilized specimens were waxed and placed in a moist room to cure.

Methods and Equipment

Triaxial Testing. The shear strength parameters, ϕ and C , were evaluated for the compacted soil in the saturated and unsaturated conditions. The quick test (unconsolidated/undrained or Q) was used to evaluate the soil in an unsaturated condition, while the consolidated-quick test (consolidated/undrained or R) was utilized to evaluate the saturated soil.

Quick tests were conducted on soil specimens at the optimum moisture content by using confining pressures of 0, 0.5, 1.0, and 1.5 kg/cm² with compressed air as the confining medium. These low confining pressures were used to minimize possible consolidation and, hence, increased saturation. Since Q tests on saturated clays show that $\phi = 0^\circ$ and $C = \frac{1}{2}$ unconfined compressive strength, the samples were not saturated and no pore pressure measurements were made. The rate of strain used for these tests was 0.05 inches/minute.

Consolidated-quick tests were conducted on soil specimens which had been saturated using a back pressure saturation system employing a pressure of 3.0 kg/cm² for 36 to 48 hours. During this saturation

interval an equal confining pressure of 3.0 kg/cm^2 was supplied through a confining medium of water to prevent any volume change in the specimen. After saturation, the back pressure was held constant while the confining pressure was increased to the appropriate consolidation pressure. The consolidation pressures ranged from 0.5 to 2.0 kg/cm^2 (actual confining pressures of 3.5 to 5.0 kg/cm^2 less back pressure of 3.0 kg/cm^2). The amount of volume change during consolidation was considered to be equal to the volume of pore fluid displaced. The volume of displaced pore fluid was measured by a burette. The change in height of the specimen was determined by measuring the height of the loading head prior to and after consolidation. Consolidation was considered to be completed when no further increase in displaced pore fluid was observed in the burette (usually 3 days). Pore pressure measurements were made using an Anteus pore pressure relay system which permitted an effective stress measurement (R test). A schematic diagram depicting the set-up employed for R and \bar{R} tests is shown in Figure 24. A very slow rate of deformation of 0.001 inches/minute was used to minimize any localized pore pressure build-up.

For all tests the load was mechanically applied by an electrically driven Karol Warner compression machine (Model 550). A Lanzi (Test Lab) triaxial cell was the triaxial apparatus utilized for testing. Two rubber membranes of 1.40 inches ID and 0.025 inch wall thickness were used. Peak stresses were selected as failure criteria for establishing strength envelopes. Moisture contents of the broken specimens were determined and revealed that testing of the R and \bar{R} tests was conducted at 100 percent saturation and at optimum moisture for the Q tests.

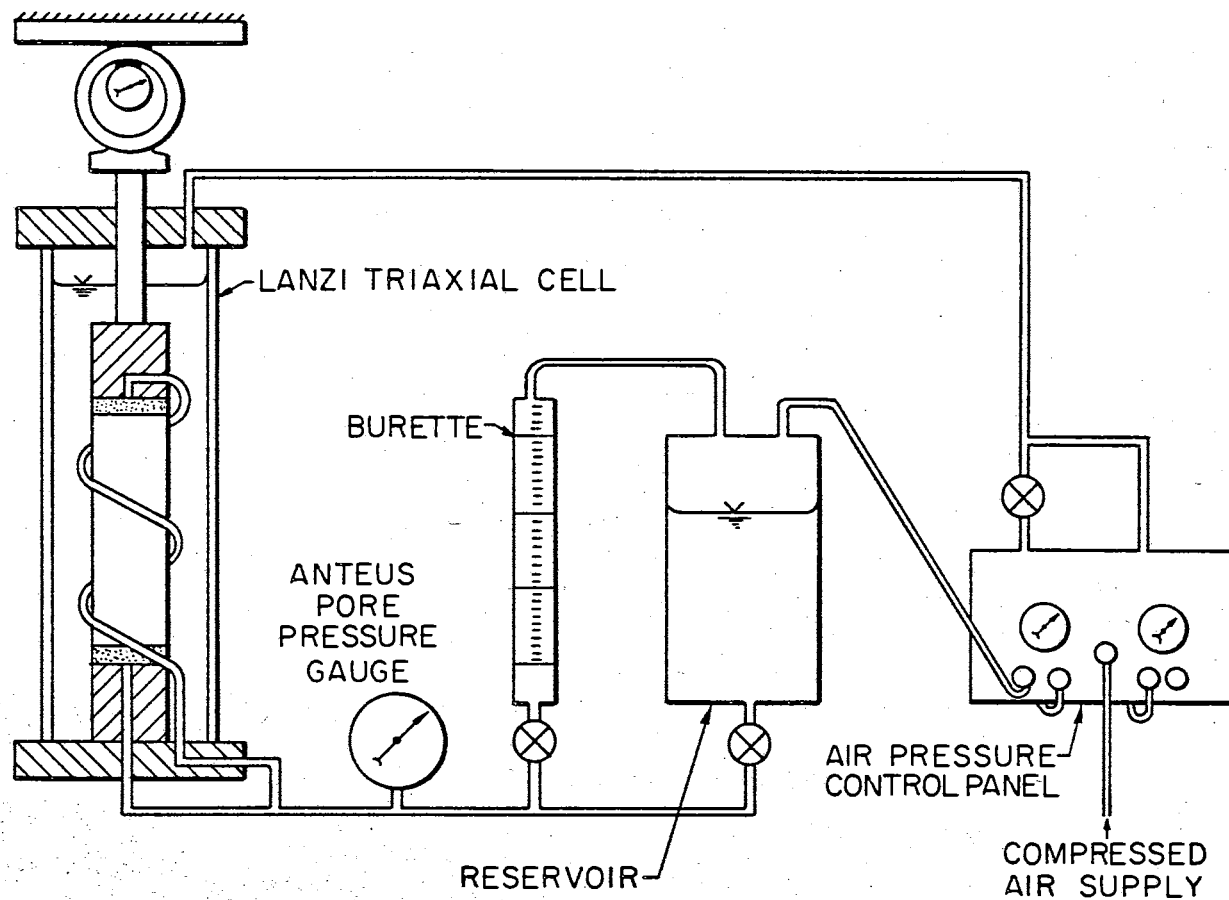


Figure 24. Schematic Diagram of Triaxial Compression Apparatus for R and \bar{R} Tests

Lime Treatment Evaluations. After the specified curing time, the soil-lime specimens were stripped of their was coatings, and their unconfined compressive strengths were evaluated. The tests were conducted at a constant deformation rate of 0.05 inches/minute on the Karol Warner compression machine. The reported results are the average of three tests. The peak stress was chosen to represent failure.

The electron micrographs of the soil-lime mixtures were prepared by Mrs. Linda Hare of The University of Oklahoma School of Geology Laboratory with a JEOLCO JSM - 2 scanning electron microscope. Prior to scanning, the samples were coated by vaporizing a gold-palladium alloy metal in a JEOLCO JEE -4C vacuum evaporator.

Experimental Results and Discussion

Compaction

The density values determined by miniature Standard Proctor compaction are listed in Table IX. These values show that despite the high content of iron and aluminum oxides, the density of the soil is quite low in comparison to normal clays of temperate regions with similar Atterberg limits. For example, Permian red clay from Stillwater, Oklahoma, with a specific gravity of 2.7 has a plasticity index of 22 percent and a Standard Proctor density of 106 pcf.

Apparently, remolding has little effect on the Standard Proctor density and optimum moisture content of the compacted soil. However, extraction of the sesquioxides results in an increase in density. This increase is interpreted as being due to the chemical breakdown of the granular structure, which allows a closer packing of the clayey particles and a lower void ratio. Although the heavy sesquioxides

TABLE IX
STANDARD PROCTOR DENSITIES
OF LATERITIC SOIL

Soil Condition	Dry Density	Opt M.C.	PL
Unremolded	84.5 pcf	35.0%	39.5%
Remolded	83.0 pcf	34.5%	40.0%
Sesquioxide Free	88.0 pcf	29.5%	32, 1%

were removed, the lower void ratio permits a greater density of the soil.

Normally, the optimum moisture content is several percentage points below the plastic limit (PL). The data presented in Table IX are consistent with this observation. In the previous chapter (see Chapter V), the decrease in plastic limit of the iron free soil was interpreted as being due to a alteration of the allophanic colloids by the extraction procedure. In a similar manner, the decrease in optimum moisture content after removal of the sesquioxides also results from changes in these soil colloids.

A comparison of the optimum moisture content and plastic limit indicates that if compaction occurred on the wet side of optimum, probably the soil would become highly plastic and complicate field compaction with heavy equipment.

Shear Strength

The results of the various triaxial compression tests are listed in Table X. The strength envelopes for these tests are presented in Figures 25 through 33. In all tests fairly high shear strength parameters were obtained, which indicates that despite the low density and high plasticity of this soil in a compacted state both the unre-molded and remolded soil possess satisfactory strength characteristics. The magnitude of the ϕ values, particularly those for the \bar{R} tests, seem to reflect the inherent granular nature of this soil.

Apparently mechanical remolding has little effect on shear strength parameters of the compacted soil. However, for Q tests the remolded soil had a slightly lower ϕ value and greater cohesion than

TABLE X
SUMMARY OF TRIAXIAL COMPRESSION TESTS
OF A LATERITIC SOIL

Soil Condition	σ_3 kg/cm	U kg/cm	$\bar{\sigma}_3$ kg/cm	$\sigma_1 - \sigma_3$ kg/cm	Strength Parameters
Unconsolidated/Undrained-Q					
Unremolded	0.0			1.40	$\phi = 26.0^\circ$ $C = .42 \text{ kg/cm}^2$
	0.5			2.07	
	0.75			2.55	
	1.50			2.96	
Remolded	0.0			1.68	$\phi = 24^\circ$ $C = .56 \text{ kg/cm}^2$
	0.5			2.50	
	1.0			3.05	
	1.75			2.91	
Sesquioxide Free	0.0			2.34	$\phi = 11^\circ$ $C = .91 \text{ kg/cm}^2$
	0.5			2.21	
	1.0			2.85	
Consolidated/Undrained-R					
Unremolded	0.0			0.893	$\phi = 17^\circ$ $C = .40 \text{ kg/cm}^2$
	1.0			1.893	
	2.0			2.76	
Remolded	0.75			1.145	$\phi = 21^\circ$ $C = .15 \text{ kg/cm}^2$
	1.00			1.592	
	1.50			1.879	
	1.75			2.337	
Sesquioxide Free	1.0			1.216	$\phi = 22^\circ$ $C = .05 \text{ kg/cm}^2$
	1.5			1.832	
	2.0			2.890	
Consolidated/Undrained (with pore pressure measurements)-R					
Unremolded	1.0	0.387	0.613	1.893	$\phi = 38^\circ$
	2.0	1.125	0.875	2.760	
Remolded	0.50	-0.07	0.57	1.732	$\phi = 37^\circ$
	0.75	0.34	0.40	1.145	
	1.75	0.95	0.80	2.337	
	2.00	0.53	1.47	4.372	
Sesquioxide Free	1.50	0.66	0.84	1.832	$\phi = 32^\circ$
	2.00	0.74	1.26	2.890	

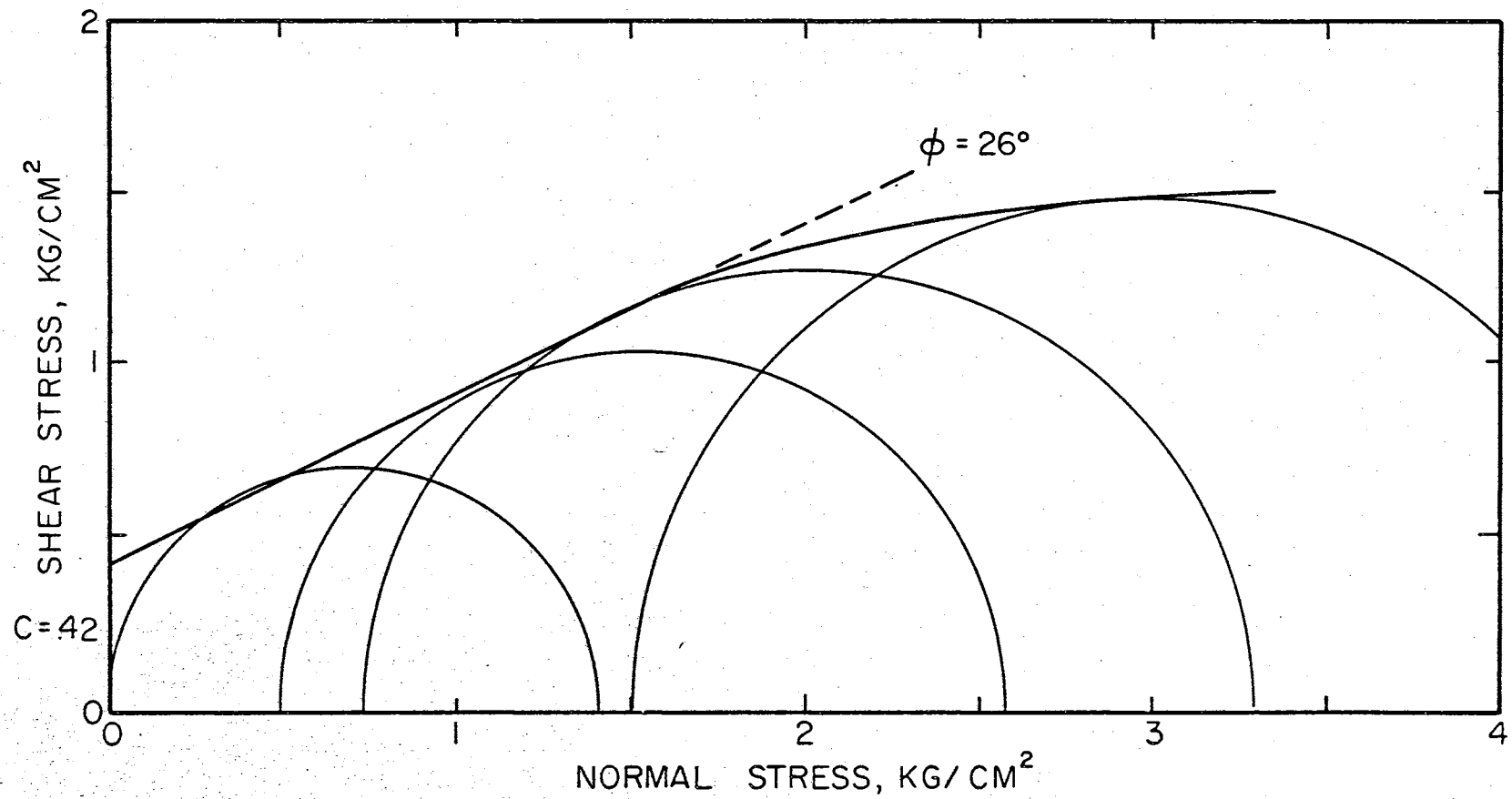


Figure 25. Strength Envelope for Quick Test on Unremolded Lateritic Soil

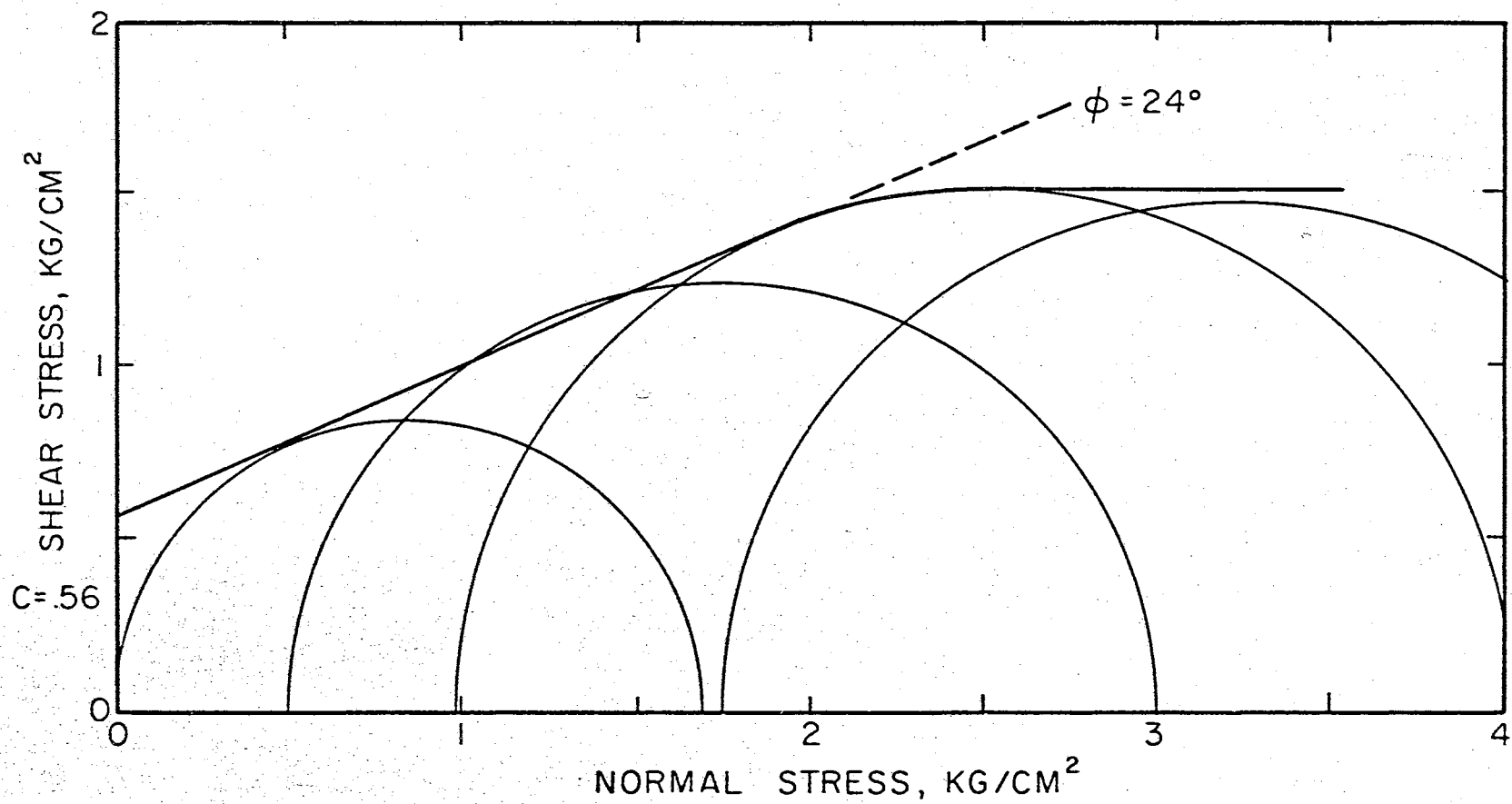


Figure 26. Strength Envelope for Quick Test on Remolded Lateritic Soil

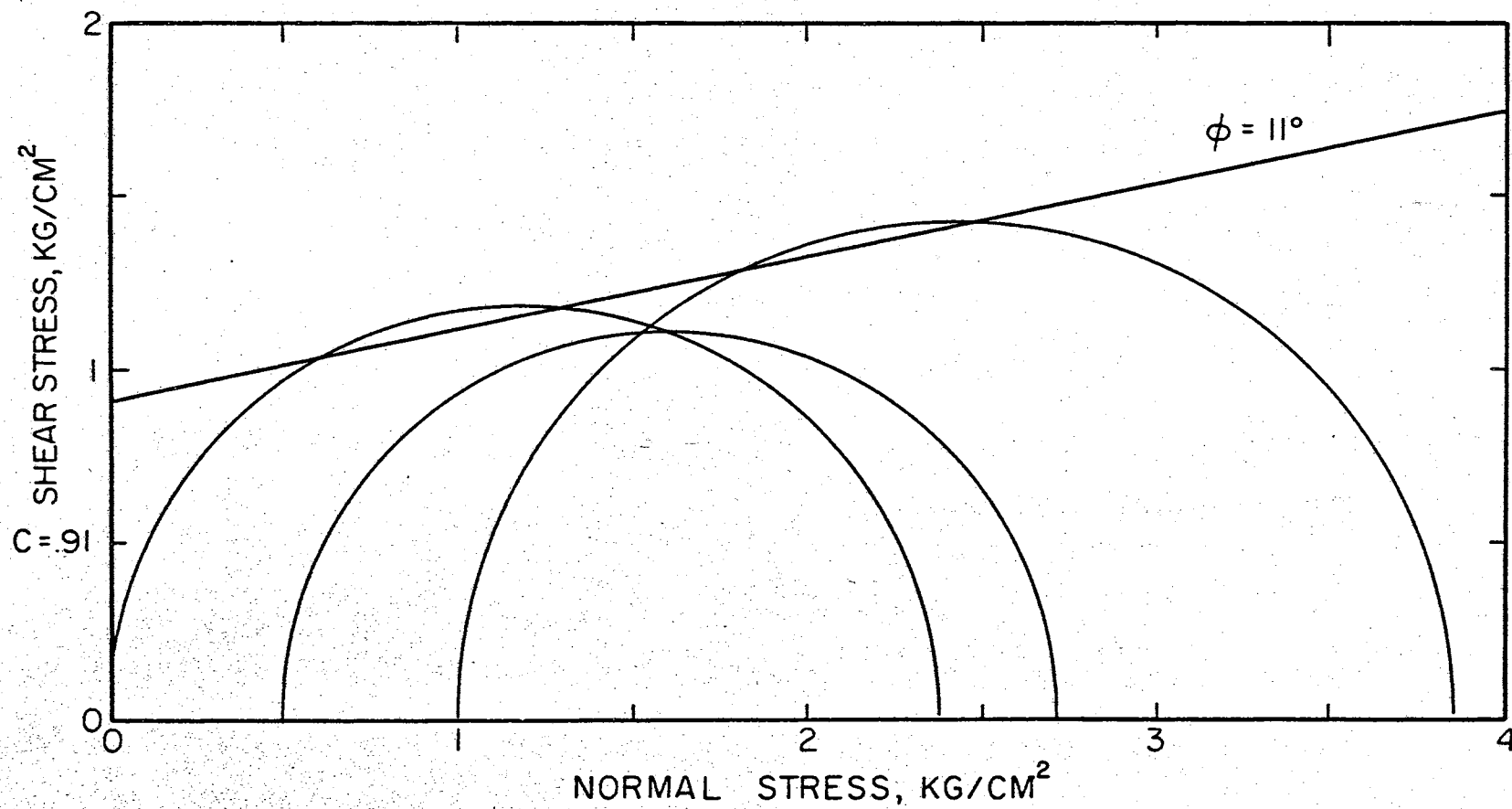


Figure 27. Strength Envelope for Quick Test on Sesquioxide Free Lateritic Soil

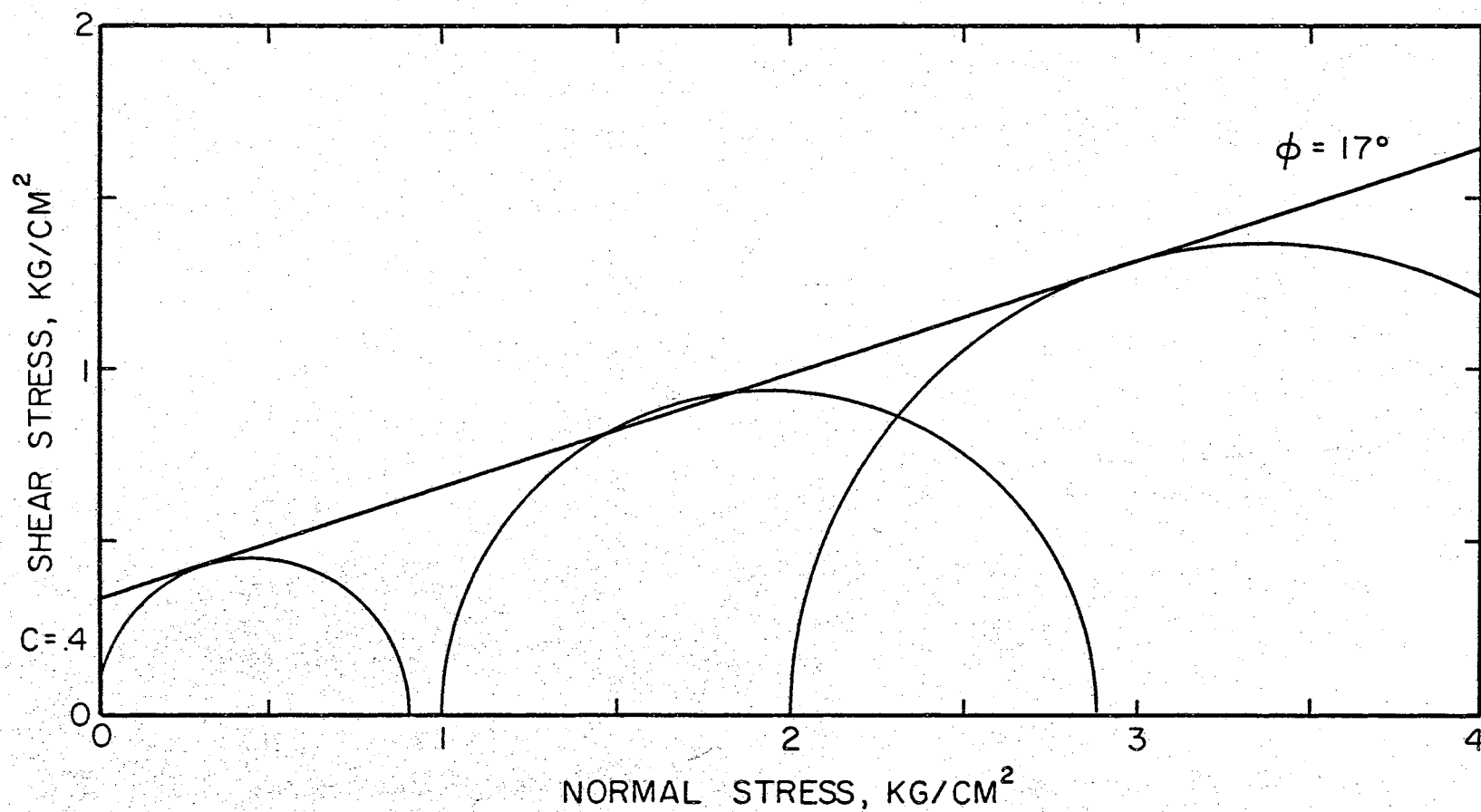


Figure 28. Strength Envelope for Consolidated-Quick (R) Test on Unremolded Lateritic Soil

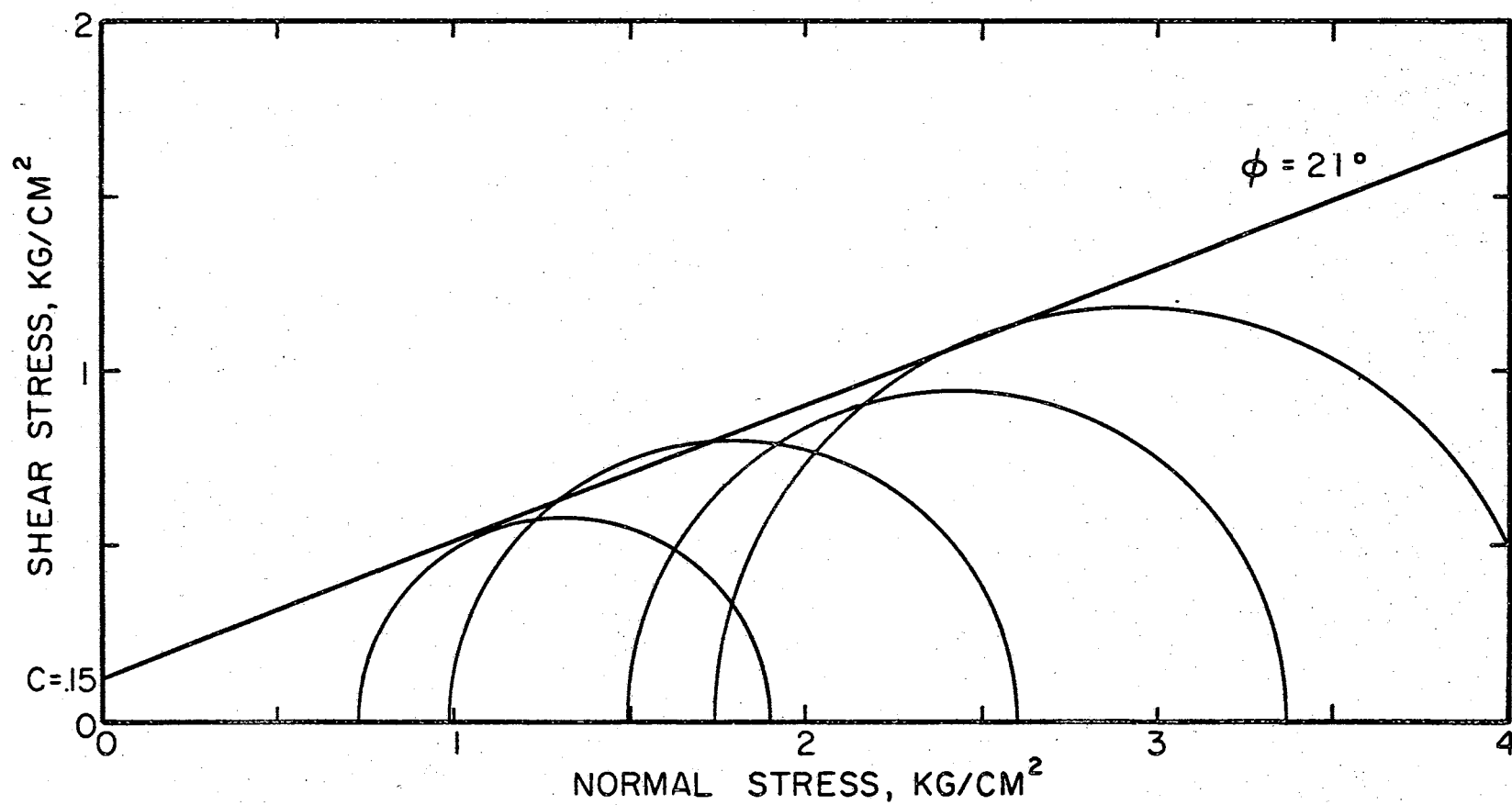


Figure 29. Strength Envelope for Consolidated-Quick (R) Test on Remolded Lateritic Soil

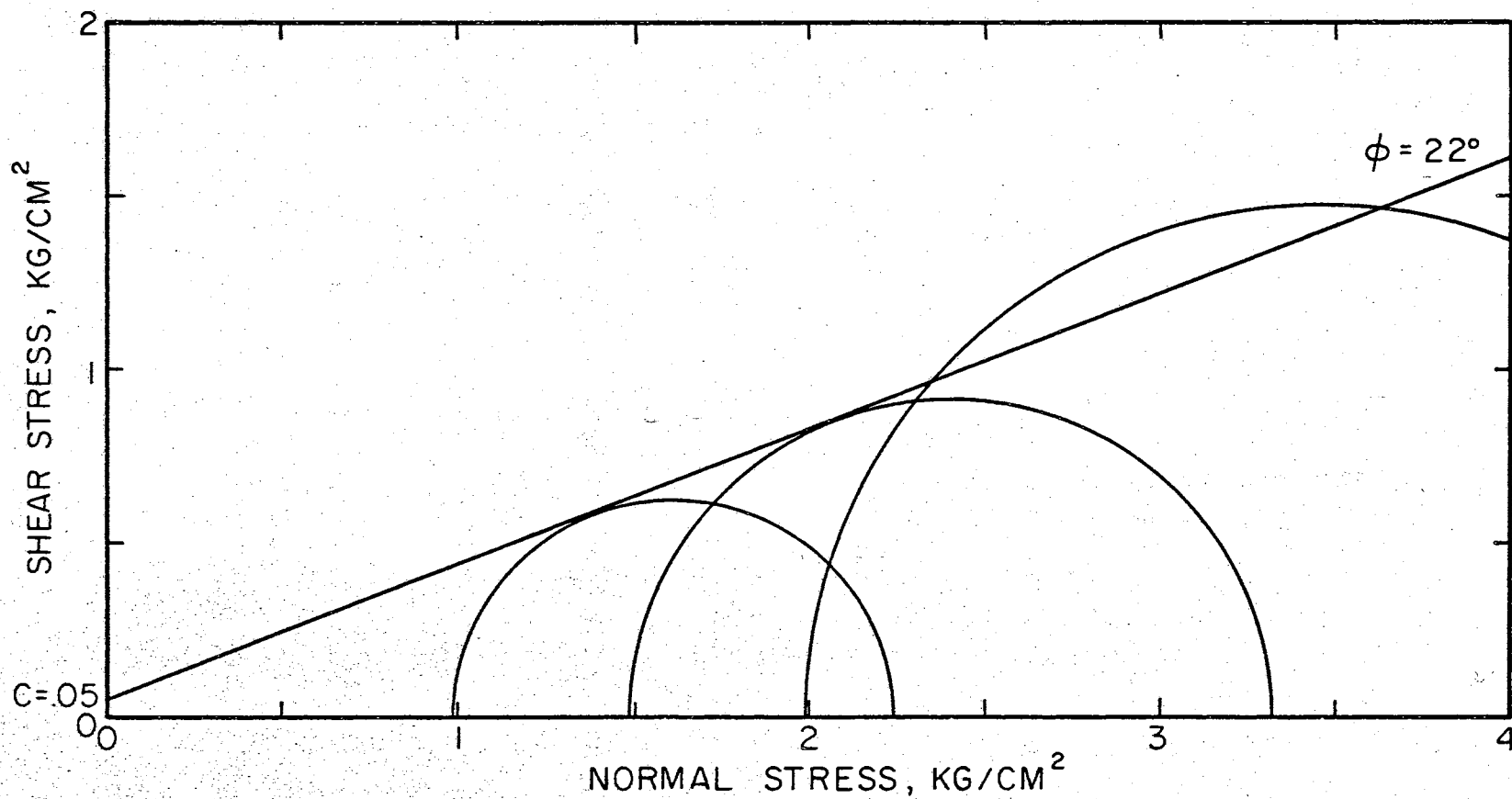


Figure 30. Strength Envelope for Consolidated-Quick (R) Test on Sesquioxide Free Lateric Soil

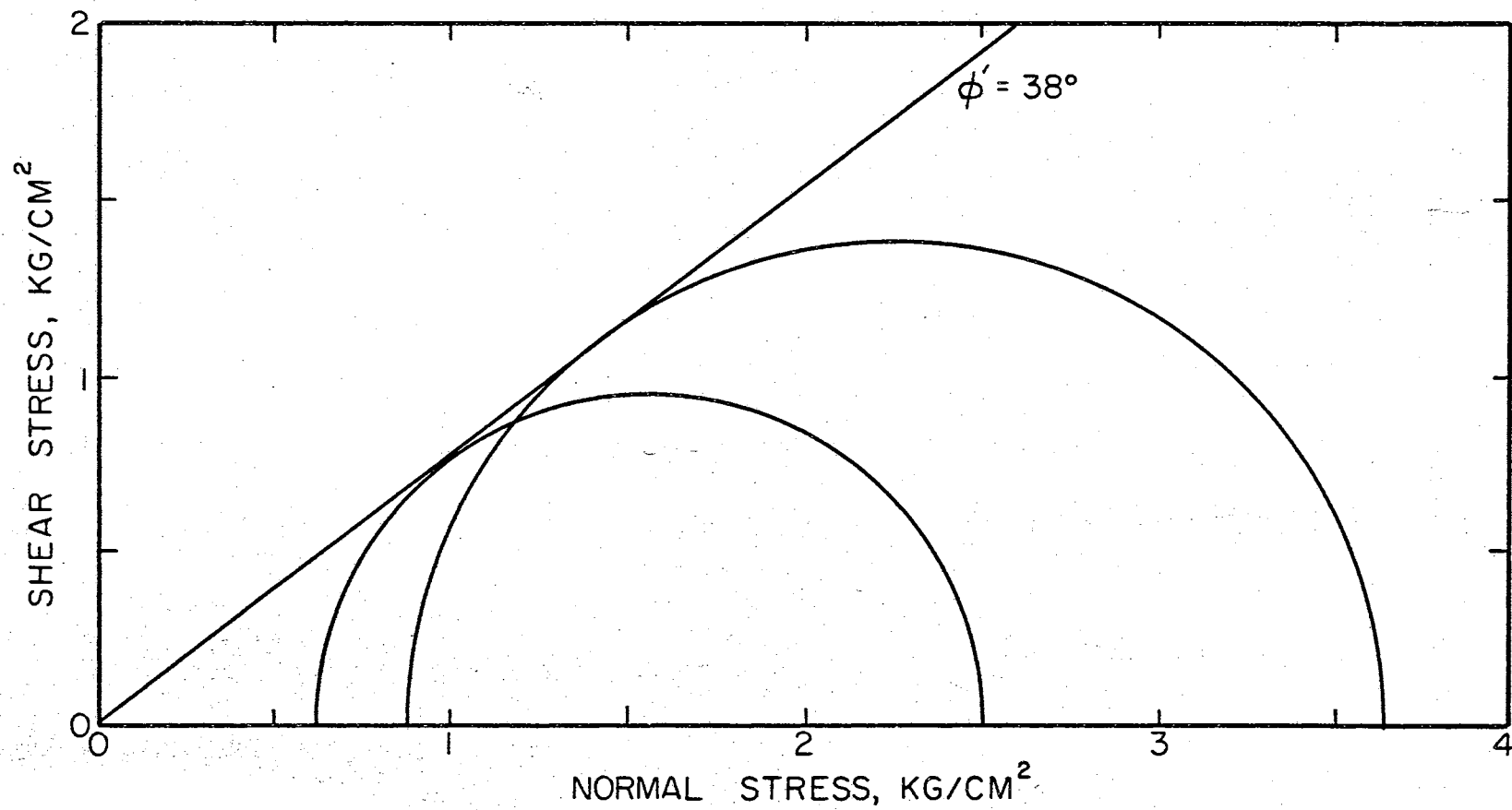


Figure 31. "Effective" Strength Envelope for Consolidated-Quick (\bar{R}) Test on Unremolded Lateritic Soil

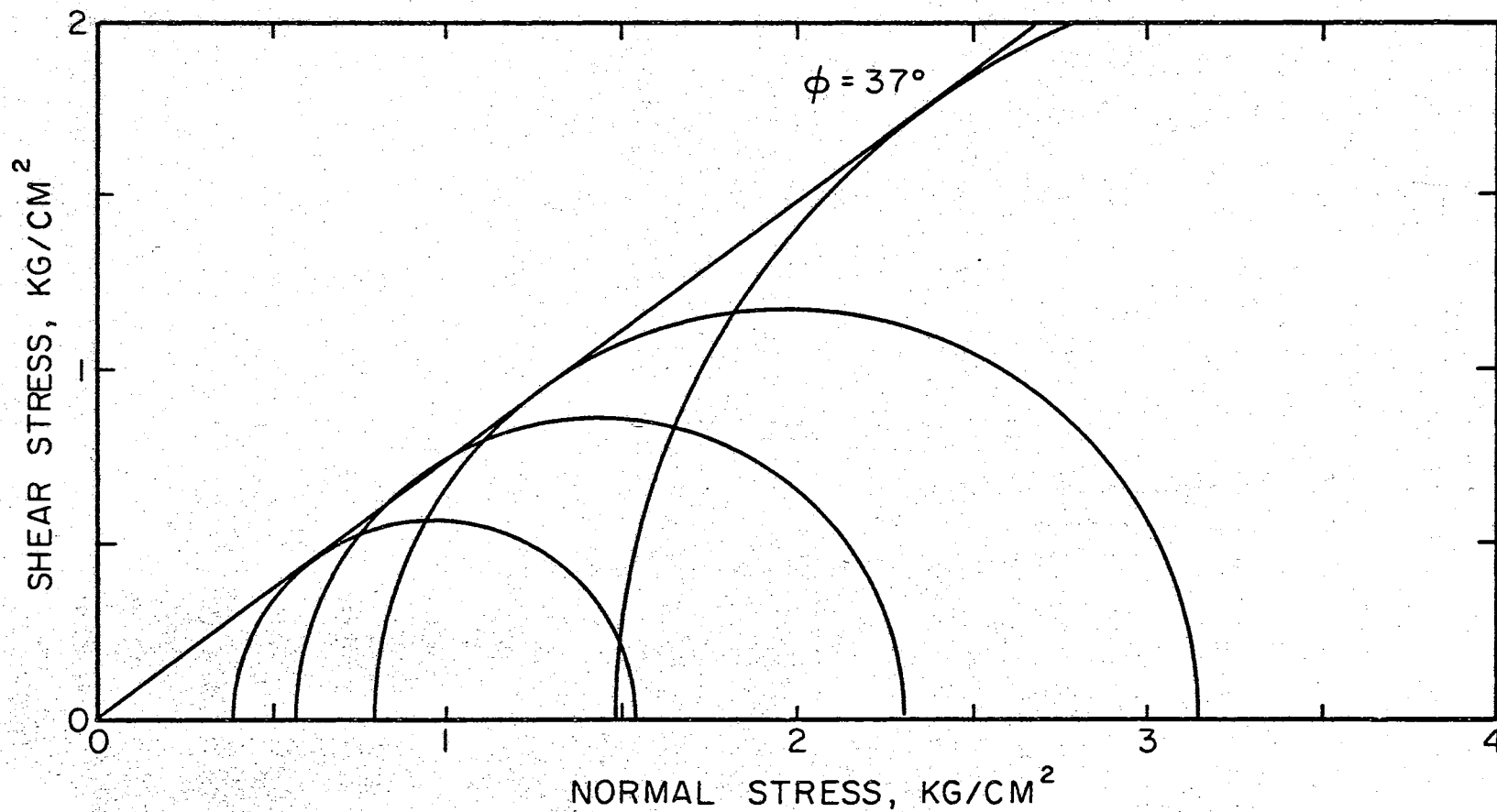


Figure 32. "Effective" Strength Envelope for Consolidated-Quick (\bar{R}) Test on Remolded Lateritic Soil

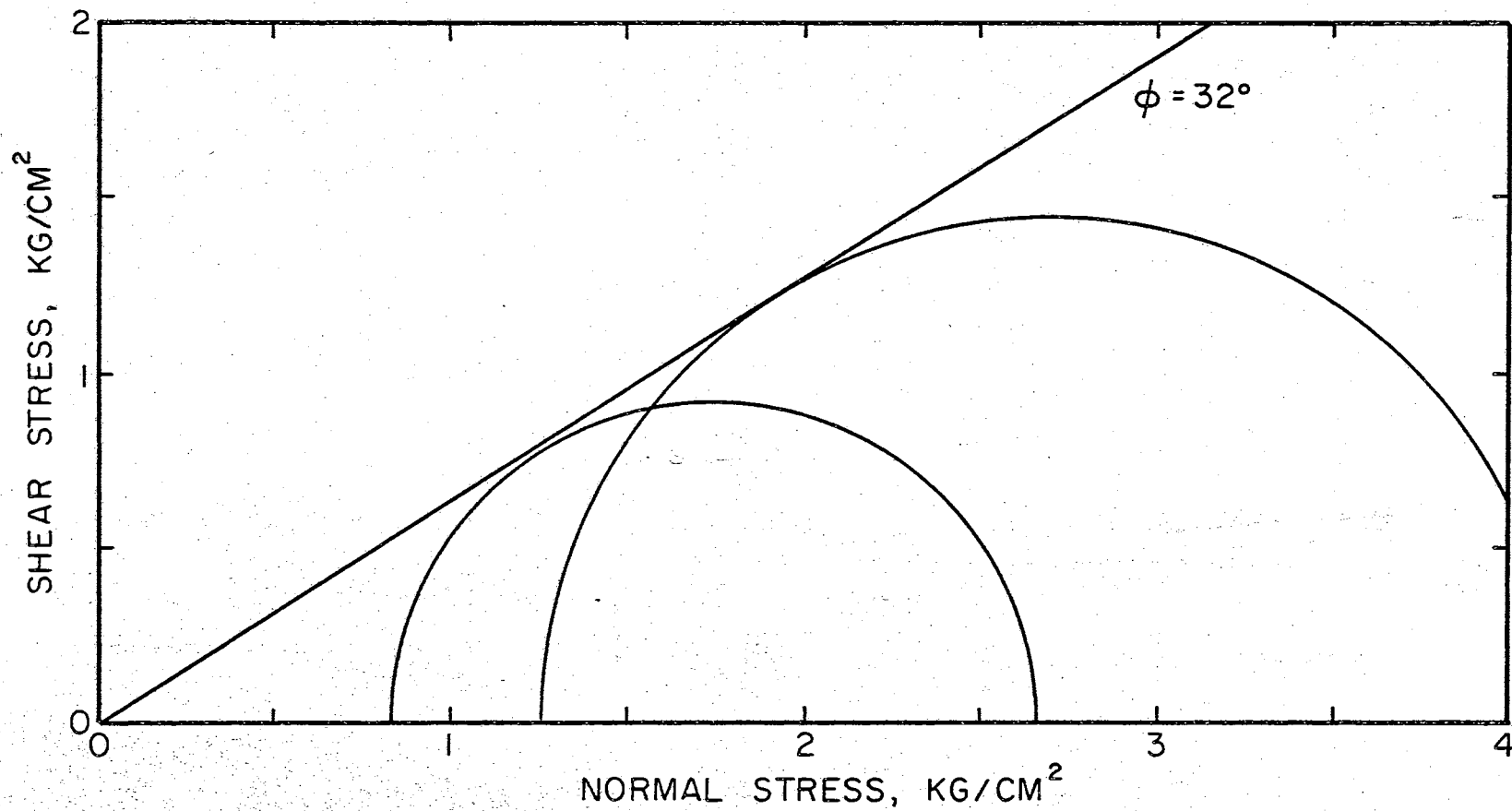


Figure 33. "Effective" Strength Envelope for Consolidated-Quick (\bar{R}) Test on Sesquioxide Free Lateritic Soil

did the unremolded soil. This trend is also indicated by comparing the strength parameters of the sesquioxide free soil with those of unremolded and remolded soils as determined by Q and \bar{R} tests. Thus, the apparent trend is that remolding decreases the $\bar{\Phi}$ values and increases the cohesion of the soil. This reduction in $\bar{\Phi}$ values is attributed to a reduced interlocking of the disaggregated soil grains, while the increase in cohesion is due to a more characteristic behavior of the disaggregated clays. However, it must be concluded that mechanical remolding is insufficient to disaggregate completely the clayey clusters and thereby significantly reduce the shear strength of compacted lateritic soil.

Lime Stabilization

The results of the lime stabilization program are presented in Figure 34. For these tests 5 percent lime was selected as the comparison percentage. On the basis of the pH test (19), 5 percent lime is approximately equal to the lime modification percentage. In a previous study (78) the stabilization percentage (lime content producing greatest strengths) was 10 percent.

Comparisons of the various curves presented in Figure 34 reveal some interesting aspects. The unremolded, remolded, and sesquioxide free soils all exhibit strength increases with time. These strength increases indicate that pozzolanic reactions are occurring. However, the variation between the magnitude and the speed of the strength gains suggests that the susceptibility of the soil to stabilization is influenced by mechanical and chemical remolding.

Since most or all of the free silica has been removed from

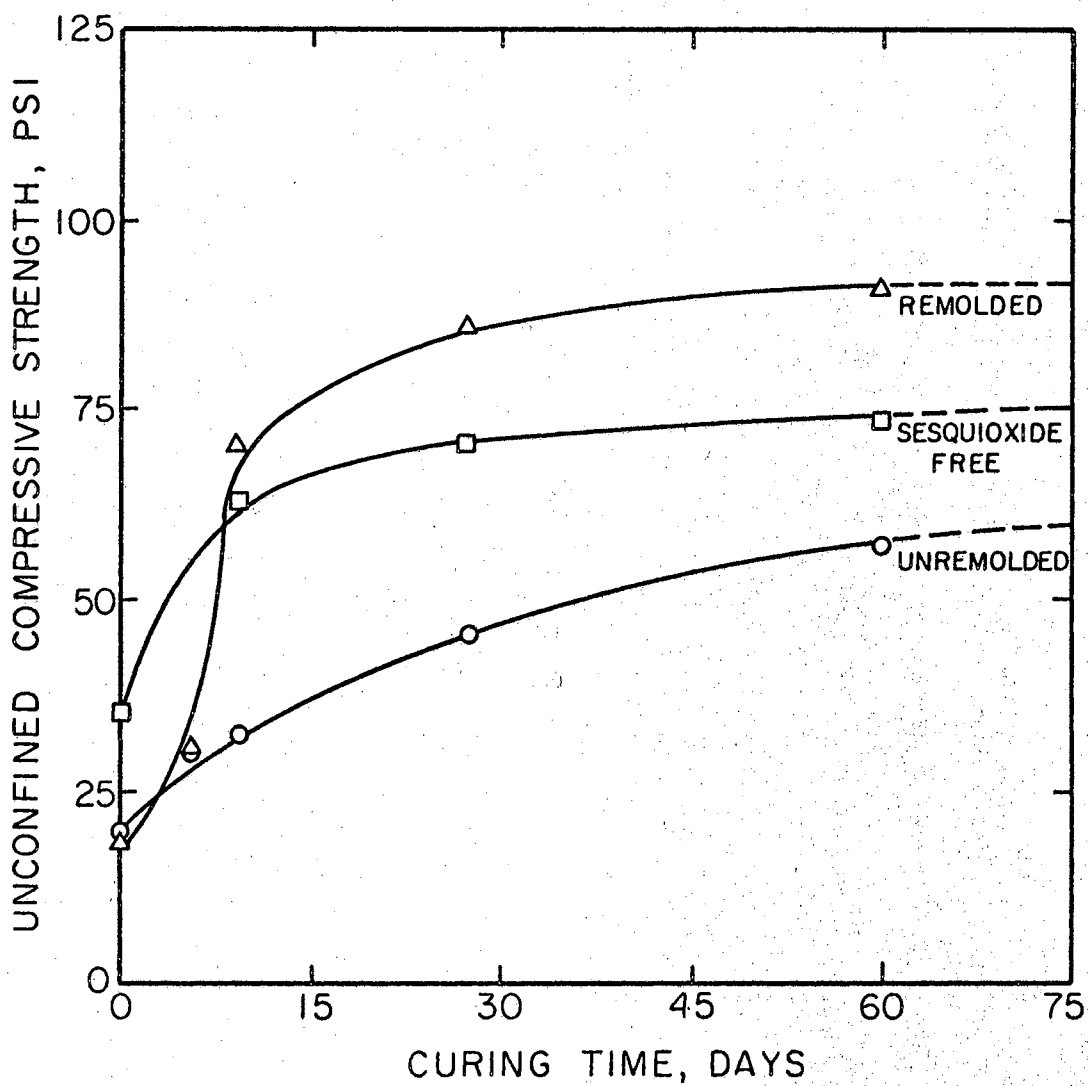


Figure 34. Effect of Curing Age on Unconfined Compressive Strength of a Lateritic Soil Plus Five Percent Lime

lateritic soils during the genesis of the soil, the primary source of silica available for pozzolanic reactions would be the clay minerals. However, the presence of sesquioxidic coatings on the surfaces of these clays would inhibit the calcium and clay (silica) reactions. Thusly, the retarded and poor response of the lime stabilized unre-molded soil in producing significant strength increases is attributed to the nonavailability of the clay minerals (silica) for reaction with the lime. The exhibited strength gains of the unremolded soil are probably limited to the reaction of the lime with small amounts of amorphous silica.

Although the clay surfaces probably remain coated by the sesquioxides after mechanical remolding, evidently the lime susceptibility of the soil is enhanced. Apparently the increased amount of finer particles produced by remolding supplies greater surface areas for soil - lime reactions. Therefore, the primary difference between the responses of the unremolded and remolded soil to lime treatments is attributed to particle size.

Generally, it would be expected that removal of the sesquioxides would produce greater strength gains than those obtained for the natural soil in the unremolded and remolded conditions. These greater strengths would result from (a) increased amount of fines due to the chemical breakdown of the clay aggregations, and (b) removal of the protective sesquioxidic coatings from the surfaces of the indigenous clay particles. Thompson (77) evaluated this concept on natural and iron free non-lateritic soils which were treated with 5 percent lime. His results showed strength increases were greater for the soil in the iron free state than for the corresponding natural condition (202 psi

to 138 psi; 270 psi to 67 psi). However, Figure 34 shows that removal of the sesquioxides from this lateritic soil produced higher strengths only when compared with the unremolded soil. Thus, the concept that the free iron and aluminum oxides retard the lime susceptibility of the soil is partially substantiated.

Comparisons of the strengths of remolded soil with those of sesquioxide free soil indicate that extraction of the sesquioxides reduces the lime reactivity of the soil. Since both the remolded and sesquioxide free samples possess similar grain size distributions (see Chapter V), it appears that the sesquioxide extraction process removes something, possibly alumina, which contributed to the stabilizing reactions. In previous chapters it has been shown that the extraction procedure greatly alters the characteristics of the amorphous materials in the soil. Therefore, it is apparent that the major contributors to the stabilizing reactions in this soil are the amorphous alumina and silica. These amorphous colloids readily enter into pozzolanic reactions because of their high specific surfaces and reactivity.

It appears reasonable to conclude that lime would be an ineffective stabilizer for lateritic soils unless a substantial quantity of amorphous allophanic colloids are available in the soil for pozzolanic reactions.

The electron micrographs shown in Figures 35 through 41 were obtained using lime treated remolded specimens which had cured for approximately 1.5 to 2.0 years in a moist room at 20°C. The micrographs were made to observe the type of various soil-lime reaction products and to determine at what lime concentration they were formed. Generally, these reaction products have been identified as hydrated

forms of calcium silicates (CSH) and/or calcium aluminates (CAH).

The calcium silicates are members of the tobermorite mineral family and appear in the literature (73) as, (a) CSH(gel), (b) CSH(I), and (c) CSH(II). Under the electron microscope, CSH(gel) appears as irregular platelets or fibers, CSH(I) appears as crumpled foils, and CSH(II) appears as long thin fibers or rolled up tubular sheets. The calcium aluminates may crystallize into hexagonal plates or cubic forms (85).

A photomicrograph of a specimen to which no lime has been added is shown in Figure 35. The granular, fluffy, clayey clusters are easily seen. The complete absence of any clay platelets indicates that the clays must exist in microaggregated form.

A photomicrograph of a specimen in which 5 percent lime has been added to the soil is shown in Figure 36. No crystal growth is visible in this figure. However, the ragged edges of the clusters suggests some chemical attack. If 5 percent lime is the lime fixation point, then logically no major crystal growth should appear at or below this lime concentration (32).

Photomicrographs of a specimen to which 10 percent lime has been added to the remolded soil are shown in Figures 37 and 38. The formation of new crystals is quite evident in these figures. The irregular platelets, whose edges appear as laths are tentatively identified as CSH(I). This identification is based upon the Ca/Si ratio and the appearance of the crystals. CSH(I) forms at molar Ca/Si ratios which are less than 1.5. The Ca/Si ratio for 10 percent lime is 0.55. The cubic crystals (grape-like bunches) in Figure 38 are thought to be lime, CaO, which has not entered into any pozzolanic reactions.

However, CAH also possess a cubic structure and these cubes may be some form of these compounds (CAH). The large quantity of iron in the soil also may have combined with the lime to produce a type of hydrogarnet, which also possesses a cubical structure. Therefore, these cubic crystals only can be tentatively identified as lime and further research is required for positive identification.

Photomicrographs of specimens in which 20 percent lime has been added to the remolded soil are shown in Figures 39 and 40. The various forms of lime reaction products is quite apparent. The rolled up tubular sheet in Figure 39 is tentatively identified as CSH(I) as CSH(II) forms at Ca/Si ratios which are greater than 1.5, and the Ca/Si ratio for 20 percent lime is only 1.0. The lath shaped crystals in Figure 40 are also tentatively identified as CSH compounds. The hexagonal platelets appearing in this micrograph are tentatively identified as CAH compounds. This identification is based upon the observation that at high lime contents the clay minerals should have undergone severe chemical attack and dissolution. The dissolution of the clay minerals would produce ragged edges, several of which can be seen in the figure. Therefore, the sharp hexagonal platelets must be reaction products; of which, CAH is the most logical.

Figure 41 is a micrograph of sesquioxide free soil. The platey structure of the clays is quite obvious. A comparison of this figure with that (Figure 35) of the natural soil strikingly shows the agglomerating effect of the sesquioxides.

These electron micrographs show that lime reaction crystals are formed in these soil - lime mixtures; however, these crystals are only formed at lime contents above 5 percent. This observation is

consistent with the lime fixation concept, and with the evidence that significant strength increases occur only at lime concentrations above the fixation percentage.

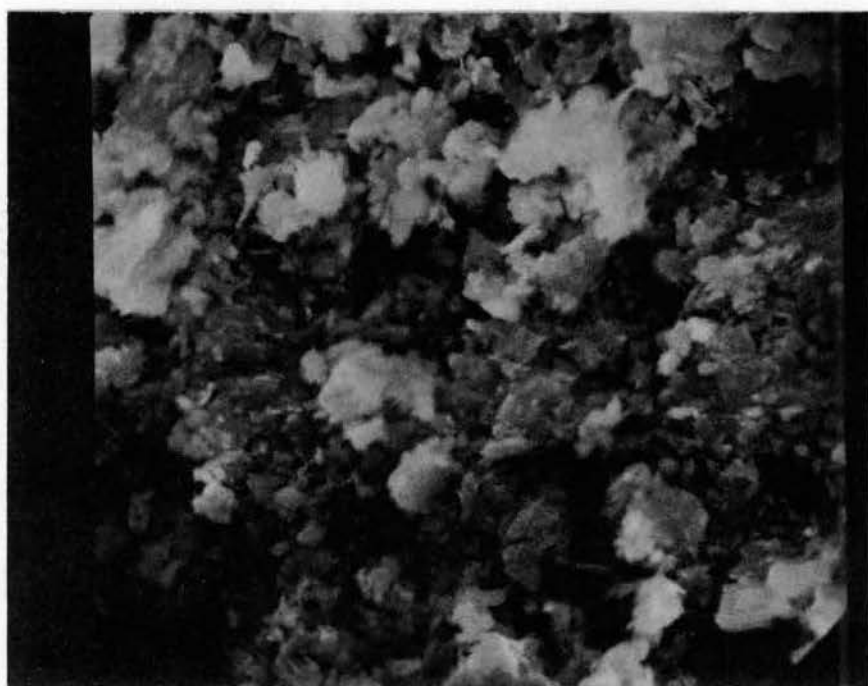


Figure 35. Electron Micrograph of Remolded Lateritic Soil (3000X)



Figure 36. Electron Micrograph of Remolded Lateritic Soil Plus Five Percent Lime (360X)

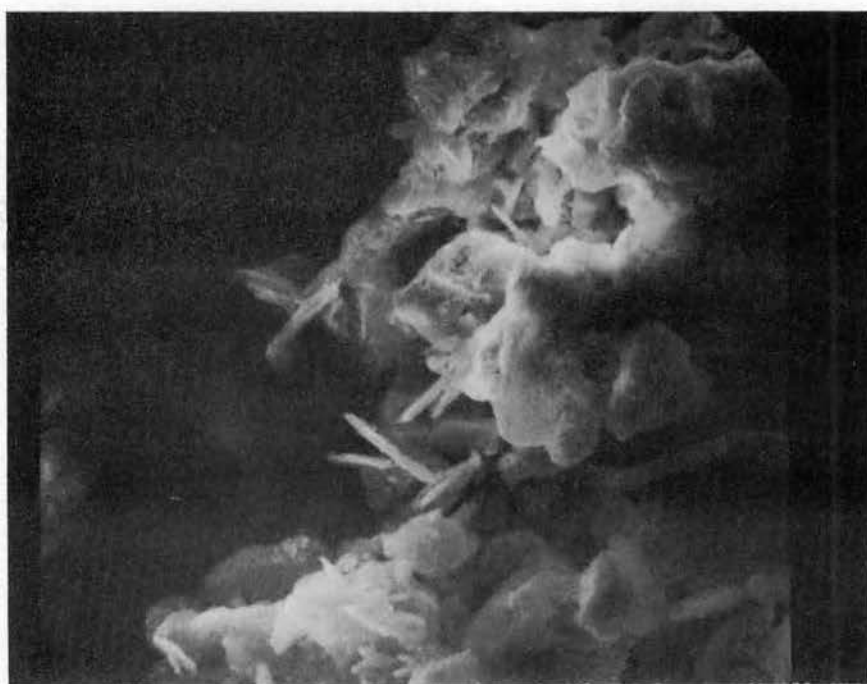


Figure 37. Electron Micrograph of Remolded Lateritic Soil Plus Ten Percent Lime (600X)

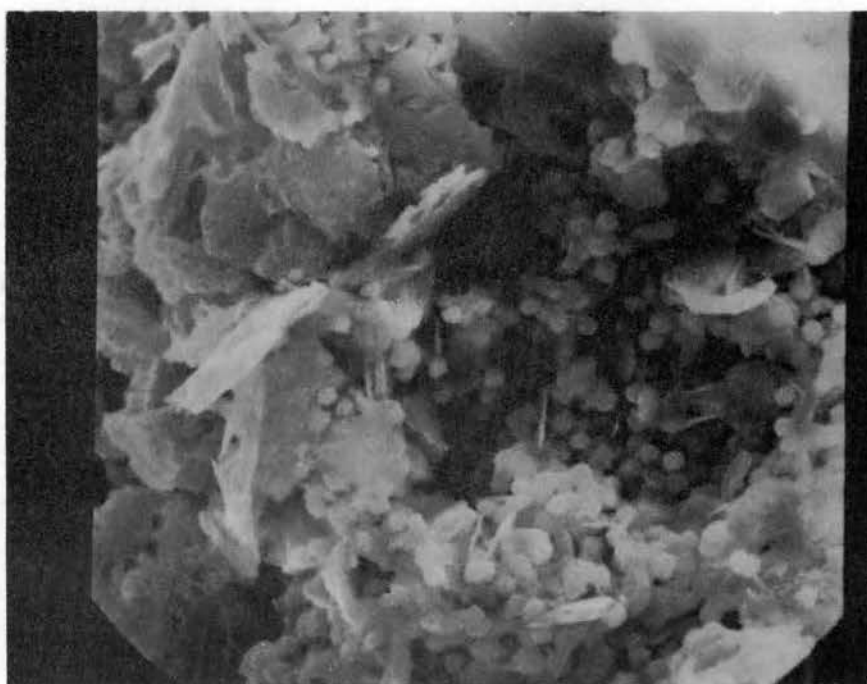


Figure 38. Electron Micrograph of Remolded Lateritic Soil Plus Ten Percent Lime (1800X)

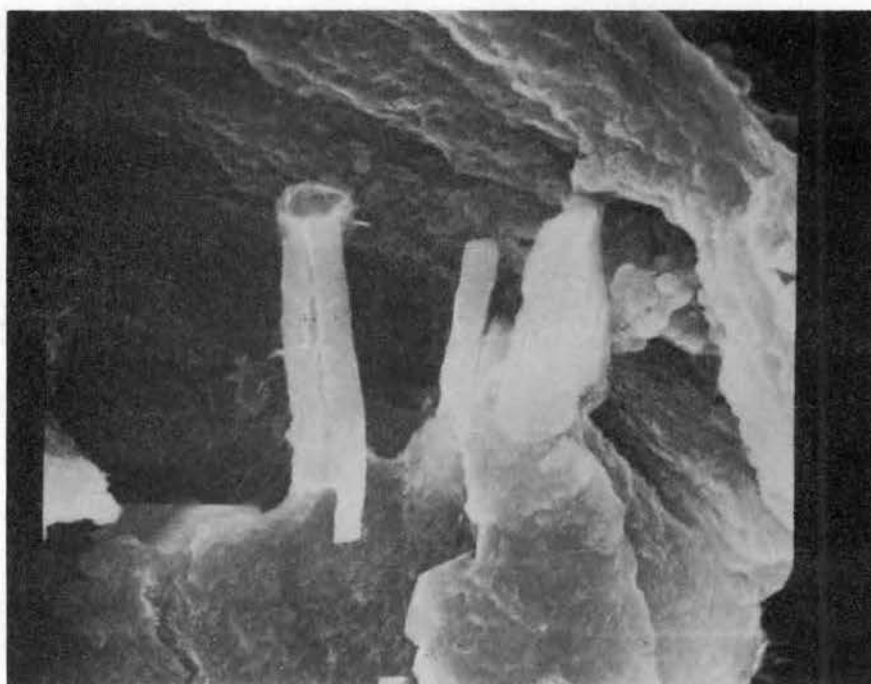


Figure 39. Electron Micrograph of Remolded Lateritic Soil Plus Twenty Percent Lime (2400X)



Figure 40. Electron Micrograph of Remolded Lateritic Soil Plus Twenty Percent Lime (3000X)

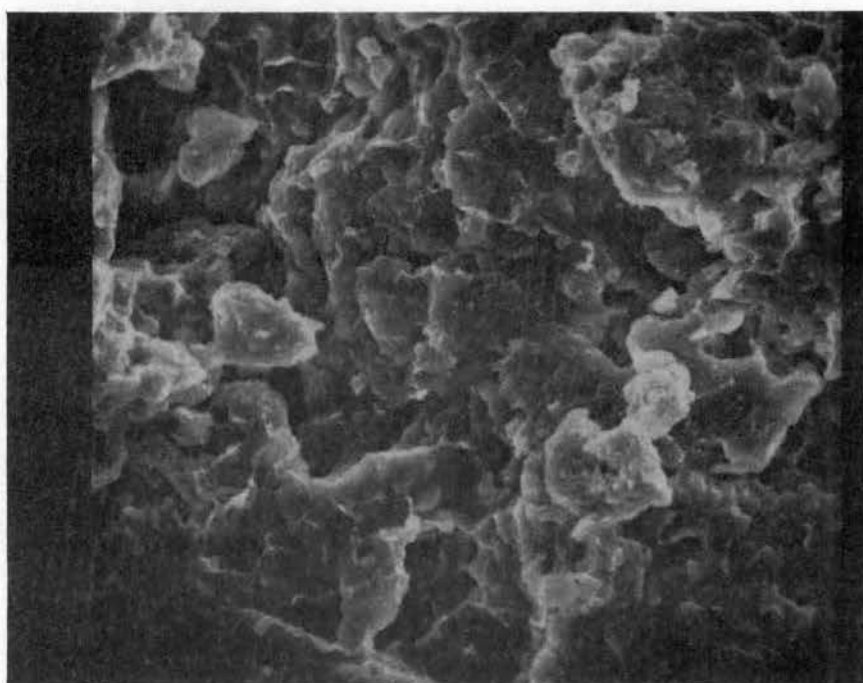


Figure 41. Electron Micrograph of Sesquioxide Free
Lateritic Soil (600X)

CHAPTER VII

SUMMARY AND CONCLUSIONS

Summary

Some basic geotechnical properties, i.e., geological formation, mineralogy, and chemical composition, of a lateritic soil were investigated with the objective of obtaining more insight into some of the unusual engineering properties of lateritic soils as reported in the literature. Concurrently, the properties were compared with those reported in the literature to ascertain if the characteristics of this soil were unique or whether it behaved in a fashion similar to other lateritic soils. Results of this investigation revealed that the sesquioxides of iron and aluminum exerted a considerable influence on the behavior of this soil. The influences of the sesquioxides on some physico-chemical and engineering properties for this soil are summarized as follows:

- (1) X-ray diffraction studies revealed the presence of sesquioxide coatings on the clay mineral surfaces of the soil (see Figures 12 and 13). These sesquioxides suppressed the diffraction peak intensity as removal of the sesquioxides resulted in increased peak sharpness. The clay mineral of this soil was a 1:1 pseudo-kaolin clay.
- (2) The sesquioxide coatings function as cementing agents and bind the finer clay sizes into coarser aggregations. These

microaggregations imparted a granular structure to the soil. Extraction of the sesquioxides increased the amount of clay sized particles from three percent to 49 percent (see Figure 23).

- (3) Substantial quantities of amorphous colloids (allophane) were present in the soil. Approximately 20 percent free silica and five percent free alumina existed in the minus No. 40 sieve fraction of the soil.
- (4) The sesquioxide coatings neutralized the exchange sites of the clay minerals, and the amorphous constituents dominated the exchange characteristics of the soil. Removal of the sesquioxides resulted in an increase in CEC from 10.5 me/100 gms to 16.0 me/100 gms in the minus No. 40 sieve fraction. However, alteration of the allophanic colloids by the extraction of the sesquioxides caused a decrease in CEC from 25.0 me/100 gms to 21.0 me/100 gms in the fine clay fraction.
- (5) The textural characteristics and plasticity of the soil were greatly influenced by remolding. Grain size analyses (see Figure 23) revealed that remolding increased the amount of clay sized particles from three percent to 45 percent. Atterberg limit determinations showed that the plasticity index was increased from 19.2 percent to 28.9 percent by remolding.
- (6) The oxide surface coatings influenced the Atterberg limit values of the soil due to their high moisture retention capabilities. Removal of the sesquioxides decreased the

plasticity index from 28.9 percent to 19.2 percent and the optimum moisture content from 35 percent to 29.5 percent. Likewise, a phenomenal reduction was obtained in peak intensity of the 100°C DTA peak (see Figure 22) after sesquioxide extraction.

- (7) Despite the presence of heavy sesquioxides, low compacted densities (84.5 pcf) were characteristic of this soil. Contradictory to normal expectations, removal of the sesquioxides increased the compacted density from 84.5 pcf to 88.0 pcf. Remolding had little effect on the compacted densities of the soil.
- (8) When compacted, this soil possessed satisfactory shear strength characteristics. The high ϕ' values (38°) reflected the granular nature of the soil. Although remolding had little effect on the shear strength parameters, ϕ and C , the apparent trend was that remolding decreased ϕ and increased C .
- (9) This lateritic soil responded quite favorably to lime treatment and produced significant strength gains (20 psi to 80 psi) with five percent lime. Remolding effected the lime susceptibility of the soil and higher strengths were obtained for remolded soil rather than unremolded soil. Electron microscopy studies showed that lime reaction products were formed with ten percent and twenty percent lime. Contrary to expectations, removal of the sesquioxides decreased the effectiveness of lime for strength increases.

Review of results reported in the literature indicated that this

lateritic soil exhibited traits similar to those of other lateritic soils. However, one possible exception was the presence of a significant amount of amorphous silica in the soil and its effect on the response of the soil to lime stabilization. The results of this study concur wholeheartedly with Winterkorn's (83) statement that "...the presence of iron in lateritic soils is one of the most important factors which influences their engineering properties."

Conclusions

In this investigation the mineralogical, chemical, physical, and engineering characteristics of a Panamanian lateritic soil were investigated. The following conclusions can be made for this type of soil and the testing procedures employed:

- (1) Mineralogically, the soil is primarily composed of remnant quartz grains and a 1:1 pseudo kaolin clay. Secondary silica, as cristobalite and chalcedony, and magnetite exist in the soil in lesser amounts. In addition to the forementioned minerals, considerable quantities of free amorphous iron, aluminum, and silica are present in the soil.
- (2) The sesquioxides of iron and aluminum influence the behavior of this soil by coating the clayey constituents of the soil and binding them into coarser aggregations. These sesquioxide coatings on the indigenous clay minerals suppress their normal behavioral characteristics while the aggregations impart a granular structure to the soil.
- (3) Remolding of the soil greatly influences the textural characteristics and plasticity of the soil. The granular structure

is quite friable and readily breaks down to increase the percentage of finer particles. The increased amounts of fines cause an increase in the plasticity of the soil. Since these index properties are affected by remolding, new or revised laboratory tests should be developed for evaluation of the properties of lateritic soils. The one-point liquid limit determination can be used for more reliable evaluation of the Atterberg limits.

- (4) The inherent granular structure of the soil influences the engineering characteristics. Despite the presence of heavy sesquioxides in the soil, the low densities are due to the high void ratio provided by the granular aggregations. The high shear strength values for ϕ' reflect a greater amount of interlocking of the granular aggregations than would normally be produced by a platy structure. Disruption of the granular structure by remolding has little effect on the shear strength of the compacted soil.
- (5) The amorphous constituents dominate the physico-chemical properties of the soil. The exchange capacity is primarily due to the amorphous colloids as the clay minerals have been neutralized by the iron and aluminum oxides.
- (6) Lime is an effective stabilizer for this lateritic soil. Its effectiveness is primarily attributed to the availability of amorphous silica rather than the clay minerals for strength producing pozzolanic reactions.
- (7) The characteristics of this lateritic soil are quite similar to those of other lateritic soils as reported in the

literature. However, a possible exception is the presence of significant quantities of amorphous silica and its effect on the lime susceptibility of the soil.

Recommendations for Future Research

It is recommended that research studies be undertaken to extend the knowledge developed from this study. The following are suggestions of possible research topics involving lateritic soils:

- (1) An investigation similar to this study, but using lateritic soils from different areas of the world. Such an investigation would determine if the conclusions of this study are applicable to a wide variety of lateritic soils.
- (2) A study of the influence of the $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio on the index properties; i.e., grain size and Atterberg limits, and engineering properties such as shear strength, volume change characteristics, and compaction. Such an investigation might well lead to the development of a more reliable engineering classification system for lateritic soils.
- (3) An evaluation of the susceptibility of various lateritic soils to stabilization by different additives. In these studies particular attention should be given to the percentages of amorphous colloids in the soil, and to the effects of remolding, method of compaction, $\text{SiO}_2/\text{R}_2\text{O}_3$ ratio, and the concentration of additive on the developed strengths.
- (4) An investigation of the influence of the sesquioxides on the volume change characteristics of various lateritic soils.

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APPENDIX A

PROCEDURES FOR CHEMICAL ANALYSES
AND TREATMENTS

Fractionation Procedure for Separating the Silt, Coarse Clay and Fine Clay Fractions

The following steps were followed for separation of the soil into the silt, 50 - 2 μ , coarse clay, 2 - 0.2 μ , and fine clay, < 0.2 μ fractions:

- (1) Test for Carbonates - Carbonates present in the soil act as cementing agents and should be removed prior to fractionation. However, in this soil several drops of 1 N HCl failed to effervesce which indicates an absence of carbonates. Therefore, this treatment was bypassed.
- (2) Removal of Organic Matter - Five ml increments of 30 percent H_2O_2 were gradually added to 5 gms of minus No. 100 U. S. Standard sieve material, until a total of 20 mls of H_2O_2 had been added. Increments of 5 mls were used in order to control the reaction rate should any violent frothing of the mixture occur. The suspension was gently warmed and frequently stirred for 2 or 3 hours, cooled, covered, and allowed to digest for 12 hours. No frothing occurred during the process which indicates a low organic content of this soil.
- (3) Removal of the Sand, > 50 μ - The organic and carbonate free suspension was washed 3 times with distilled water to remove the H_2O_2 prior to wet sieving to separate out the sand fraction. The soil suspension was dispersed in an electric mixing cup with a dilute Na_2CO_3 solution (pH 9.5, 2 gms of Na_2CO_3 in 18 liters of water) as a deflocculating agent. Dispersion of latosols, rich in kaolinite, halloysite, and

sesquioxides requires a solution of pH 8 to 10 (35). The suspension was then wet sieved through a U. S. Standard No. 270 sieve, which removed the sand fraction.

- (4) Separation of the Silt Fraction, $50 - 2\mu$ by Sedimentation -
The suspension of silt and clay material was placed in a 1000 ml cylinder, mixed by shaking, and allowed to sit undisturbed for 3.5 hours. This time is the required time for 5 cms of gravity sedimentation of 2μ particles at 25°C , assuming a specific gravity of 2.65. A depth of exactly 5 cms was siphoned off after this time lapse, and the suspension was remixed. This procedure was continued until the top 5 cms were a clear solution after 3.5 hours of settling. Dilute Na_2CO_3 was continually added at various intervals to maintain a pH of 9.5 in the suspension. The remaining material in the cylinder was the silt fraction, while the decanted suspension was the clay fraction.
- (5) Separation of the Fine Clay, $< 0.2\mu$ from the Coarse Clay, $2 - 0.2\mu$ - A Sharples Supercentrifuge with a bowl speed of 33,000 rpm and a flow rate of 600 ml/min was utilized to separate these two fractions.

Sesquioxide Removal Treatment

A maximum of about 5 gms of Fe_2O_3 and/or Al_2O_3 can be removed in a 40 ml portion of citrate reagent (51). For calculating the quantity of reagents required for this procedure, 10 percent Fe_2O_3 was assumed to be present in this lateritic soil.

Reagents

1. 0.3 M Sodium Citrate (88 gms of $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2 \text{H}_2\text{O}$ per liter)
2. 1.0 M Sodium Bicarbonate (84 gms of NaHCO_3 per liter)
3. Sodium Dithionite, $\text{Na}_2\text{S}_2\text{O}_4$

Procedure

A suitable amount of soil containing 0.5 gms of extractible Fe_2O_3 is added to a solution of 40 mls of 0.3 M citrate and 5 mls of 1.0 M bicarbonate. The suspension is placed in a water bath and the temperature is brought to $70^\circ - 80^\circ \text{C}$ (NOT MORE!). Higher temperatures can cause sulfur precipitation. Then 1.0 gms of solid dithionite is added to the suspension and stirred continually for one minute. The suspension is allowed to digest for 5 minutes and a second 1.0 gms portion of dithionite added. After a second digestive period of 5 minutes, the suspension is centrifuged and the supernatant decanted for future analyses, if so desired.

The soil after the initial addition of the dithionite turned a light gray color and began settling beneath a dark yellowish green supernatant. Four washings with distilled water were used to remove the reagents which were used in the treatment prior to testing of the "sesquioxide free" soil.

Free Silica and Alumina, and

Kaolinite Determinations

Procedure

The percentages of free silica and free alumina were determined after heating the soil to 110°C by a procedure of selective dissolution

and colorimetric analyses for Si and Al. The percentages of kaolinite present in the soil were determined after destruction of the kaolin by heating to 560°C , colorimetrically determining the percentages of Si and Al, and calculating the amount of kaolin based upon its formula weight.

Selective Dissolution (35)

A 0.100 gm soil sample is weighed and transferred to a nickel beaker. Then 100 mls of boiling 0.5 N NaOH (2 gms of reagent NaOH pellets in 100 mls of water) are poured into the beaker and boiling continued on a hot plate for exactly 2.5 minutes. The beaker and its contents are then rapidly cooled in a cold water bath. The supernatant is promptly removed by centrifuging and the Si and Al percentages determined immediately to avoid possible contamination from glassware or polymerization of the Si and Al.

Colorimetric Determination of Silica (41)

Reagents

1. Sodium Molybdate (50 gms of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ in 1 liter of water)
2. Oxalic Acid (5 gms in 100 mls of water)
3. Stannous Chloride (25 gms of $\text{SnCl}_2 \cdot \text{H}_2\text{O}$ in 50 mls of conc. HCl) Warm if necessary for complete solution. Dilute to 500 mls with distilled water for a 1.2 N stock. Just before using, dilute 1:1 to obtain a 0.6 N final working solution. This working solution should be fresh and the stock solution should be no more than 2 weeks old.

4. Sodium meta Silicate (exactly 6.27 gms of $\text{Na}_2\text{SiO}_3 \cdot 9 \text{H}_2\text{O}$ in 1 liter of 0.5 N NaOH) Heating of the salt to 100°C removes $6\text{H}_2\text{O}$ for formula weight calculations. The 6.27 gm concentration develops a stock solution of 1000 ppm Si in NaOH.

Procedure

1. To polyethylene tubes which have been rinsed with 100 percent methanol just prior to use add 10 mls of sodium molybdate and 1 ml of 6 N HCl. If the solution turns yellow the tubes are contaminated and must be rewashed with methanol.
2. Add an aliquot of the solution to be measured. The solution should contain 100 micrograms or less of Si. Then mix the contents.
3. Add 1 ml of 5 percent oxalic acid and mix.
4. Add 3 mls of 6 N HCl and dilute to 40 mls with distilled water and mix.
5. Add 1 ml of stannous chloride as the dilute working solution and mix.
6. Finish dilution to 50 mls and mix. If the blank or standard solution turns a deep blue the stannous chloride solution should be remade or insufficient acid is present in the solution.
7. Read the optical density at 810 or 660 millimicrons.
8. Prepare the standard calibration curve by using sodium meta silicate. Since the salt is in NaOH, insufficient acid may be present in the mixture and, therefore, the blank should be made with distilled water.

Calculations

$$\text{Percent Si} = \frac{(\text{ppm})(\text{dilution factor})}{\text{original weight}}$$

$$\text{Percent SiO}_2 = (\text{percent Si})(2.14)$$

$$\text{Percent Kaolinite} = \frac{(\text{Percent SiO}_2 \text{ after } 560\text{C}) - (\text{Percent SiO}_2 \text{ after } 110\text{C})}{46.5} \times 100$$

Colorimetric Determination of Aluminum

Reagents

1. pH 4.2 buffer. Neutralize 1 liter of 1 percent NaOH with 60 mls of glacial acetic acid and adjust to pH 4.2.
2. Aluminon solution. Weigh 0.5 gms of aluminon reagent into 1 liter of pH 4.2 buffer.
3. Standard aluminum solution. Weigh 0.5 mls of 6 N HCl and dilute to 1 liter. For a 5 microgram/ml solution, take 10 mls of stock solution (step 3) and dilute to 1 liter.

Procedure

Adjust pH of the solution containing the unknown aluminum to pH 4.2 with the buffer solution and dilute to volume (usually 50 mls). Place a 10 ml portion of the pH 4.2 buffer solution in a 50 ml flask. Add 10 mls of distilled water followed by exactly 10 mls of 0.4 percent aluminon reagent and mix. Next, add 5-20 mls of the unknown solution, bring to volume, and allow the mixture to develop its color for 25 minutes. Determine the percent optical density at 520 millimicrons.

Calculations

$$\text{Percent Al} = \frac{(\text{ppm})(\text{dilution factor})}{\text{original weight}}$$

$$\text{Percent Al}_2\text{O}_3 = (\text{percent Al})(1.89)$$

$$\text{Percent Kaolinite} = \frac{(\text{Percent Al}_2\text{O}_3 \text{ after } 560\text{C}) - (\text{Percent Al}_2\text{O}_3 \text{ after } 110\text{C})}{39.5}$$

X 100

Cation Exchange Capacity by Calcium

Saturation and Displacement

with Sodium

Reagents

1. Standard calcium solution. Dissolve 0.5005 gms of pure dried CaCO_3 in 0.2 N HCl and dilute to 1 liter. Solution is 0.01 N Ca.
2. pH 10 ammonium chloride-ammonium hydroxide buffer. 100 mls of 1 N NH_4Cl and 500 mls of 1 N NH_4OH (conc. NH_4OH is 14.8 N).
3. Standard versene. Dissolve 2 gms of EDTA salt in 900 mls of water, then add 50 milligrams of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. The normality is approximately 0.01 N, but this normality should be determined by titration of several portions of the Ca standard (0.01 N).

Procedure

1. The various soil fractions are calcium saturated by 4 washings with 1 N CaCl_2 .

2. The excess calcium ions are removed by approximately 4 washings with distilled water. Periodic checks for chlorides can be made with AgNO_3 . When no white precipitate is observed in the supernatant after adding AgNO_3 , the excess calcium ions are assumed to have been removed.
3. The exchanging is accomplished by 4 washings of 1 N NaCl or 1 N NaOAc . The supernatants of each washing should be decanted and saved for analysis of calcium content.
4. The exchanged calcium is determined by a versenate titration of the sodium extract. The titration is accomplished by adding 10 mls of pH 10 buffer plus several drops of Eriochrome Black T to an aliquot of the solution containing the exchanged calcium. The titration versene used is EDTA. The end point is a change from a wine red color to a bright blue.
5. The CEC is calculated as follows:

$$\text{me}/100 \text{ gms} = \frac{(\text{ml versene})(\text{normality of versene})(100)}{\text{sample weight}}$$

APPENDIX B

STATISTICAL ANALYSES

Coefficient of Variation Results for

Percentages of Free Silica

and Alimina

Soil Fraction	Result No.	Free Silica (%)	Result No.	Free Alumina
Unremolded (minus No. 40)	1.	25.50	1.	5.46
	2.	15.47	2.	7.82
	3.	18.21		
	Ave. = 19.93		Ave. = 6.64	
	s = 5.18		s = 1.67	
	C.V. = $\frac{19.93}{5.18} = 26\%$		C.V. = 25%	
Remolded (minus No. 40)	1.	22.85	1.	4.44
			2.	3.23
			Ave. = 3.38	
			s = 0.85	
Silt (50-2 μ)	1.	10.27	1.	3.54
	2.	7.02		
	Ave. = 8.64			
	s = 2.30			
Coarse Clay (2-0.2 μ)	1.	21.06	1.	6.27
	2.	16.86		
	3.	16.86		
	4.	13.03		
Fine Clay (< 0.2 μ)	Ave. = 16.95			
	1.	10.70	1.	4.40
	2.	14.48	2.	3.65
	Ave. = 12.59		Ave. = 4.03	
	s = 2.67		s = 0.53	
	C.V. = 21%		C.V. = 13%	

Coefficient of Variation Test Results

for Cation Exchange Capacities

Soil Fraction	Result No.	CEC-Natural Soil me/100 gms	Result No.	CEC-SiO ₂ -Free Soil me/100 gms
---------------	------------	--------------------------------	------------	---

Unremolded (minus No. 40)	1.	10.81	1.	15.93
	2.	10.71		

Ave. = 10.49
s = .2048
C.V. = 1.95%

Remolded (minus No. 40)	1.	10.93	1.	17.02
	2.	10.65	2.	16.81

Ave. = 10.79
s = .0392
C.V. = 0.36%

Ave. = 16.91
s = 0.220
C.V. = 0.13%

Silt (50-2μ)	1.	10.09	1.	12.86
	2.	12.33	2.	13.29

Ave. = 11.21
s = 2.5088
C.V. = 22.4%

Ave. = 13.09
s = .0924
C.V. = 0.706%

Coarse Clay (2-20 μ)	1.	15.27	1.	17.80
	2.	17.50	2.	20.50

Ave. = 16.43
s = 2.7144
C.V. = 16.5%

Ave. = 19.15
s = 3.645
C.V. = 19.03%

Fine Clay (< 0.2 μ)	1.	26.44	1.	20.74
	2.	23.68	2.	21.29

Ave. = 25.06
s = 3.8808
C.V. = 15.2%

Ave. = 21.01
s = .1512
C.V. = 0.72%

$$s_p^2 = \frac{13.1866}{9} = 1.4562$$

$$C.V. \text{ (pooled)} = \frac{1.2104}{16.0155} = 7.56\%$$

Analysis of Variance Test Results
for Cation Exchange Capacity

Hypothesis: No difference exists between independent estimates of the variance. The estimates of variance are chosen so as to lump the smallest variances into one group and the widest dispersion in another group

GROUP I	GROUP II
.2048	2.7144
.0392	3.8088
2.5088	.1512
.0924	3.6450
.0220	
<hr/>	<hr/>
Total = 2.8672	Total = 10.3195

$$s_p^2 = .5734$$

$$s_p^2 = 2.5799$$

$$F \text{ (calculated)} = 4.50$$

$$F (\alpha = .05, 5, 4) = 6.26$$

Conclusion: Accept Hypothesis. No difference exists between estimates of variance. Therefore, these variances can be pooled

$$s_p^2 = 1.4652.$$

t Test Results—Influence of Remolding

on CEC of the Minus 40 Fraction

Hypothesis: No difference exists between the CEC values for the minus 40 fractions of the unremolded and remolded soil

Unremolded Soil me/100 gms	Remolded Soil me/100 gms
1. 10.81	1. 10.93
2. 10.17	2. 10.65
t (calculated) = .2478 t ($\alpha = .05, 9$) = 2.262	

Conclusion: Accept hypothesis. Remolding has no effect on the CEC values of the unremolded and remolded.

t Test Results—Influence of Sesquioxide

Removal of the CEC Values

Hypothesis: No difference exists between the CEC values of the natural and sesquioxide free silt fraction.

Natural Soil (me/100 gms)	Sesquioxide Free Soil (me/100 gms)
1. 10.09	1. 12.86
2. 12.33	2. 13.29
t (calculated) = 1.553	t ($\alpha = .05, 9$) = 2.262

Conclusion: Accept hypothesis. Removal of the sesquioxides has no effect on the CEC values of the silt fraction.

Hypothesis: No difference exists between the CEC values of the natural and sesquioxide free coarse clay fractions.

Natural Soil (me/100 gms)	Sesquioxide Free Soil (me/100 gms)
1. 15.27	1. 17.80
2. 17.67	2. 20.50
t (calculated) = 2.2472	t ($\alpha = .05, 9$) = 2.262
	t ($\alpha = .10, 9$) = 1.833

Conclusion: Reject hypothesis at 90% confidence level. Removal of the sesquioxides causes an increase in the CEC values of the coarse clay fraction.

Hypothesis: No difference exists between the CEC values of the natural and sesquioxide free fine clay fractions.

Natural Soil (me/100 gms)	Sesquioxide Free Soil (me/100 gms)
1. 26.44	1. 20.74
2. 23.68	2. 21.29
t (calculated) = 3.346	t ($\alpha = .05, 9$) = 2.262

Conclusion: Reject hypothesis. Removal of the sesquioxides causes a decrease in the CEC values of the fine clay fraction.

Least Squares Regression Analysis for

Liquid Limit Flow Curve

80/80 LIST

00000000011111111122222222233333333334444444445555555556666666667777777778
 1234567890123456789012345678901234567890123456789012345678901234567890

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C      LEAST SQUARES REGRESSION FOR LIQUID LIMIT IS A PROGRAM WHICH
C      FITS A LEAST SQUARES REGRESSION OF DATA TO GIVE A FLOW CURVE
C*****
C      NTEST IS IDENTIFICATION NUMBER OF TEST
C      NBLWS IS THE NUMBER OF BLOWS
C      YMC IS THE MOISTURE CONTENT, ENTERS SAME CARD AS NBLWS
C      ENTRY CODES FOR DATA ARE FIRST TWO CARDS FOR PROB IDENT COL 0-80
C      THIRD CARD IS FOR TEST NUMBER, COL 0-5, COL 5-80 IS FOR IDENT
C      FOURTH CARD ENTERS NUMBER OF TRYS IN COLS 5-10
C      FIFTH CARD ENTERS DATA NBLWS IN COL 5-10, MC IN COL 10-15 ETC
C      LAST CARD IS LEFT BLANK TO STOP THE PROGRAM
1      FORMAT (20A4)
2      FORMAT (/ , 5X, 20A4)
3      FORMAT (15, 5X, 20A4)
4      FORMAT (// , 5X, 11HTEST NUMBER, 15, 20A4)
5      FORMAT (5X, 5(15, F5.4) )
6      FORMAT (5X, 15)
7      FORMAT (/ , 10X, 26HTHE REGRESSION EQN. IS Y = , F8.4, 5HPLUS, F8.4)
8      FORMAT (/ , 10X, 19HTHE LIQUID LIMIT IS, F10.4)
9      FORMAT (/ , 10X, 23HTHE PLOTTING POINTS ARE, F10.4,
1     17HFOR TEN BLOWS AND, F10.4, 15HFOR FORTY BLOWS)
      DIMENSION AN1(40), AN2(17), NBLWS(50), YMC(50), XBLWS(50), YHAT(50)
      WRITE (6, 12)
      FORMAT ('1')
12     READ (5, 1) (AN1(N), N = 1, 40)
      PRINT 2, (AN1(N), N = 1, 40)
20     READ(5, 3) NTEST, (AN2(N), N = 1, 17)
      PRINT 4, NTEST, (AN2(N), N = 1, 17)
      IF (NTEST) 999, 999, 30
30     READ(5, 6) NTRYS
      READ (5, 5) (NBLWS(I), YMC(I), I = 1, NTRYS)
      SUMX = 0.0
      SUMY = 0.0
      SUMX2 = 0.0
      SUMXY = 0.0
      DO 10 I = 1, NTRYS
        X = NBLWS(I)
        XBLWS(I) = ALOG10(X)
        SUMXY = SUMXY + XBLWS(I) * YMC(I)
        SUMX = SUMX + XBLWS(I)
        SUMY = SUMY + YMC(I)
        SUMX2 = SUMX2 + XBLWS(I) ** 2.0
10     CONTINUE
      X2SUM = SUMX2 - (SUMX) ** 2.0 / NTRYS
      XYSUM = SUMXY - (SUMX) * (SUMY) / NTRYS
      BONE = XYSUM / X2SUM
      BZERO = (SUMY / NTRYS) - (BONE) * SUMX / NTRYS
      DO 11 I = 1, 3
        XBAR = 10. + 15. * (I - 1)
        YHAT(I) = BZERO + BONE * ALOG10(XBAR)
11     CONTINUE
      WRITE(6, 7) BZERO, BONE
      WRITE(6, 8) YHAT(2)
      WRITE (6, 9) YHAT(1), YHAT(3)
      GO TO 20

```

00000000011111111122222222233333333334444444445555555556666666667777777778
 1234567890123456789012345678901234567890123456789012345678901234567890

```

999      WRITE (6, 12)
        STOP
        END
$ENTRY   TRIAL PROGRAM
        THESE DATA ARE JUST FOR TESTING OF AN EXAMPLE
1        LIQUID LIMIT OF TRIAL DATA
4
60 1000   30 1270   10 1680   5 1950

```

\$IRSYS

VITA

2
Frank Charles Townsend

Candidate for the degree of

Doctor of Philosophy

Thesis: THE INFLUENCE OF SESQUIOXIDES ON SOME PHYSICO-CHEMICAL AND
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